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UNIVERSITY OF OKLAHOMA

GRADUATE COLLEGE

VISCOELASTIC MEASUREMENTS OF FRACTURING FLUIDS FOR
PROPPANT TRANSPORT APPLICATION

A Dissertation

SUBMITTED TO THE GRADUATE FACULTY

in partial fulfillment of the requirements for the

degree of

Doctor of Philosophy

By

NAVAL GOEL

Norman, Oklahoma

2001

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VISCOELASTIC MEASUREMENTS OF FRACTURING FLUIDS FOR
PROPPANT TRANSPORT APPLICATION

A Dissertation APPROVED FOR THE
MEWBOURNE SCHOOL OF PETROLEUM AND GEOLOGICAL ENGINEERING

BY

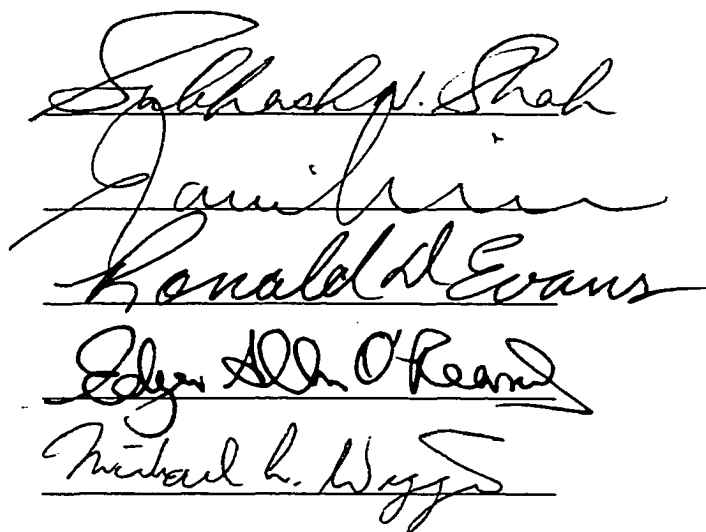
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To my Parents

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ABSTRACT

One of the primary functions of a fracturing fluid is to transport and distribute proppant through a fracture and to keep the particles suspended till reservoir closes on the fracture. The proppant when suspended and uniformly distributed in the fracture improves the productivity of the reservoir. Therefore, a question is frequently asked regarding the minimum fluid viscosity required for satisfactory proppant transport through a fracture. The knowledge of this viscosity is useful for evaluating fluids prior to their use in the stimulation treatment and for demonstrating the effectiveness of new fluid formulations for fracturing application. The present study addresses this very important question.

The study is performed by characterizing crosslinked guar gels for their viscoelastic and suspension properties. The gels are then evaluated for their proppant transport capabilities in two slot models using a field-scale test facility. These characterizations are performed in fracturing slurries prepared at low as well as high sand concentrations and evaluated at ambient as well as elevated temperature conditions. These characterizations are performed for borate-crosslinked gels and then, validated with zirconate-crosslinked gel. The results show that the proppant transport capability of crosslinked gels correlates with the fluid's elasticity and not with its viscosity. Based on these results, a minimum elastic modulus criterion required for a satisfactory performance of crosslinked fluids in a fracturing treatment is proposed.

The criterion proposed in this work is developed from the proppant transport behavior evaluated using a field-scale test facility. This criterion also provides a useful and simple method to evaluate fracturing fluids in a laboratory without the need for extensive fluid testing.

CHAPTER 1

INTRODUCTION

Hydraulic fracturing is widely used to stimulate oil and gas production from a reservoir.¹ This technique improves well productivity by removing near wellbore damage and by increasing reservoir permeability in low as well as high permeability formations.^{2,3} In addition to such formations, the fracturing treatment has been used in many other unique reservoirs;⁴⁻⁶ due to the recent developments in the fracturing technology, it has also been used to produce gas from coalbed,^{7,8} and from wells exceeding 300°F.^{9,10} These current developments and the future advances in the hydraulic fracturing technique will continue to open new avenues for this technique to stimulate oil and gas production around the world.

Hydraulic fracturing is a simple treatment that comprises of creating a fracture in a formation and then placing proppant in the created void. The proppant keep the fracture open and provide a highly conductive path, which allows oil and gas to flow easily to the wellbore. The fracturing treatment is performed by a viscous fluid pumped at high enough pressure to initiate and propagate fracture in the formation. The fluid also keeps the proppant in suspension during its flow from surface equipment to the fractured formation.

A desirable property in the fracturing fluid is its viscosity. A high fluid viscosity helps to create wider fractures, increase fracture area and thus extend the fracture into the formation.¹¹ It also assists to distribute proppant uniformly across the fracture by keeping them in suspension. A uniform distribution and proper placement of the proppant in the

fracture are considered the most important variable for a stimulation treatment, as after the treatment they determine the final geometry of the propped fracture.^{11,12} The significance of the fracturing fluid viscosity is exemplified in several case histories that demonstrate improved well production through design of effective fluid systems.¹³⁻¹⁵ It is estimated that almost one billion gallon of fracturing fluid is pumped per year in North America alone,^{16,17} and 90% of the fracturing fluids are water-based fluids.¹⁸ If one million gal of the fluid is used in each fracturing treatment, then industry performs almost three stimulation treatments per day with water-based fracturing fluid. These figures suggest that a successful stimulation of a formation requires a thorough understanding of the fracturing fluid characteristics.

Guar is the most popular polymer for preparing water-based fracturing fluids. Its wide use is due to its low cost and is because less than 1% of the polymer quantity can increase by several hundred-fold the water characteristics required for fracturing application. The guar based fracturing fluids, however, have one huge disadvantage. The guar polymer is a very large molecule and so, it cannot be completely broken down into small molecules. After a fracturing treatment, it leaves an insoluble polymer residue in the proppant pack inside the fracture and in the reservoir formation around the fracture.³ This residue reduces the permeability of the reservoir and the propped fracture. Hence, it damages the formation and limits the full potential that could be achieved with the stimulation operation.

The disadvantage of guar is being overcome by chemically treating the guar polymer so that the polymer residue is minimized. The chemical treatment of guar, however, increases the price of the polymer, offsetting its cost advantage. Another

method used to reduce polymer residue is to crosslink guar with a crosslinking agent.¹⁹ The fracturing stimulations performed earlier with non-crosslinked 100 lb/Mgal guar are now performed only with crosslinked 35 lb/Mgal guar, and even less guar in some cases. Currently, guar is crosslinked mainly with either boron or zirconate-based crosslinking agent; of which, borate-crosslinked guar gels are used in almost 75% of the fracturing treatments.²⁰ Even though the guar concentration used in fracturing has been reduced, the polymer residue is still a problem. This problem has intensified efforts to critically monitor fracturing fluid quality and evaluate fluid properties that correlate with the fluid performance during a fracturing treatment.²¹ One of the questions which was not considered critical earlier but has become extremely important is the minimum fracturing fluid viscosity required to satisfactorily transport proppant through a fracture.

The guar fracturing fluid is currently characterized for its viscosity, which is also used to determine its proppant transport behavior through a fracture. The guar gel exhibits non-Newtonian fluid characteristics, where its viscosity varies as a function of shear rate. This shear sensitivity implies that the guar properties observed under static fluid conditions are different from those under fluid flow conditions. Therefore, the guar viscosity should describe the fluid capability to transport proppant while the fluid is flowing during a fracturing treatment, and to suspend proppant particles while it is static after the fracturing treatment. Several studies have, however, shown that viscosity is insufficient to describe suspension capabilities of a fluid. A two-fold increase in the fluid viscosity is observed to result in several-fold increase in its proppant suspension properties.²² A difference of as much as 375% has been seen between the values calculated with the viscosity-based correlation and those determined in the experiments

on particle settling in crosslinked guar gels.^{23,24} In spite of these anomalies, the use of fluid viscosity to correlate proppant suspension in a fracture is continued. The drawbacks of the fluid viscosity demonstrate a great need to identify a characteristic property of guar gel that can more accurately describe its proppant suspension and transport through a fracture.

The proppant transport capability of a guar based fracturing fluid is considered to be due to the entanglements formed in a non-crosslinked polymer or to crosslink bonds created in a crosslinked gel.^{10,25} These bonds provide a strong network structure to the fluid and strengthen the guar polymer to keep proppant in suspension. Thus, the network structure formed in a fluid should be correlated with its particle suspension behavior. The discrepancy in describing particle suspension with fluid viscosity shows that the viscosity does not correctly depict the network structure formed in the fracturing fluids. The guar fluid exhibits, besides non-Newtonian behavior, viscoelastic characteristics having both elastic and viscous components.^{1,21} The fluid elastic properties are considered to better describe the network structure formed in these fluids,²⁶⁻²⁹ and might provide an alternative technique to better understand the suspension capabilities of a fracturing fluid.³⁰⁻³² However, very few attempts have been made to correlate fluid elasticity with the proppant suspension.³³⁻³⁷ These attempts have, however, been confined to understand the settling either of single particle or in single slurry concentration under mostly static fluid condition. None of these studies correlated the fluid elasticity evaluation under more realistic slurry flow condition. This gap in the current state of the research is attempted to fill in the present investigation by

understanding and correlating the fracturing fluid elasticity with its capability to satisfactorily transport proppant through a fracture.

This study will provide more accurate characterization of the existing fracturing fluids, and will also help to understand and develop new generation of viscoelastic fracturing fluids.³⁸ In addition to the significance of this work for hydraulic fracturing treatments, the study will be useful in several other processes in the industry where the fluids are used to suspend and transport particles. Few areas in the petroleum industry include particle suspension in drilling fluids and cement slurries, gravel packing in sand production control, wellbore clean out operation, sand removal in deviated and horizontal wells etc.³⁹⁻⁴³

The problem of understanding particle suspension in non-Newtonian and viscoelastic fluid is a classical fluid mechanics problem. The particle suspension in Newtonian fluid was successfully described about 150 years ago,⁴⁴ but the same problem in non-Newtonian and viscoelastic fluids remains unsolved. The particle suspension in viscoelastic fluids has been a focus of tremendous research in recent years.⁴⁵⁻⁵⁰ The current state of the research shows that the viscoelastic fluids exert drag reduction as well as drag enhancement on the particle settling in them.^{51,52} The particle settling in viscoelastic fluid is fundamentally different from that in non-elastic fluids.^{53,54} The particle suspension in these fluids cannot be studied by modifying the correlations developed in non-elastic Newtonian fluids, as has been done in the hydraulic fracturing simulators available today. But, it is important to understand particle-particle and hydrodynamic interactions in such complex fluids.⁵⁵ Therefore, the knowledge gained in

understanding the suspension characteristics of fracturing fluids will help to further comprehend particle suspension in non-Newtonian and viscoelastic fluids.

The present study undertakes a systematic approach by dividing the research into several tasks and building from the results obtained at each stage. A critical rheological parameter was first identified at ambient temperature from an extensive characterization of borate-crosslinked gels in laboratory and from the gel capabilities to transport proppant slurries prepared at low sand concentration. The proppant transport tests were performed with a large-scale test facility that was representative of a field-scale fracturing treatment. Then, the identified parameter was confirmed by evaluating the gels at elevated temperature and for proppant transport of slurries prepared at higher sand concentration. The rheological property identified with the borate-crosslinked gels was finally evaluated for a zirconate crosslinked gel system at conditions completely different from those used with the borate-crosslinked gels.

The research results show that the fluid elasticity provides better correlation between a crosslinked fracturing fluid and its proppant transport behavior. A rheological criterion is proposed which is based on the minimum fluid elastic modulus beyond which a fracturing fluid would satisfactorily transport proppant during a fracturing treatment.

The investigation is described in detail in following chapters. Chapter 2 provides a review of the current state of research on the fracturing fluids characterization for proppant transport. Chapter 3 describes the experimental procedure and equipment used in the investigation. Chapter 4 justifies the approach taken by evaluating the fracturing fluid viscosity on the existing fracture models. Chapter 5 examines the suspension characteristics of borate-crosslinked gels through their rheological and atomic force

microscopic characterizations. Chapter 6 establishes and confirms the critical rheological property for borate-crosslinked gels. Chapter 7 validates the identified fluid property and the elastic modulus criteria with zirconate-crosslinked gel. Chapter 8 discusses the implications of the research for particle settling characteristics in non-Newtonian fluids. Chapter 9 finally concludes the study and discusses the limitations of the work and the directions for future work. Chapter 10 lists the references included in the study. Two appendices are also included with supplemental data. Appendix A provides a listing of the several computer algorithms prepared to handle the huge data gathered during the research. Appendix B summarizes the procedure used in the two-dimensional GdK fracture model.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

This study has four aspects that must be addressed and understood to identify the rheological property of a fracturing fluid for its capacity to satisfactorily transport proppant through a fracture. These areas are fracturing fluid, fluid characterization, proppant transport, and suspension applications. A better understanding of the proppant transport and suspension behavior requires an insight into the fracturing fluid chemistry and the fluid characterization techniques available today.

An aqueous fracturing fluid comprises of a water-soluble polymer. The polymer is either used as a non-crosslinked linear solution or crosslinked with a crosslinking agent to prepare a crosslinked gel. The polymer and the crosslinking agent have been used for a long time, so large information is available in the literature on the chemistry of the polymers and crosslinking agents used in this study. A thorough research on their chemistry would help to select the fracturing fluid for the study and also, to understand the relationship between the fluid behavior and its suspension capabilities. It would also assist to identify various fluid properties that reflect its performance prediction. The identified properties can then be correlated with the laboratory measurable parameters that industry can use to evaluate fracturing fluid prior to performing fracturing stimulation.

The most commonly characterized property of a fluid is its viscosity. The most common use of the fluid characterization is the understanding that a fluid with a higher viscosity creates a wider fracture. A similar understanding of other fluid properties that

can be measured with rheological instruments would help to characterize the fracturing fluid. Furthermore, the identification of the fluid rheology would help to develop a minimum criterion for predicting a fluid capability to transport proppant through a fracture.

In addition to the fluid rheology, another aspect of this research is to understand the proppant transport behavior of a fluid through a fracture. A review of the current industry methods to test for proppant transport behavior would help to use them in the present research for evaluating the minimum fluid rheology criteria. This would also help to disseminate the findings of the present research to the industry. Finally, since suspensions are used in every sphere of life, a review of the research performed in other disciplines would be useful to understand the problem on proppant transport characteristics of fracturing fluids.

An exhaustive literature search was performed on the fracturing fluid chemistry, fluid mechanics of particle settling and fluid rheology to understand the various aspects of the present study. This literature survey is briefly described below.

2.2 FRACTURING FLUIDS

Water based polymers have been in use in the petroleum industry for many years. Their wide use comes from the readily available source of water around the world and from the requirement of very small quantity of polymer, less than 1% by weight, for most applications in the petroleum industry.⁵⁶

For the fracturing treatment, aqueous fluids were introduced in 1953 with the use of sugar-based polymers. These polymers found tremendous support because of their

characteristics to increase water viscosity and reduce friction pressure and to be handled safely.⁵⁷ Among the different polymers, guar gum and its derivatives are the most extensively used polymers in the fracturing fluids.

2.2.1 Guar Gum

Guar Source and Structure. Guar gum is obtained from guar plant called *Cyamopsis tetragonolobus*.⁵⁸ The guar plant is a pod bearing plant containing 6-9 seeds per pod with each seed composing of hull (15%), germ (40%), and endosperm (40%).¹⁶

The endosperm contains the majority of galactomannan, the guar polymer having galactose and mannose monomer units. The galactose and mannose are simple sugar units, so the guar gum is also called a polysaccharide.⁵⁶ The mannose unit forms the backbone to which the galactose unit is bonded as a branch. Two mannose units and one galactose unit form the monomer giving guar gum a branched copolymer structure as shown in Fig. 2.1. The figure shows the two mannose units are bonded internally between each other and externally with another monomer unit giving the guar polymer an endo β 1 $\rightarrow$ 4 and an exo β 1 $\rightarrow$ 4 linkage between mannose-mannose units.⁵⁹ Similarly, the mannose unit is bonded to the galactose unit at α O-6 linkages between the mannose-galactose units.⁶⁰

Figure 2.1 shows the guar polymer to have a mannose to galactose group ratio of 2:1. A ratio of about 1.6:1 has been actually observed in the guar polymer.⁵⁷ The difference in the two ratios is because of random substitution of galactose side group on the mannose backbone throughout the polymer molecule.⁶¹⁻⁶² As a result, a guar polymer is highly polydisperse.^{58,63-65} The guar gum obtained from different sources have different

mannose to galactose ratio and dissimilar distribution of galactose side groups along the mannose backbone.⁶⁰ The commercial polysaccharides, therefore, differ in the molecular weight and molecular weight distribution. Some molecular parameters of a typical guar polymer are summarized in Table 2.1.

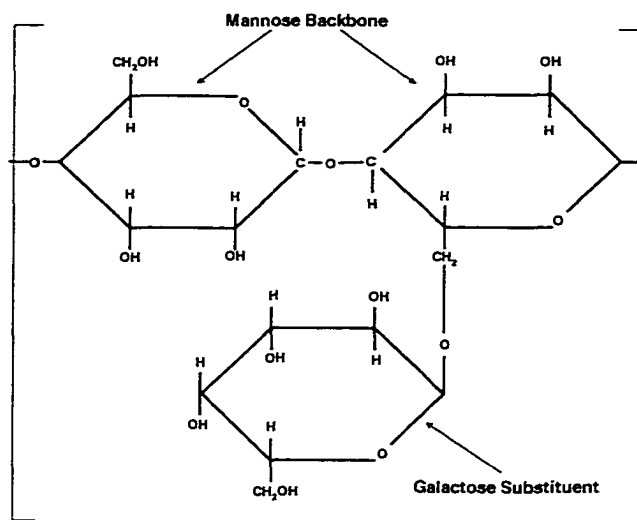


Figure 2.1—Structure of Guar Gum

Table 2.1- Molecular Parameters of Guar Polymer^{60,64,66}

Molecular Parameter	Value
n_r , Number of segments of monomer units	8000 (approx.)
l_r , Length of a segment of monomer unit	0.54 nm
l_c , Contour length ($n_r l_r$)	4.3 μm
l_p , Persistence length ($n_r^{1/2} l_r$)	48.3 nm
C , Characteristics Ratio	12.6
R_g , Radius of Gyration ($C n_r^{1/2} l_r$)	0.17 μm

The linkages in the guar gum are unstable and can be easily broken by high temperature, mild acid solutions or enzymes designed to attack any of the three linkages.^{56,59,61} The guar gum is also susceptible to biological attack from aerobic bacteria and sulfate-

reducing bacteria. The biological degradation of guar is prevented by adding biocides that inhibit the growth of microorganisms in the solution.

Because of the ease with which guar polymer can break, the guar solutions in this work were prepared in water having biocides. These solutions were stored under ambient temperature and at neutral pH condition. The solutions were formulated for one or two days within which new batch of the solution was prepared. On the other hand, their susceptibility to acid was utilized to break the guar solutions before discharging them into the Norman City disposal system.

Guar Hydration. The guar polymer easily dissolves in water and readily establishes hydrogen bond with the water molecule and gets hydrated.^{56,57} The hydration of the polymer particle causes it to swell, exposing more sites on the guar to establish more hydrogen bonds with the water molecule. The hydration of the polymer continues till each guar molecule is well bonded with the water molecule. Since hydration of few sites on the polymer exposes more sites for bonding, the guar hydration is a time-limited process where the time required to bond depends on the time taken to expose these sites. The hydration rate can be enhanced by either raising the temperature, increasing the mixing rate of guar particles in water, or by simply allowing more time for guar and water to interact.⁶⁷ The hydration rate is monitored with guar solution viscosity which varies as an exponential function of the time for hydration. When no further increase in the viscosity is observed, the guar hydration is considered to be complete.

During the hydration, the guar particles should be well dispersed. The dispersion helps to form a homogeneous polymer solution and allows all guar particles to hydrate

uniformly. The uniform dispersion prevents formation of balls of gel mass. The gel balls are formed if the external polymer particles are hydrated preventing the internal guar particles to come in contact with the water. This restricts further hydration and leaves a ball of partially hydrated guar. These gel balls are also called fish eyes and are undesirable. They reduce the concentration of guar in the solution and can plug off small openings during a fracturing treatment.

The formation of gel balls is prevented by increasing the surface area through a very fine grinding of the guar powder and by subjecting the solution to high shear as the guar is mixed in water. A more innovative method to prevent formation of fish eyes is to coat the guar powder with a small amount of a borate-crosslinking agent.^{68,69} Yet another technique used to prepare homogeneous solution is to heat the guar solution at 175°F for 30 min.⁷⁰

The crosslinker coated guar powder requires a special procedure to mix guar in water. Before adding the guar powder to water, the water pH is raised to about 10. The powder disperses easily under the alkaline condition but does not get hydrated or form a gel ball. At high pH, the guar and the borate-crosslinker react to form a complexation compound leaving no guar to react with water. This compound is broken by lowering the solution pH to 7. At neutral pH, the well-dispersed guar particles are free from the crosslinker and are available for hydration, preparing a homogeneous guar solution. Because of the advantages of this special procedure, it was used in the present work to hydrate guar and prevent the fish eyes formation in the guar solutions used.

Guar Sensitivity. The guar solution is influenced by temperature, shear environment and presence of salts in water.

The guar solution is compatible with monovalent salts like sodium chloride (NaCl) and potassium chloride (KCl) etc. Its compatibility with polyvalent salts, found in sea water, depends on the salt concentration.⁵⁶ Special precautions are therefore needed to prepare guar solution in sea water for fracturing application in offshore wells.⁷¹ The presence of salts in the guar solution screens the charge on the polymer.⁷² This screening causes polymer chains to coil together thereby decreasing the radius of interaction of the polymer molecule and increasing the overlap concentration of the guar in solution.

The salts are sometimes added to the guar solution because they prevent swelling of water-sensitive clays in the formation. In a fracturing treatment, the guar solution is usually prepared in 2% KCl water for fracturing treatment.¹⁰ No KCl was however added to the guar solution used in the present research. Its absence does not affect the research objective of correlating fluid rheology with proppant transport through a fracture.

The chemical bonds on the different units on the guar polymer are sensitive to temperature, so the guar solution is sensitive to temperature. The guar performs less effectively at high temperatures.¹⁰ A higher guar concentration is therefore required at elevated temperature to obtain similar fluid performance to that at low temperature.

The guar polymer hydrates to form a flexible random coil configuration.^{30,60,63} This configuration is influenced by shear, so the guar solution exhibits a non-Newtonian fluid behavior.^{73,74} At low shear rates, guar polymer chains resist molecular orientation; so, the guar solution exhibits a Newtonian viscosity behavior at these shear rates. When

the shear rate is slowly increased from the Newtonian plateau region, the chains begin to align with the applied shear. This alignment results in an increase in the end to end distance of the polymer and a corresponding increase in the solution viscosity with shear rate.⁷⁵ This shear-thickening behavior is very typical characteristics of guar polymer and is observed in a very small regime just before the viscosity begins to decrease with shear. As the shear rate is further increased, the solution viscosity decreases and a shear-thinning behavior is observed. A typical viscosity-shear rate curve of guar solution is shown in Fig. 2.2. The figure shows Newtonian plateau, shear-thickening and shear-thinning regions in the guar viscosity curve. Thus, the shear environment influences the characteristic properties of the guar and possibly, its structure.

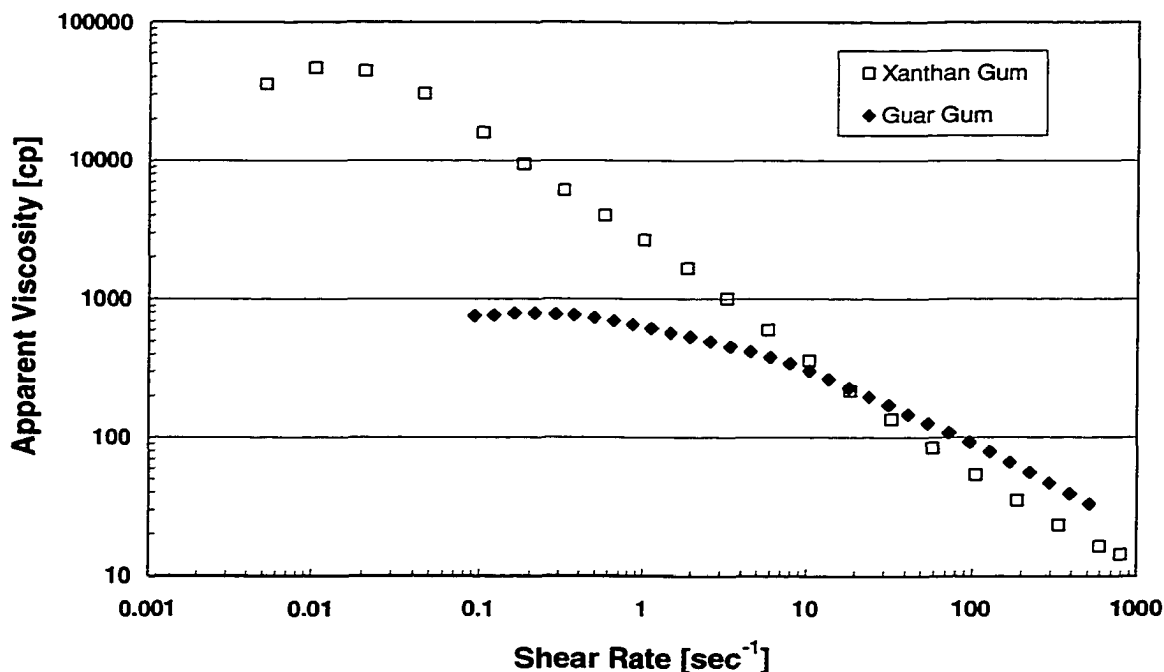


Figure 2.2—Comparison of Viscosity of 40 lb/Mgal Guar and Xanthan Solutions

The flexible coil configuration of guar in solution makes guar ideal for fracturing application. The flexibility in the guar polymer imparts it less sensitivity to shear than that of a xanthan polymer, which has a rigid helical configuration. As a result, the guar solution has lower viscosity than that of xanthan at low shear rates, but has higher viscosity than that of xanthan at high shear rates. The viscosity of guar solution is compared to that of xanthan solutions is shown in Figure 2.2 for similar concentration of the polymers. For fracturing, a high fluid viscosity is required to keep proppant particles in suspension when fluid is flowing through a fracture. Whereas for drilling applications, a high viscosity is required to keep drill cuttings in suspension under static fluid conditions during a tripping operation. The viscosity curves as shown in Fig. 2.2 provide a very good indication of a fluid performance in the field.

The above discussion explains that a fundamental understanding of the polymer structure helps to describe the behavior of the polymeric solution under conditions applicable to the petroleum industry.

Disadvantages of Guar. Guar is one of the cheapest water-soluble polymer used in the petroleum industry.⁵⁶ It, however, has one huge disadvantage. The guar cannot be completely broken down into its constituent sugar units. As a result, it leaves undesirable polymer residue in the propped fracture and in the surrounding formation where it leaks off during the fracturing treatment. This residue reduces the permeability of the reservoir and the proppant pack, and restricts oil and gas flow into the wellbore. The insoluble residue in guar is about 6 to 8% by weight.⁵⁷ Since millions of gallons of fluid are pumped in a fracturing treatment, a large amount of the residue can be left behind which

can severely damage the formation after the treatment. Proppant conductivity impairments of 15 to 75% have been observed from guar based fracturing fluids.^{76,77}

Several techniques have been developed to minimize the damage to the formation from the polymer residue. The guar polymer is chemically treated with propylene oxide and chloroacetic acid to prepare derivatized forms of guar, such as hydroxypropyl guar (HPG) and carboxymethyl guar (CMG).⁷⁸ These forms can be further treated to prepare carboxymethyl hydroxypropyl guar (CMHPG).⁷⁹ The derivatised forms leave less polymer residue than the guar polymer. The HPG solution leaves 2 to 3% residue, and CMHPG solution leaves only 1 to 2%.^{56,57,80} These chemical treatments have helped to reduce the polymer residue, but they also have offset the cost advantage of the guar polymer because of the additional expense of chemically derivatizing the polymer.

Another method to reduce polymer residue is to crosslink the guar polymer.¹⁹ The crosslinking can be performed at lower guar concentrations, so the crosslinked guar leaves less residue in the formation. Furthermore, the crosslinking of guar imparts better fluid characteristics than those obtained in the non-crosslinked guar solutions by increasing the polymer concentration.

The crosslinked guar were earlier prepared in guar concentrations of 30 to 50 lb/Mgal. These crosslinked guar gels were designed for a wider temperature range application.²⁵ In recent years, the crosslinked gels have been prepared at low guar concentrations of 20 to 25 lb/Mgal.^{25,81,82} The newer gels are however prepared for a smaller temperature variation of 10 to 20°F, so they are formulated for specific applications. This narrow range of application requires a critical understanding of the crosslinked gel performance and its characteristic properties.

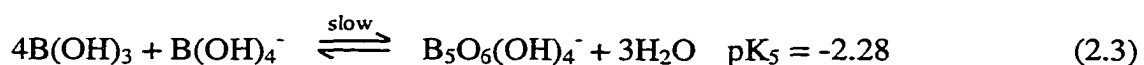
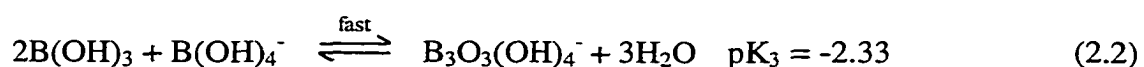
2.2.2 Crosslinking Agent

Guar based polymers are crosslinked most commonly with either of the two crosslinking agents, borate or zirconate based systems. The borate-crosslinked gel accounts for almost 75% of the fracturing fluids.²⁰ The borate systems are also the more studied crosslinking species. Whereas, very little information is available in the open literature on zirconium crosslinking agents.^{16,83-85} Even the crosslinking mechanism between the zirconium crosslinker and guar polymer is not well understood. A detailed information on the zirconium crosslinker solutions is presented in Chapter 7. The following section describes only the borate based crosslinker solutions.

The borate-crosslinking agent is obtained from boron compounds. The characteristic properties of various boron compounds are used to prepare special formulations of borate-crosslinked gels for fracturing application. The main function of the boron compounds is to release mono-borate ions that establish crosslink bonds with hydroxyl groups on the guar polymer, and form borate-crosslinked guar gels.

Boron Compounds Dissociation. The boron compounds dissociate in alkaline solutions to form two primary species: boric acid, $B(OH)_3$, and mono-borate ions, $B(OH)_4^-$. These two species are only present at low concentration of the compounds. As the boron compound concentration is increased, the crosslinker solution may dissociate further to form polyborate ions. The polyborate ions are considered to be formed in alkali borate solutions at concentrations above 0.2M.⁸⁶ The presence of polyborate ions has been confirmed by several researchers, the identification of the individual polyborate species has been difficult because of the presence of multiple ions whose stability and

concentration vary with the experimental condition.^{87,88} The dissociation studies performed using Nuclear Magnetic Resonance (NMR),^{88,89} Raman Spectroscopy,^{90,91} and potentiometric techniques⁹² have shown that two polyborate species are predominantly present in the solution. They are tri-borate, $B_3O_3(OH)_4^-$, ions and penta-borate, $B_5O_6(OH)_4^-$, ions.⁹³ The boron compounds dissociate according to the following equilibrium reactions,^{89,91-95}



The dissociation constants (K 's) of the three reactions at 75°F are also shown in the equations. The dissociation constant of the first reaction is well established. But those of the second and the third reactions are reported to be different, varying from 1.9 to 2.6.^{89,91,92,95} Because of this variation, the dissociation constants reported by several studies were averaged in the above equations. Equations 2.1 and 2.2 are fast reactions, so borate ions and boric acid are in rapid equilibrium with each other, whereas Eq. 2.3 is a slow reaction.

Influence of Boron Dissociation on Crosslinking with Guar. The crosslinking ion in the reaction between the boron compound and the guar polymer is the mono-borate ions. As guar is crosslinked, the concentration of borate ions is reduced in solution and the equilibrium dissociation of boric acid is disturbed. This shift in the equilibrium

concentration results in the release of more mono-borate ions from the boric acid and polyborate ions. The released borate ions can then further crosslink with guar. Since the polyborate ions dissociate back to mono-borate ions, their presence in boron compounds does not reduce the concentration of mono-borate ions during borate-crosslinking with guar.¹⁰ Therefore, the isolation of individual polyborate species is not critical in the crosslinking reactions. The availability of mono-borate ions determines the performance of borate-crosslinked gels, so the important function of boron compounds is to provide sufficient mono-borate ions that can crosslink with guar polymer chains during a fracturing treatment.

Equation 2.1 shows that the concentration of borate ions increases at higher hydroxide concentration or higher fluid pH. The dissociation constants of reactions in Eqs. 2.2 and 2.3 suggest a decrease in the polyborate ions concentration at high pH.^{90,91} These reactions indicate that similar concentration of mono-borate ions can be achieved in a smaller concentration of boron compound in high pH solution as that in a higher concentration solution prepared at low fluid pH.^{10,82} Therefore, borate-crosslinked gels having similar borate ions concentration can be prepared by changing the solution pH and the borate solution concentration.

The dissociation of boric acid to borate ions is an exothermic reaction. The dissociation equilibrium Eq. 2.1 shifts backwards as the temperature is increased. This shift would result in a decrease in the mono-borate ions concentration at a given boron concentration.⁹² Since a petroleum reservoir exists under geothermal conditions, the concentration of borate ions would decrease as the temperature increases from ambient in

the surface equipment to elevated condition in the reservoir. Therefore, the concentration of borate-crosslinking agent should be adjusted to the reservoir temperature.

The concentration of borate ions in a crosslinker solution can be determined at any temperature using temperature dependency of the dissociation reaction between boric acid and borate ions.^{96,97} The dissociation constant K_I is empirically correlated to the temperature by⁹²

$$\log K_I = \frac{1573.2I}{T} + 28.6059 + 0.012078T - 13.2258 \log T + (0.325 - 0.00033T)I - 0.0912(I)^{3/2} \quad (2.4)$$

where T is temperature in °K, and I is molal concentration of KCl in solution. Eq. 2.4 is valid for I from 0 to 1 molal, and T from 273 to 573 °K. The dissociation constant is negligibly dependent on pressure up to 2000 psi,⁹² so the concentration of borate ions should not be a function of the pumping pressure during a fracturing treatment. Equation 2.4 can be used to determine fraction of borate ions in a crosslinker solution at different pH and temperature. Figure 2.3 shows the borate ions fraction as a function of the fluid pH and temperature. The figure displays an increase in the borate fraction with an increase in the fluid pH and a decrease in the fraction with an increase in the solution temperature.

Borate-Crosslinker Compounds. The most widely used boron compounds for fracturing application are boric acid and borax. The solubility of these compounds is however low at ambient temperature conditions under which a fracturing fluid is formulated. Because of the low solubility, these compounds provide low concentration of borate ions at the surface facilities.

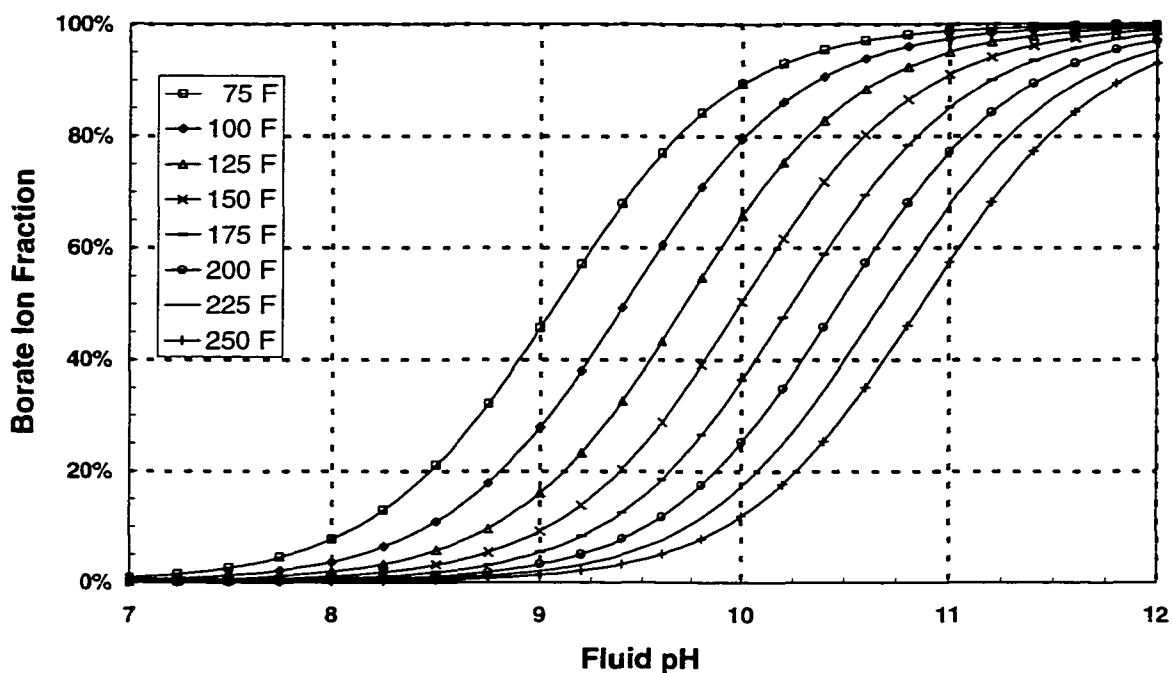


Figure 2.3—Variation of Borate Ion Fraction with Fluid pH and Temperature

The low solubility of these boron compounds is improved by adding borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, to saturated boric acid solution or vice versa, and forming polyborates in the solution. The polymerization allows more borax or boric acid to dissolve in water.⁹⁴ This technique allows manufacturing of disodium octaborate tetrahydrate, $\text{Na}_2\text{B}_8\text{O}_{13} \cdot 4\text{H}_2\text{O}$ (US Borax, CA, Trade Name POLYBOR™) by spray drying mixtures of borax and boric acid.⁹⁸ The solubility of this compound at 20°C is about 10 wt.%, which is two to three times that of either boric acid or borax.⁹⁴ In the present work, POLYBOR was used as the source of the crosslinking ions for preparing borate-crosslinked guar gels.

The sodium-borate compound, disodium octaborate tetrahydrate, is amorphous, so its constituents, boric acid and borate ions, concentrations in solution are difficult to ascertain. This compound contains about 14.5 wt.% Na_2O and 66.3 wt. B_2O_3 . The ratio

of Na_2O to B_2O_3 is about 0.22, a value which is very close to 0.25 for sodium pentaborates.^{88,98} So the concentration of boric acid and borate ions in POLYBOR can be approximated from the constituents of sodium pentaborate.^{88,93} Dr. Bob Smith (US Borax) mentioned that POLYBOR contains almost 95% by wt. boric acid,⁹⁹ and Phil Harris (Halliburton) suggested to approximate that each pound of POLYBOR is equal to 1.8 lb of boric acid.¹⁰⁰ One mole of this compound can be considered equivalent to 8 moles of boric acid. Thus, the boric acid equilibrium reactions given by Eqs. 2.1 to 2.3 can be used for this commercial compound.

The above discussion on the borate crosslinker agent shows that the concentration of borate ions is influenced by fluid pH, temperature and crosslinker solubility. This in turn means that the borate crosslinking with guar is a function of these parameters. Hence, a close control of borate chemistry is important to understand fracturing fluid characterization and proppant transport behavior in a fracture.

2.2.3 Borate-Guar Crosslinking

Why crosslink? The characteristics properties of guar solution come from the large molecular weight and entanglement links in the guar polymer. The greater the number of links in the guar polymer, the better is the fluid capability to suspend proppant. In a non-crosslinked guar, these links are induced by increasing the guar concentration, so that the individual chains in the polymer are entangled with one other. The entanglements create large number of couplings in the polymer chain and thus impart strength to the guar.

The entanglement couplings can also be induced by chemically creating the associations between the chains.¹⁰¹ The chemical association also called crosslinking

increases the polymer molecular weight, which has the similar effect on the gel properties to that obtained with an increase in the polymer concentration.

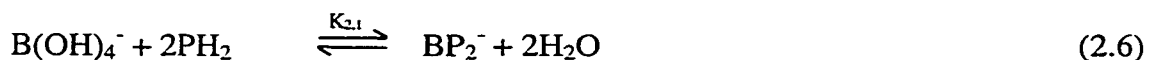
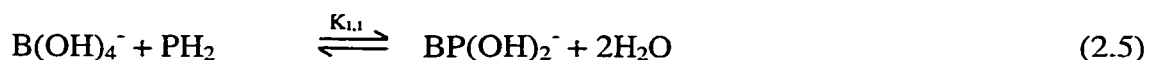
The enhanced capabilities of the crosslinked polymer have numerous applications. Borate crosslinking with starch is used to prepare adhesives that have very good fluid properties and very high strength. These adhesives find applications in carton sealing, corrugated box board, and in textiles for binding together fibers for denim, sateen etc.^{98,102,103} The crosslinking association has helped to increase the viscosity and elasticity of the polymer.^{1,104} It improves the strength of the crosslinked polymer without requiring to increase the polymer concentration. As a result, the crosslinking of guar has been a very useful technique to reduce formation damage from the fluids pumped during the hydraulic fracturing treatments.

What is crosslinking? Crosslinking links the polymer chains together. These links are established either within the same chain, intramolecular crosslinking, or between the different chains, intermolecular crosslinking. The intramolecular crosslinking only links the same chain, so it does not help to increase the molecular weight of the polymer. It contributes little to the elasticity of the crosslinked gel,²⁸ so is undesirable. The intermolecular crosslinking, on the other hand, links together different polymer chains, so it increases the molecular weight and elasticity of the polymer. As a result, the intermolecular crosslinking is desirable, and is aimed for in borate-crosslinking of guar.

Several studies have shown that hydroxyl groups present on polyols compounds are the possible sites for crosslinking with boron compounds. The hydroxyl groups on

the same monomer or on adjacent monomer can crosslink to form a complexed compound.¹⁰⁵ The complexation of small, model polyols with boron compounds has been studied using Raman spectroscopy and Nuclear Magnetic Resonance NMR. These studies have concluded that the borate-crosslinking occurs only above pH 7.^{101,106} This pH is also the limiting value above which boric acid begins to dissociate into monoborate ions as shown in Figure 2.3. It means that the effective crosslinking species in the complexation reaction between the boron compounds and polyols is the monoborate ions. This is confirmed further from the oxygen-oxygen separation distance in the tetrahydroxy monoborate anion of 2.4 to 2.44 Å, which compares well with the separation and conformation of hydroxyl groups in the polyols.¹⁰⁵ Thus, the guar crosslinking with borate ions is a complexation of adjacent polymer molecules.¹⁰⁷⁻¹⁰⁹ Among the different hydroxyl groups present on a guar molecule, shown in Fig. 2.1, *cis* conformation of the hydroxyl group is a better orientation than *trans* for complexation with borate ions.¹⁰⁵ As the *cis* conformation is available on both galactose and mannose unit of a guar molecule, both units are possible sites for crosslinking with guar.¹¹⁰

The mechanism of crosslink bonding in the complexation reaction is considered through either electron sharing, because both borate ions and polymer crosslink sites are negatively charged,³⁰ or hydrogen bonding between the species.^{106,111} The crosslink bond between one borate ion and a pair of hydroxyl group on the polyol results in the formation of 1:1 monodiol compound. The monodiol can further complex with another hydroxyl group pair to form a 2:1 didiol compound. These complexation reactions are represented as



where PH_2 represent the polyol compound, and $K_{1,1}$ and $K_{2,1}$ are dissociation constants of the 1:1 and 2:1 complexation reaction. The dissociation constants for the complexation between boric acid and different polyols are summarized by Sanderson.¹⁰⁵

A polyol having low diol concentration complexes with borate ions to predominantly form 1:1 monodiol compound.¹¹² In a large polyol such as guar, the diol concentration is so high that the number of 2:1 didiol complexed compounds becomes significant. The 2:1 didiol compound is formed on the same guar chain from intramolecular crosslinking or on different guar chains from intermolecular crosslinking. It is the di-complexed compounds that contribute to the macroscopic properties required for satisfactory proppant transport during a fracturing treatment.¹⁰

Factors that Influence Crosslinking. The borate-crosslinking with guar is influenced by the parameters which control the availability of monoborate ions and hydroxyl group, and those that affect the crosslinking. The parameters controlling the monoborate ions are fluid pH, temperature and boron compound concentration, and those influencing the guar polymer are shear, temperature and presence of salts. These parameters were discussed earlier in the previous sections. So only those conditions that affect the crosslinking reaction itself are discussed below.

The crosslinking reaction is affected by orientation and expandability of the guar chains, and the energy required to form or break crosslink bonds. Guar is a flexible random coil molecule and its chains configuration changes with shear. This change

exposes the hydroxyl groups on the polymer for possible crosslinking with borate ions. When a borate-crosslinked gel is subjected to low shear rates, the guar chains unwind resulting in an acceleration of the complexation reaction with the borate ions. The increase in the number of complexes formed causes the gel viscosity to increase with shear. Consequently, the gel exhibits shear thickening behavior at low shear rates.^{113,114} The alignment of the guar chains with shear is also seen in the Atomic Force Microscopy (AFM) images of the crosslinked gels subjected to low shear rates.¹¹⁵ The reason for the shear thickening behavior at low shear rate is theoretically described to be due to a higher probability of forming new crosslink bonds than that for the existing bonds to break off from an increased tension during the shearing.¹¹⁶

In addition to changes in the guar chains with shear, the guar molecule can expand in length as guar complexes with borate ions. At low borate ions concentration, the guar forms intramolecular crosslink which causes the chains to contract. But as the chains are further crosslinked with borate ions, the complexation imparts a negative charge to the crosslinked guar. The charged molecules repel each other causing chains to expand.^{112,117} This repulsion decreases the formation constant of mono-complexed compound, prevents further crosslinking in guar and thus, limits the total number of crosslinked sites formed in guar.¹¹⁸ The limitation in the number of complexed compound formed on the guar molecule means that no further improvement in the crosslinked guar characteristics can be achieved despite further increase in the crosslinker concentration.

The enthalpy for the crosslinking reaction between hydroxyl groups and borate ions is -5 kcal/mol, and the energy required to break individual crosslink bonds is 6

kcal/mol.¹⁰⁹ These low energy values make it easy for crosslink bonds to break and reform under shear. Secondly, the lifetime of interaction between borate ions and polymer is considered to be 1 millisecond,⁸⁵ which is extremely small as compared to the duration of a fracturing treatment. Since shear is readily applied during mixing and pumping a fracturing fluid and the lifetime of the crosslink bonding is small, the crosslinked bonds break and reheal, and the crosslinked gel characteristics continuously change during a fracturing treatment. So the shear environment as well as the duration of the shear are significant parameters that influence the characteristics of a borate-crosslinked gel. The shear sensitivity of borate-crosslinked gels implies that these gels be prepared and characterized with a careful consideration of the shear conditioning of a fracturing fluid in the surface equipment and tubular prior to their flow through a fracture. Therefore, it is important to incorporate shear history, both shearing value and shearing time, while formulating crosslinked fluids for fracturing applications.^{119,120}

Temperature affects the performance of borate-crosslinked guar gel in several ways. It affects the borate ions concentration through changes in the boron compound dissociation, as described earlier in Section 2.1.2. It can also reduce the number of didiol complexes by shifting the equilibrium reaction between the borate ions and hydroxyl groups.^{109,121} Thirdly, it can break guar polymer chains through attack on the acetal linkage between the sugar units on the guar molecule thereby decreasing the chain length.¹²¹ All these factors result in a decrease in the number of crosslinked bonds formed on a borate-crosslinked gel as the temperature is increased. Since crosslinking imparts strength to the gel, these factors also affect the gel capability to suspend and transport proppant through a fracture.

A unique feature of borate-crosslinked guar gels is that they exhibit demixing or phase separation.¹²² This behavior causes the crosslinked guar to separate from solvent through a diffusion or expulsion of water from the polymer-rich phase. The demixing is described to be due to a competition between the energy gained when a gel crosslinks and the entropy decreased from an increase in the polyol concentration in the solution.¹²³ The demixing indicates formation of a fully crosslinked gel.¹⁰ This phase separation is, however, undesirable, as it results in ineffective crosslinking. A phase-separated gel does not have sufficient viscosity to create a fracture, and cannot transport proppant because of its non-homogeneous characteristics.

The gel demixing can be reversed to a favorable crosslinked gel by either heating the gel or adding a smaller polyol compound, which can preferentially complex with borate ions. The two methods reduce the availability of borate ions for crosslinking with guar molecule, thereby, decrease the probability for phase separation in the crosslinked guar gel.

2.2.4 Importance of Fracturing Fluid Chemistry

A very relevant question at this stage would be the reason for understanding the chemistry of fracturing fluid in the present research. There are several good reasons for understanding the fluid chemistry. Foremost among them is the use of this knowledge to carefully control fluid formulations for successfully stimulating a reservoir. This understanding has helped to develop and use water-based crosslinked guar gels for fracturing formations at temperatures of 300°F and beyond.^{9,10,124}

Secondly, the guar solution leaves a polymer residue in the proppant pack. So large efforts are being made to reduce the polymer concentration used in a fracturing treatment. An understanding of the chemistry of crosslinked gels has been useful to prepare low polymer loading gels and minimize damage to the stimulated formation. These low polymer gels have been used to perform thousands of fracturing treatments around the world.^{25,81,82,125}

One recent example of this understanding is a unique modification for guar.¹²⁶ This modification proposes addition of an anion to the guar which charges the polymer molecule such that the modified units repel each other causing guar to expand and exposing more hydroxyl groups for crosslinking.^{127,128} The proposed modification has reduced the amount of polymer in a derivatised guar for the crosslinking reaction.¹²⁵

Another application of an understanding of the chemistry is to delay the crosslinking reaction between the hydroxyl groups and borate ions. This application is described below in detail.

Delayed Crosslinking. A fracturing fluid is subjected to high shear in the surface equipment and during its flow through the wellbore and perforations. Because of the shear sensitivity of crosslinked gels, the guar should be crosslinked only after the fluid has passed the high shear environment. This method where the guar is not crosslinked prior to its flow through a fracture is called delayed crosslinking.

The delay in crosslinking has several advantages. The crosslinked gel does not degrade in the high shear environment and thus retains its characteristic properties for a satisfactory performance in a fracture. Secondly, a non-crosslinked gel has lower friction

losses through tubular than a crosslinked gel.^{129,130} Lower losses reduce the surface pressure required to fracture a formation and thus, decrease the hydraulic horsepower of the pumping equipment used during a fracturing treatment. The pumping cost being a major portion of the fracturing operation,¹⁸ the delay in crosslinking improves the economics of stimulating a well.

There are number of methods by which the crosslinking reaction can be delayed. Since the crosslinking reaction is a bonding of mono-borate ions with hydroxyl groups, a controlled release of borate ions can satisfactorily delay the guar crosslinking. This method is the most widely used technique in the industry, and has been the focus of several patent applications. The release of borate ions can be controlled by either manipulating the dissociation of boric acid using Eq. 2.1, or crosslinking the ions with a smaller polyol compound which is more active but less stable than guar.

The dissociation of boric acid is controlled through either the fluid pH or the boric acid source. Mondshine¹³¹ proposed a slowly soluble borate source like alkaline earth metal borate salts whose solubility increases with temperature and which releases borate ions only at high temperature. Nimerick¹³² described a buffer solution that exhibits little changes in pH with temperature and thus, the borate ion concentration is least affected by the temperature changes. Harris et al.¹³³ buffered their formulation to a pH between 8 to 9 and thus, minimized the effect of temperature on fracturing fluid. Harris and Heath¹³⁴ prepared an overcrosslinked gel mass and used its low solubility in solution to delay the borate crosslinking with guar. Nelson et al.¹³⁵ encapsulated the borate ion source with a polymer coating and added a polyol for delaying the crosslinking and stabilizing the fluid at high temperature. Dawson,¹³⁶ similarly, described the use of an aldehyde-based

additive, and Card et al.¹³⁷ proposed a different polyol having at least two hydroxyl groups. These additives chemically bond with borate ions at ambient temperature but break down under downhole conditions, thereby delaying the release of borate ions for crosslinking with fracturing fluid.

The delayed crosslinking of guar has reduced the pumping cost without affecting the performance of fracturing fluid in the stimulation operation.^{129,138} This delay methodology is important for borate-crosslinked gels where the crosslink bonds break and reheal quickly and are reversible. It is even more important in zirconate-crosslinked gels, which form irreversible gels. The high shearing rate permanently damages, on a time scale of fracturing treatment, the zirconate-crosslinked gels and impairs their characteristic properties.

2.3 FRACTURING FLUID CHARACTERIZATION

The fluid characterization provides an easy and inexpensive method to evaluate the fluid performance before using it in the stimulation of a well. The fluid must, therefore, be characterized under conditions that are representative of the stimulation environment which a fluid experiences during a fracturing treatment.

There are two aspects of characterizing a fracturing fluid. First is to prepare the sample as it would be prepared in the field, and the second is to evaluate the fluid under the shear conditions of a fracturing operation. These two aspects are discussed below in detail.

2.3.1 Mixing and Shear Preconditioning

Mixing. Fracturing fluid contains several chemicals that are added to perform specific functions in the fluid. These chemicals should be added in the same order for the characterization study as would be used in a field operation.

The borate-crosslinked guar gels are prepared by mixing borate crosslinker solution to a guar solution and adjusting the resultant mixture pH to a value where the crosslinker solution would release borate ions for crosslinking. The fluid mixing procedure is simple and straightforward, but the chemicals and their addition sequence depend on the formulation procedure recommended by the company from which the chemicals are purchased. The recommended procedure should be carefully followed to prepare consistent gels in different test runs.

The crosslinker and guar polymer should be homogeneously mixed for uniform crosslinking of guar.²⁹ Guar crosslinked under dynamic mixing conditions exhibits homogeneous crosslinking, whereas batch mixed crosslinked gel contains gel domains having varying crosslink densities.¹³⁹ The gel therefore should be kept under static conditions for a minimal duration from the time the gel is crosslinked to when it is characterized.¹⁴⁰

Shear Preconditioning. The bonds between the borate ions in the crosslinker and the hydroxyl groups on guar are labile, and they break and reheal under shear. Several studies have shown that the viscosity of guar gel is a strong function of shear conditioning prior to the viscometric measurements of the gel.^{119,140-142} The borate-crosslinked gel is observed to have much higher viscosities when the gel is not shear

conditioned as compared to when it is conditioned even for one min. Moreover, the gel shear pre-conditioning not only simulates the field operation but also helps to improve the replicability and reproducibility of the crosslinked gel characterizations.¹⁴³

Power et al.¹¹⁵ observed microscopic images of the gel prepared under different shear history conditions and found that the strength of the crosslinked gel was enhanced when pre-conditioned under moderate level of shear. They described the increase in gel strength to an alignment and ordered arrangement of the gel structure under the applied shear. When the gel was pre-conditioned at high shear rates, they observed the gel composed of globular structures and had poor strength.

Goel et al.¹⁴⁴ compared laboratory measurements with field-scale characterization of borate-crosslinked gels. The laboratory scale simulations were performed using viscometers available at three service companies test facilities, and the field-scale operation was simulated at the University of Oklahoma. The comparison showed huge difference in the measured viscosities even though identical chemicals and similar fluid formulation and characterization procedure were used in all laboratory facilities. These differences were found to originate from the dissimilarities in the method of sample mixing, mode of transferring the sample to the viscometer, rate of sample heating, and magnitude of the shear rates during the sample preconditioning and heating. The viscosities measured with the viscometers differed 25 to 100% from the values obtained on the field-scale fracture simulator.

Because of the importance of the shear histories in the crosslinked gels, American Petroleum Institute (API) recommends two shear preconditioning rates.¹⁴⁵ To simulate a fracturing treatment of a reservoir at temperatures less than 200°F, API recommends

shear conditioning the fluid at 675 sec^{-1} for a period of 2.5 min., and for temperatures above 200°F , it recommends to shear at 1350 sec^{-1} for 5 min.

2.3.2 Fluid Rheology

After preparing and shear preconditioning the fracturing fluid, it is evaluated for its characteristic behavior under fluid flow conditions. This evaluation is accurately performed using fluid rheology. The rheology measurement helps to predict the response of the fluid under the stresses imposed during a fracturing treatment.

The relationship between the applied stress and the resultant strain in fluids like water or oil is linear. Such fluids are called Newtonian fluids. In fracturing fluids, however, the stress and strain rate are non-linearly related; so these fluids exhibit non-Newtonian behavior. The ratio of stress to strain rate, called viscosity, is constant in the Newtonian fluid; the same is a function of strain rate in the non-Newtonian fluids.

Viscosity. Because of the non-Newtonian behavior, the fracturing fluid characteristics change during different stages of the stimulation treatment. Its viscosity is constant at low shear rate, increases in a very narrow shear rate region and then decreases in the intermediate shear rate regime. The gel thus exhibits a Newtonian plateau at low shear rates, a shear thickening behavior in the intermediate region, and a shear thinning region at high shear rates.

The shear-thinning region is the most important characteristics of a fracturing fluid, as it corresponds to the fluid response in a fracture. As a result, the fluid viscosity

is largely studied in this region. The shear-thinning response is represented by a Power law model, given by

$$\mu_a = k \left(\dot{\gamma}_w \right)^{n-1} \dots\dots\dots (2.7)$$

where μ_a is apparent viscosity at a wall shear rate of $\dot{\gamma}_w$, and k and n are consistency index and power law index of the model. Though the Power law model is widely used in the industry, it has certain limitations. This model presents an incomplete picture of the fluid response. It does not describe the Newtonian plateau and the shear-thickening regions of the crosslinked guar's non-Newtonian behavior. It overpredicts the fluid viscosities at low shear rates. Despite these limitations, the Power law model is extremely popular in the industry because of its simplicity. It is also used in the computer models used to design and simulate hydraulic fracturing treatment.^{1,146,147}

The fracturing fluid characteristics are affected by temperature and shear history. The power law model incorporates the effect of temperature on viscosity through an Arrhenius type correlation.¹⁴⁸ The temperature is correlated through a dependency of model parameters, n and k , on temperature.¹⁴⁹ This model, however, does not incorporate the shear history of the fluid into its viscosity.

Recently, Goel et al.¹²⁰ proposed a unique method to incorporate the shear history into the viscosity measurements. Their model correlates the viscosity of a crosslinked gel to wellbore shear conditioning, reservoir temperature, and fracture shear rates. The model is based on the method of reduced variables, and is given as

$$\frac{b_T}{a_T} \mu_a = K \left(a_T \dot{\gamma}_w \right)^{A \log \left(a_T \dot{\gamma}_w \right) + B} \dots\dots\dots (2.8)$$

where K , A , and B are empirical constants, and a_T and b_T are horizontal and vertical shift factors, which are correlated to temperature and fluid shear history as¹²⁰

$$\log a_T = \frac{-c_1(T-T_o)}{(c_2+T-T_o)} * \frac{1}{1+c_3N_s} \dots\dots\dots (2.9)$$

$$\log b_T = \frac{-d_1(T-T_o)}{(d_2+T-T_o)} * \frac{1}{1+d_3N_s} \dots\dots\dots (2.10)$$

In these eqns. c_i 's and d_i 's are empirical constants, T the reservoir temperature and T_o a reference temperature at which the fluid viscosity is independent of the shear history. N_s is a shearing number, which describes the shear history of a fluid in terms of its pressure drop across the tubular. The proposed model describes the power law as well as shear thickening regions in the viscosity-shear rate curve of a borate-crosslinked gel. It, however, has some limitations. The model was developed from experimental data gathered on certain fluid formulations, so its wider applicability is not established. Like Power law model, it does not predict the low-shear Newtonian viscosity of the fracturing fluid.

The viscosity of a fracturing fluid is not just important in the shear-thinning region, it is significant in the low shear region also.¹⁵⁰⁻¹⁵⁴ It is observed that the proppant settling velocities observed experimentally under stationary fluid conditions are higher than the calculated values; while the settling velocities observed in the fluid flow conditions are lower than those calculated with the Power law based correlation. The significance of low shear properties, during the fracture closure, makes it important to

consider and incorporate viscosity at low shear rates to describe the proppant suspension behavior.¹⁵¹⁻¹⁵³

Viscosity Measuring Instruments. Fracturing fluid viscosity is measured using different viscometers; pipe, rotational, and slot viscometers are the most common of them.¹⁴⁸ The selection of a particular geometry is based on the sample amount available, ease of operation, shear rate range for the application, and the geometry which represents the fluid flow: wellbore by pipe and fracture by slot.

The rotational viscometers are the most widely used instruments. They require small sample volumes for evaluating fluid behavior, and are easy to operate. These instruments are equipped with the computer software that fully automates their operation. The rotational viscometers are available with different geometries such as Couette (concentric-cylinders), cone and plate, and parallel plate.^{154,155} The couette arrangement is recommended by API to evaluate fracturing fluids,¹⁴⁵ and hence is widely used in the industry. This geometry, however, induces a varying shear rate across the sample contained between the cylinders. The shear gradient subjects a crosslinked gel to non-homogeneous shear and hence, can produce non-homogeneous gel domains in the crosslinked gel, resulting in inaccurate viscosity measurements. This shortcoming is overcome by using a narrow gap between the concentric cylinders, so that the shear gradient across the sample is small. The shear rate across the gap of the cone-and-plate is uniform,¹⁵⁵ so it is a better geometry to characterize fracturing fluids.

Besides the rotational viscometers, pipe and slot viscometers are also available. The viscosity of crosslinked gels at high shear rates can be more accurately measured

using pipe viscometer.^{25,142,156} Moreover, the pipe geometry is representative of flow through tubular and thus can also be used to simulate shear history of a fracturing fluid prior to the viscosity measurements.¹⁴⁸ A fracture can be assumed a parallel geometry, so the slot viscometer simulates this geometry better than a rotational viscometer. The shear rates representative of flow through a fracture can thus be measured with a slot viscometer.^{157,158}

API Viscosity Measurement Procedure. American Petroleum Institute recommends an operating procedure for measuring the viscosity of crosslinked fluids.¹⁴⁵ This procedure intends to provide a standardized testing for characterizing crosslinked gels which are shown to exhibit dissimilar viscosities even when measured on similar chemicals using identical viscometer geometries.^{143,144}

The API procedure requires using a rotational type, pressurized viscometer such as Fann Viscometer Model 50C, Nordman Model 5001, or equivalent. The recommended viscometer requires small sample volume and has provisions to pressurize with nitrogen gas to prevent boiling off the solvent from the water-based fracturing fluid. It is equipped with a rotor-bob configuration designated R1-B5 or R1-B5X, with bob shaft and measurement fixtures being fabricated with either 316 stainless steel or Hastelloy C. The bob designation “X” represents a cylinder having length longer than normal such that the shear stress in the conical bottom is equal to that in the concentric-cylinders annulus of the viscometer.¹⁵⁹ The fluid is thus sheared at the same rate in the annulus as that in the space below the inner cylinder and the cup.¹⁶⁰

The API procedure recommends a shear rate ramp at ambient temperature. The ramp is performed by changing the rotor speed in seven steps to impart shear rates from 100-75-50-25-50-75-100 sec^{-1} and measuring the resultant shear stress. The sample is then continued to shear at 100 sec^{-1} while the fluid is heated to the desired temperature. The sample is heated at a rate such that its temperature rises to the test value within 20 min. After achieving a temperature of 90% of the desired value, another shear rate ramp is performed to measure initial fluid viscosities at the target temperature. Then, the fluid sample is continued to shear at 100 sec^{-1} with shear ramps performed after every 30 min. for the duration of the test. After the test, the sample is cooled down to the ambient temperature and its pH and condition is observed and recorded.

The results of the API viscosity test are reported as Power law parameters from which fluid viscosity at different shear rates are calculated. The API procedure has been used to report crosslinked gel viscosities at 100 sec^{-1} measured after every 30 min.^{16,82,125} This procedure has been used for several years to characterize fracturing fluids prior to their use in the fracturing treatments. It is also used to evaluate the performance of new fracturing fluids for which patent applications are filed with the US Patent and Trade Office.

Limitations with Viscosity Measurements. There are several limitations associated with the viscosity measurements. Foremost among them is the difficulty in measuring the viscosity of crosslinked guar gels.

The crosslinked gels like other viscoelastic gels exhibit normal stresses and display Weissenberg effect.¹⁴⁸ Because of this effect, the crosslinked gel samples do not

stay in the rotational viscometers. At high shear rates, the gel either is thrown out of the viscometer or climbs out of the measuring geometry. Hence the gel viscosity is difficult to measure with the rotational viscometers at shear rates corresponding to the fracture flow conditions.

Secondly, the crosslinked gels are observed to exhibit wall slippage characteristics where the gel exhibits a finite velocity at the bob wall surface.¹⁶¹ The viscosities measured through shear rate at the wall are lower than the actual values in the bulk of the crosslinked gel sample.¹⁶² Another anomalous behavior exhibited by crosslinked gels is reversible stress transformation. These gels display multiple shear stress values for a single shear rate, and hence have multiple viscosities for the same shear rate.^{159,163} Lastly, the power law model used in the API viscosity measurements does not correctly predict the viscosity-shear rate behavior of crosslinked gels. The model overpredicts the fluid behavior at low shear rates,³³ thereby providing an inaccurate characterization of the crosslinked gels.

Because of the limitations of a rotational viscometer to characterize crosslinked fluids, Harris²⁵ proposed an alternative approach to characterize these gels. He suggested loading crosslinked gel samples simultaneously onto three pipe viscometers, set to shear fluid at three different rates. The samples are then evaluated for viscosities, at three shear rates, as a function of time. The measured values have been used to calculate the flow behavior index n as a function of time. This method was later used to correlate n values with the crosslink density and thus, to describe the proppant transport behavior of borate-crosslinked gels.²⁵ This technique though better than the previous approaches has limitations. It does not consider the low shear viscosity of the gels, and it completely

relies on the viscosity as the sole parameter to describe the proppant suspension capability of a fracturing fluid. Several studies have shown that the fluid viscosity does not describe the crosslink density, which is shown to be better indicated in the fluid elasticity.⁶⁴

Viscosity Averaged Molecular Weight. Before closing discussion on the viscosity, there is one important aspect that can be described with the fluid viscosity.

The fracturing fluid characterization requires a fundamental understanding of the fluid's molecular properties. Among the several properties of a polymeric fluid, the most important parameter is its molecular weight, a parameter on which all of its physical characteristics depend on. Since viscosity is a measure of fluid resistance to flow and hence of interactions between the different polymer chains, it provides an easy and simple method to calculate the molecular weight of a polymer. This method is however relative as it uses empirical correlation of viscosity with the molecular weight. Such correlations are established with absolute methods like osmotic pressure and light scattering;⁶⁴ hence, the molecular weight measured using viscosity is semi-empirical.

In dilute polymer solutions, the viscosity, μ , is related to the polymer concentration, c_p , by a Taylor series as

$$\mu = \mu_s \left(1 + [\mu]c_p + k[\mu]^2 c_p^2 + \dots \right) \quad (2.11)$$

where μ_s is viscosity of the solvent used to prepare the polymer solution, and $[\mu]$ is called intrinsic viscosity, which is defined as

$$[\mu] = \lim_{c_p \rightarrow 0} \frac{\mu - \mu_s}{\mu_s c_p} \quad (2.12)$$

The intrinsic viscosity describes the polymer-solvent frictional forces, as at infinite dilution, there is negligible interactions between the polymer-polymer chains.⁶⁴ The intrinsic viscosity is calculated from two semi-empirical forms of Eq. 2.11; one is Huggins Equation

$$\frac{\mu - \mu_s}{\mu_s c_P} = [\mu] + k' [\mu]^2 c_P \dots\dots\dots (2.13)$$

and other is Kraemer Equation

$$\frac{\ln\left(\frac{\mu}{\mu_s}\right)}{c_P} = [\mu] + k'' [\mu]^2 c_P \dots\dots\dots (2.14)$$

These two equations when extrapolated to zero polymer concentration yield the intrinsic viscosity.

When the logarithmic term in the Kraemer's equation is expanded, constants k'' in Eq. 2.14 correlates with k' of the Huggins equation as

$$k'' = k' - 1/2 \dots\dots\dots (2.15)$$

Equations 2.13 and 2.14 are used to determine k'' and k' , and intrinsic viscosity of a polymer. The two constants are then checked with Eq. 2.15 to validate the calculated value of the intrinsic viscosity.

The intrinsic viscosity is then used to determine molecular weight of the polymer using Mark-Houwink-Sakurada equation,

$$[\mu] = K M_v^a \dots\dots\dots (2.16)$$

where K and a are empirical constants that depend on the polymer, solvent, and temperature. M_v is called viscosity average molecular weight as it is determined using the viscosity correlation. These empirical constants for guar galactomannan are available

in the literature. Robinson et al.⁶⁰ observed the intrinsic viscosity, in dl/g, is related to the guar molecular weight by the following relationship

$$[\mu] = 3.8 * 10^{-4} M_v^{0.723} \dots\dots\dots (2.17)$$

Doublier and Launay⁶⁵ observed a slightly different relationship

$$[\mu] = 7.76 * 10^{-6} M_v^{0.98} \dots\dots\dots (2.18)$$

Thus, Eq. 2.17 and 2.18 can be used to calculate the viscosity average molecular weight of the guar used in the present research. The calculations are shown in Chapter 3.

Viscoelasticity. Crosslinked guar gel displays viscoelastic behavior having both viscous and elastic properties. The elastic characteristic means that the gel remembers its original shape, like an elastic material does. When the gel is deformed under shear, it has an ability to return to its unique shape once the imposed shear is discontinued. Because of its viscous properties, however, it cannot recover completely to its original state. The macroscopic performance of a crosslinked gel is thus a competition between its elastic and viscous properties. The presence of these two characteristics suggests that the crosslinked gel be evaluated for both these properties.

The viscoelastic characteristics of a material can be evaluated with different experimental techniques; creep, stress relaxation, and oscillatory measurements are the most common of them.¹⁵⁴ In the creep tests, a sample is subjected to a constant stress and its deformation is measured as a function of time. In the stress relaxation measurements, the sample is deformed instantaneously to a desired strain and then, the stress required to maintain the strain is measured as a function of time. In the oscillatory measurements, a small amplitude oscillatory stress is applied to the sample and the material response is

monitored for several frequencies of oscillations. The oscillatory measurement, also called dynamic mechanical analysis, is the most useful technique to evaluate the viscoelastic response of a material.⁶⁴ Hence, it was used in the present research to evaluate the crosslinked gels.

Theory of Viscoelastic Measurements.^{64,154,164} In an oscillatory experiment, a fluid sample is subjected to a sinusoidal shear strain and the resulting fluid response is measured. The applied strain is given by

$$\gamma = \gamma_o \cos \omega t \dots\dots\dots (2.19)$$

where γ_o is the strain amplitude and ω is the angular frequency. The resulting stress response is given by

$$\tau = \tau_o \cos(\omega t - \delta) \dots\dots\dots (2.20)$$

where τ_o is the amplitude of the stress response and δ is the phase lag between the input strain and the resulting stress. For a purely viscous fluid, the stress would lag behind the strain at a phase angle of 90° . For an elastic fluid, the strain and stress would be in-phase with each other at a phase angle of 0° . Whereas in a viscoelastic fluid, the stress would lag behind strain at a phase angle between 0 and 90° . Therefore for a viscoelastic fluid, the resulting stress can be separated into an in-phase and an out-of-phase response to the applied strain. Equation 2.20, on expansion, yields

$$\tau = \tau_o \cos \omega t \cos \delta + \tau_o \sin \omega t \sin \delta \dots\dots\dots (2.21)$$

which can be rewritten in terms of an in-phase and out-of-phase component as

$$\tau = \gamma_o \left(\frac{\tau_o}{\gamma_o} \cos \delta \right) \cos \omega t + \gamma_o \left(\frac{\tau_o}{\gamma_o} \sin \delta \right) \sin \omega t \dots\dots\dots (2.22)$$

and to

$$\tau = \gamma_o (G' \cos \omega t + G'' \sin \omega t) \dots\dots\dots (2.23)$$

where, G' is the in-phase component and G'' is the out-of-phase component. The in-phase component is called storage modulus as it signifies the energy stored in the fluid through its elastic forces. The out-of-phase component, G'' , is the loss modulus as it describes the energy dissipated through viscous fluid flow. These moduli are related to the phase angle δ by

$$\tan \delta = \frac{G''}{G'} \dots\dots\dots (2.24)$$

and to a complex viscosity as

$$\mu^* = \frac{\sqrt{G'^2 + G''^2}}{\omega} \dots\dots\dots (2.25)$$

The viscoelastic theory described above is applicable when both the stress and the strain are infinitesimal, and in an experiment, the ratio of stress to strain is a function of frequency alone and not of the stress magnitude.¹⁶⁵ This range of application is called to be the linear viscoelastic limit of the fluid response.

Oscillatory Response of a Viscoelastic Material. A schematic of the oscillatory response of a viscoelastic material is shown in Figure 2.4. The figure shows the viscous and elastic moduli of the material as a function of frequency of oscillation ω . The loss modulus, G'' , begins at a low value, slowly increases with frequency, reaches a maximum value, decreases and finally increases at the higher frequencies. The elastic modulus, G' , also begins at a low value, but unlike G'' , it increases with an increase in the frequency. For a viscoelastic material, G' is proportional to ω^2 and G'' is proportional to ω at low

frequencies. The linear proportionality of the viscous modulus with frequency corresponds to the Newtonian plateau in the steady shear viscosity curve of a fluid.

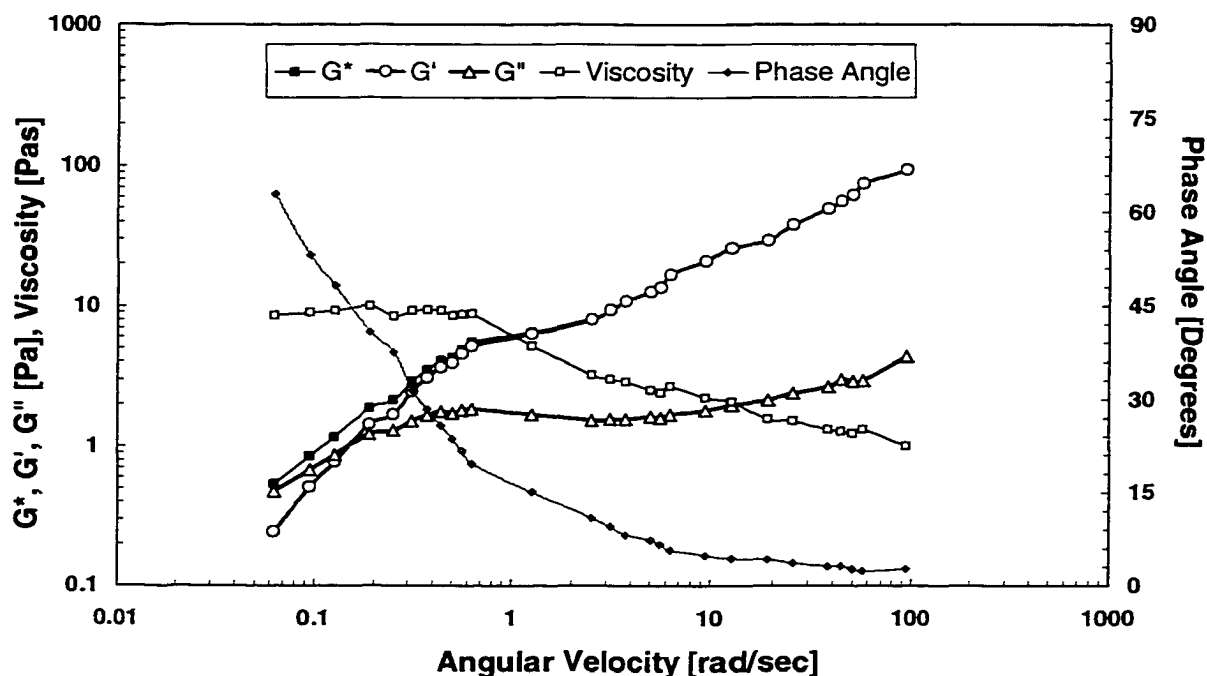


Figure 2.4—Oscillatory Response of a Viscoelastic Gel
(Borate-Crosslinked 35 lb/Mgal Guar, 1.75 lb/Mgal Crosslinker, pH 9)

The elastic modulus-frequency curve of a viscoelastic fluid can be divided into three regions: terminal, plateau, and transition. The terminal region corresponds to the low frequency region on the curve. In this region, the time scale for measuring the response of a polymer is large, so the polymer chains can diffuse and disentangle during the measurements. They can thus release their strain and thereby exhibit low stress value. Since polymer chains translate relative to one another, the molecule does not recover to its former length but relaxes to a new equilibrium state. At low frequencies, a

viscoelastic gel thus relaxes like a polymer solution and the sample undergoes flow. This region is hence called terminal flow region.

As the frequency is increased, plateau region is achieved where the elastic modulus is independent of the frequency. In this region, the polymer chains cannot translate as a whole molecule, due to strong interactions between the neighboring chains. These interactions come from entanglement couplings in a linear polymer, or from crosslink bonds in a crosslinked polymer. Because of these interactions, the chains can only undergo short-range diffusional motions in the polymer segments between the entanglement couplings. The value of the plateau modulus depends on the molecular weight of the polymer segments between the entanglements or the crosslink points, where the motion of the chains is severely constrained. In a crosslinked rubber, the crosslinked bonds remain intact even when the chains are allowed to relax for a longer period. So the chains do not exhibit any motion relative to each other and the terminal flow region is not observed, and the plateau region extends to very low frequencies in a rubber. The plateau region is however not clearly seen in the viscoelastic response of a crosslinked guar gel in Fig. 2.4.

The glass transition region corresponds to the high frequency region. In this region, the chains motion is sluggish and retarded. Here, the response of a crosslinked polymer gel is similar to that of the polymer solution.

Importance of Viscoelastic Measurements. Harris¹⁰ ascribed the proppant suspension capability of borate crosslinked gels to a strong network structure formed in these gels. Several studies concluded that the formation of molecular structure in

crosslinked gels is better studied through their dynamic rheological measurements, where these gels are characterized for their viscous as well as elastic moduli.^{21,148,166,167} The importance of viscoelastic measurements is highlighted in the characterization of crosslinked gels. Kramer et al.²⁹ observed that the HPG solution crosslinked with two different titanium crosslinker had similar viscosities but dissimilar dynamic rheological measurements. They recommended that the crosslinked gel rheology be studied with dynamic oscillatory measurements as it provides better insight than viscosity about the molecular characteristics of fracturing gels. They suggested that since the two gels formed different molecular structure, they would display dissimilar proppant transport behavior in a fracture.

Clark et al.¹⁶⁸ could not explain the settling velocities in crosslinked gels with viscosity alone. They observed large deviations in the settling velocities calculated with the viscosity correlations from that measured in the experiments. Acharya³³ concluded that the fluid elasticity completely overshadows the shear-thinning viscosity in viscoelastic fluids. These studies point to the significance of viscoelastic characterization of crosslinked gels for comprehending their proppant suspension characteristics.

Limitations of Oscillatory Measurements. The oscillatory measurements are made in the linear regime of the stress-strain relationship. This regime is identified as the region where the applied stress results in infinitesimally small displacement gradients in the fluid. Hence, these measurements are also called linear viscoelastic (LVE) characterization. The LVE measurements cannot explain the shear rate dependent behavior observed in non-Newtonian fluids.¹⁵⁴ They cannot also describe the normal

stress phenomena observed in the crosslinked gels; the normal stress being non-linear effects vary with the applied shear. Most importantly, these measurements are restricted to motions with small displacement gradients. Thus, any prediction of the fluid behavior in high shear region with the oscillatory measurements, which are made in low shear environment, should be cautiously carried from one polymeric system to another.

2.4 PROPPANT TRANSPORT AND SAND SUSPENSION

Hydraulic fracturing treatment is performed to create a fracture in a reservoir. The fracture provides a highly conductive flow path for reservoir fluids to flow to the wellbore. This path is created by a fracturing fluid, but is maintained open by proppant particles, which generate higher permeability than that of the reservoir. The longer the fracture, the deeper it penetrates into the reservoir, and the higher the permeability it generates. Moreover, the larger the propped area of the fracture the larger is its flow capacity. Therefore, productivity increase from a stimulation treatment depends on how well the fracture is filled with the proppant.

The fracture is filled with the proppant by a fracturing fluid. The proppant are mixed in the fluid at the surface, pumped down the wellbore, and transported into the fracture. The proppant particles can settle down in the fracture forming a pack at the bottom or can remain suspended in the fluid.¹⁶⁹ Between the deposited and the suspended particles, only those proppant that remain in front of the perforations provide high permeability flow path for the reservoir fluids.

The proppant particles must remain uniformly distributed in the fluid during the pumping as well as after the pumping stage of the fracturing treatment. During the

pumping stage, they should be in suspension so that the entire length of the fracture is filled with proppant. Once the fluid pumping is stopped, the reservoir closes on the void created during the fracturing. The fracture closure occurs over a period of several hours, so the particles must remain suspended or deposited across the perforations till the reservoir closes on them.^{17,24,151} This is because the final proppant distribution describes the conductivity of the proppant pack in the created fracture, and the fracture conductivity determines the production increase from the stimulated formation.¹⁷⁰⁻¹⁷²

During the stimulation treatment, the fracture conductivity and the proppant distribution in a fracture can be controlled through adjustments in the proppant concentration, fluid pump rate, or fracturing fluid characteristics.¹⁷³⁻¹⁷⁶ But after the treatment and during the fracture closure, they can be adjusted only through the fluid characteristics. Therefore, the important characteristic of the fluid is its capability to prevent particles from settling and to keep them in suspension. Therefore, the significant factor in obtaining production increase from a fracturing treatment is to understand the proppant transport phenomena in a fracture and particle settling in fracturing fluids.

Summary of Proppant Transport Investigations. The particle transport behavior has been studied numerically as well as experimentally. The numerical models available today provide a detailed description of the fracturing operation. They utilize viscosity-based correlation to describe proppant transport and settling in fracturing fluids. These correlations are developed from the Stokes law, which is based on single particle settling in an infinite medium. This law is modified to incorporate the non-Newtonian fluid characteristics of the fracturing fluids and to include the presence of other particles

on single particle settling through hindered settling relationships. The experimental studies, on the other hand, provide useful insight into the physical processes occurring during the proppant suspension and transport. These studies become the basis for development of models to theoretically describe the experimentally observed phenomena.

Kern et al.¹⁷³ reported the first few experimental observations on the proppant transport behavior in a vertical slot, which was used to model a fracture. They observed a gelled oil could support sand particles under static condition, but under fluid flow conditions, the particles settled down near the wellbore. Lowe and Huitt¹⁷⁴ observed the slurry to flow in three regions; top, middle and bottom section. The top-most layer comprises of suspended particles with each particle acted upon by the fluid. In the middle layer, the particles move as bulk materials with each particle hindering the movement of the other particles in the layer. The lowest layer consisted of deposited particles having negligible movement from the flowing fluid.

Domselaar and Visser¹⁷⁷ developed a numerical model to calculate proppant concentration and final fracture shape after a fracturing treatment. Their model assumed negligible settling of the proppant particles because of the use of extremely high viscosity fracturing fluids. They however did not specify how much viscosity was sufficient for negligible settling. Daneshy¹⁷⁸ presented a more comprehensive numerical model for describing proppant transport in a fracture. He correlated fluid viscosity with particle settling, and described the perfect proppant transport to be a condition where there was very little particle settling during the fracturing treatment. His analysis, however, did not consider the shear thinning characteristics of fracturing fluid.

Novotony²⁴ described the importance of monitoring proppant transport under fluid flow conditions. He observed that the settling velocities measured in stagnant non-Newtonian fluids could not reliably predict proppant transport in a fracture. He argued that the particles suspended in a stagnant fluid would settle down in flowing condition due to shearing of the fluid. Moreover, the particles redistribute when flowing as slurries. At low particles concentration, Kirby and Rockefeller¹⁵² and Clark et al.¹⁷⁹ observed particles clustered together and then settled faster than single particle settling. At high solids concentration, however, the particles settled slowly because of the hindered settling from other particles in the slurry. McMechan and Shah¹⁸⁰ observed the particles clustering occurred in linear guar gels at sand concentrations less than 10 lb/gal, and hindered settling effects dominated at higher sand concentrations.

Shah¹⁸¹ explained the particle-settling behavior under dynamic fluid conditions with a viscosity-based correlation developed from static settling data. He evaluated the slurries prepared only in linear guar solutions and not in crosslinked gels, which are used in the fracturing treatments performed today. Clark et al.¹⁶⁸ studied particles settling velocities in gels crosslinked at different crosslinker concentrations, and observed very large deviation in the experimentally observed settling velocity from those calculated with the Stokes law based correlation. Hannah and Harrington¹⁸² even argued against using the non-Newtonian form of the Stokes law to describe settling in non-Newtonian fluids. Sievert et al.²² observed a two-fold increase in the viscosity resulted in several fold increase in the transport capability of the fluid. Several studies concluded that the crosslinking modified the fracturing fluid in such a way that factors other than viscosity described the proppant transport characteristics of crosslinked gels.^{22,168,183}

Unable to identify the parameter to describe settling in crosslinked gels, Clark and Guler²³ proposed an empirical coefficient to account for the difference in the settling velocity from the viscosity-based correlations. They suggested the coefficient was a function of molecular entanglements in the crosslinked gels. Similarly, Acharya³⁴ attributed the excellent proppant suspension characteristics of crosslinked gel to a network structure formed in these gels. de Kruijf et al.¹⁸⁴ described that the crosslinked gels had elasticity which help them to stretch their molecular network under shear conditions, and prevented gel from breaking up, thereby keeping the particles in perfect suspension.

Acharya³³ highlighted the importance of fluid elasticity to describe single particle settling in viscoelastic fluids. He observed the fluid elasticity overshadowed shear-thinning viscosity of these fluids. Clark et al.¹⁵⁰ Roodhart,¹⁵¹ Kirkby and Rockefeller,¹⁵² described the importance of incorporating zero shear viscosity in the viscosity correlations to understand static settling velocities of proppant. Jin and Penny³⁷ studied the impact of viscous and elastic properties on single particle settling in different fracturing fluids. These studies highlighted the settling of proppant suspension under static fluid conditions, but they did not explain the proppant transport characteristics under fluid flow conditions. Since the fluid characteristics must be evaluated under fluid flow conditions, there is a void in the current-state-of-research in using fluid elasticity to understand fluid's capability to suspend and transport proppant.

Laboratory Evaluation of Fracturing Fluids. In addition to understanding the proppant transport behavior of crosslinked fracturing fluid, there is a need to identify a

criterion to evaluate these fluids prior to their use in the field. The industry uses two methods to predict an acceptable proppant transport behavior. One method is based on a minimum viscosity of the fracturing fluid, and the second is based on the proppant settling velocity in the fluid.

Brannon and Ault⁹ observed proppant transport behavior of their new borate-crosslinked in a transparent slot. They assigned the perfect transport to the condition where the proppant settling was less than ½ in. (1.25 cm) during 30 min. flow of the fluid through the slot model. They confirmed the successful use of their new formulation to fracture Red Fork formation in Oklahoma and Wilcox formation in Texas. Later on, Brannon and Tjon-Joe-Pin⁶² considered proppant settling less than ¼ in. (0.6 cm) in 30 min as a very good sand transport behavior of zirconate-crosslinked gels. Davies et al.¹⁸⁵ considered settling velocities less than 2-3 cm/min to be acceptable for successful proppant transport through a fracture. They argued that this settling velocity would result in the formation of 1-2 meter high bed during the fracture closure period. A similar justification was proposed by Powell et al.,⁸¹ who suggested that the settling of proppant particles under static conditions could predict the height to which proppant would settle over the period of the fracturing treatment.

de Kruijf et al.³⁵ described three static velocity regimes: a perfect proppant suspension at velocities less than 0.5 cm/min, marginally acceptable suspension at velocities in between 0.5 and 5 cm/min, and unacceptable settling at velocities over 5 cm/min. They, however, did not explain the basis of their criteria. Unlike Brannon and Ault,⁹ they did not show confirmation of their criteria with proppant transport studies under fluid flow conditions or with case histories on successful fracturing treatments.

Besides the settling velocity based criteria, a fluid viscosity based criterion is also considered to describe acceptable proppant transport characteristics. Armstrong et al.⁴ and Nimerick et al.⁸² utilized a minimum viscosity of 100 cp at a shear rate of 100 sec⁻¹ for three hrs or more at temperatures to 240°F for adequate proppant transport and sand placement in a fracture. Similar criterion is available on the viscosity at other shear rates. The viscosity criterion is, however, observed to be not applicable in all cases. Dawson et al.¹²⁵ observed acceptable settling characteristics in fluids having viscosities of 322 and 461 cp at 40 sec⁻¹ but fair in fluids with viscosity of 521 cp. They did not explain why the gel having higher viscosity exhibited fair characteristics whereas the lower viscosity gels displayed acceptable behavior.

Proppant Convection Effects. One controversial phenomenon that is argued a lot in the proppant transport studies is the presence of convection and encapsulation. Based on their study on Karo syrup and mineral oil, Cleary and Fonseca¹⁸⁶ concluded that the convective downward motion of slurry dominated particle settling, and encapsulation of viscous fluid by a low viscosity fluid accelerated the convective settling process. Barree and Conway¹⁷⁶ provided more reasonable explainable of this phenomena as they concluded that the convection was important when the density gradients in the pad fluid and the proppant-laden slurry exceeded the viscous forces in the fracturing fluid. Shah and Asadi¹⁸⁷ confirmed this observation in their study with a large-scale fracturing simulator. They found that an increase in the viscosity with linear or crosslinked guar gel over that of water overrode the density effects, inhibited encapsulation and, prevented convective downward motion of the slurry. Similar observations have been made in a

series of studies performed by Clark and his team.^{188,189} These studies showed the convection was an insignificant phenomena in proppant transport with the crosslinked gels used today.

2.5 PARTICLE SETTLING IN NON-NEWTONIAN VISCOELASTIC FLUIDS

The problem on particle settling in non-Newtonian fluids is not just important for the petroleum industry, but is an area of considerable significance for numerous industrial operations. This problem is synonymous to suspension settling in almost every sphere of life. Some of the examples of suspensions are particles in salad dressing, concrete and asphalt in construction activities, silt carried by river streams, coal slurries transported hydraulically through pipelines, sewage disposal, etc. In all these examples, it is important to understand the characteristics of the carrier liquid for keeping the particles suspended in it. This is because the particles settling could lower the effectiveness of the slurry and create undesirable handling problems.

In addition to the importance for numerous industrial applications, the particle settling in static viscoelastic fluids is described to serve as a model problem to validate numerical codes against the experimental observations in this problem.⁵²

Summary of Particle Settling Investigations in non-Newtonian Fluids. Wasserman and Slattery¹⁹⁰ used variational methods to develop upper and lower bounds for the drag coefficient of a slow moving sphere through a Power law model fluid. They found poor agreement between the theoretical coefficients and the experimental data on sphere settling in non-Newtonian fluids. Similarly, several studies have shown that there are

fundamental differences in the particle settling in non-Newtonian and Newtonian fluids. Certain characteristics are observed in particle settling in the non-Newtonian fluids that are not seen in the Newtonian systems.

One such behavior is particle migration in fluid flow conditions. Highgate and Whorlow¹⁹¹ observed that the settling particles did not distribute in Newtonian fluids, but exhibited small migration in low normal stress fluids and a rapid migration towards the low shear regions in high normal stress fluids. The particles migration increased when the shear rate on the fluid was increased. Gauthier et al.¹⁹² observed particle migrated towards the higher shear rate regions in shear-thinning fluids; whereas in viscoelastic fluids, the particles migrated in opposite direction from the high shear to the low shear rate regions. Karnis and Mason¹⁹³ ascribed this particle migration in viscoelastic fluids to a combined effect of normal stresses and velocity gradient in the fluid.

Another interesting phenomena observed in the viscoelastic fluids is during the simultaneous settling of two spheres. Riddle et al.⁴⁹ observed that two spheres converged when dropped within a small initial separation distance, but diverged when the separation distance was increased. The converging of the spheres is similar to the clustering phenomena seen in the proppant particles settling in fracturing fluids.¹⁸⁰

In addition to the influence of the separation distance, the time between the successive dropping of particles was also observed to be a critical parameter in the settling in viscoelastic fluids. The particle was observed to accelerate when dropped within a short time span of the previous particle.^{50,194} Cho et al.¹⁹⁴ ascribed the increase in the velocity of the second sphere to a network of depleted polymer molecules behind the falling sphere causing polymer molecules from the nearby region to diffuse to fill up

the void and creating flow disturbances behind the first sphere. Ambeskar and Mashelkar⁴⁵ further evaluated the explanation on the depletion of polymer layers behind the particles, and concluded that the role of molecular phenomena needed further understanding.

Gheissary and Brule⁵² described two types of particle-particle interactions: near field and far-field. The near-field interaction was influenced by the viscoelastic properties of the fluid. The far-field interaction was caused by the wake or hole formed behind the settling sphere, but could not be explained from the fluid viscoelastic properties.

Joseph et al.⁵⁴ found the particles dropped side by side reoriented from across to along the stream. This side by side settling was also dependent on a critical separation distance and was similar to the critical separation between the spheres dropped one after another. They argued that the particles aggregated in the elastic liquids and hence, fluid elasticity is an important property for understanding particle settling in viscoelastic fluids. Allen and Uhlherr¹⁹⁵ concluded that the elastic response overshadowed the shear-thinning behavior in the settling of concentrated slurries. They further suggested not to assume homogeneous distribution of particles to model suspensions flow with viscoelastic fluids. Liu and Joseph⁵³ concluded that particle settling in viscoelastic fluids was a competition between the viscoelastic stress, viscous stress, and fluid inertia. They also confirmed that the particles clustering overshadowed the single particle settling in viscoelastic fluids.

Becker et al.⁴⁷ observed that the particle-settling velocity in the viscoelastic fluids overshoot to transient velocities up to 50% before the spheres approached a final constant value. Bobroff and Phillips⁵⁵ also observed time dependent suspension settling under

static fluid conditions. They found the settling velocity of the suspension was faster initially than in the later stages of the settling.

Differences in Particle Settling in Viscoelastic Fluids. Besides the differences in the experimental observations on the particle settling in Newtonian and non-Newtonian fluids, there are large discrepancies in the settling velocities within different viscoelastic fluids.

Chmielewski et al.⁴⁸ studied spheres settling in two Newtonian elastic fluids: polyisobutylene (PIB) in polybutene and polyacrylamide (PAA) in corn syrup. They observed the drag forces on the spheres settling in PAA were lower than that in PIB, causing particles to settle faster in PAA. They concluded that the particle settling in viscoelastic fluids involved both reduction and enhancement of drag on the particles. Solomon and Muller⁵¹ observed different drag enhancements in two polystyrene based Newtonian viscoelastic fluids prepared in different solvents. The fluid having poorer polymer-solvent contacts exerted lower drag on the settling particles. Their study exhibited the importance of determining the fluid molecular architecture to determine the macroscopic response of a viscoelastic fluid.

The differences in the particle settling in dissimilar polymer fluid imply that the polymer configuration is an important parameter for settling in viscoelastic fluids. Tirtaatmaja et al.⁴⁶ suggested separately measuring the rheological parameters in each fluid. Solomon and Muller⁵¹ discussed the influence of fluid expandability and extensibility on drag on the settling particles. They emphasized the importance of

solution microstructure, polymer molecular weight and polymer solvent interactions to understand the macroscopic behavior of spheres settling in complex fluids.

Bush¹⁹⁶ proposed to consider elongational as well as shear-thinning properties of the fluid for studying particle settling in viscoelastic fluids. van den Brule and Gheissary¹⁹⁷ also highlighted the influence of fluid elasticity to reduce the settling velocity in non-Newtonian viscoelastic fluids. They further concluded that the elasticity became even more significant as the particle experienced higher shear rates.

Bobroff and Phillips⁵⁵ reasoned for the fundamental differences in the sedimentation in Newtonian and viscoelastic fluids, and strongly refuted to modify Newtonian theory on sedimentation to describe sedimentation in non-Newtonian fluids. They proposed to develop a theory for settling in non-Newtonian fluid requires a better understanding of particle-particle interactions in complex viscoelastic fluids.

2.6 APPROACH TAKEN FOR THE RESEARCH

Guar leaves undesirable polymer residue that reduces the permeability of the reservoir and proppant pack, and restricts oil and gas flow into the wellbore. Because of the residue, the guar based fracturing fluids are observed to impair the proppant conductivity from 15 to 75%. One method to reduce this formation damage is to crosslink the guar polymer. The crosslinking can be performed at lower guar concentrations, reducing the damage from the fracturing fluid. The crosslinking of guar, moreover, imparts better fluid characteristics than those obtained in the non-crosslinked guar solutions by increasing the polymer concentration.

The crosslinking modifies the fracturing fluid in such a way that factors other than viscosity describe the proppant transport characteristics of crosslinked gels. A two-fold increase in viscosity is observed to result in several fold increase in the proppant transport capability of the fluid. A large deviation is also observed in the settling velocity calculated with the viscosity based correlation from that measured in the experiments. Secondly, the proppant settling observed under static fluid conditions cannot describe the proppant transport characteristics under fluid flow conditions. Thus, there is a void in the current state of the research on understanding the fluid capability to suspend and transport proppant.

In addition to understanding the proppant transport behavior, there is a need to identify a criterion to evaluate the fluids for proppant transport during the stimulation treatment. Currently, the Stokes law on single particle settling in Newtonian fluid is modified to incorporate the non-Newtonian characteristics of the fracturing fluids. Several studies, however, have described fundamental differences in the particle settling in non-Newtonian and Newtonian fluids. Even single particle settling in different Newtonian viscoelastic fluids exhibits contrasting characteristics which are difficult to explain. To develop a theory for settling in the non-Newtonian fluid would require a fundamental understanding of the polymeric structure formed in real fracturing fluids under realistic field conditions. The experimental investigations can provide useful insight into the physical processes occurring during the proppant transport through a fracture, and can therefore become the basis to theoretically describe the actual phenomena.

Borate-crosslinked gels account for almost 75% of the fracturing treatments. The proppant suspension characteristics of these gels are considered to be due to the formation of 2:1 complexation compound between the borate ions in the crosslinking agent and the polyol groups on the guar polymer. The more the number of complexes formed the better is the suspension capability of the crosslinked gel. It is impossible to count the number of these complexes formed in crosslinked guar gels, and so it is difficult to correlate them directly to the proppant suspension. The correlation can however be found through a macroscopic property that reflects the molecular structure formed in the crosslinked gels and thus explain the proppant transport behavior. For the last several decades, the fluid viscosity has been considered to be such a property. It however does not explain the proppant settling in fracturing fluids. Thus, there is a need to identify a better fluid property to evaluate these fluids for proppant transport behavior.

The fluid property can be identified by preparing different crosslinked gels having similar number of complexes and so, identical macroscopic fluid characteristics. Equation 2.6 shows that the number of complexed compound formed depends on the borate ions and the guar concentrations. Gels having similar number of complexes can be formed by controlling the borate ions or guar concentrations. The borate ions concentration can be varied through fluid pH and boric acid concentration, as per Eq. 2.1. Therefore, either fluid pH or boric acid concentration or guar concentration can be regulated to prepare similar number of 2:1 complexes in different borate-crosslinked gels. These gels having same crosslinked sites would then presumably have similar proppant transport behavior and identical fluid rheological properties.

2.7 RESEARCH OBJECTIVES

There are two principal objectives of the investigation: (1) to fundamentally understand the relationship between the fracturing fluid characteristics and its suspension settling and slurry transport behavior, and (2) to determine the minimum fluid rheology required to obtain satisfactory proppant transport through a fracture.

CHAPTER 3

EQUIPMENT AND EXPERIMENTS

3.1 INTRODUCTION

The experimental study was performed in two phases. The fluids were first prepared and evaluated in the laboratory. The laboratory results were then further evaluated on a field-scale experimental facility. These two phases of the fluid evaluation are discussed in the following chapter. The chapter describes the equipment, instruments and procedure used to perform the experimental investigation.

3.2 FLUID PREPARATION

The fluids used in the research were guar-based fracturing fluids. They were prepared as linear guar solutions, which were denoted non-crosslinked gels. These solutions were crosslinked with borate-crosslinker solutions and called crosslinked gels. All fluids were prepared in Norman City tap water.

A desired quantity of the water was measured in a mixer, and a bactericide solution added to it. The bactericide prevents degradation of guar polymer during the experiments and helps to store the prepared solution for a day. The bactericide solution (Halliburton, Duncan OK, Part No. 516000060) was added at a ratio of one liter per thousand gallon of water. The water pH was then raised to a value between 9 to 10 by adding sodium bicarbonate powder (Halliburton, Duncan OK, Part No. 070151860) at a concentration of 2.5 lb per thousand gallon of water (lb/Mgal). By raising the solution

pH, the water could interact with individual guar grains allowing guar powder to mix homogeneously and hydrate completely.⁶⁸

After raising the pH, a desired quantity of guar powder (Economy Polymer, Houston TX, Batch No. 716001) was added to the water and mixed to prepare a homogeneous guar solution. The solution at pH 10 dispersed the guar into water but did not let it hydrate. The guar hydration required a pH close to neutral. So, the solution pH was lowered to slightly less than 7 by adding fumaric acid (Halliburton, Duncan OK, Part No. 070152660) at a concentration of 2.5 lb/Mgal. As the guar began to hydrate, an antifoaming agent (Halliburton, Duncan OK, Part No. 070157670) was injected into the solution to prevent air bubbles from entrapping in the hydrating guar solution. The resulting solution was left to hydrate. After an hour, viscosity of the guar solution was measured. The measured values were compared with the published data to establish that an acceptable guar solution was formulated. The measured values were recorded to compare the guar solutions prepared on different test runs in the study.

All linear guar solutions were prepared using the procedure described above. These solutions were then used for two series of tests. In the first series, the linear solutions were prepared at different guar concentrations, and were denoted linear or non-crosslinked guar. These solutions were characterized for their viscosities, viscoelastic properties and proppant slurry settling. The linear guar solutions were prepared at guar concentration from 1 to 100 lb/Mgal.

In the second series of tests, guar concentration was fixed at 35 lb/Mgal. This concentration was selected because of its wide use in fracturing treatments, and because this concentration was higher than the overlap concentration of the guar used in this

research, as explained in Chapter 4. The guar solution pH was adjusted to three values: 9, 10, and 11. The solution pH was adjusted using sodium hydroxide and muratic acid solutions, and monitored with a calibrated pH meter (Cole Parmer, Model pH 10 series). The pH-adjusted solutions were then crosslinked with borate-crosslinker solution, and the resultant mixtures were denoted crosslinked gels.

The borate crosslinker solution was prepared in Norman city water by mixing 1 lb of disodium octaborate (US Borax, California, POLYBOR™, Batch No. 6E16 02) in a gallon of water. At this concentration, the crosslinker solution is saturated at ambient temperature.⁹⁴

The formulated non-crosslinked and crosslinked solutions were then characterized in the laboratory for their rheological properties and static suspension capabilities, and in the field-scale facility for their proppant transport behavior. For both laboratory and field-scale evaluations, the guar solutions were prepared using identical procedure with the chemicals added in the similar order. Their characterizations in the laboratory and field-scale facility are described next.

3.3 LABORATORY TESTING

The purpose of the laboratory testing was two-fold: to identify a guar concentration for preparing crosslinked guar and to provide a laboratory measurable property that describes the proppant transport behavior of viscoelastic guar gels.

The guar solutions were prepared in one gallon capacity mixer (Kitchen Aid Proline, St. Joseph MI, Model No. KSM5). These solutions were then either used directly as non-crosslinked gels or crosslinked with the crosslinker solution. The non-

crosslinked gels being shear history insensitive were not subjected to shear conditioning prior to their characterization. On the other hand, the crosslinked gels being highly shear sensitive were shear conditioned before their characterization. The non-crosslinked guar solutions were studied at ambient temperature only. Whereas the crosslinked guar gels were characterized at ambient as well as elevated temperatures.

3.3.1 Fluid Shear Preconditioning and Heating

The linear guar solutions were crosslinked and shear preconditioned in a blender (Waring, NewHartfort, CT, Model 31BL92 7011). One liter of the guar solution was transferred to the blender and its pH was adjusted to 9, 10, or 11 for crosslinking it. The solution was then stirred at low speed to create a vortex in the solution without introducing air into it. Into this vortex, a desired quantity of the crosslinker solution was injected, and the solution was continued to stir to allow crosslinker to react with the guar. This crosslinked gel was stirred for three min to simulate shear conditioning of the fluid down wellbore during a fracturing treatment.

Blender Shearing. The crosslinked guar gel is extremely shear-sensitive, and its characteristic properties change with shear and time. This shear-sensitivity implies that it is important to not only incorporate shear conditioning prior to characterizing crosslinked gel but to carefully monitor and control the shear imparted to it.

The high shear environment of the tubular was simulated in the laboratory by shearing the gel in the blender. After injecting the crosslinker solution to the linear guar solution, a specially designed cover was placed on the gel to prevent it from trapping air

during the shear conditioning in the blender.¹⁹⁸ Then, the blender speed was increased to condition the gel at a shear rate of 1000 sec⁻¹. This shear rate was read on a rate indicator (Red Lions Control, York, PA, Model Ditak-7) which received pulse signals from a Hall-Effect Pick-up (Dart Controls, Zionsville, IN, Model PU-E Series). The pick-up was attached to the blender rotor shaft; it sent signals at a rate of one pulse per revolution of the rotor. In the present work, the rotation pulse was programmed into the rate indicator to convert the rotor speed N (rpm) into shear rate $\dot{\gamma}$ (sec⁻¹). The shear rate was calculated using the following relation,¹⁹⁹

$$\dot{\gamma} = 10 * N \quad (3.1)$$

The blender speed reading on the rate indicator was calibrated with a strobe light (Shimpo Digital Stroboscope, IL, Model DT-301-A2).

Thus, by monitoring the blender speed and maintaining similar shear value, the crosslinked guar gels were prepared at different crosslinker concentrations and subjected to identical shear conditioning of 1000 sec⁻¹ for three min. The shear-conditioned samples were then either loaded onto the rheometer for rheological measurements or transferred to a glass cylinder for sand settling tests.

Elevated Temperature Tests. After formulating the guar solution in the mixer, the solution was transferred to the blender and its pH adjusted to the desired value for either ambient or elevated temperature evaluation. For ambient temperature tests, the solution was crosslinked in the blender as such at room temperature.

For elevated temperature tests, the pH adjusted guar solution was transferred to a tripour beaker. The beaker was then placed in a hot water bath (Blue M Electric Company, Blue Island, IL, Model MW-130A-1), preheated to the test temperature. The guar solution was heated in the bath for 30 min., which was maintained similar during the heating of different gel samples. The heated guar solution was then transferred back to the blender for crosslinking.

Besides preheating the water bath, glass jar of the blender and the glass cylinder used for suspension settling tests were also preheated in the water bath. This preheating was performed to minimize thermal losses during crosslinking and mixing of the heated guar solution. Furthermore, to reduce heat loss from the guar solution during elevated temperature slurry tests, sand particles used for preparing slurry were also preheated separately in an electric oven (Photolastic Inc, Malvern, PA).

The heated guar solution was crosslinked and shear preconditioned in the blender for 3 min. The crosslinked gel was then either loaded to the rheometer for viscoelastic measurements or transferred to the glass cylinder for the sand settling tests. The glass cylinder containing slurry was transferred to the preheated electric oven for observing static settling at elevated temperature.

3.3.2 Fluid Rheology Measurements

The fluid rheology was obtained using different viscometers. The linear guar solutions were characterized using a capillary viscometer, Fann viscometer, and Bohlin rheometer. The crosslinked gels were analyzed with Bohlin rheometer and Nordman viscometer. The rheology measurements and the viscometers are described below in detail.

Capillary Viscometer. The capillary viscometer was a pipe viscometer containing a single diameter tube. It measured the fluid viscosity from the fluid's resistance to flow through the capillary tube. The liquid head provided the driving force for the fluid flow. The viscosity was calculated from the time taken by the fluid to flow through a fixed distance on the tube.

The capillary viscometer used in the present work was a Cannon-Fenske instrument having a capillary diameter of 0.63 mm (Capillary number 100).²⁰⁰ This viscometer measured the kinematic viscosity in a range from 3 to 15 centiStokes. The kinematic viscosity was converted to the absolute viscosity by multiplying the viscometer-measured value with the fluid density, determined with an API hydrometer.

The capillary viscometer was primarily used to measure viscosity of guar solutions prepared at low polymer concentrations. These solutions had low viscosity, which was difficult to measure accurately with a rotational viscometer. The capillary viscometer being ideal for such solutions was used to measure viscosity of linear guar solutions prepared at 1 to 10 lb/Mgal concentrations.

Fann Viscometer. The Fann viscometer was a rotational instrument and had concentric cylinders geometry. It worked as a controlled strain device where the strain was imposed on one cylinder and the resultant stress was measured on the second cylinder.

The Fann viscometer used in this study was a model 35. The instrument contained a rotating outer sleeve and a stationary inner cylinder or bob to which a helically-wound torsion spring was attached. The outer sleeve was rotated at a constant angular velocity that induced motion in the fluid contained in the annular space between

the rotor and the bob. The shear transmitted through the fluid exerted drag on the inner cylinder, which was held stationary by applying torque from a spring. The spring torque was measured as a deflection on a calibrated dial on the viscometer. From the sleeve rotation and the spring torque, the shear rate and shear stresses were calculated from which the fluid viscosity was determined.²⁰¹

The sleeve had an inner radius of 1.8415 cm and the inner bob had an outer radius of 1.7245 cm. The bob was 3.8 cm long and thus, provided a very large surface area for accurate measurements of the fluid drag. The sensitivity of the Fann viscometer was further improved by using two separate torsion springs designated No. 1 and 1/5th, where the second spring was more sensitive to the small torque in the low viscosity fluids.

The fluid viscosities were calculated at several shear rates by rotating the outer sleeve at different rates. The measured viscosity-shear rate data were fit with power law model to determine fluid behavior at shear rates of interest in a fracturing treatment.

The Fann Model 35 viscometer was a very rugged and durable instrument, and was helpful to provide a quick check on the fluid behavior. So it was used for the preliminary evaluation of the linear guar solutions. The measured viscosities were recorded for comparison of the prepared guar solutions between different test runs.

The Fann Model 35 was, however, suitable for viscosity measurements at ambient temperature only. For elevated temperature viscosity measurements, Fann model 50 or Nordman rheometer was a more suitable instrument and was used in this study.

Nordman Rheometer. Like Fann Model 35, Nordman rheometer was a controlled rate instrument and had a concentric-cylinders geometry. Here also, the shear rate was

applied through the rotation of the outer sleeve, and the viscous drag was measured from the tension in the spring attached to the inner bob.

Unlike Fann Model 35, the Nordman viscometer had a closed rotating sleeve, which worked as a cup. Being a closed device, the viscometer could be pressurized and heated, making the viscometer capable of measuring fluid properties at 1000 psi pressure and 500°F temperature. The viscometer could simultaneously heat the fluid sample and measure its viscosity. The possibility of applying high pressure on the sample made this viscometer suitable for the fracturing application where water-based gels are used. The high pressure offsets any increase in fluid vapor pressure with temperature and thus, prevents water-based fracturing fluids from boiling off during their viscosity measurements at elevated temperature.

The Nordman rheometer was available with a single dimension of the outer cup, but it worked with different dimensions of the inner cylinder/bob. The instrument was available with three different bob sizes designated B1, B2X, and B5X. The dimensions of these bobs are given in Table 3.1.

Table 3.1 Dimensions of Fixtures on Nordman Viscometer

Rotor Designation	Radius (cm)	Length (cm)
Inner Bob B1	1.7245	7.62
Inner Bob B2X	1.2308	8.529
Inner Bob B5X	1.5987	9.19
Outer Cup R1	1.8415	-

The designation “X” means an extended bob whose length is more than that of a standard bob of 7.62 cm length. The purpose of using a longer bob is to maintain similar shear

rates in the fluid contained below the inner cylinder and that in the annular space between the concentric-cylinders. The sensitivity and accuracy of the viscometric measurements with the Nordman rheometer could be improved by using different bob sizes.

The Nordman viscometer was fully computerized and was programmed to perform several types of tests. These tests included calibration, standardized and custom tests. The calibration tests involved checking the performance of the viscometer with oils of known viscosities. The standardized test was the API recommended viscosity test, described in Section 2.2.2, for evaluating the fracturing fluids. The custom test was designed based on the user requirements.

In the present work, the Nordman viscometer was used to measure viscosities of the different crosslinked guar gels at ambient and elevated temperatures using the API recommended test procedure. These measurements provided a direct comparison of the viscosities of the gels prepared here with those available in the literature.

The Nordman viscometer or Fann Model 50 was an advanced version of the Fann Model 35. Thus, it carried not just the advantages of the Model 35 but also the shortcomings of the older model. The Nordman viscometer could not be used to measure viscoelastic characteristics of the fracturing fluids. For such measurements, Bohlin rheometer was equipped with the necessary capabilities and was a more suitable device.

Bohlin Rheometer. Bohlin rheometer, unlike the previous two controlled rate viscometers, was a controlled stress instrument. The controlled stress device applies stress with a motor and measures the resultant shear rate with an optical encoder. This mechanism is more sensitive to the fluid response at very low shear rates than a

controlled rate device. Hence, the controlled stress rheometer provides a better insight into the gel structure formed in polymeric fluids.

The Bohlin rheometer worked with a unique drag cup motor that applied torque to the fluid. This torque was transmitted from the motor to the measuring geometry by frictionless air bearings. The geometry, in turn, applied the torque to the fluid sample. The applied torque induced shear in the sample whose displacement was measured with a highly sensitive optical angular position transducer. The unique air-bearings and the optical sensors made this an advanced instrument for characterizing the fracturing fluids.

The Bohlin rheometer was a very versatile instrument as it was capable of measuring both viscosity and elastic properties of a fluid using several measuring geometries. The rheometer was programmed to perform various experiments such as yield stress, viscosity, oscillatory and creep tests. These tests could be performed with three available geometries: concentric-cylinders, parallel plate, and cone and plate.

In the present research, a cone and plate fixture was used because it applies a uniform shear rate across the sample.¹⁵⁵ This fixture had two plates with a flat fixed lower plate of 60 mm diameter, and a conical rotating upper plate of 4° cone angle and 40 mm diameter. The gap between the plates was filled with the test fluid.

The Bohlin rheometer was used to measure viscosity and viscoelastic properties of the non-crosslinked guar solutions and crosslinked guar gels. These measurements were made at ambient as well as elevated temperature.

The Bohlin rheometer was equipped with a separate temperature bath that had provisions for cooling and heating the test samples from 5 to 300°F. The bath controlled the sample temperature with oil circulating through the measuring system. The oil was

maintained at a desired temperature with a refrigeration and heating unit. The bath was electronically attached to the computer controls used for the rheometer operation, which allowed control of the bath oil temperature during the rheological characterization.

The heating of test fluid with the bath oil had some limitations. This method did not allow precise control of the fluid temperature because of indirect heating. As a result, temperature variations up to 10°F were observed during the elevated temperature evaluation of the crosslinked gels. Moreover, the measuring geometries available with the Bohlin rheometer were open to atmosphere. This limited the upper temperature to below the boiling point of water-based fluids.

The water-based crosslinked gels could evaporate during the rheological measurements. So, a thermal enclosure was used around the measuring geometries and oil was placed around the test sample to prevent evaporation of these gels. Since the measuring geometries were open, the rheometer was not used to directly heat the test sample. The sample was separately heated in a water bath as described in Section 3.3.1. The rheometer bath was used to maintain the sample temperature during the rheology measurements. Therefore, the rheometer was preheated to the test temperature using its oil bath. The upper conical plate was also preheated in a separate water bath prior to the measurements. This method of heating the sample was found to provide a more representative characterization of the crosslinked gels at elevated temperatures for fracture flow conditions.

3.3.3 Static Settling Tests

The static settling characteristics of the slurries prepared in non-crosslinked and crosslinked guar gels were observed in a glass cylinder (Kimble USA). The cylinder was of 1000 ml capacity, and was 400 mm high and had 60 mm I.D.

The sand and the guar solution volume were calculated to prepare one liter fracturing slurry. The calculated volumes were used to mix sand and guar solution in the Waring blender. The slurries prepared in the non-crosslinked guar solutions were prepared directly in the hydrated guar solution. For slurries mixed in the crosslinked gels, the guar solution pH was adjusted to a desired value before adding sand to it. The sand guar slurry was well mixed in the blender and a known amount of the crosslinker solution was added to the mixture. The slurry in the crosslinked gel was then shear conditioned in the blender for 3 min at 1000 sec^{-1} . The slurries thus prepared were poured into the glass cylinder to observe their suspension characteristics under static fluid conditions.

The glass cylinder was back-lighted by a portable fluorescent utility light (Ace Hardware, Oak Brook, IL, Model 30819) to observe sand particles in the slurry, and to identify a front that separated the slurry from sand-free liquid. The movement of front was observed, and the time taken by the front to move a height of 35 mm, corresponding to a cylinder volume of 100 ml, was recorded with a stopwatch. The recorded time and the distance slurry settled were used to calculate settling velocity of the suspensions.

The suspensions settling was observed at ambient conditions and at elevated temperature. The elevated temperature settling tests were performed inside the electric oven, as described in Section 3.3.1.

The static settling tests were performed on 2 ppg slurry only. The settling of higher concentration slurries was difficult to monitor in the glass cylinder.

3.4 FIELD-SCALE EVALUATION

The purpose of the field-scale evaluation was to characterize fluids under condition that was representative of an actual fracturing treatment. This evaluation was performed at the large-scale test facility of the University of Oklahoma. The facility contained mixing and blending equipment, coiled tubing, heat exchanger, and fracture models. A schematic of the test facility is shown in Figure 3.1. The various equipment shown in the schematic are described in the following sections.

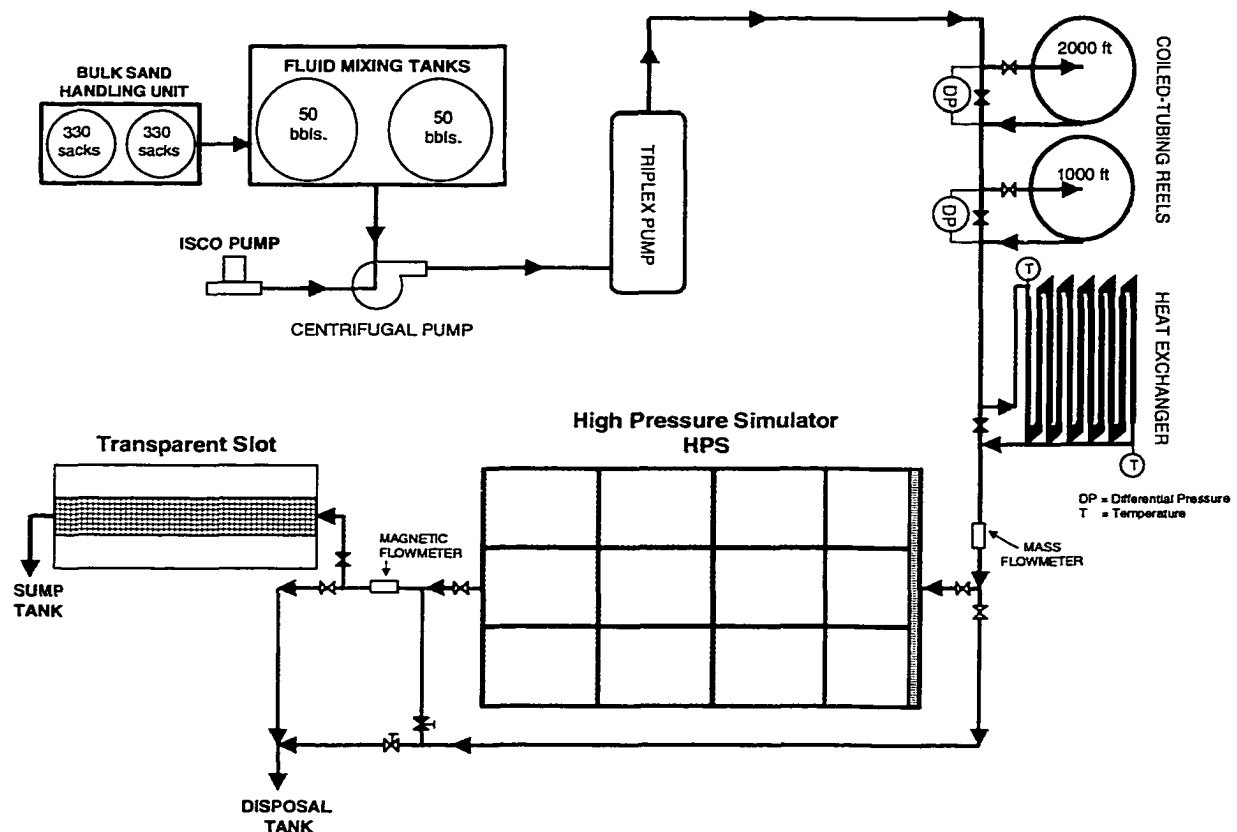


Figure 3.1—Schematic of the Field-Scale Test Facility at the Well Construction Technology Center, The University of Oklahoma

3.4.1 Fluid Preparation

The guar solutions were prepared in two 50-barrel (bbl) capacity tanks. The solutions were prepared using similar chemicals added in the same order to that used in the laboratory characterization. The solution was hydrated in the tanks and then, its pH was adjusted to a desired value. The mixing tanks had pneumatic control panels to adjust blender speed and to operate valves for diverting guar solution from each tank to the test facility. The control panel was manned during the experiments to maintain an uninterrupted supply of the guar solution.

The guar solution thus prepared was pumped through the coiled tubing and fracture models to fill up the test system with the gel before introducing slurry into the test setup. The purpose of filling up the system with the gel was to reduce convection-induced settling of slurry in the slot models.¹⁸³ Also, by pumping out some gel into the setup, the guar solution level was reduced in one tank to allow mixing sand and prepare 50 bbl slurry in the tank. To prepare the slurry, the sand was pneumatically transferred from two bulk sand handling units each of 300 sack capacity (100 lb per sack) and blended into the guar solution.

3.4.2 Fluid Conditioning

The guar solution containing slurry was crosslinked in a centrifugal pump. The pump had provisions to inject crosslinker solution while the guar solution flowed through the pump impellers. This method of crosslinker injection allowed uniform crosslinking of the guar solution under fluid flow conditions.

The crosslinker solution was injected using two syringe pumps (ISCO Inc., Lincoln, NE, Model 500D). The two pumps worked alternately: one pump injected the crosslinker solution, and the other filled up the syringe. These pumps operated automatically through solenoid operated valves. Since the crosslinker injection was a critical aspect of the large-scale evaluation, the crosslinker pumping station was manned to maintain smooth operation of the syringe pumps.

After crosslinking in the centrifugal pump, the crosslinked guar gel entered a triplex pump. The triplex pump was a reciprocating device, which was capable of pumping fluids up to 5000 psi pressure and 300 gallons per min (gpm) flow rate. In the present research, the triplex pump was operated to pump crosslinked gel at 40 gpm. This flow rate was selected to maintain a coiled tubing shear of 1000 sec^{-1} , which was similar to the rate maintained in the samples shear-conditioned in the laboratory.

The triplex pump transported the test fluid through a coiled tubing system used to simulate high shear fluid transport through a wellbore. The coiled tubing had a nominal diameter of 1.5 in. (tubing I.D. 1.188 in.) and was 3000 ft long. The tubing length simulated the depth of the reservoir to be fracture treated, and allowed to shear precondition crosslinked gel for four min at 1000 sec^{-1} .

For the ambient temperature tests, the crosslinked gel after shear conditioning was directly pumped from the coiled tubing to either of the two slot models for observing proppant transport behavior in a fracture. For elevated temperature tests, the gel after shear conditioning was heated in a heat exchanger prior to its flow through either slot model.

The heat exchanger was a 500 ft long concentric-pipes exchanger. It consisted of a 2 in. diameter tube inside a 3 in. diameter shell. The test fluid flowed through the inner tube that was heated by hot water circulating in the tube-shell annulus. The fluid temperature was controlled with the hot water circulation. The temperature of the hot water was separately controlled with a hot oiler truck, rented for the test. The heat exchanger was used in this research to heat crosslinked gels to a maximum temperature of 150°F.

3.4.3 Slot Models of a Fracture

A fracture is a crack in the formation, so it can be assumed a parallel wall geometry. In the present study, fracture was simulated with a slot having a very narrow gap width as compared to its length and height. Two such devices were used in the present research: one called high pressure simulator (HPS) and the other transparent slot.

High-Pressure Simulator. The high-pressure simulator (HPS) was a variable gap width parallel plate slot model of a fracture. It was 7 ft high and 9-1/3 ft long, and had provisions to adjust gap widths from zero to 1¼ in. A photograph of the HPS is shown in Figure 3.2.

One wall of the slot was formed from 12 individual, movable steel platens, each 28 in. square, laid out in a 3 by 4 matrix. The opposite wall was formed from 12 separate fixed platens. The gap between the slot walls was adjusted with hydraulic actuators on each of the 12 platens on the movable wall. The actuators allowed setting the gap width

to a selected value, and helped to compensate deformation in the supporting structure under high pressure and temperature.

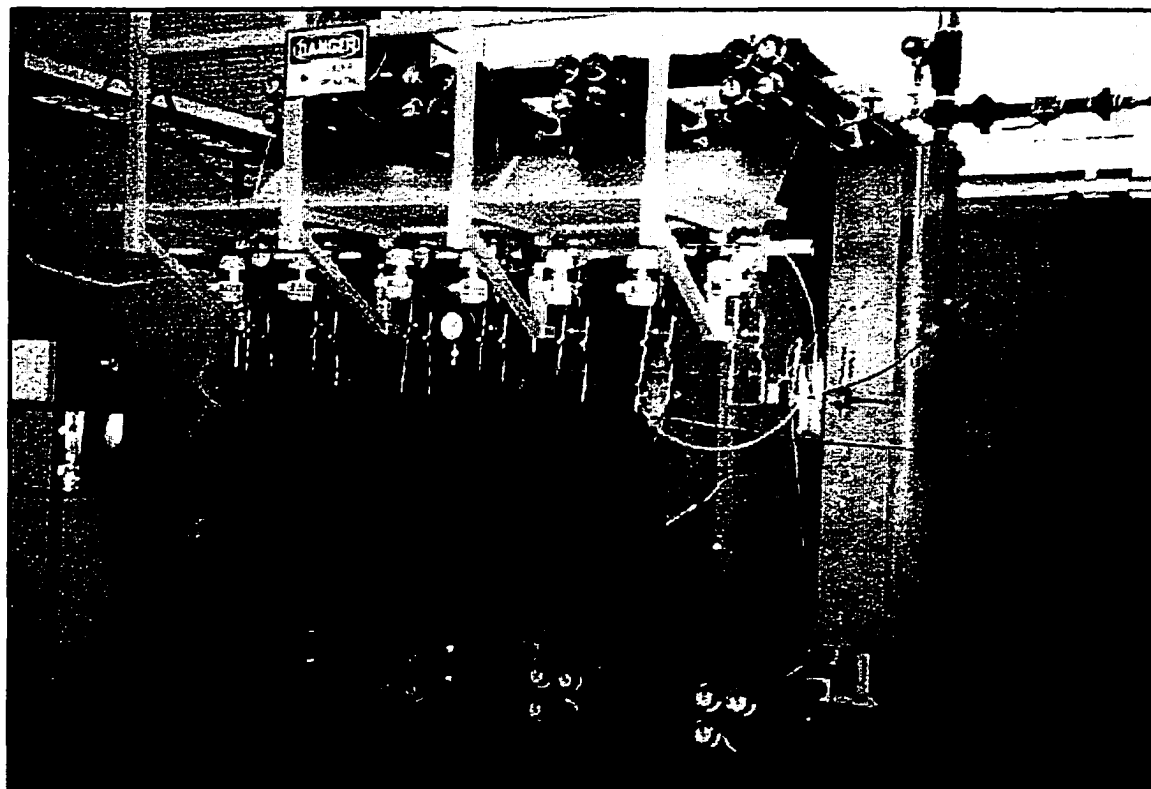


Figure 3.2—High-Pressure Simulator (HPS)

Each of the square platens on the movable and the fixed walls contained 1 in. thick synthetic resin material. These walls were equipped with instrumentation such as fiber optics vision system, light emitting diodes (LED) embedded to monitor proppant transport characteristics of the test fluid. A photograph of the two walls on an open HPS are shown in Figure 3.3.

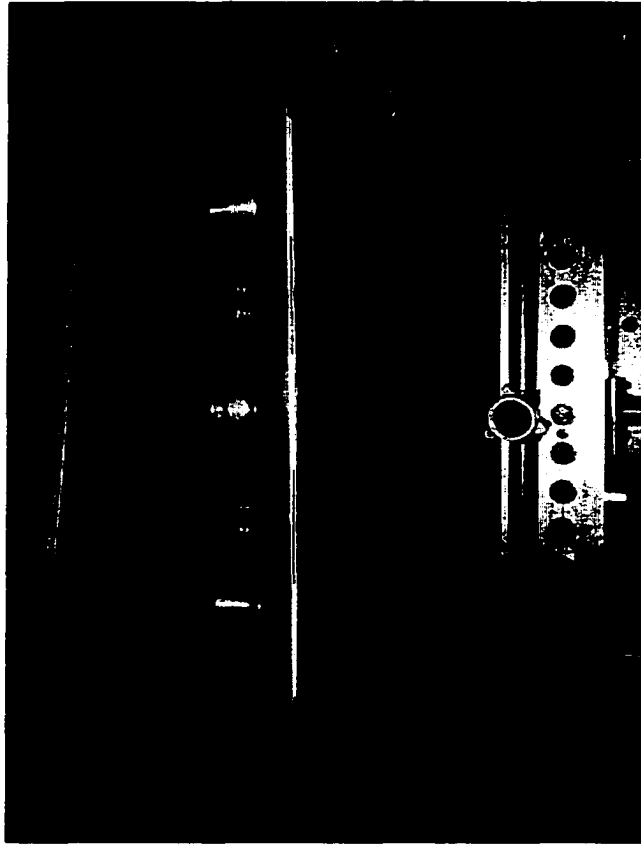


Figure 3.3—LEDs Embedded in One Wall of the HPS

The fiber optics vision system provided a means to see inside the steel-walled HPS. The system consisted of an array of light emitting diodes embedded on one wall to illuminate the gap in the slot. The light generated by the LEDs was received by fibers placed on the opposite wall. The LED system contained 121 discrete HP-2000 diodes molded with the synthetic facing on each platen. With an array of 121 LEDs on each facing and nine facing on each wall, 1089 LEDs illuminated the HPS. Each LED was aligned across the slot gap to a receiving fiber giving a total of 1089 fibers on the opposite wall of the slot. A schematic of the LEDs installed on the HPS is shown in Figure 3.4.

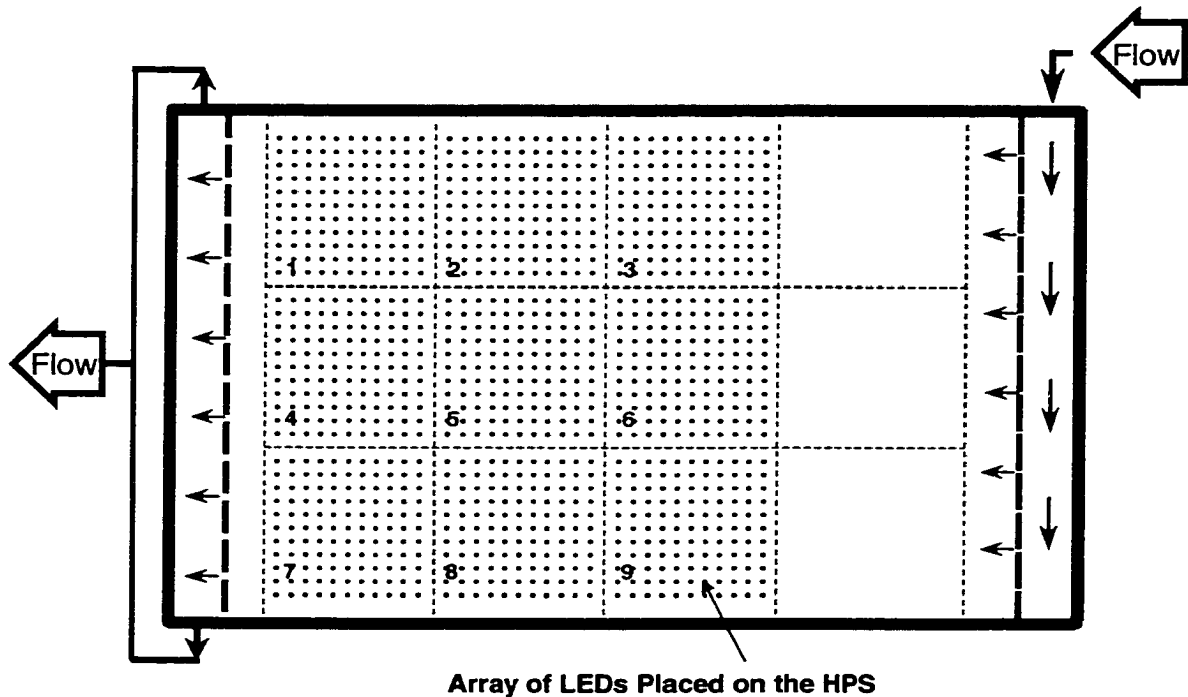


Figure 3.4—Schematic of the LEDs Placed in Nine Platens of the HPS

When fluid flowed through the gap, the light received by the fibers was shadowed and a fluid image was captured by the fibers. The light received by the fibers was similarly affected by the sand particles contained in the fluids and a shadow was observed. For each image-receiving facing of the slot, 121 fibers were grouped together to form an 11 by 11 square bundle of fibers. This bundle, shown in Figure 3.5, was one in. on each side and was constructed in a spatial order similar to the fibers in the facing. The bundle conveyed the image from the slot wall to a window outside the HPS. From the window, the image was captured on a black and white video camera. For nine facings, nine cameras were installed on the HPS in a 3 by 3 array to acquire a complete picture of the fluid flow behavior in the slot.

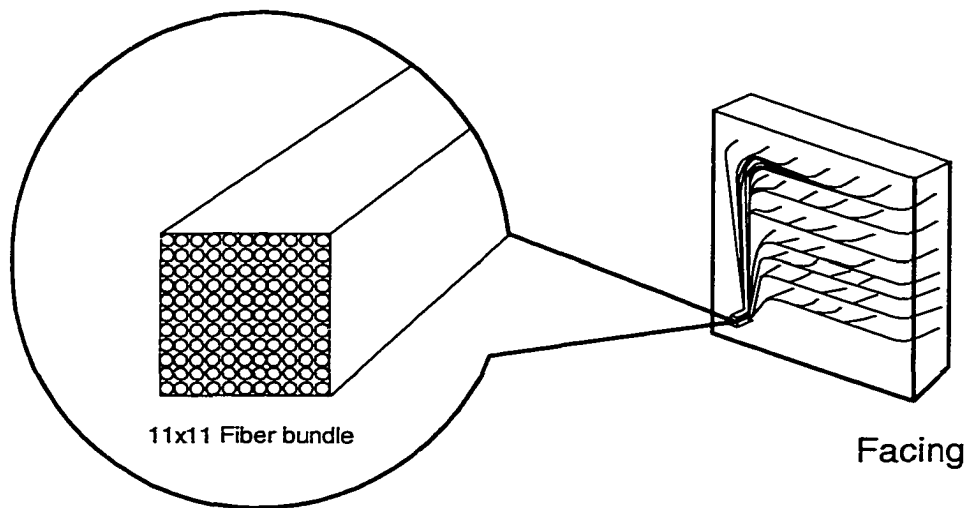


Figure 3.5—11 by 11 Bundle of Fibers to Capture Image Across Each Platen

The nine cameras gave an output image in National Television System Committee (NTSC) video signals at a rate of 30 frames per second. The video signals were driven by a nine-channel gain controller device through cables to a data acquisition system. A schematic of the data acquisition system is shown in Figure 3.6. The individual image from the nine cameras was incorporated into a composite image signal using a video multiplexer, which generated 2.7 frames per second. The composite image was named raw image and was simultaneously displayed on a monitor and recorded on a 8 mm video cassette (Sony Electronics, Oradell, NJ, Model Hi8). The raw image was digitized by a digital signal processing (DSP) frame grabber and displayed by an enhanced display board (EDB). The DSP allowed the system to process one image as it grabbed another, and the EDB displayed the processed image in real time. The digitized image was processed to obtain light intensities, which were then converted to sand concentration using a mathematical model.²⁰² The processed image was displayed on a monitor and recorded on an 8 mm videotape.

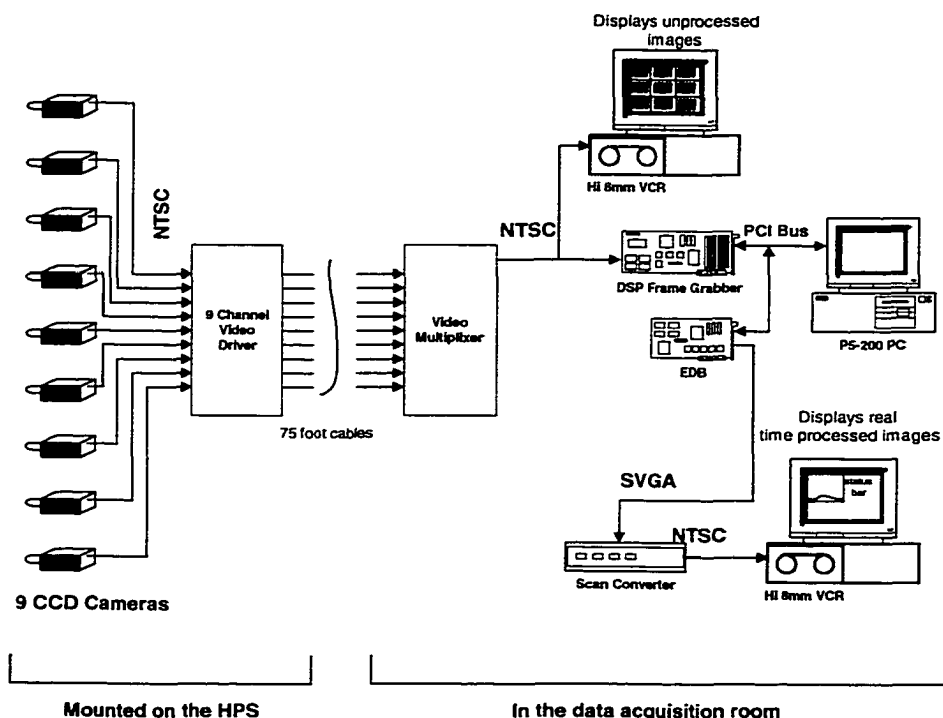


Figure 3.6—Schematic of the Data Acquisition System for the Vision System²⁰²

The inlet manifold on the HPS comprised of a cylindrical wellbore with 22 equally spaced openings for uniform fluid flow across the slot height. These openings were $\frac{3}{4}$ in. in diameter, 3 in. long, and spaced 4 in. apart. The alternate openings were inserted with $\frac{1}{2}$ in. diameter pipe to simulate a perforation tunnel, and the balance were plugged off. The fluid exited the HPS through 22 outlet openings identical in size to the one in the HPS inlet, and entered a manifold of similar size as that in the inlet manifold. The outlet openings were not equipped with the $\frac{1}{2}$ in. inserts, and none of them was plugged off. A schematic of the perforations and platen layout is shown in Fig. 3.4.

After shear conditioning through the coiled tubing, the test fluid was pumped at 10 gpm flow rate through the HPS, set to a gap width of 0.375 in. The balance fluid was

disposed off. The low flow rate through the HPS was maintained to simulate a fracture shear rate of 20 sec^{-1} .

Transparent Slot. This fracture model was also a parallel plate slot having a fixed gap width. It was 12 ft long, 20 in. high, and 0.25 in. wide. This model was made of Plexiglas and so, called transparent slot because it allowed direct visual observations on the proppant distribution in it. A photograph of the transparent slot is shown in Fig. 3.7.

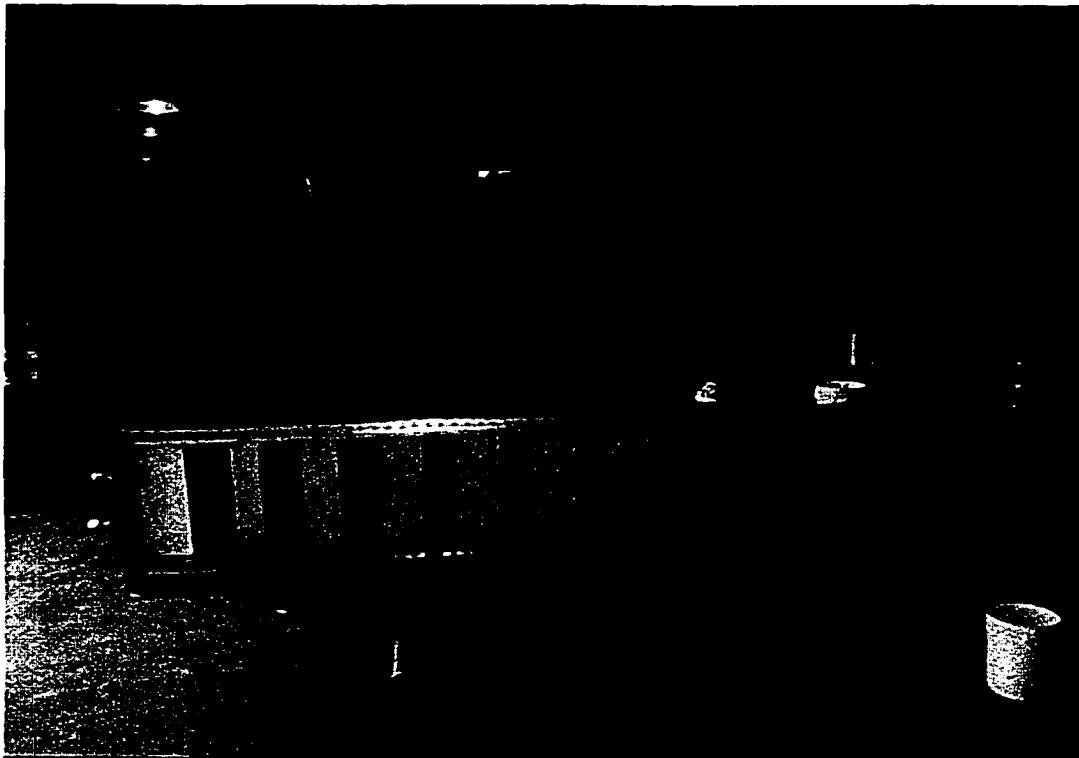


Figure 3.7—Transparent Slot

The proppant distribution in the transparent slot was observed and recorded with a video camera (Panasonic AG-195 P), and the captured images were stored on a videocassette.

A few frames of these video images were digitized, and are shown in the following chapters to describe the proppant transport behavior of crosslinked gels through the transparent slot.

The test fluid was pumped through the transparent slot at 10 gpm, which corresponded to a shear rate of 185 sec^{-1} . Thus, the test fluid was alternately pumped through the HPS and the transparent slot with low shear rate of 20 sec^{-1} maintained in the HPS, and a higher shear of 185 sec^{-1} in the transparent model. These two shear rates allowed simulation of a wider range of fracture flow conditions to evaluate proppant transport capabilities of the test fluids.

3.5 INSTRUMENTATION

The fluid evaluation experiments performed on the field-scale test facility were expensive. It was, therefore, important to gather maximum information with minimum number of tests performed; this required that each experiment be well planned and carefully performed. Also, all data gathered during the experiments were displayed in real time so that any corrective action might be taken immediately during the test. The data were logged and recorded so that they could be analyzed after the test. The data logging was performed with an efficient data acquisition system.

The large-scale experiments also required several instruments to measure the various parameters during the fluid evaluations. These parameters, fluid flow rate, temperature, density and pressure drop across the coiled tubing, were monitored with a state-of-the-art instrument for each parameter. They were then transmitted with a reliable communication medium to the data acquisition system.

Data Acquisition System. The data generated during the field-scale evaluation of the test fluids were acquired and recorded using a Hydra data acquisition system (Fluke Corporation, Everett, WA, Model No. 2625A). This system contained stand-alone data loggers and PC-based front ends. This arrangement allowed accuracy and flexibility in capturing and storing the data.

The hydra had 21 analog measurement channels, 4 alarm output channels, 8 digital input or output channels, and a totalizer channel. A brief specification of the Hydra system is given in Table 3.2.

Table 3.2 Fluke Hydra System Specifications

Channels	40 Analog Inputs
Alarms	4 TTL
Digital Input/Output	8 TTL
Telemetry	RS 232 at 19.2 kbaud; 8 data bits, no parity bit, one stop bit
Sample Rate	Up to 10 samples per second
Calibration	User controlled (linear fit " $mx+b$ ") User controlled Gain and Offset
Dynamic Range	3mV to 300 volts AC or DC 4-20 milliamps current loop

The Hydra Data logger communicated with a host computer via a wireless modem radio link. The wireless tool allowed up to 1200 ft of separation between the remote logger and the computer. The remote operation was necessary owing to the use of various field size equipment in the experiments. The computer controlled all operations on the acquisition system, the base station sent commands and queries to the remote Hydras, and the remote system responded to it with the status and the measured data.

The Hydra system contained two high speed Flukes capable of monitoring individual test parameters. They had a very high common mode rejection, and were unaffected by ground loops or shorts that might occur in the electrical system. The hydras digitized the data signals close to its acquisition location which allowed much shorter signal lines and thus a minimum signal of noise ratio on all experimental parameters. This system provided a common time stamp on all data to facilitate matching and processing data immediately after the experiments.

Fluid Flow Measurement. The fluid flow rates were measured at two locations; the rates through the coiled tubing and the slot models. The flow through the slot models was measured with a magnetic flowmeter and that through the coiled tubing with a mass flowmeter.

The necessity of monitoring the flow with two different flowmeters arose from the dissimilar flow conditions in the coiled tubing and the fracture models. These conditions created separate operational problems. The slurry was easily transported through the coiled tubing, so the flowrate could be monitored with the mass flowmeter. However, at the lower flow rate through the slot models, the sand particles would settle down during the experiments, which made it difficult to monitor the flow rate using the mass-based instrument. The flowmeter working on the fluid velocity was observed to perform better under low fluid flow conditions. Therefore, the magnetic flowmeter was used for measuring the fluid flow through the fracture models, and the mass flowmeter for flow through the coiled tubing.

The magnetic flow meter (Foxboro Inc., Foxboro, MA, Model 281H-SABA-TB) worked on the Faraday's law of electromagnetism. The fluid flowed at right angle through a magnetic field and thus, induced a voltage across the flow tube in the instrument. The induced voltage was correlated with the liquid flow velocity from which the fluid flow rate was calculated in gal per min.

The mass flowmeter (Micro Motion, Boulder, CO, Model No. DS150S 14 35UR) contained a sensor having a flow tube and a signal processing transmitter. This instrument worked according to the Newton's Second Law of Motion. It had an electromagnetic drive coil located at the center of a bend in the flow tube. The coil caused flow tube to vibrate at its natural frequency. When the fluid flowed through the vibrating tube, the fluid mass resisted the tube motion and thus, induced twist in the tube. The twist in the tube, called Coriolis effect, was correlated to the mass flow rate of the fluid flowing through the tube. The instrument was rated to measure a flow rate of 2800 lb/min, and to a pressure of 1500 psi and temperature of 400°F. The flow meter had an accuracy of 0.12 gal/min at the 40 gal/min used in the present research.

In addition to the flow rate, the mass flowmeter measured the fluid density and temperature. The fluid density was a useful parameter to monitor the slurry concentration in the fluid flowing through the system.

Differential Pressure Transducers. The pressure drops were monitored across the coiled tubing and the HPS. The differential pressure across the coiled tubing was measured to estimate friction loss in the tubular and to monitor the shear preconditioning of the crosslinked gel. The pressure drop in the HPS was used to calculate the fluid

viscosity under fracture flow conditions and to compare the calculated viscosities of different fluids flowing through the slot model.

The pressure drop in the coiled tubing was measured with two microprocessor based differential pressure transmitters (Honeywell, Ft. Washington, PA, Model STD170). The two instruments were placed across two coiled tubing: 2000 and 1000 ft long. They were calibrated to a span of 1000 psi across the 1000-ft coil, and 2000 psi across the 2000-ft coiled tubing. The instrument accuracy included the combined effects of linearity, hysteresis, and repeatability, and was within 0.15% of the calibrated span.

The pressure drop in the HPS was also measured with a microprocessor based differential pressure transmitter (Honeywell, Ft. Washington, PA, Model STD120). It was measured across 56 in. section of the HPS length. The instrument was rated to a pressure drop of 400 in. of water, and was calibrated to a span of 10 psi. The instrument accuracy was within 0.0625% of the calibrated span.

Visual Basic Programs. Enormous amount of data were collected during the field-scale evaluation as well as the laboratory testing of this research. The analysis of these data was very cumbersome. Therefore, several visual basic programs were written to analyze the data and present them in a form from where meaningful interpretation could be made. The algorithms of these visual basic macros are given in Appendix A. These algorithms describe the procedure for analyzing the light intensity data gathered from the vision system and the viscoelastic data measured with the Bohlin rheometer.

CHAPTER 4

JUSTIFICATION FOR THE EXPERIMENTAL EVALUATION

4.1 INTRODUCTION

The purpose of this investigation is to understand the fluid rheological property that describes the proppant transport characteristics of fracturing fluids. Since fluid viscosity is currently used to describe the proppant transport behavior, an obvious first step is to understand the shortcomings of this rheological property. The shortcomings in fluid viscosity can be evaluated using the Fracture models widely used today and can also be observed through experimental investigations.

The fracture models were used to study the influence of fluid viscosity on fracture dimensions and stimulated reservoir productivity. This study was performed by stimulation treatment of two different example reservoirs. The first treatment was evaluated with a two-dimensional fracture model, and the second treatment was evaluated with a three-dimensional model of a commercially available fracture simulator. In both treatment simulations, the fluid viscosity was varied while maintaining constant all other stimulation and reservoir parameters.

The experiment investigation was performed by observing the proppant transport behavior through the transparent slot model. This investigation was performed in two crosslinked gels that had very similar fluid viscosities. These gels were evaluated for their capabilities to transport 2 ppg slurry through the slot model.

The fluid evaluations with the fracturing simulator and the slot models showed number of observations which could not be explained with the fluid viscosity. The results of these evaluations are described in this chapter.

The shortcoming of viscosity in explaining the observed proppant transport behavior through fracture requires search for a better rheological parameter. Another measurable fluid rheological parameter is its elasticity. Because the current fracturing simulators are not equipped to handle fluid elasticity, the elasticity evaluation can only be performed with experiments. These experiments should however be performed on the fluids used in the actual fracturing treatments. The experimental evaluation requires selection of fracturing fluid concentrations and the test parameter that is representative of an actual fracturing treatment. The parameters selected for the experiments and the reasons for their selection are justified in this chapter.

4.2 FRACTURE DESIGN USING 2-D FRACTURE MODEL

The influence of fluid rheology on fracturing treatment was first studied using the two-dimensional Geertsma-deKlerk (GdK) model.¹

The GdK model assumes fracture height is constant but fracture width and length change during the fracturing treatment. The width is expressed as a function of the fracture length. This model considers the fracture face near the wellbore has a rectangular shape in the vertical plane, and the fracture has an elliptical configuration in the horizontal plane.¹

In the present study, the GdK model was used to study the effect of fluid viscosity on the productivity of a stimulated reservoir. The petrophysical properties of the

reservoir and the design parameters of the stimulation treatment are summarized in Table 4.1. The procedure for the fracture design evaluation is summarized in Appendix B.

Table 4.1 Example Reservoir and Fracturing Treatment Parameters

Static Reservoir Pressure P_r	1800 psi
Reservoir Permeability k	0.1 md
Reservoir Porosity ϕ	8%
Depth to Producing Formation	4025 ft
Young's Modulus E	3.5×10^6 psi
Poisson's ratio ν	0.3
Vertical Stress Gradient S_v	1.15 psi/ft
Well Spacing A_c	80 acres
Wellbore radius r_w	2.5 in.
Producing Formation height h	50 ft
Fracturing Fluid	VERSAGEL 1300
Fracturing Fluid Type	30 lb/Mgal HPG
Fluid Rheological Model	$\mu = k \left(\dot{\gamma} \right)^{n-1}$
Base Case	
Power law exponent n	0.62
Consistency Index k	0.009 lbf sec ⁿ /ft ²
Fluid Volume V_{fluid}	40000 gal
Pad Volume V_{pad}	10000 gal
Fluid Pump Rate Q	15 bbl/min
Fluid Loss Coefficient C	0.00147 ft/min ^{1/2}
Spurt Loss V_{sp}	0.0 gal/ft ²
Proppant Size and Type	20/40 mesh Sand
Average Proppant Concentration	3 ppg
Closure Stress on Proppant Bed	2000 psi
Maximum Wellhead Pressure	4500 psi
Static Bottom hole Temperature	100°F

The fracturing fluid was assumed to exhibit non-Newtonian characteristics, and that a Power law model described its viscosity. The effect of fluid viscosity on the productivity was studied by changing separately power law index, n , and consistency index, k . The

values of n were varied from 0.5 to 1, and those of k from 0.005 to 0.03 lbf secⁿ/ft². The results of the influence of viscosity are shown in Fig. 4.1 and 4.2.

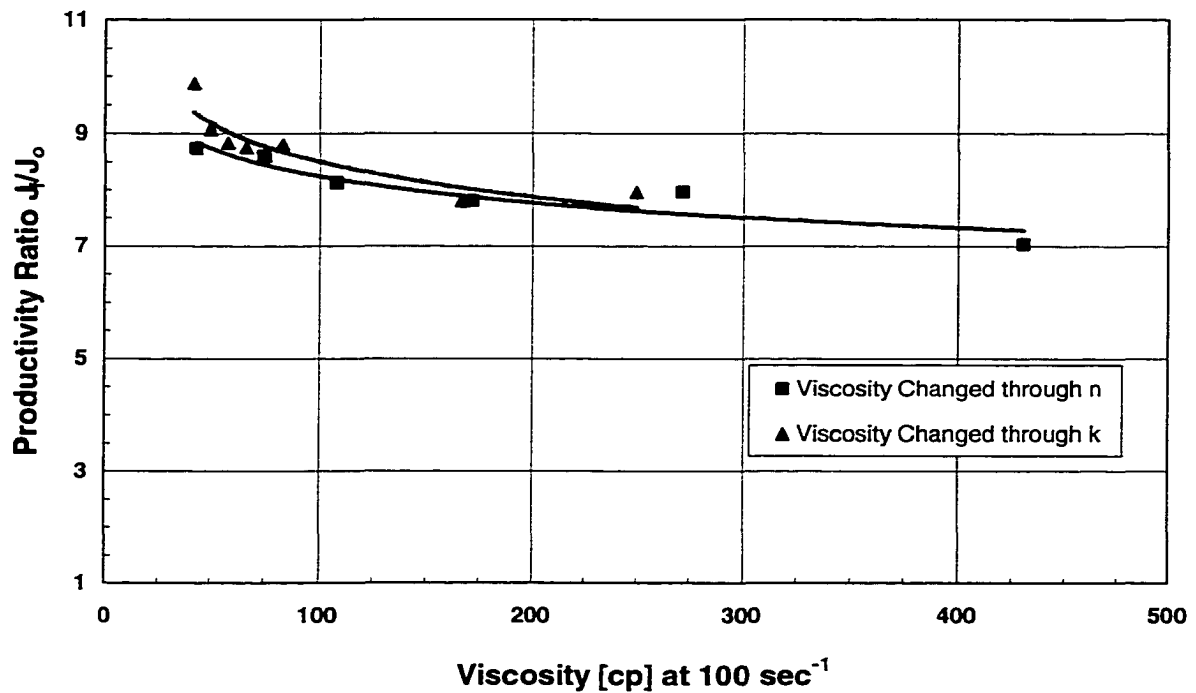


Figure 4.1—Effect of Fluid Viscosity on Productivity Ratio using the 2-D Model

Figure 4.1 shows the fluid viscosity at 100 sec⁻¹ as a function of productivity ratio of the stimulated to non-stimulated reservoir, J/J_0 . The figure shows the productivity ratio dropped from 10 to about 7 as the fluid viscosity was increased from 40 to 400 cp. This decrease in the productivity ratio with an increase in viscosity was due to a decrease in the fracture dimensions, as shown in Fig. 4.2. The figure depicts decrease in the fracture length and propped height with an increase in the fluid viscosity. This observation is contrary to what one would expect. As fluid viscosity increases, the proppant suspension characteristics of a fracturing fluid would improve and so, the propped length of the fracture should increase. However, the expected behavior is not seen in Figs. 4.1 and 4.2.

The two-dimensional GdK model, hence, does not accurately depict the effect of fluid viscosity on the fracture dimensions. The poor depiction also means that this model cannot provide a critical viscosity value above which the fluid would exhibit transport of proppant through a fracture. This value can be used prior to a fracturing treatment to evaluate if a fracturing fluid would perform satisfactorily during the treatment.

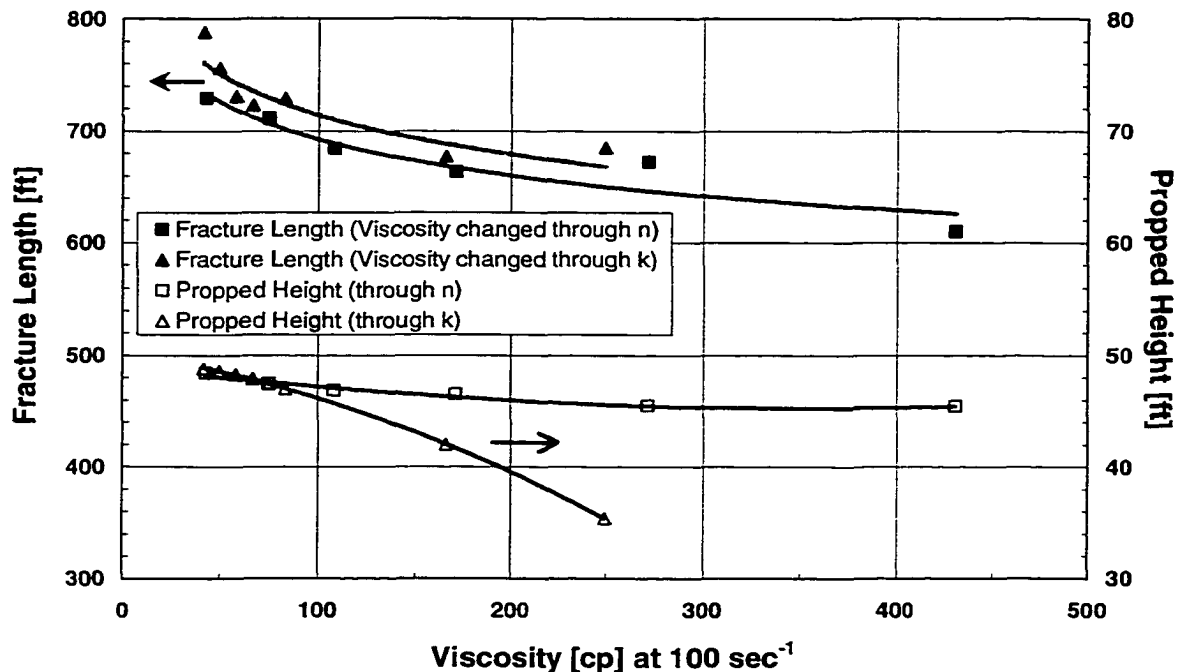


Figure 4.2—Effect of Fluid Viscosity on Fracture Dimensions using the 2-D Model

Figures 4.1 and 4.2 display that the effect of changing n and k is not similar. At similar viscosities, the productivity ratio and the fracture dimensions are dissimilar because of selection of different parameters of the Power law model. This difference highlights the importance of understanding the influence of each characteristic of the fluid on the stimulation treatment.

The influence of fluid viscosity on the reservoir productivity was further evaluated using a commercially available fracture simulator. This evaluation is described below in detail.

4.3 FRACTURE DESIGN USING FRACPRO™ SIMULATOR

The effect of fracturing fluid viscosity on reservoir performance was studied on the commercial simulator called FRACPRO™. The fracture design was run on a reservoir whose parameters were taken from the tutorial example described in the simulator manual.²⁰³

The fracture simulations were performed using the three-dimensional model of FRACPRO 99. This model was not described in the user manual, so its characteristic features are not known. The manual indicated that the simulator model considered spatial variations in the reservoir stress, rock modulus, pressure, and flow distribution; but it did not calculate the variations at specific points within the fracture. It further considered the non-Newtonian effects of the fracturing fluid, hindered settling rates, and settled bank build-up. These features of the 3-D simulator model described in the FRACPRO 99 manual are similar to those considered in the 2-D fracture model described in Section 4.2.

The example reservoir for the simulations was Sawyer 144A No. 5 well from the Sutton County in Texas.²⁰³ The well was stimulated in the middle and upper Canyon Sands. The simulations runs described in the present study were performed on the middle sand only. These runs were made by varying the fracturing fluid viscosity parameters in the simulator, while keeping all other reservoir and treatment parameters identical to those in the tutorial example.

The parameters input into the simulator are summarized in Tables 4.2 through 4.6, and the results of the simulation runs are shown in Figures 4.3 through 4.5. Table 4.2 lists the petrophysical and reservoir fluid properties, Table 4.3 the well completion parameters, Table 4.4 the formation stress profile, and Table 4.5 the fracture treatment schedule. Table 4.6 lists the miscellaneous options selected in the FRACPRO for the simulation runs.

Table 4.2-Reservoir Parameters for FRACPRO Simulation

Reservoir Parameter	Parameter Value
Permeability	From 5933 to 6008 ft – 0.01 md From 6008 to 6152 ft – 0 md From 6152 to 6227 ft – 0.01 md
Porosity	7.5%
Formation Type	0 to 6375 ft – Sandstone Beyond 6375 ft – Shale
Reservoir Temperature	170°F
Reservoir Pore Pressure	2010 psi
Average Pressure in Fracture	5350 psi
Pore Fluid Compressibility	0.0005 psi ⁻¹
Leakoff Fluid Permeability Ratio (K_p/K_t)	10.0
Leakoff Fluid Viscosity	1 cp (cold) 0.5 cp (hot)
Pore Fluid Viscosity	0.02 cp (cold) 0.01 cp (hot)

Table 4.3-Well Completion and Tubular Configuration for the Simulation

Well Parameter	Parameter Value
Annulus Segment 0 to 5609 ft	Casing ID 4.892 in., Tubing OD 2.6 in., and ID 2 in.
Casing Segment up to 6186 ft	Casing ID 4.892 in.
Perforated Interval Bottom	6186 ft
Number of Perforations	200
Perforation Diameter	0.33 in.
Perforation Intervals	One
Wellbore Orientation	Vertical
Wellbore Fluid	Slick water

Table 4.4-Formation Stress Profile for FRACPRO Simulation

No.	Depth (ft)	Stress (psi)	Young' Modulus (psi)	Poisson's Ratio
1	0	4100	$5 * 10^6$	0.2
2	5695	4400	$5 * 10^6$	0.2
3	5805	4650	$5 * 10^6$	0.2
4	5917	4250	$5 * 10^6$	0.2
5	6023	4950	$5 * 10^6$	0.2
6	6060	4250	$5 * 10^6$	0.2
7	6230	4900	$5 * 10^6$	0.2
8	6255	5200	$5 * 10^6$	0.2
9	6277	4900	$5 * 10^6$	0.2
10	6325	5150	$5 * 10^6$	0.2
11	6375	5350	$5 * 10^6$	0.2
12	6470	5450	$7.5 * 10^6$	0.22

Table 4.5-Fracturing Treatment Schedule for the Simulation

No.	Slurry Rate (bpm)	Proppant Concentration (ppg)	Stage Length (min)	Proppant Type
1	27	0	23	-
2	27	1	3	Brady Sand 20/40 mesh
3	27	2	2.5	-- do --
4	27	3	4	-- do --
5	27	4	3.5	-- do --
6	27	5	5	-- do --
7	27	6	4.5	-- do --
8	27	7	4.8	-- do --
9	27	8	2	-- do --
10	27	0	3.5	-
11	0	0	40	Shut-In

Table 4.6-Miscellaneous Simulation Options Selected for the Runs

1	Run from Job-Design Data
2	FracPro 3D Model
3	No temperature modeling
4	Proppant Convection
5	Allow Fracture Growth at Shut-in
6	Model Wellbore and perforation
7	FRACPRO Fluid Path Tubular Configuration
8	Run Fracture and Wellbore Models
9	General Iteration
10	Vertical Fracture
11	General Reservoir
12	Ignore Back Stress Effects
13	Calculate C-Total from Permeability
14	Producing Bottomhole Pressure 2614 psi
15	Calculate Fluid Volume from Stage Length
16	Simulator Start Time 0, End Time 100 min with Time step of 0.5

The fracturing treatment was performed with a borate-crosslinked 35 lb/Mgal guar gel. This gel was listed in the FRACPRO fluid library as HL_BOR_G35_1, manufactured by Halliburton as BoraGel. The fluid for the pad and the slurry stages of the treatment was maintained similar. The fluid viscosity data given in the simulator library are summarized in Table 4.7. The table lists the viscosity data as the Power law model parameters at several temperatures, at an initial time and after four hr. For the simulation runs, the FRACPRO linearly interpolated the fluid library data to the reservoir temperature conditions.

The fluid library data were first used to perform an initial simulation run; this run was named Base Case 1. The fracturing fluid viscosity data were manipulated by changing either the consistency index, k , or the power law index, n , to perform several fracturing simulation runs. These runs were performed by varying the individual power law model parameter as a percentage of the base case while keeping all other reservoir

and fracturing parameters in the simulations similar. The fluid formulations thus generated and their viscosity data are summarized in Table 4.8. These fluid formulations simulated fluid viscosities from 50 to 300 cp at 100 sec⁻¹.

Table 4.7-Viscosity Data of BoraGel from the FRACPRO Fluid Library

Temperature °F	Power law exponent <i>n</i>		Consistency Index <i>k</i> lb _f sec ^{<i>n</i>'} /ft ²	
	Time = 0 hr.	Time = 4 hr.	Time = 0 hr.	Time = 4 hr.
75	0.32	0.40	0.323	0.0999
100	0.33	0.41	0.274	0.0837
125	0.34	0.43	0.226	0.0681
150	0.35	0.44	0.185	0.0548
175	0.37	0.46	0.149	0.0435
200	0.39	0.48	0.117	0.0336
225	0.42	0.51	0.0874	0.0249

Table 4.8-Summary of Viscosity Data at 170°F for Different Simulation Runs

Cases		Power Law Exponent <i>n</i>		Consistency Index <i>k</i> lb _f sec ^{<i>n</i>'} /ft ²		Viscosity cp at 100 sec ⁻¹
		at 0 hrs	at 4 hrs	at 0 hrs	at 4 hrs	after 3 hrs
1	Base	0.366	0.456	0.156	0.04576	220
2	1.1 <i>n</i>	0.403	0.502	0.156	0.04576	268
3	0.9 <i>n</i>	0.33	0.41	0.156	0.04576	179
4	1.1 <i>k</i>	0.366	0.456	0.1716	0.05	240
5	0.9 <i>k</i>	0.366	0.456	0.1404	0.0412	197
6	0.5 <i>k</i>	0.366	0.456	0.078	0.0229	110
7	0.7 <i>k</i>	0.366	0.456	0.109	0.032	153
8	0.5 <i>n</i>	0.183	0.228	0.156	0.04576	81
9	0.3 <i>n</i>	0.11	0.137	0.156	0.04576	54
10	0.3 <i>k</i>	0.366	0.456	0.0468	0.01373	65

The results of the fracturing simulation runs for all ten cases were then incorporated into reservoir simulation mode of the FRACPRO simulator package. The reservoir simulator was then used to study the stimulated reservoir production performance over a period of

ten years. The reservoir and the economic parameters for the reservoir simulation runs are given in Table 4.9.

Table 4.9-Input Parameters for the Reservoir Simulation Runs

Input Parameter	Parameter Value
Depth to Middle of Pay	6136 ft
Gross Pay Thickness	200 ft
Net Pay Thickness	100 ft
X-Y Extent of the Reservoir	1000 ft by 2500 ft
Gas Price	3 \$/MSCF
Price Escalation	3% per year
Discount Rate	15%
Operating Cost	\$5000 per month
Cost Escalation Factor	5% per year
Initial Investment	\$ 650,000
Stimulation Cost	\$ 85,000
Total Production Period	10 years

The results of the fracture simulation runs are shown in Figures 4.3 and 4.4, and that of the reservoir simulation runs are shown in Figure 4.5. Figure 4.3 shows the effect of fluid viscosity on the fracture dimensions. The figure presents the changes in the viscosity through adjustments in the n values as filled marker points and those through adjustments in the k values as unfilled points. The fracture dimensions obtained with the Base Case fluid are shown by larger size markers.

Figure 4.3 displays a small change in propped height and length of the fracture, and fracture width at perforations when the fluid viscosity was changed from 50 to 250 cp. The small change in the fracture dimensions indicates a poor dependence of the fracture geometry on the fracturing fluid viscosity. This result implies an insignificant contribution of the fluid rheology on the stimulation treatment of a reservoir. It is, however, not true as discussed below.

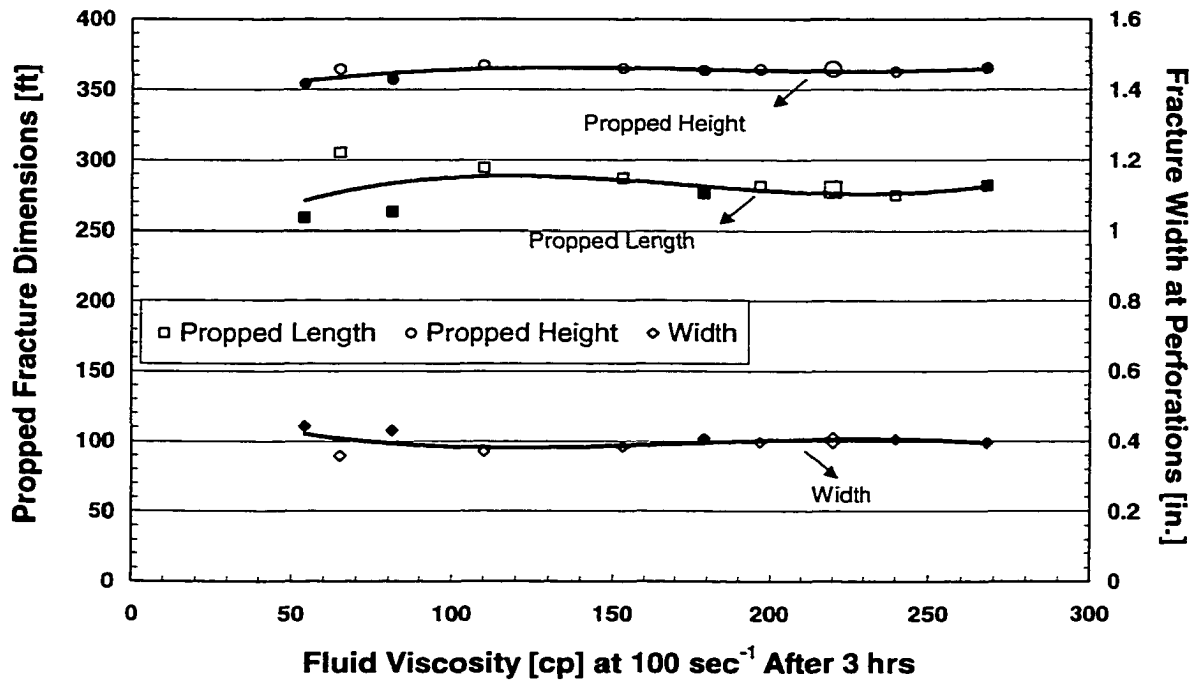
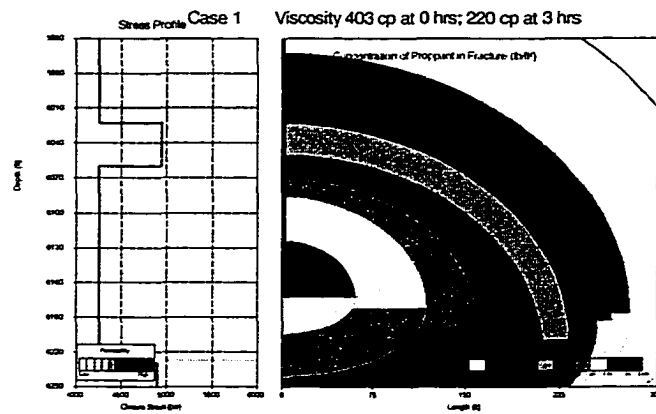


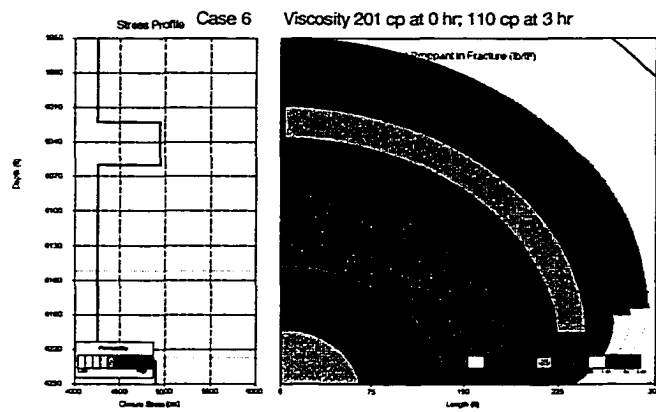
Figure 4.3—Effect of Fluid Viscosity on Fracture Dimensions using FracPro Model

Figure 4.3 shows that there is not much difference in the fracture dimensions whether the fluid viscosities were manipulated with either n or k . There is thus no difference in the viscosity with either of the power law model parameters. But when the simulation results are plotted as proppant concentration in the fracture, a different picture evolved.

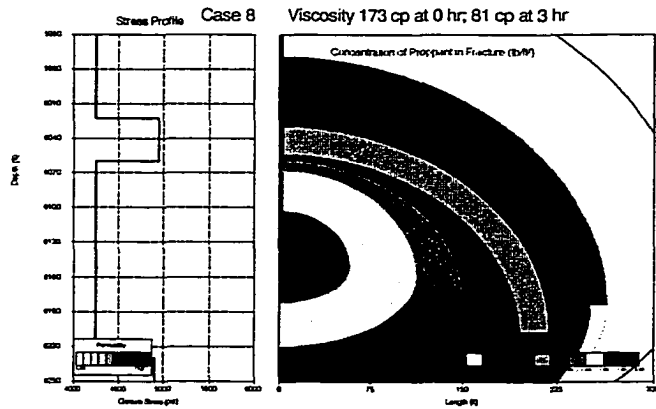
Figure 4.4 shows the proppant concentration in the fracture (lb/ft²) for the three cases 1, 6, and 8 described in Table 4.8. The figure presents first the proppant distribution obtained with the base case fluid, followed by that with Cases 6 and 8. The Base Case 1 described the stimulation with a 220 cp viscosity fluid, Case 6 with 110 cp and Case 8 with 81 cp viscosity. The figure on the right track of each case displays the proppant concentration after each of the 11 stages of the fracturing treatment. On the left track, it presents the permeability and stress profile as a function of the formation depth.



(a) Base Case 1 Viscosity 403 cp at 0 hr: 220 cp at 3 hr



(b) Case 6 Viscosity 201 cp at 0 hr: 110 cp at 3 hr



(c) Case 8 Viscosity 173 cp at 0 hr; 81 cp at 3 hr

Figure 4.4—Effect of Fluid Viscosity on Proppant Concentration in the Three Cases

The proppant concentration in Figure 4.4 is a ratio of weight of the proppant placed in fracture to the fracture face area and, was calculated from the product of the fracture length and height.

Figure 4.4 (a) shows the proppant concentrations were higher in the upper half of the fracture for the Base case fluid of 220 cp viscosity. The figure also depicts the proppant concentration increased near the wellbore because the higher concentration slurries were pumped in the later stages of the treatment. Since the concentration of proppant was higher in upper half of each stage, it is safe to conclude this fluid successfully transported proppant through the fracture and that the stimulation treatment was successful with the Base case fluid. Based on the similar arguments, the performance of Case 6 fluid formulation having 110 cp viscosity, after 3 hours, cannot be called successful from Fig. 4.4 (b). The figure shows that the sand settled down in the fracture because the proppant concentrations were higher in the bottom of the fracture than those in front of the perforations, 6086 to 6186 ft. The concentrations were lower than those obtained in the Base case. These observations suggest unsatisfactory performance of the Case 6 fluid. The poor stimulation treatment with Case 6 would be attributed to the lower viscosity of this fluid as compared to that of the Base case. A further reduction in fluid viscosity however depicts contrasting behavior. Figure 4.4 (c) shows the proppant concentration in the fracture when Case 8 fluid having 81 cp viscosity was used to stimulate the formation. The figure displays the proppant concentration was uniform in the upper and lower half of the fracture, and the proppant were well distributed in the fracture. Figure 4.4 thus points that the proppant transport and suspension with Case 8 fluid was as good as that with the base case, and was better

than that with Case 6 fluid even though the fluid viscosity in Case 8 was lowest of the three cases.

The dissimilar performances of the three cases are difficult to explain with the fluid viscosity alone. The difference between the fluid formulations of Case 6 and 8 was that in Case 6, the consistency index of the fluid was 50% of the Base case value, whereas in Case 8, the power law index of the fluid was 50% of the base case. The unsatisfactory performance of Case 6 fluid depicts a greater dependence of consistency index on the three dimensional model used in the FRACPRO simulator.

A higher proppant concentration in the fracture means higher fracture conductivity and higher permeability of the stimulated formation. Thus, the productivity index of the reservoir stimulated with the fluid formulations of the Base case and Case 8 would be higher than that obtained with the Case 6 fluid.

Figure 4.5 shows the effect of fracture on the performance of the reservoir. The figure displays a negligible difference in the cumulative gas production in 10 years, or in the net present value of the stimulated formation when the fluid viscosity was changed from 50 to 250 cp. For similar change in the viscosity, there was negligible improvement in the productivity index of the stimulated formation. Thus, Figure 4.5 suggests no impact of the fluid viscosity on the performance of the stimulated reservoir.

The fracture design simulations performed with the FRACPRO simulator and GdK model indicate that the viscosity is not a critical parameter during a hydraulic fracturing treatment. Figure 4.4 however signifies the role of fluid rheology on the stimulation treatment, because the proppant distribution in the fracture were observed to be dissimilar in the three cases, which differed only in the rheological parameters of the

fluids used for the stimulation. The proppant distribution seen in Figure 4.4 cannot be explained through fluid viscosity. Therefore, the three-dimensional commercial simulator was as unsuccessful as the two-dimensional model for evaluating a fracturing fluid and to identify a critical viscosity above which the fluid would satisfactorily transport proppant through a fracture in a stimulation treatment.

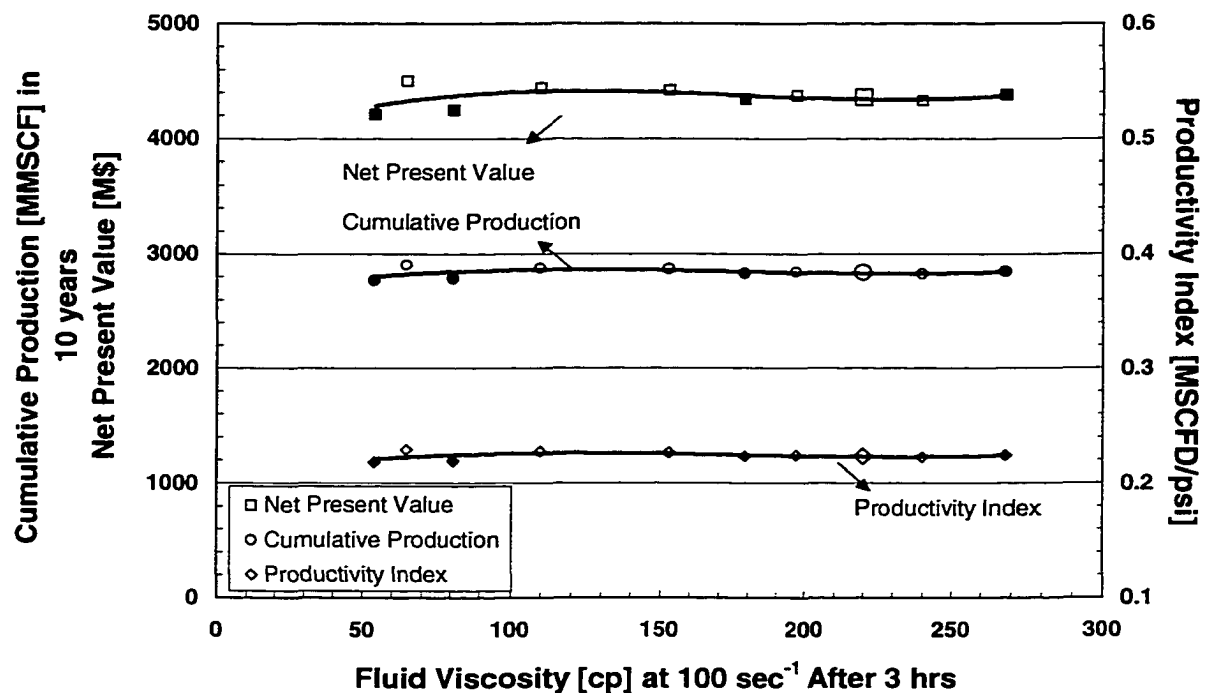


Figure 4.5—Effect of Fluid Viscosity on the Stimulated Reservoir Performance

Several case histories on hydraulic fracturing treatments have shown that the fluid viscosity has a significant effect on the fracture created in a reservoir.²⁰⁴ The viscosity is observed to be an important parameter to effectively transport proppant in the near-wellbore region,¹⁵ a critical region for stimulating reservoir productivity. The importance of fluid viscosity is evident in the field but is absent in the fracture models available

today. Therefore, only experimental investigation can evaluate the fluid rheology for satisfactory proppant transport through a fracture. The experiment evaluation offers an opportunity to understand the relationship between the fluid rheology and proppant transport and to identify the rheological property that can differentiate a successful stimulation from an unsuccessful one without actually performing the fracturing treatment.

4.4 EXPERIMENTAL INVESTIGATION

The experimental investigation required selection of a physical model to represent a fracture, and needed identification of the test parameters that would simulate fluid flow conditions of an actual fracturing treatment in the selected physical model.

The American Petroleum Institute (API) recommends shear conditioning a fracturing fluid at a shear rate of 650 or 1350 sec^{-1} for a period of 2.5 or 5 min.¹⁴⁵ Therefore, in the present research, a shear rate of 1000 sec^{-1} was used to condition the fracturing fluid prior to its flow through a fracture. This shear conditioning was simulated for a period between 2.5 to 5 min., so that the measurements were within the API recommended values.

A coiled tubing of 1.5 in. nominal diameter, 1.188 in. ID, and 3000 ft length was available at the University of Oklahoma. So to simulate 1000 sec^{-1} shear pre-conditioning, the fluid was pumped through the coiled tubing at 40 gpm. This flowrate allowed fluid residence time of less than 4 min. in the coiled tubing. The residence time represented the time to pump the fluid down the wellbore during a fracturing treatment.^{69,205,206}

By using the flow rate of 40 gpm through the shear-conditioning loop, an upper limit was set on the fluid flow rate through the slot models.

Fracture Model. A fracture can be assumed to have parallel faces, so it may be simulated with a slot model. The slot dimensions should be selected that they represent a real fracture. The parallel walls of the slot model should be separated at a gap equal to the width of the fracture created during a stimulation treatment. The slot height and length should be selected to simulate fluid flow through the model under laminar flow conditions as in a flow through a fracture. Furthermore, the slot model should be long enough to allow fluid to reside in the slot for a sufficient period such that the proppant transport characteristics of the fluid can be satisfactorily evaluated.

In a typical fracturing treatment, the fluid is pumped at about 10 to 50 bbls per min (bpm). The fracture created in the formation has gap widths from 0.25 to 1 in. and height from 50 to 300 ft. Thus, the velocity of the fluid flowing through a fracture can be estimated by the following equation, in field conditions,

$$v\left(\frac{cm}{sec}\right) = 2052 \frac{Q(bpm)}{w(in.)H(ft)} \dots\dots\dots (4.1)$$

and the shear rate through the slot is given by

$$\dot{\gamma}(sec^{-1}) = \frac{6v}{w} = 8I \frac{Q(bpm)}{w^2(in.^2)H(ft)} \dots\dots\dots (4.2)$$

For the typical fracturing conditions, the fluid velocity in the fracture would vary from about 100 to 5000 cm/min, and the shear rate from 1 to 500 sec⁻¹.

The fracture was simulated in two slot models, the transparent slot and the high-pressure simulator described in Chapter 3. These models were not designed for the

present work but were available at the university. Hence, the dimensions of the models were fixed; the fluid flow rates were adjusted such that the fluid velocity through them was within the range of the velocities obtained in the field treatments. Equations 4.1 and 4.2 were simplified for the known dimensions of the available slot models. The velocity through the transparent slot, set to a gap width of 0.25 in., was given by

$$v\left(\frac{cm}{sec}\right) = 120q\left(\frac{gal}{min}\right) \dots\dots\dots (4.3)$$

and that through the high pressure simulator, set to a gap width of 0.375 in., was given by

$$v\left(\frac{cm}{sec}\right) = 19q\left(\frac{gal}{min}\right) \dots\dots\dots (4.4)$$

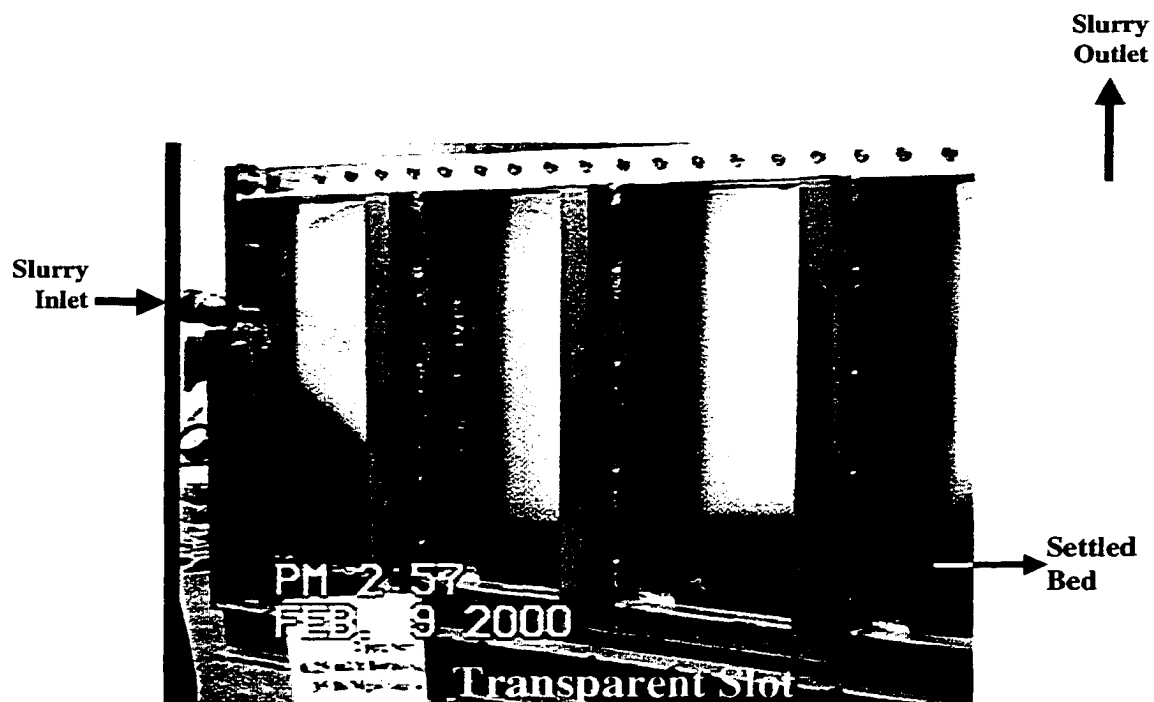
The velocity equations suggested that several flow rates below 40 gpm could be selected for fluid flow through the slot models. Hence, the fluid flow rates were first varied experimentally to identify a minimum rate that could be easily controlled and that would cause least erosion in the equipment if the proppant slurry was pumped through the slot models. The analysis showed that a minimum rate of 10 gpm was ideal for the experimental evaluation. This flow rate corresponded to fluid flow velocity of 200 and 1200 cm/min, and slot shear rates of about 20 and 200 sec⁻¹. These values lie within the range observed under the field conditions.^{69,206} Therefore, a flow rate of 10 gpm was selected to evaluate the proppant transport behavior of the test fluids.

Preliminary Experimental Evaluation. The selected experimental conditions were used first to investigate the significance of fluid viscosity on the proppant transport through a fracture. The investigation was performed in borate-crosslinked 35 lb/Mgal guar gel, which was also used in the evaluation with the FRACPRO model.

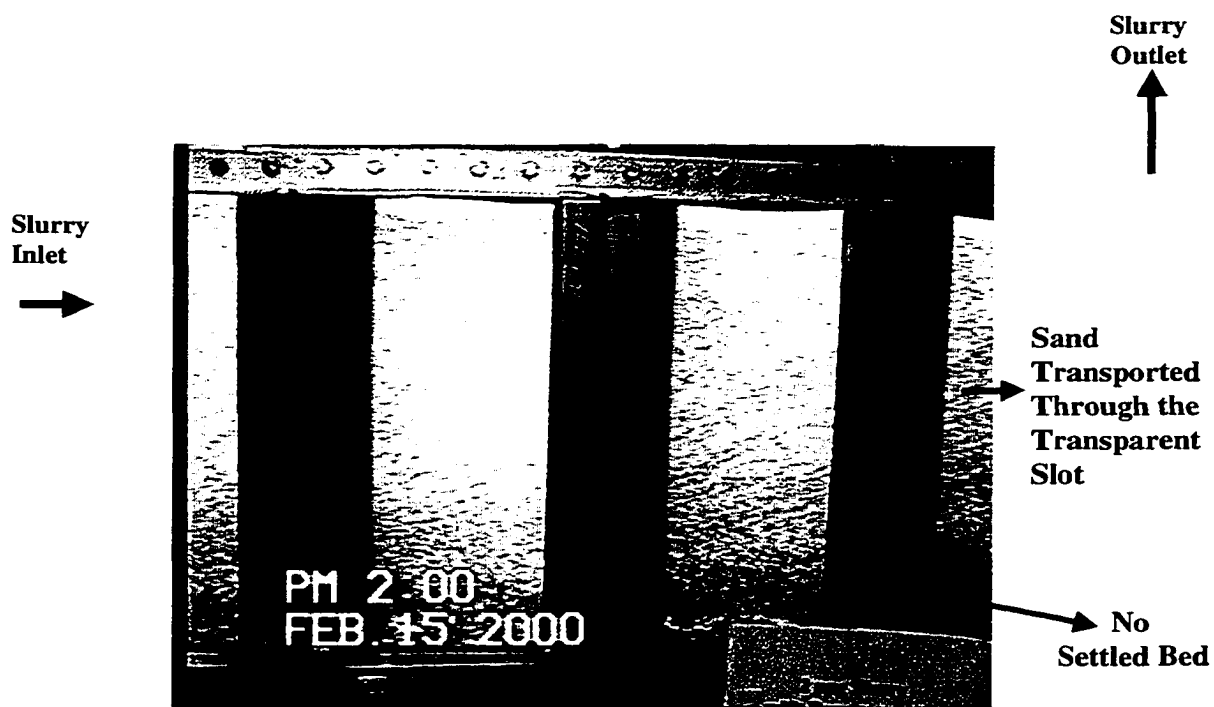
The crosslinked guar gel was prepared in two borate-crosslinker concentration of 0.25 and 0.45 lb/Mgal of guar solution. The gel prepared at 0.25 lb/Mgal concentration had an initial viscosity of 200 cp at 100 sec^{-1} and 135 cp at the end of the three-hour API viscosity test (see Figure 5.1 for the viscosity data). Similarly, the gel prepared at 0.45 lb/Mgal concentration had an initial viscosity of 260 cp, which stabilized to 150 cp after three hours. These values suggested that the two gels had very close viscosities, and so their proppant transport behaviors should also be similar. It was however not so. Figures 4.6 and 4.7 describe the sand suspension behavior of the two gels.

Figure 4.6 shows a settled bed formed in the transparent slot model when a 2 ppg slurry was pumped with the gel prepared at 0.25 lb/Mgal crosslinker concentration. This bed was formed as soon as the slurry was introduced into the slot. The figure further points that the bed was formed below the inlet opening on the slot. This meant that the proppant concentration and the fracture conductivity would be poor in front of the perforation. A lack of proppant particles across the perforation would undermine the success of the stimulation treatment, because the proppant keep the fracture open and provide a highly conductive path for the reservoir fluids to flow. The performance of the guar gel prepared at 0.25 lb/Mgal concentration would therefore be unsatisfactory.

On the other hand, the performance of the gel prepared at 0.45 lb/Mgal crosslinker concentration was satisfactory as shown in Figure 4.7. The figure shows the proppant were uniformly distributed across the height and length of the slot. No settled bed was seen in the slot. This fracturing gel was therefore able to successfully transport sand particles through the fracture model.



**Figure 4.6—Proppant Transport in Transparent Slot with
0.25 lb/Mgal Borate-Crosslinked Guar Gel**



**Figure 4.7—Proppant Transport in Transparent Slot with
0.45 lb/Mgal Borate-Crosslinked Guar Gel**

Figures 4.6 and 4.7 show that the gel prepared at 0.45 lb/Mgal performed better than the gel at 0.25 lb/Mgal crosslinker concentration even though both had similar viscosity values. . Thus, the fluids having similar viscosities do not exhibit identical proppant transport characteristics. The fluid evaluations performed with the fracture simulator models and described in Section 4.3 showed that the fluid having lower viscosity exhibited better proppant distribution in the fracture. The lack of correlation between the proppant transport and viscosity implied that there was some other fluid property, instead of viscosity, that would better describe the particle suspension behavior of the fracturing fluids. This fluid property cannot be identified with the fracture simulators available today because these models utilize viscosity-based correlations to describe proppant settling. The new fluid property may be identified with an experimental investigation of the proppant transport characteristics of fracturing fluids.

Several studies have shown that fracturing fluids exhibit not just viscous properties but elastic characteristics also. Hence, these fluids should also be evaluated for their elastic properties. The difference in the suspension characteristics of the two gels shown in Figures 4.6 and 4.7 might be better described with their elastic properties.

One method to estimate the elastic properties of a fluid is through oscillatory measurements. These measurements are made in linear viscoelastic limit. Therefore, the first task in characterizing the fracturing fluid for their elasticity was to identify the linear viscoelastic limit. This identification would further require selection of an appropriate fracturing fluid for evaluation. The most common fracturing fluid used today is guar based gels, so the guar solution was selected as the base polymer.

The selection of the guar concentration is described in Section 4.5. This concentration was then used to establish the linear viscoelastic limit for the oscillatory measurements. Section 4.6 describes the procedure for establishing the linear viscoelastic limit.

4.5 LINEAR GUAR SOLUTIONS PROPERTIES

The identification of the guar concentration required preparing and evaluating guar solutions at concentrations from 1 to 100 lb/Mgal. These solutions were characterized in the laboratory for their viscosities and suspension settling properties.

The viscosity of the guar solutions prepared at concentrations less than 10 lb/Mgal were measured with the Cannon-Fenske capillary viscometer and with the Bohlin rheometer, and those at higher concentrations were measured with the Bohlin rheometer only. This selection was discussed in Section 3.3.2.

The suspension characteristics of the guar solutions were evaluated in a 1000 ml capacity glass cylinder by testing the fluid's ability to suspend 2 ppg slurry prepared with 20/40 mesh Brady sand.

Molecular Weight of Guar. The viscosity values of the guar solutions measured with the capillary viscometer are given in **Table 4.10**. The viscosities of the solutions measured with the Bohlin rheometer are shown in **Figure 4.8**. The viscosities in the Newtonian plateau region of the curves shown in Figure 4.8 are also given in Table 4.10. The table and the figure show an increase in guar viscosities with an increase in the polymer concentration. The viscosities of the guar solutions exhibit a typical non-

Newtonian behavior, with Newtonian plateau at low shear rates and shear-thinning characteristics at higher shear rates. The transition from the Newtonian plateau to the shear-thinning region changed as the polymer concentration was increased. The transition moved to lower shear rates at higher guar concentrations.

Table 4.10-Low Shear Viscosity of Linear Guar Solutions

Guar Concentration [lb/Mgal]	Viscosity [cp] from Capillary Viscometer	Zero-Shear Viscosity [cp] from Bohlin Rheometer	Static Settling Velocity [cm/min]
0	1.0927	0.551	420
1	1.3087	0.696	336
2	1.58172	0.812	280
3	1.7177	0.977	240
4	2.081	1.739	210
5	2.31	1.586	187
6	2.72	2.615	153
7	3.062	2.946	153
10	4.634	7.082	140
12	6.61	10.45	129
15	10.35	19.94	112
16	-	20.06	105
18	-	36.98	105
20	-	53.15	70
22	-	45.02	51
25	-	88.89	48
30	-	187.2	37
35	-	451.5	9
40	-	754.9	7
50	-	2143	2
70	-	8337	0.27
80	-	15989	0.19
100	-	42482	0.15

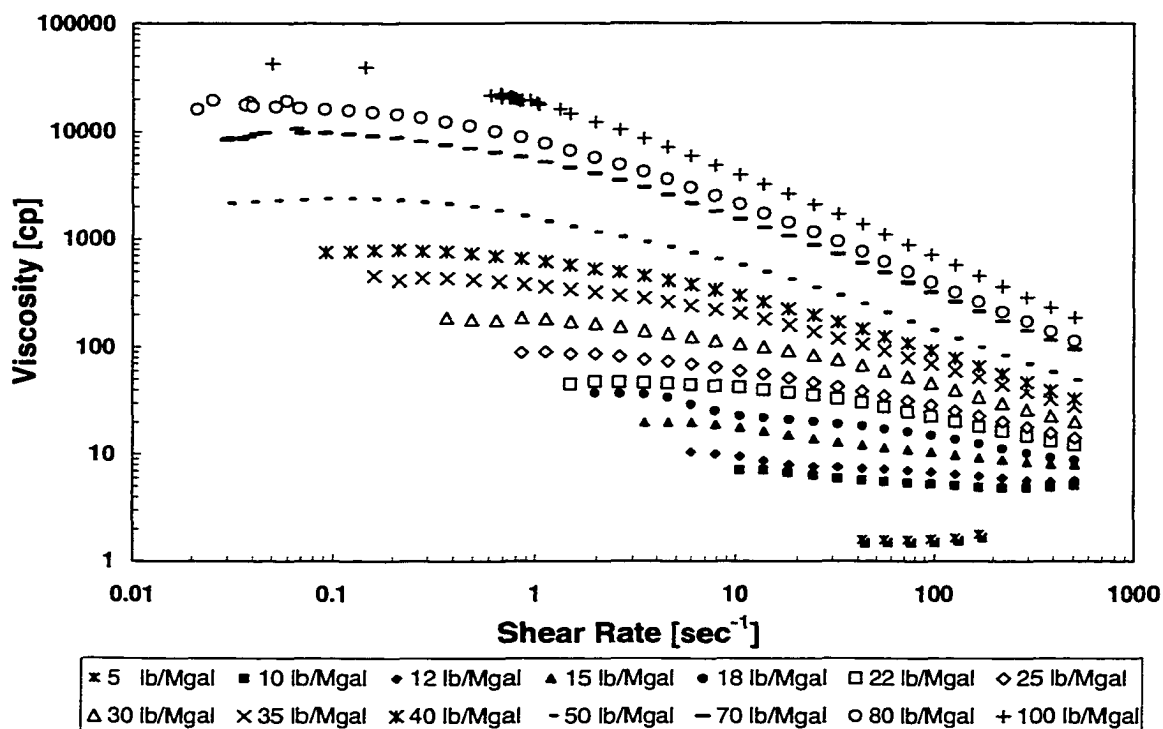


Figure 4.8—Viscosity-Shear Rate Curves for Different Guar Concentrations

Equations 2.12 and 2.16 showed that the calculation of molecular weight of the guar polymer required calculation of its intrinsic viscosity, which required a plot of the relative viscosity as a function of polymer concentration.

The plot of the relative viscosity of the guar solutions prepared at concentrations less than 10 lb/Mgal is shown in **Figure 4.9**. The viscosity values were fitted to the Huggins equation, given by Eq. 2.13, and to the Kramer's equation, Eq. 2.14, and the parameters for the two equations were determined. The data fitted the Huggins equation as

$$\frac{\mu - \mu_s}{\mu_s c_P} = [\mu] + k' [\mu]^2 c_P = 1.49 + 0.8718 c_P \dots\dots\dots (4.5)$$

and the Kramer's equation as

$$\frac{\ln\left(\frac{\mu}{\mu_s}\right)}{c_P} = [\mu] + k''[\mu]^2 c_P = 1.4958 - 0.2959 c_P \dots\dots\dots (4.6)$$

The same viscosity data when fitted to a third-order polynomial yielded

$$\frac{\mu - \mu_s}{\mu_s c_P} = 1.49 + 1.146 c_P - 1.2734 c_P^2 + 0.9806 c_P^3 \dots\dots\dots (4.7)$$

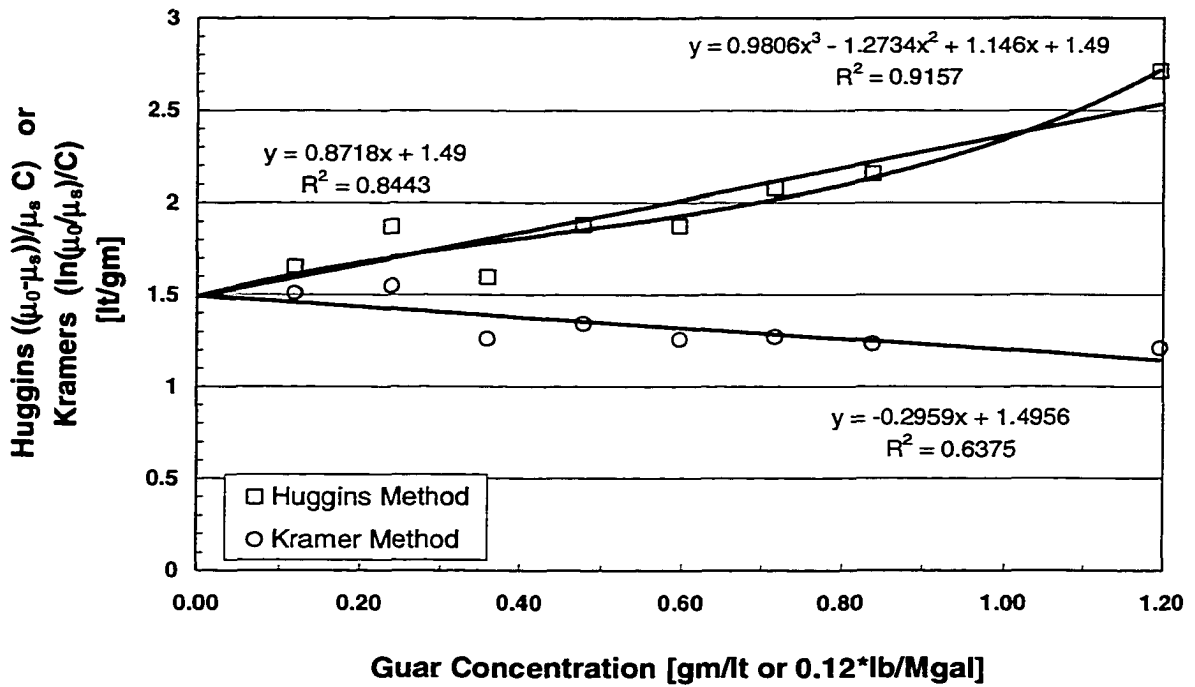


Figure 4.9—Relative Viscosity versus Polymer Concentration of Guar Solutions

Equations 4.5 through 4.7 show the intrinsic viscosity, $[\mu]$, to be equal to 1.49 l/gm or 14.9 dl/gm. From these equations, the constant k' for the Huggins equation was found to be 0.3927, and k'' for the Kramer equation was -0.1323 . The difference between these two constants, $k' - k''$, is 0.52, which is close to 0.5, a value shown in Eq. 2.15 confirms the accuracy of the intrinsic viscosity calculated from the viscosity data.

The intrinsic viscosity was then used to calculate viscosity average molecular weight, M_v , of the guar polymer used in the present study. Equations 2.17 and 2.18 were used for this calculation. Equation 2.17 yielded the molecular weight to be 2.26×10^6 Kg/Kg-mol, and Eq. 2.18 yielded it to be 2.6×10^6 Kg/Kg-mol.

The calculated intrinsic viscosity and molecular weight of the guar were compared with those available in the literature. The values are shown in Table 4.11. The table shows the calculated values agreed with the published data.

Table 4.11-Comparison of the Molecular Properties of Guar Gum

Solvent	$[\mu]$ dlt/gm	Molecular weight 10^{-6} kg per kg mol	Method	c_p^* lb/Mgal	Additional data	Ref.
Water	12.6	2.2	Size exclusion chromatography	-	-	58
Deionized water	4.5 to 12.5	0.44 to 1.65	Light scattering	-	At $c < c^*$ $\mu \propto c^{1.3}$; and at $c > c^*$ $\mu \propto c^{5.1}$	60
Water	2.3 to 11.7	0.38 to 2.0	Light scattering and viscosity	9.2	At $c < c^*$ $\mu \propto c^{1.17}$; and at $c > c^*$ $\mu \propto c^{4.1}$	65
Distilled water	7.86	1.0	Viscometer	-	-	79
Deionized water	16.0	4.5	Light scattering	8.33	At $c < c^*$ $\mu \propto c^{1.1}$; and at $c > c^*$ $\mu \propto c^{4.0}$	123
Deionized water	16.8	2.6	Viscometer	7	-	207
Norman City tap water	14.9	2.26	Viscometer	10.4 to 12.5	At $c < c^*$ $\mu \propto c^{1.09}$; and at $c > c^*$ $\mu \propto c^{4.1}$	This work

Normalized Viscosity Curve for Guar. The viscosity curves at different concentrations can often be reduced to a single curve by normalizing the shear rate with a characteristic time constant for each concentration of the guar solution.¹⁵⁴ The time constant is defined as $\frac{(\mu_o - \mu_s)M_v}{c_p RT}$ where μ_s is the viscosity of solvent, here water. This time constant was calculated for each guar concentration, c_p , analyzed in the study and a normalized curve was obtained. The curve is shown in Figure 4.10. The normalized viscosity curve fitted a modified Carreau model as

$$\frac{\mu}{\mu_o} = \left(1 + \left[1.4 \frac{\dot{\gamma}(\mu_o - \mu_s)M_v}{c_p RT} \right]^2 \right)^{-0.34} \dots\dots\dots (4.8)$$

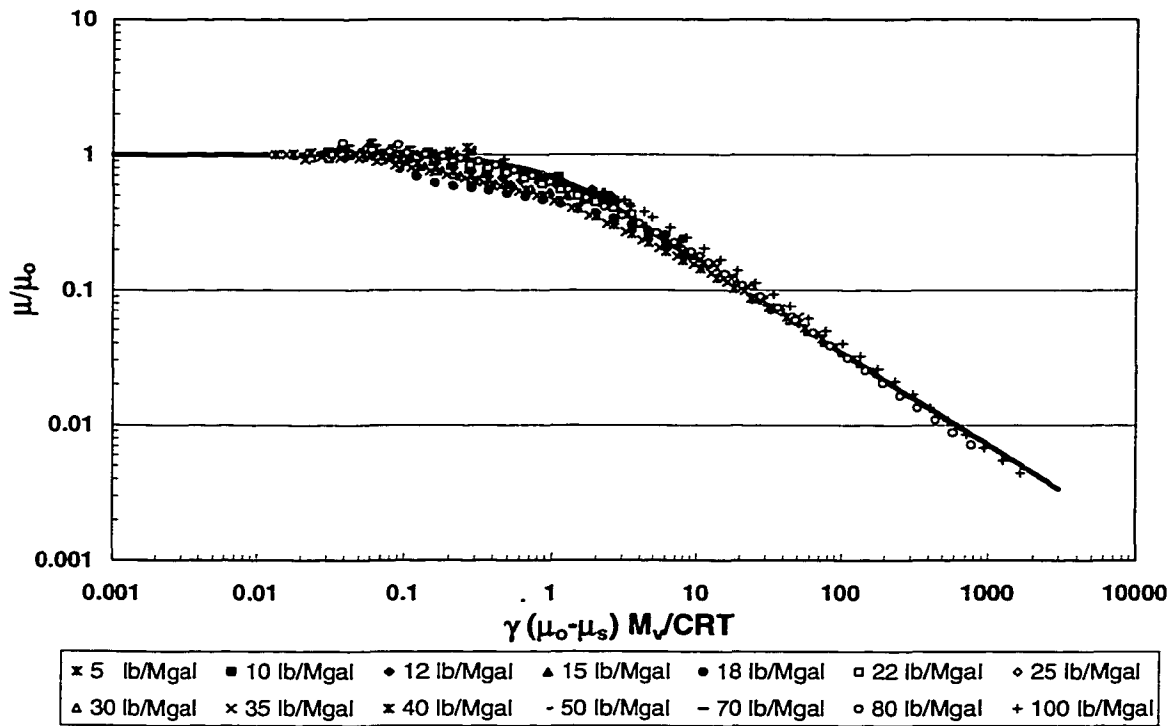


Figure 4.10—Normalized Viscosity Curve for all Guar Concentrations

Overlap Concentration of Guar. The viscosity of the guar solutions can also be used to determine the overlap concentration of the polymer.

The significance of the overlap concentration c_p^* is well known. At concentrations below c_p^* , the polymer chains are in dilute solution with little interaction among the individual chains. At concentrations above c_p^* , the polymer chains begin to overlap with each other and entanglements among chains become important. This overlap influences subsequent crosslinking, as chains are more likely to come close to each other and establish interchain crosslinks when a crosslinker solution is added to the polymer.

The viscosity values in the Newtonian plateau region of the curves were used to determine the overlap concentration of the guar used in the present work. These values are plotted in Figure 4.11 as a function of the polymer concentration. The figure shows a typical viscosity-concentration plot of a polymeric fluid. As the guar concentration was increased, the fluid viscosity increased. The viscosity increase could be separated into two distinguishable regions. At low guar concentrations, the viscosity increase was small, and the viscosity-concentration curve had a slope of 1.096. Whereas at higher concentrations, the viscosity increase was larger with a slope of 4.12. These two curves intersected at c_p^* . The overlap concentration was 12.33 lb/Mgal when the viscosity values obtained with the Bohlin rheometer were used. This concentration was 10.45 lb/Mgal when the capillary viscometer measured values were used. These two concentrations compare well with the published data as shown in Table 4.11.

Figure 4.11 shows the viscosity of a polymer solution is a function of the polymer concentration. As the polymer concentration is increased beyond the overlap

concentration, the entanglement couplings between the polymer chains cause a large increase in viscosity with a small increase in the polymer concentration. The influence of entanglements on the fluid viscosity implies that they would also influence other macroscopic properties of the guar polymer. Analogous to the overlap concentration, there should be a critical concentration beyond which there would be a large decrease in the particle settling characteristics for a small increase in the polymer concentration.

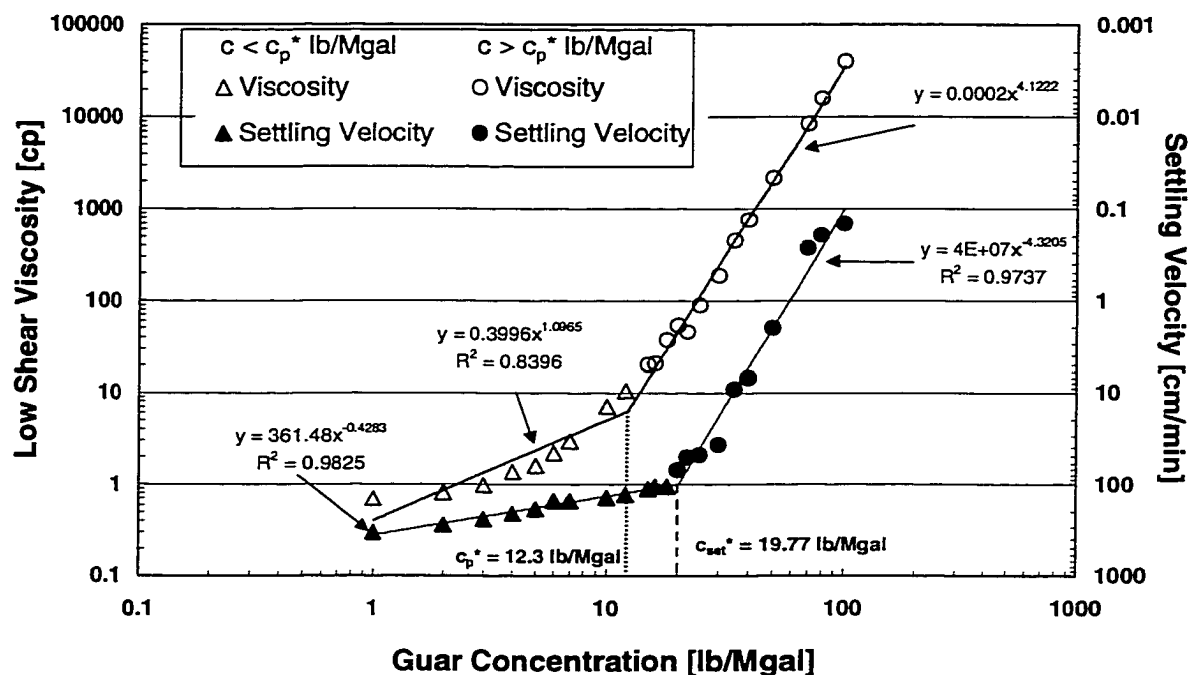


Figure 4.11—Comparison of Low Shear Viscosity with Settling Velocity of 2 ppg Slurry

Therefore, the settling velocity of 2 ppg slurry prepared in different guar solutions were determined. The settling velocities are shown in Table 4.10, and plotted against the polymer concentration in Figure 4.11. The figure shows the settling velocity decreased with an increase in the guar concentration. The plot displays two distinguishable regions

in the settling velocity data, as was seen in the viscosity-concentration plot. At lower polymer concentrations, the slope of the settling velocity curve was -0.428 , and at higher concentrations, the slope was -4.32 . These two regions intersected at a critical concentration for settling c_{set}^* , 19.75 lb/Mgal. This concentration was much higher than the overlap concentration c_p^* calculated from the low shear viscosity values.

The difference in the critical concentrations, c_p^* and c_{set}^* , could be because the shear rates around the settling sand particles were not in the zero-shear rate region for all guar concentrations. The agreement between these two concentrations might be improved if the guar viscosities were calculated at the shear rates of the suspended particles.

The characteristic shear rate of the suspension settling is defined as,⁵⁵

$$\dot{\gamma} = \frac{u_{suspension}}{d_p(1-\chi)} \dots\dots\dots (4.9)$$

where $u_{suspension}$ is the maximum settling velocity of the slurry of volume fraction χ and of mean particle diameter d_p . Equation 4.9 was used to calculate the shear rate from the settling velocity of suspension in each guar solution. The shear rate was then used to determine the guar viscosities at the settling conditions in the glass cylinder. These viscosity values were plotted along with the settling velocities against the guar concentration in Figure 4.12. This figure gives a critical guar concentration c_{crit}^* of 18.75 lb/Mgal, which is much higher than the overlap concentration, and closer to the critical settling concentration. Figure 4.12 shows that the critical settling concentration in the linear guar solutions could be closely estimated from the viscosity calculated at the suspension settling shear rates.

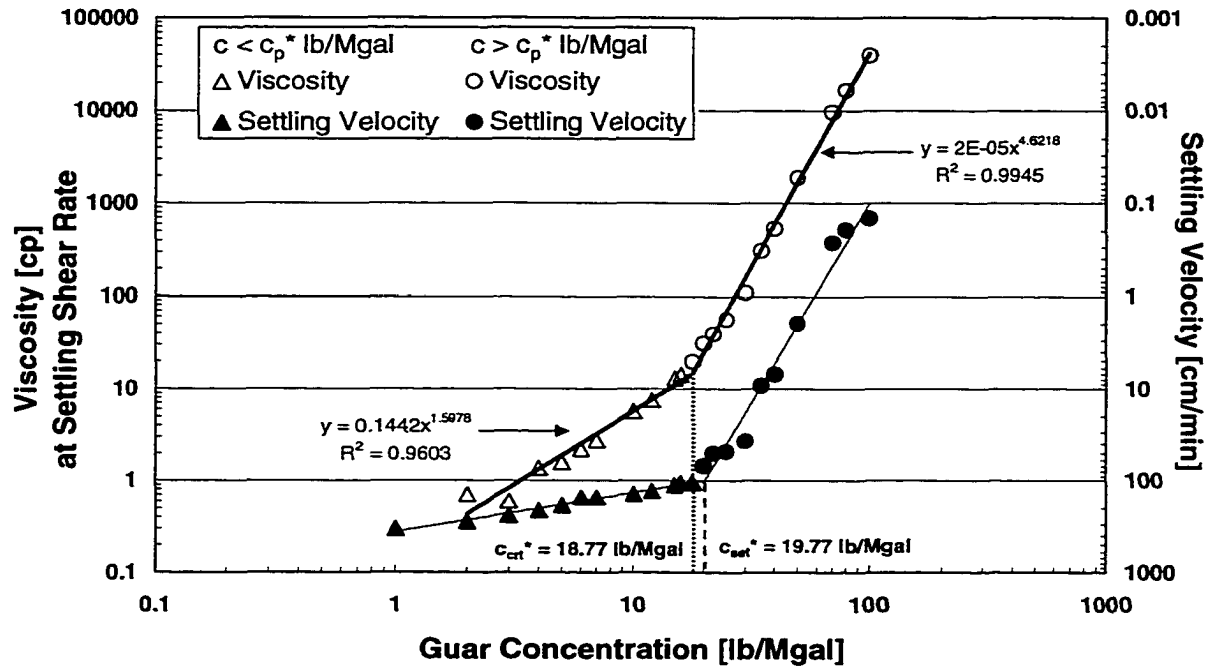


Figure 4.12—Comparison of Viscosity at Suspension Shear Rate with Settling Velocity of 2 ppg Slurry

The good correlation between the c_{crit}^* and c_{set}^* values for the guar solutions does not mean that the viscosity is also a good predictor of settling rates in crosslinked guar. Since the characteristic times of breaking and reforming of the bonds in orate-crosslinked are probably not similar to those of entanglements in the linear guar, the suspension characteristics of the crosslinked and non-crosslinked guar would not be similar. Moreover, the guar solutions exhibit low elasticity, whereas crosslinked guar gels are highly elastic. And as the elasticity is known to affect particle settling in viscoelastic fluids, it would significantly influence the particle settling in crosslinked gels. Therefore, it would be necessary to characterize crosslinked gels for their elastic characteristics to understand their proppant transport behavior.

Selection of Guar Concentration. Figure 4.11 showed that the overlap concentration of the guar solution was 12.33 lb/Mgal. Above this concentration, the guar polymer chains can establish crosslink bonds across the chains. Figure 4.12 showed that the critical settling concentration of the guar solution was 19.77 lb/Mgal. Above this concentration, the guar solution had better suspension characteristics. Therefore, to study particle suspension in crosslinked guar gels, it would be better to prepare the gels in guar having concentrations higher than these two critical concentrations.

A guar concentration of 35 lb/Mgal, twice the critical concentrations, was therefore selected to prepare crosslinked guar gels for studying the proppant transport behavior. This concentration was also one of the guar loading widely used today in the hydraulic fracturing treatments of the reservoirs at 225°F.^{10,82}

4.6 ESTABLISHING THE LINEAR VISCOELASTIC LIMIT

The elastic properties of a crosslinked polymer are more than those of a non-crosslinked polymer, both prepared at similar guar concentration. The linear viscoelastic limit identified with a non-crosslinked guar solution would be applicable for all crosslinked gels prepared using that guar concentration. Since a guar concentration of 35 lb/Mgal was selected to prepare crosslinked guar gels, the linear viscoelastic limit was identified with this concentration only.

The linear viscoelastic limit was determined using the stress sweep test on the Bohlin rheometer. This test was performed at four frequencies, 0.01, 0.1, 1.0 and 10 Hz, to cover the frequency range where the oscillatory measured were made. The results of the test are shown in Figure 4.13. The figure shows the stress was linearly dependent on

the strain. This linear dependence is maintained in the measurements of all frequencies at a strain of 0.05; if this strain were maintained during the oscillatory measurements, the viscoelastic properties of all crosslinked gels would be within the linear viscoelastic limit.

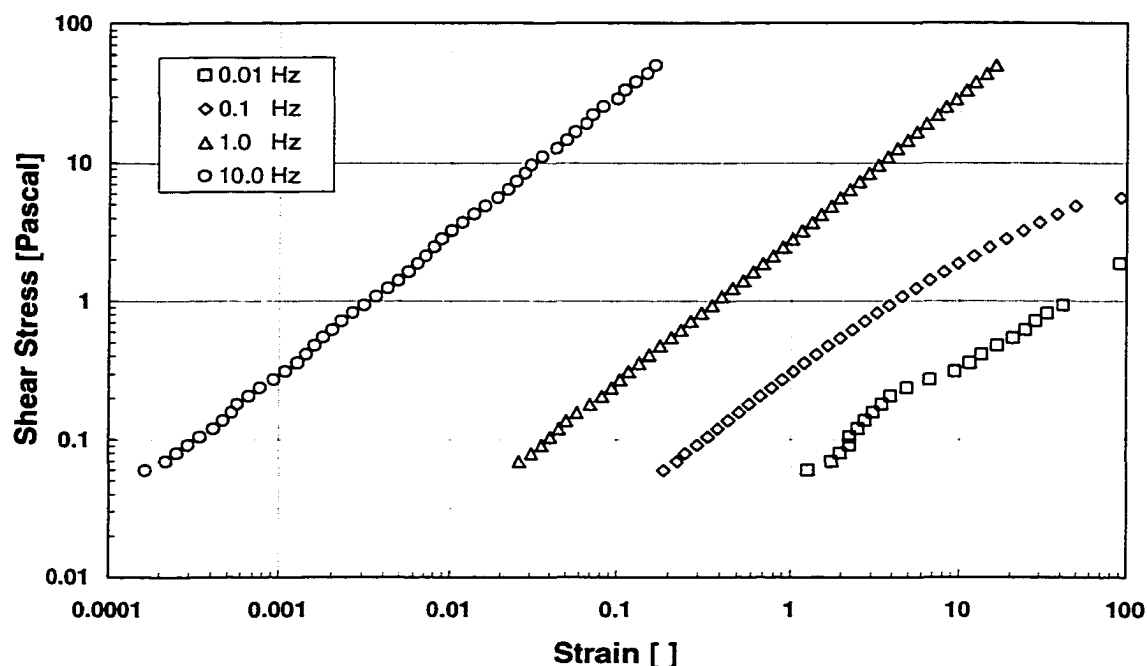


Figure 4.13—Evaluation of Linear Viscoelastic Limit of 35 lb/Mgal Guar, 75°F

The Bohlin rheometer used air bearing to minimize friction losses during the transmission of torque from its motor to the measuring geometry. This bearing imposes a lower limit on the shear stress that could be applied on the sample. The lower limit of the instrument used in the present research was 0.06 Pascal. At this stress limit, Figure 4.13 shows that the strain values could be higher than 0.05 at some frequencies during the oscillatory measurements. The figure suggests that the viscoelastic measurements at strains corresponding to 0.06 Pa stress would still be in the linear viscoelastic regime. Hence, all viscoelastic measurements were made by setting the strain to 0.05 and activating the 'Auto Stress' function on the Bohlin rheometer.

CHAPTER 5

UNDERSTANDING RELATIONSHIP BETWEEN FLUID AND ITS PROPPANT TRANSPORT CHARACTERISTICS

5.1 INTRODUCTION

Since their introduction in 1950s, the fracturing fluids have been continuously improved from simple oils to sophisticated water-based polymer gels.²⁰ During these years, numerous patent applications have been filed describing the unique chemistry used to prepare these fluids. These unique fluids have been subsequently used around the world.^{81,82} Though several fluid formulations have been developed over the years, the method to evaluate the fluid for its capability to suspend and transport proppant has remained unchanged.^{82,126,135} Currently, viscosity is used as a parameter to evaluate fracturing fluid for its sand suspension characteristics. An established criterion for a fluid's capability to transport proppant is that the fluid should have a minimum viscosity of 100 cp at a shear rate of 100 sec^{-1} over a three hour test performed at a desired temperature.^{82,126,135} This viscosity is measured using the API procedure described in Section 2.2.2.¹⁴⁵

Section 4.4 described that the crosslinked gels having similar viscosities had dissimilar proppant transport behavior. This observation suggests a need for more comprehensive evaluation of crosslinked gels to understand the discrepancies in using viscosity. This need requires a fundamental understanding of the relationship between the microscopic structure of crosslinked guar gels and their proppant transport behavior. The fundamental understanding would help to comprehend the relationship between a

fluid and its suspension characteristics, and then to identify a fluid property other than viscosity that would describe the fluid performance for fracturing treatments.

Thus, borate-crosslinked guar gels were formulated by varying the crosslinker concentration while keeping all other fluid parameters constant. The gels were evaluated for their capabilities to transport 2 ppg slurry through the transparent slot and the HPS models. All gels were then rheologically characterized using oscillatory measurements and imaged using Atomic Force Microscope (AFM) to understand the influence of microscopic structure of the crosslinked gels on their macroscopic performance in the slot models.

One unique aspect of this characterization is the use of AFM imaging to understand microscopic structures formed in the crosslinked gel. Several imaging techniques have been used earlier to understand polymer gel structures,²⁰⁸ but rarely to better understand those of the crosslinked guar gels,^{115,209} and never to comprehend the suspension characteristics of a borate-crosslinked guar gel. Thus, by including AFM characterization, this study uses a fundamental approach to correlate fluid behavior with its suspension characteristics.

5.2 EXPERIMENTAL SECTION

Sample Preparation. The guar solutions were prepared at pH 11, using the procedure described in Chapter 3. The prepared guar solutions were transferred to one liter Waring blender for mixing and shear conditioning the gel. Then, the crosslinker solution was added at a concentration of 0.0, 0.25, 0.45, 1.0, and 3.0 lb/Mgal of the guar solution to prepare five crosslinked gel samples. These samples were then characterized

at ambient temperature in the laboratory for their rheology and AFM imaging, and in the field scale test facility for their sand transport behavior.

For the rheological characterization and AFM imaging, the borate solution was injected into one liter of the guar solution while it was mixed and sheared in a blender. The guar gel thus prepared was further sheared for an additional three minutes to shear-precondition it prior to its characterization.¹⁴⁵ The sample was then loaded immediately on to a viscometer for API-recommended viscosity tests, or to a rheometer for oscillatory measurements. For the AFM measurements, the gel sample was sheared further for few more seconds beyond the shear-conditioning period. Then, a freshly cleaved natural muscovite mica (V-4 grade, Asheville-Schoonmaker Mica, Newport News, VA) was dipped into the gel to transfer the sample onto the mica surface. The mica was then cleaned around the edges to remove any excess gel, transferred to a closed chamber containing a desiccant, and left to dry. All crosslinked gel samples were prepared at the same time and dried in the closed chamber for two days before making the AFM measurements.

The crosslinked gels were directly used in the AFM characterization and were neither diluted nor imaged under alcohol as had been done earlier to image polysaccharides.²¹⁰ The present approach was taken because the earlier studies had used non-crosslinked gels whose structures were probably not influenced by the presence of water and alcohol. Whereas in this study, borate-crosslinked gel was used, and it can be affected by dilution through changes in the gel pH. Since the fluid pH strongly influences the crosslinking chemistry of a borate-crosslinked gel, any change in pH

would affect the gel structure and alter its characteristics. Therefore, the gel samples were not interacted with any other chemicals after formulating them in the blender.

Atomic Force Microscopy Measurements. AFM images were acquired with a NanoScope III MultiMode Scanning Probe Microscope (Digital Instruments, Santa Barbara, CA), which was capable of mapping sample surface in three dimensions.²¹¹ The instrument was operated in contact mode as well as TappingMode. Both these modes have been successfully used earlier to image polysaccharide samples.^{115,210,212} In the present study, the images obtained with the contact mode are only shown. The samples were imaged with a sharpened triangular silicon nitride probe having force constant in the range from 0.01 to 0.6 N/m and a nominal tip radius between 5 and 40 nm.

The crosslinked gels were first imaged with an optical microscope (scale 900 microns) to observe global features of the sample. Then they were imaged with the scanning probe microscope to capture detailed surface features not visible under the optical microscope.

API Viscosity Tests. The viscosity tests were performed on the Nordman Viscometer using the API test procedure described in Chapter 3. The API viscosity test was performed for three hours at a shear rate of 100 sec^{-1} . During the single shear rate test, a shear rate ramp of 100, 75, 50, 25, 50, 75 and 100 sec^{-1} was performed for a short duration every thirty minutes.¹⁴⁵ The shear rate ramp was used by the instrument to report the test results as power law model parameters, from which the gel viscosity at a

shear rate of 100 sec^{-1} was calculated as a function of time.^{145,160} These calculated values are presented here.

Oscillatory Measurements. The viscoelastic measurements were made on the Bohlin rheometer using the cone-and-plate geometry with a cone angle of 4° and diameter of 40 mm. These measurements were made at a shear strain of 0.05, which was in the linear viscoelastic regime of the fluid.

Proppant Transport/Suspension Tests. The proppant transport tests were performed using the large scale test setup shown in Figure 3.1. These tests were performed using the procedure described in Section 3.3.

The proppant transport tests were performed with 2 ppg slurry prepared in borate-crosslinked guar gels at pH 11. The slurry was pumped at 40 gpm through the coiled tubing to simulate a shear-conditioning rate of 1000 sec^{-1} . Then, the slurry was diverted at 10 gpm alternately through the transparent slot and the HPS, and the balance fluid was pumped off to a disposal tank. After pumping through the slot, the gel was disposed off into a sump tank.

5.3 RESULTS AND DISCUSSION

API Viscosity Tests. Figure 5.1 shows the apparent viscosity at a shear rate of 100 sec^{-1} for the five crosslinked gel samples. The figure shows the viscosities as average of the values obtained from different test runs. It also shows error bars, which are drawn to describe the range of the viscosity measured during the various runs.

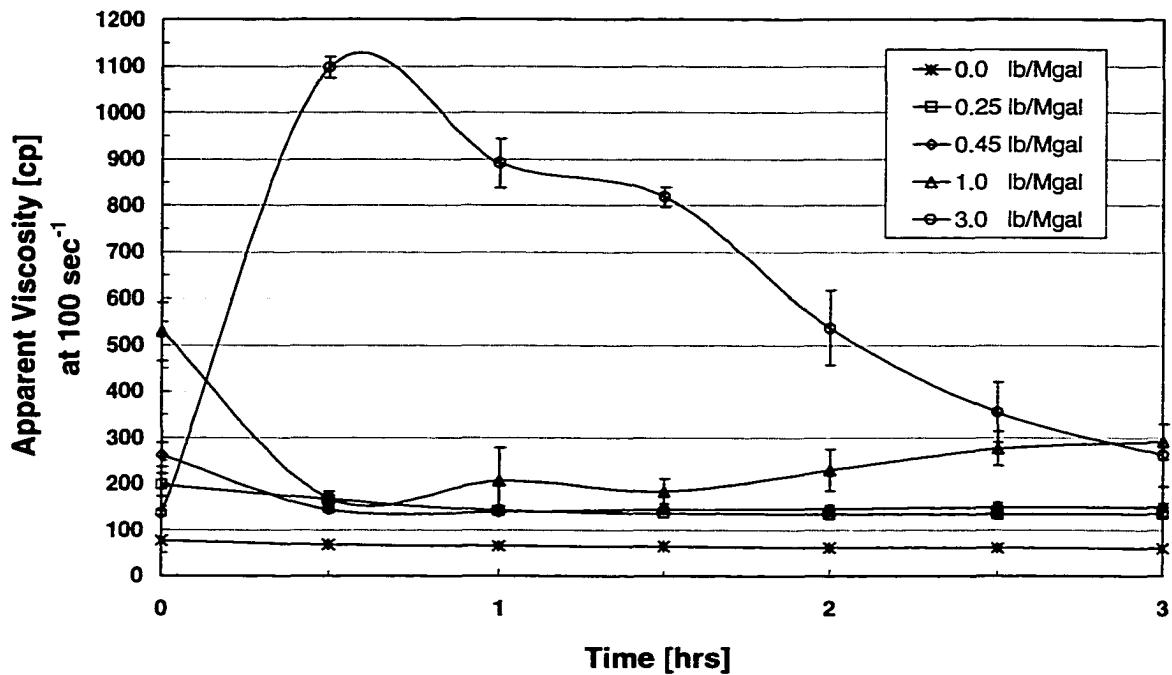


Figure 5.1—API Viscosity Values as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 11 (35 lb/Mgal Guar, 75°F)

Figure 5.1 shows the guar gel prepared without crosslinker solution had an initial viscosity of 77 cp and 60 cp at the end of the three-hour test. The guar solution thus had lower viscosity than 100 cp. Its viscosity increased beyond 100 cp when the crosslinker solution was added to it. At 0.25 lb/Mgal crosslinker concentration, the initial viscosity of the crosslinked gel was close to 200 cp, which after three hours reduced to 135 cp because of the shear degradation of the gel. Similarly at 0.45 lb/Mgal, the initial viscosity was 260 cp, which stabilized after three hours to 150 cp, which was close to that of 0.25 lb/Mgal crosslinked gel. This trend of increase in the gel viscosity with the crosslinker concentration was continued when the crosslinker concentration was increased to 1 lb/Mgal. At this concentration, it can be seen that the gel had higher initial

as well as three-hour viscosities than those of the previous three gels. The viscosity of 1 lb/Mgal gel, however, did not stabilize in three hours as it did in the other two gels. This could be because of a labile nature of the crosslinking bonds in these gels. The crosslinked bonds between the monoborate ions in the crosslinker solution and cis-hydroxyl groups on the guar polymer break and reform continuously with the applied shear.^{84,109,213} Also, the lifetime of their interaction is only one millisecond.⁸⁵ So the structure and viscosity of the crosslinked guar gels change with both the shear rate and time. An equilibrium was established between the crosslink bond breaking and reforming during the three hour evaluation of 0.25 and 0.45 lb/Mgal gel, but not that of the 1 lb/Mgal gel. So the observed behavior in Fig. 5.1.

A similar change is observed in the viscosity of the gel prepared at 3 lb/Mgal, except that this gel had a low initial viscosity. The low viscosity could be because of a very high crosslinker concentration that over-crosslinked this gel.¹⁷ When this gel was sheared in the high shear environment of the blender, it broke down into small gel domains, of poor gel strength and low initial viscosity. But when transferred to the low shear conditions of the viscometer, these domains realigned,¹¹⁵ and crosslinked back together, thereby increasing the gel viscosity. As the gel was sheared further in the viscometer, it began to shear degrade and as a result, its viscosity began to decrease with time. Besides decreasing the fluid viscosity, the high crosslinker concentration in this gel caused it to phase separate. The gel, when kept unsheared for 24 hrs, separated into two phases: a crosslinked polymer mass and a water phase containing almost negligible guar polymer. This phase separation of the gel indicates that a fully crosslinked guar gel was formed at 3 lb/Mgal crosslinker concentration. A similar phase separation was not

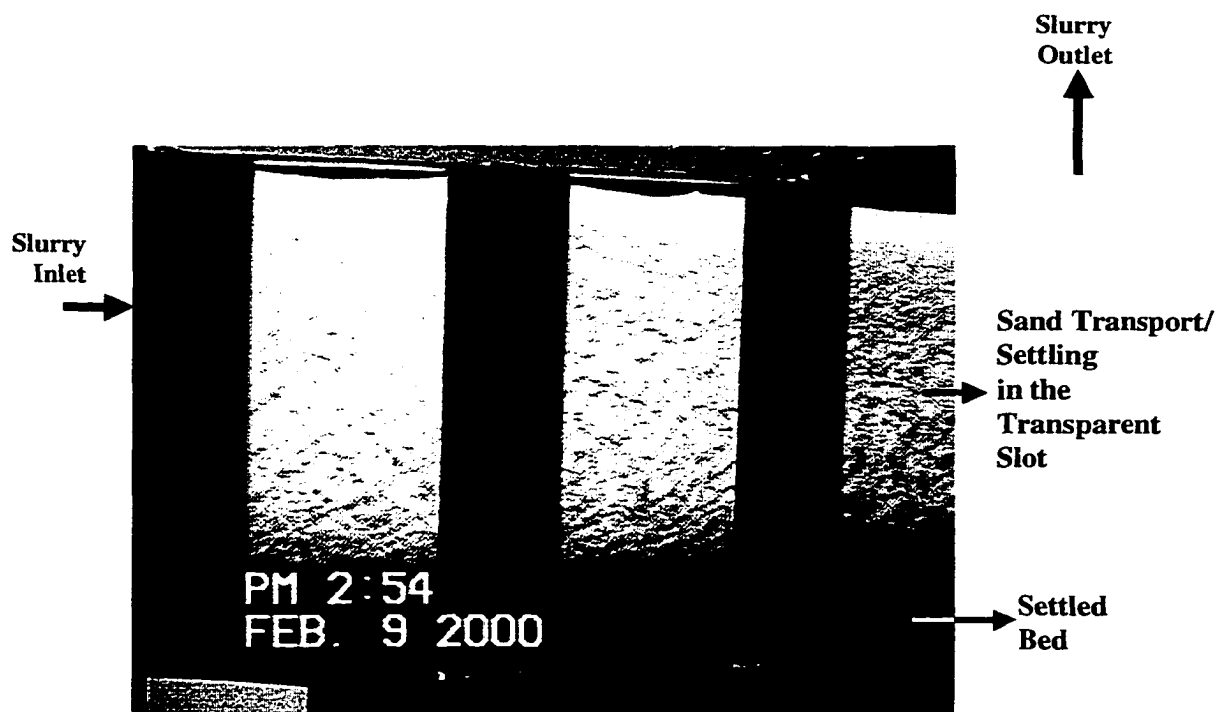
observed in any other crosslinked gel prepared in this study indicating that the other gels were not completely crosslinked.

Yet another observation is worth mentioning here. The borate-crosslinked guar gel exhibits normal stresses, so it climbs the viscometer shaft when sheared in a rotational viscometer. After the API viscosity tests, a small gel volume was observed resting on the top of the bob. Since a fixed quantity of the gel sample was loaded in the viscometer, the gel that climbed up the bob came from the annulus between the bob and the cup. Because of the gel climbing, an insufficient volume of gel would have remained in the annulus, and so the viscometer measured a lower viscosity. This reduction in the gel viscosity could not be correctly identified. However, as the measured value was less than the actual viscosity of the crosslinked gel, it would not affect the minimum viscosity criterion used for the solids suspension in the fluid.

Figure 5.1 shows that except for the non-crosslinked gel, all other gels had viscosities greater than 100 cp during the three-hour test. Since these gels had higher viscosities than the minimum reported for satisfactory proppant transport,^{82,135} they should be able to suspend and transport sand through the slot.

Proppant Transport/Suspension Tests. Figure 5.2 depicts one frame of the video images obtained when a 2 ppg slurry of 0.25 lb/Mgal borate-crosslinked guar gel was pumped through the slot. A settled bed at the slot bottom is evident in the images. The bed formed as soon as the sand slurry was introduced into the slot. The formation of the settled bed implied that the gel was unable to keep particles in suspension and that the proppant transport through the slot was inadequate. This undesirable settling displays an

unsatisfactory performance of the 0.25 lb/Mgal crosslinked guar gel as a carrier fluid. This behavior was observed despite the gel having higher viscosity than the minimum required for satisfactory proppant transport. A similar behavior was observed for slurry flow through the HPS as shown in Figure 5.3.



**Figure 5.2—2 ppg Proppant Slurry Transport through Transparent Slot with
Borate-Crosslinked Guar, 0.25 lb/Mgal Crosslinker,
pH 11 (35 lb/Mgal Guar, 75°F)**

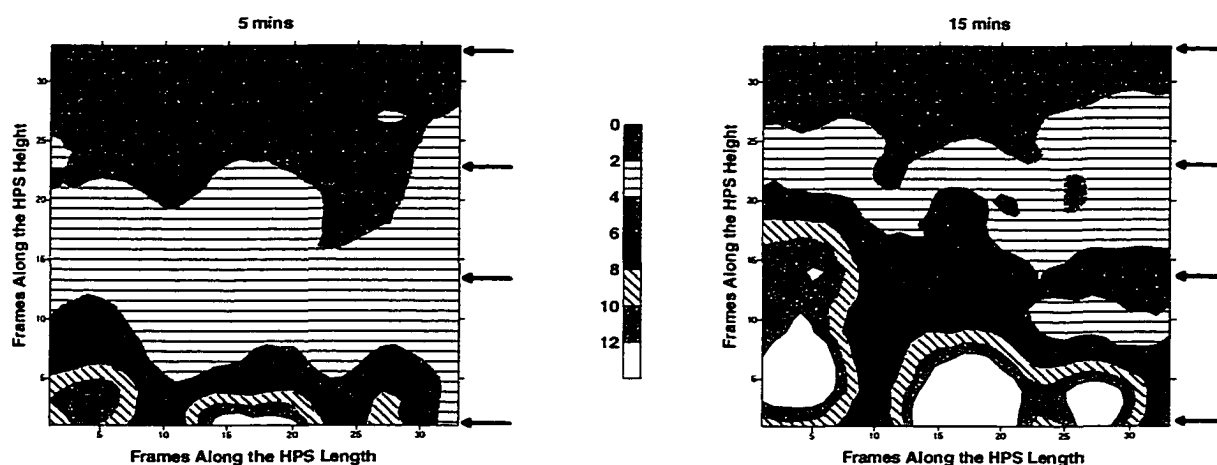
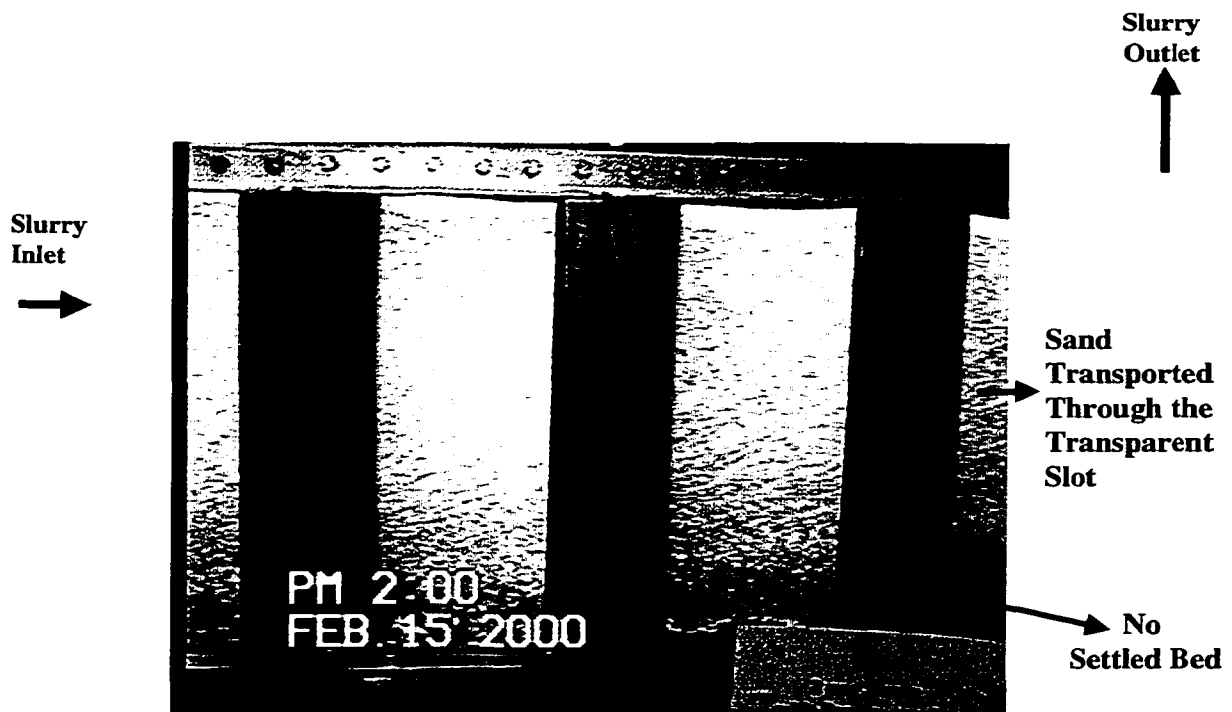


Figure 5.3—Proppant Concentration (ppg) in the HPS after 5 and 15 min of 2 ppg Slurry Transport with Borate-Crosslinked Guar, 0.25 lb/Mgal Crosslinker, pH 11 (35 lb/Mgal Guar, 75°F)

The proppant transport capability of the crosslinked gel can be improved if the crosslinker concentration were increased. Therefore, another test was performed where the crosslinker concentration was raised to 0.5 lb/Mgal, twice that of the previous test. At the new crosslinker concentration, the sand was observed uniformly distributed throughout the slot. Since the gel prepared at 0.5 lb/Mgal exhibited satisfactory performance, the crosslinker concentration was reduced to 0.45 lb/Mgal and the slurry flow was continued through the slot. At this crosslinker concentration also, the crosslinked gel was able to transport sand particles through the slot with no settled bed formed. This is evident from the sand distribution images shown in Figure 5.4. Because the 0.45 lb/Mgal crosslinked gel transported sand through the slot, the crosslinker concentration was further reduced to 0.4 lb/Mgal to identify the lowest crosslinker concentration that would carry sand particles through the model. In the 0.4 lb/Mgal crosslinked gel, the sand particles began to separate from the gel displaying that this gel

was losing its ability to transport 2 ppg slurry through the slot. The diminishing capability of this gel indicated that 0.45 lb/Mgal was the minimum acceptable crosslinker concentration in this borate-crosslinked guar gel at pH 11 to satisfactorily transport 2 ppg slurry through the slot. The proppant transport behavior through the HPS was also satisfactory at 0.45 lb/Mgal crosslinker concentration. The behavior is shown in Figure 5.5.



**Figure 5.4—2 ppg Proppant Slurry Transport through Transparent Slot with
Borate-Crosslinked Guar, 0.45 lb/Mgal Crosslinker,
pH 11 (35 lb/Mgal Guar, 75°F)**

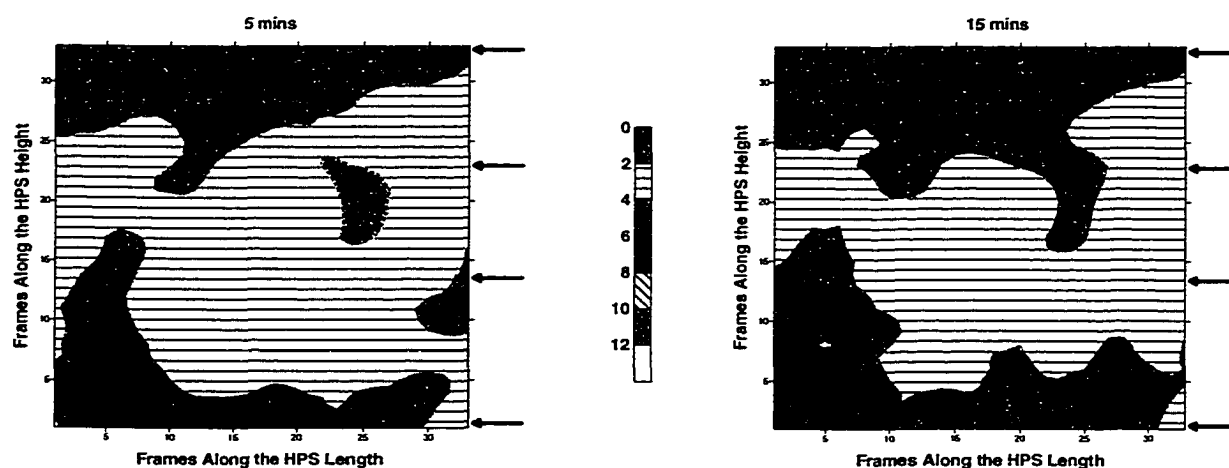


Figure 5.5—Proppant Concentration (ppg) in the HPS after 5 and 15 min of 2 ppg Slurry Transport with Borate-Crosslinked Guar, 0.45 lb/Mgal Crosslinker, pH 11 (35 lb/Mgal Guar, 75°F)

Figure 5.1 shows that the viscosity of the 0.45 lb/Mgal crosslinked gel was higher than 100 cp at 100 sec^{-1} , which was also the case for the gel at 0.25 lb/Mgal. These two gels had similar viscosities but dissimilar suspension capabilities. The 0.45 lb/Mgal gel satisfactorily transported sand particles through the slot whereas the 0.25 lb/Mgal gel did not. The measured viscosity was able to correctly predict the suspension capability of the 0.45 lb/Mgal gel but not that of the 0.25 lb/Mgal gel. The viscosity criterion was therefore partially successful in predicting the performance of the carrier fluid. This implied that the currently used viscosity criterion is not completely reliable and is insufficient for characterizing suspension properties of the crosslinked gels.

Since the 0.25 lb/Mgal crosslinked gel could not satisfactorily transport proppant through the slot, the gel prepared without crosslinker, i.e. guar polymer solution, would also perform unsatisfactorily as a carrier fluid. Since the 0.45 lb/Mgal borate-crosslinked

gel described the minimum crosslinker concentration to transport a 2 ppg slurry through the slot model, a gel prepared at 1.0 lb/Mgal would transport the same slurry. Furthermore, the API viscosity values for the gel prepared at 1.0 lb/Mgal were higher than those of the 0.45 lb/Mgal gel as shown in Fig. 5.1. The gel prepared at 3.0 lb/Mgal concentration sheared into small gel domains and exhibited phase separation, so it would be an unacceptable carrier fluid formulation. Hence, the borate-crosslinked guar gels prepared at 0.0, 1.0, and 3.0 lb/Mgal were not examined for their proppant transport behavior through the slot models.

The question now is why the 0.25 lb/Mgal crosslinked gel exhibited poor suspension characteristics while the 0.45 lb/Mgal gel could satisfactorily transport 2 ppg slurry through the slot. To understand this, these crosslinked gels were further characterized for their viscoelastic properties, and their network structures imaged using the AFM.

Oscillatory Measurements. Figure 5.6 shows the elastic and viscous moduli of various borate crosslinked gels investigated. It shows that the elastic and viscous moduli increased as the crosslinker concentration was increased from 0.0 to 3.0 lb/Mgal. The increase in the viscous moduli with concentration was seen only at low frequencies, whereas the increase in the elastic moduli was seen in almost the entire frequency range. This increase in the moduli could be attributed to an increase in the crosslink density of the guar gel. At higher crosslinker concentrations, the solutions had more borate ions that could further attach to the cis-hydroxyl groups on the guar polymer to establish more crosslinked bonds between the polymer chains.^{101,109} An increase in the number of

crosslinked bonds resulted in an increase in the gel moduli at higher crosslinker concentrations.

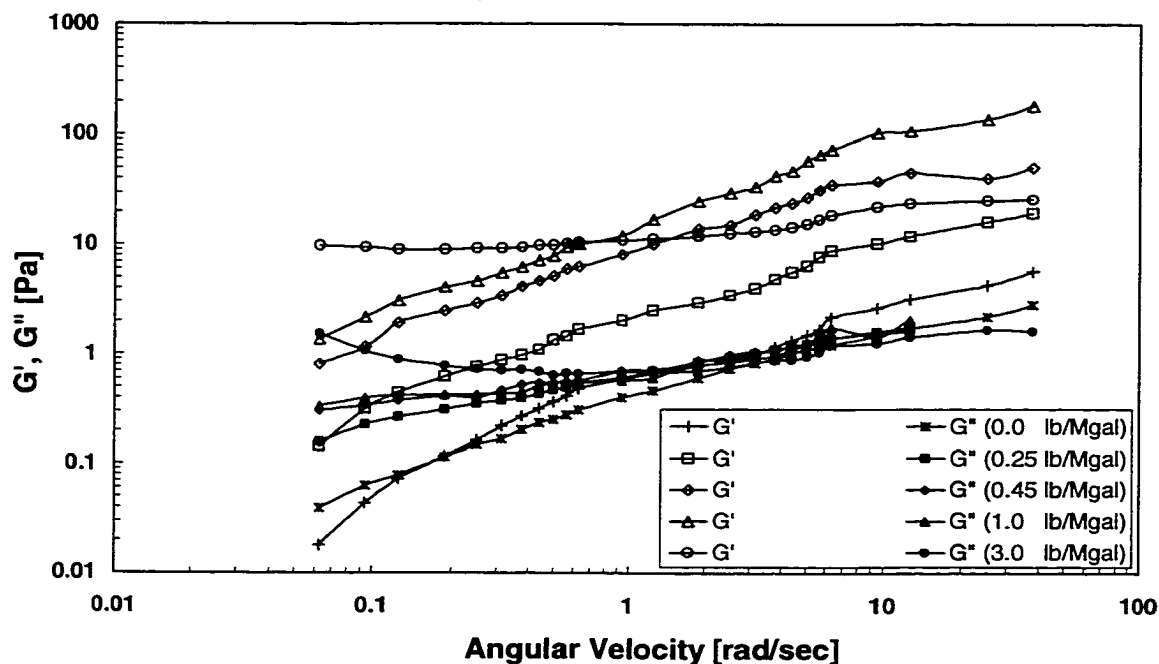


Figure 5.6—Oscillatory Measurements as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 11 (35 lb/Mgal Guar, 75°F)

Figure 5.6 further shows that except for the gel prepared at 3 lb/Mgal, the elastic modulus curves of all other gels did not clearly exhibit plateau moduli. The absence of the plateau moduli in the borate-crosslinked gels analyzed here could be explained from a low enthalpy of formation of the crosslink bonds between the borate ions and hydroxyl groups. This low enthalpy means the crosslink bonds can break easily with the energy supplied during the gel shearing. Therefore, when the gel was shear preconditioned in the blender, these bonds broke with the shear, and the bonds could not reestablish when the gel was characterized for oscillatory measurements immediately after the shear

conditioning. As a result, the gels did not exhibit plateau modulus in the rheological measurements performed immediately after shearing in the blender.²¹⁴ Since the plateau modulus, which indicates a solid-like characteristic, could be used with rubber elasticity theories to estimate the crosslink density, its absence made it difficult to quantify crosslinking from the elastic moduli of these gels. The oscillatory measurements could only suggest that the elasticity and viscosity of a borate-crosslinked gel increased with an increase in the crosslinker concentration. In the 3 lb/Mgal crosslinked gel, however, the crosslinker concentration was so large that despite the shearing in the blender, a constant elastic modulus G' was observed at even the lower frequencies. In this gel, G' did not increase at higher frequencies but remained constant at a value similar to that at the lower frequencies. The lower G' of this gel at the higher frequencies, as compared to those of the gels prepared at 0.25, 0.45 or 1 lb/Mgal, indicated the presence of wall slippage behavior during the oscillatory measurements.¹⁰⁹

Figure 5.6 further shows that the borate-crosslinked gels have higher G' than G'' values in the entire frequency range where the gels were evaluated. The higher elastic than viscous modulus indicates the formation of polymer chains network in the borate-crosslinked guar gels.²¹⁵ This well structured polymer network would not have formed in the 0.25 lb/Mgal gel as it was unable to suspend sand particles in the slot. The network structure might have existed in the 0.45 lb/Mgal and higher concentration gels because of the satisfactory proppant transport behavior in the slot. Thus, by merely observing a higher elastic modulus than the viscous modulus, it is difficult to suggest that a network structure exists in borate-crosslinked gels, and that the gel would exhibit satisfactory suspension characteristics.

Because of these limitations in identifying gel structure with rheological measurements, these gels were further characterized using the AFM measurements.

AFM Characterization. For the AFM measurements, the borate-crosslinked gels were transferred to the mica surface by dipping the mica substrates into the gel, and were left to air dry. These gels dried to form films on the substrate. The films were then imaged with an optical microscope and a scanning probe microscope. The optical microscope could not identify structures in the gel films, so AFM was used to explore microscopic features in the gel.

The AFM images depicted that the gel dried into three distinguishable regions: a thin-film region left behind by the evaporated water containing nominal amount of polymer, a polymer-rich region where the guar fibers were distinctly seen in the image, and a thick-film region where the gel dried into grains containing fractals of polymer mass. These three regions are shown in the AFM images of Figure 5.7 for 35 lb/Mgal guar solution.

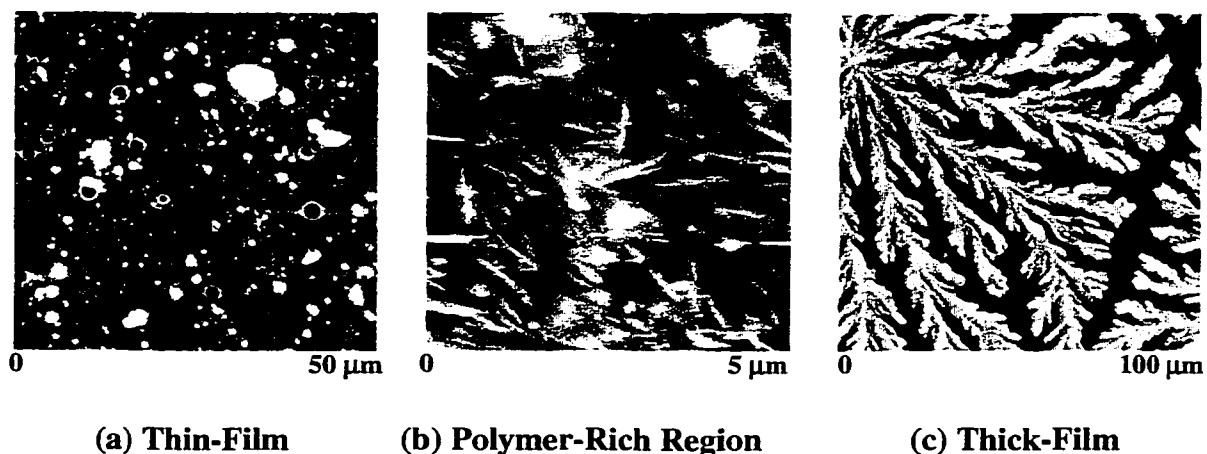


Figure 5.7—Atomic Force Microscopy Images of 35 lb/Mgal Guar, pH 11, 75°F

Figure 5.7 (a) shows the thin-film region, which has no polymer fiber. The figure depicts this film to have a number of tiny pinholes, which were uniformly distributed on the image. Similar pinholes were observed in the AFM images of all gel samples. At some locations on the mica substrate, these pinholes were visible with the AFM, as seen in Fig. 5.7 (a), and at others, they were covered by guar fibers and hence could only be seen under the fibers in the AFM images of the crosslinked gels. The presence of pinholes in all samples indicated that the thin film could be covering the entire mica surface. This thin film might have formed from the water used to prepare the guar solution. Since this water contained some chemicals added for uniform dispersion of guar in the solution, it dried to leave a thin film. This film might contain some guar polymer and chemicals that were deposited due to different affinities of the guar polymer and solvent to the mica substrate. This film could also be formed from an incomplete molecular dispersion of guar polymer in the solution, as that was been seen in water soluble polymers.⁶⁰ The significance of this film is not well understood at this point.

Figure 5.7 (b) shows the polymer-rich region where the guar polymer fibers were randomly oriented small and short fibrils. These fibrils are seen to overlap, indicating that the guar concentration in the prepared solution was higher than its overlap concentration. The fibrils seemed to have strong interaction with the AFM probe, resulting in undesirable noisy lines in the image.

Figure 5.7 (c) shows the thick-film region where the guar fibers are not individually visible but are seen as a large polymer grain of fractal structures. The grain had a nucleus from which leafy branches were spreading out radially. The thick-film region contained several similar grains with each grain having a nucleus and leaves and

being separated from other grains at a boundary. This boundary seemed to result from depletion of guar and not from cracking of guar film on drying, because the leafy branches in the neighboring grains were oriented in the opposite directions. The fractals were about 3 microns in thickness, and had a wide range of size and density. The number density of these fractal grains decreased in the gels as the crosslinker concentration was increased. The decrease in their densities indicated that the number of grains increased so much in the gel that the boundaries between them were not clearly demarcated, and so the leafy structures became unidentifiable. The diminishing fractal pattern in the thick-film region indicated differing structures in the gels prepared at different crosslinker concentrations.

As compared to the thick-film region, the polymer-rich region provided more distinguishable features on the crosslinked gel samples. The polymer-rich regions were therefore identified on these samples and were then used to understand the relationship of the network structures formed in the gels with their sand transport characteristics.

Figure 5.8 depicts the polymer-rich region on a crosslinked gel sample. It shows this region to be near the rim of a thin-film region. This image displays a unique separation between the thin film and the fibers, and demonstrates the location to identify the polymer-rich region on borate-crosslinked guar gels.

Figure 5.9 shows AFM images in the polymer-rich region of the gels prepared at different crosslinker concentrations. Figure 5.9 (a) describes the image of the gel prepared at 0.25 lb/Mgal crosslinker concentration, and depicts that the addition of the crosslinking agent had maintained fibrous structure in the guar. Since the size of the fibers in this image was larger than that of the guar solution, the fibers would have come

from the aggregation of fibrils. The crosslinking agent joined the guar together into a network of polymeric fibers. Underneath this network of fibers, the thin film region containing pinholes can be seen at the bottom right corner of the image. The AFM image further points that the crosslinker concentration in this gel was not at a high concentration as the fibers were not completely joined together and some of them had dangling ends that were not crosslinked to any other fiber. The image also presents void spaces between the fibers, indicating low crosslink density in the sample. Because of the low fiber density, the AFM image suggests that this gel might not have sufficient strength to sustain the weight of the sand particles. This gel was, therefore, unable to transport proppant through the slot models.

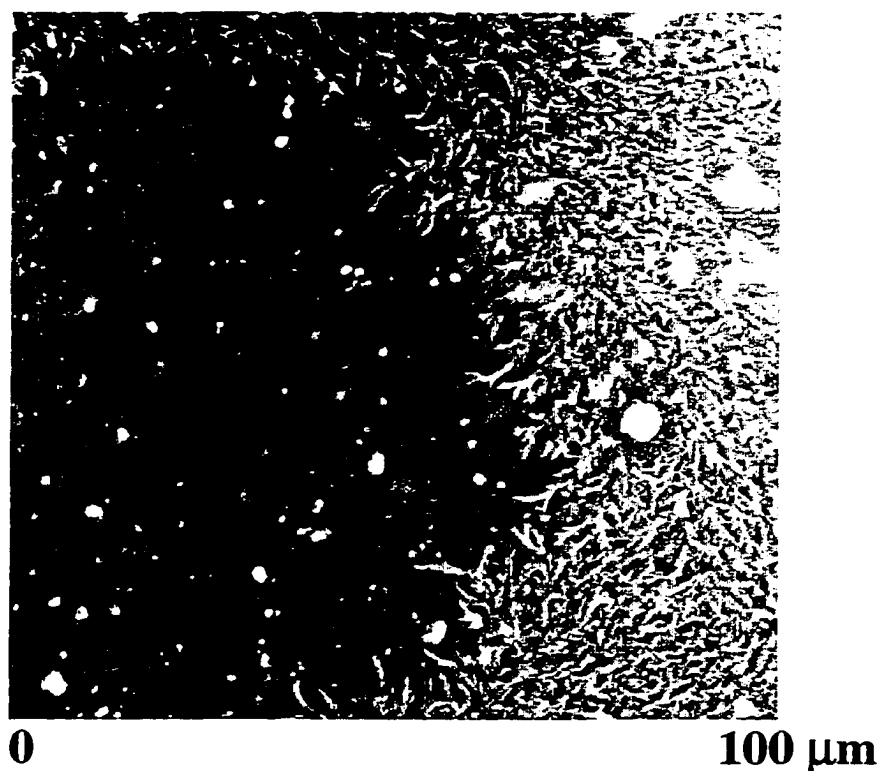
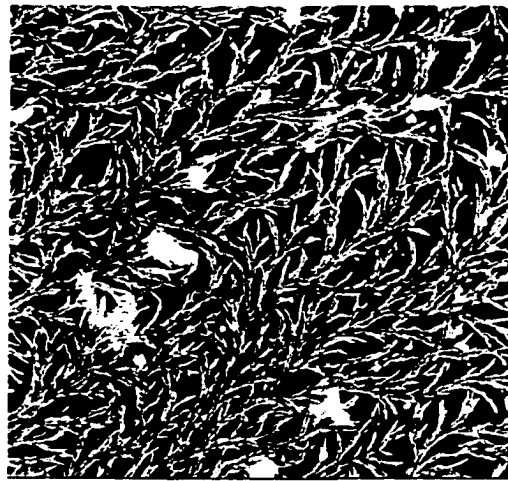
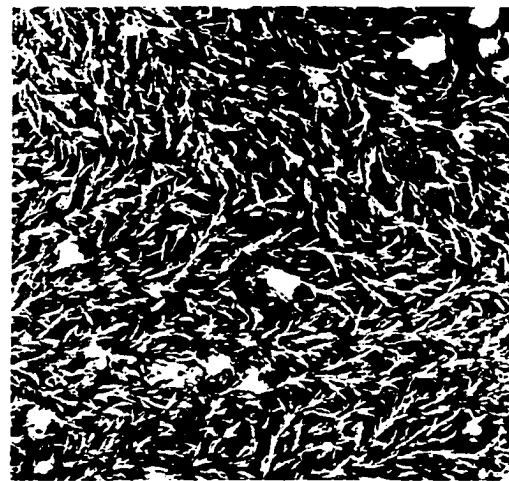


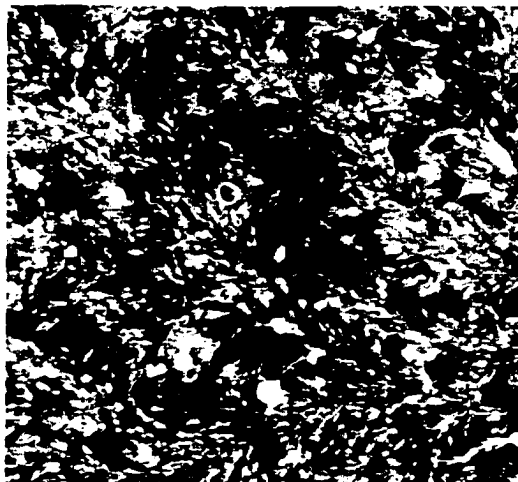
Figure 5.8—Polymer-Rich Region on Borate-Crosslinked 35 lb/Mgal Guar, pH 11



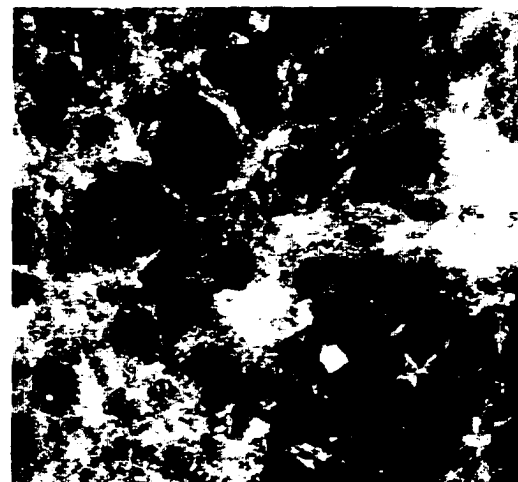
(a) 0.25 lb/Mgal Crosslinker Concentration



(b) 0.45 lb/Mgal Concentration



(c) 1.0 lb/Mgal Crosslinker Concentration



(d) 3.0 lb/Mgal Concentration

Figure 5.9—AFM Images as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 11 (35 lb/Mgal Guar, 75°F)

When the crosslinker concentration was increased to 0.45 lb/Mgal, more fibers combined together, as seen in the AFM image of the gel in Fig. 5.9 (b). The fiber density increased in this gel, leaving very little void between the fibers, and little exposure of the lower

layer under the fibers. The higher fiber density strengthened the gel and improved its ability to suspend proppant through the slot. As a result, the gel was observed to transport 2 ppg slurry through the slot models.

Figure 5.9 (b) further shows that the guar fibers though were more crosslinked than 0.25 lb/Mgal gel were distinguishable as fibers in the image. The fibers formed in 0.45 lb/Mgal gel were still in a two-dimensional network structure. This implies that the gel prepared at 0.45 lb/Mgal had enough strength to transport 2 ppg slurry, but might be unable to suspend slurries prepared at higher sand concentrations. Since fracturing fluids are used to transport proppant at concentrations up to 10 to 12 ppg, another gel having crosslinker concentration greater than 0.45 lb/Mgal would be required to suspend and transport high concentration slurries through the slot.

Unlike the AFM image for 0.25 lb/Mgal gel, where the thin film layer was visible under the fiber, Fig. 5.9 (b) does not reveal this film. The thin film can, however, be seen near the rim of the polymer fibers as shown in Fig. 5.8.

To exhibit changes in the gel structures at higher crosslinker concentrations, a gel was prepared at a crosslinker concentration of 1 lb/Mgal, which is about twice the minimum concentration (0.45 lb/Mgal) required to transport 2 ppg slurry through the slot model. The AFM image of this borate-crosslinked gel is shown in Fig. 5.9 (c). It shows that the individual fibers could not be recognized here as could be in the image for 0.45 lb/Mgal gel. At 1 lb/Mgal concentration, the guar gel structure had wide and long fibers. The fibers seemed to be connected with one another to form a thicker and multi-layered structure, which was uniformly distributed throughout the image. This AFM image suggested that the gel prepared at 1 lb/Mgal would have better strength than the gel

prepared at the lower crosslinker concentrations. As the crosslinker concentration was further increased, the gel continued to evolve into a three-dimensional network, as seen in the AFM image of 3 lb/Mgal gel in Fig. 5.9 (d). At this crosslinker concentration, the image shows the crosslinked gel to be concentrated into small polymer rich regions, which were interspersed with water phase regions. This image also indicates the fiber aggregates to be attached together by threads of fibers, giving a tissue-like structure to the gel.

While taking the AFM images, the crosslinked gels prepared in the blender were set aside in beakers for any distinguishable features in these gels. After two days, when a mica disc was dipped into these gel samples, interesting observations were made. In the gels prepared at lower crosslinker concentrations of 0.25 and 0.45 lb/Mgal, the mica disc could be easily inserted in the gel and pulled out with the gel intact in the beaker, showing a good mobility of the guar fibers in these gels. On the other hand, in the gel prepared at 1 lb/Mgal, the mica disc when pulled out of the gel lifted the gel along with it as if the gel was a single unit of polymer mass. The gel was lifted to a substantial height before it bounced back into the beaker. This behavior indicated that the guar fibers were well connected with one another and formed a three-dimensional structure at this crosslinker concentration. In 3 lb/Mgal gel, a very different behavior was observed. At this concentration, the gel exhibited phase separation with a solid gel mass floating over a polymer-free solution. This solution was checked for its viscosity and found to have similar viscosity to that of water. The absence of guar in the phase separated water indicated that the entire guar was concentrated into the gel mass. When the mica disc was inserted into this gel mass, the mica could cut through the gel. Even when the mica

was pulled out, the gel neither pulled up with the mica nor rehealed the cut, suggesting that an immobile gel network had formed in 3 lb/Mgal gel. This immobility pointed to diminishing viscous characteristics of the gel.

The visual observations made on the borate-crosslinked gels pointed that the gel changed from a viscous fluid to a viscoelastic material and finally, to a solid gel when the crosslinker concentration was increased from 0.25 through 1 to 3 lb/Mgal.

Limitations of the AFM Measurements. The AFM measurements were made on the dried gel samples, so the images do not correctly depict the actual structure formed in the gel whose suspension characteristics were evaluated in the slot. The AFM images do describe the gel behavior and thus help to understand their suspension capabilities.

In the guar solution, the images suggested that the polymer fibrils overlapped. When the crosslinking agent was added to the guar solution, the fibrils crosslinked together into fibers. These fibers aggregated and integrated into a strong network as the crosslinker concentration was increased. At even higher crosslinker concentration, the fibers merged together to form wider leaves, making it difficult to identify individual fibers in the image. The fibrous structure continued to develop in the crosslinked gel till the polymer fibers were concentrated into gel mass leaving water as a separate phase.

Fluid Property for its Suspension Capability. The viscosity is seen to insufficiently describe proppant transport behavior of borate-crosslinked gel, a question may be raised regarding a better way to characterize fracturing fluids in a laboratory.

The AFM images of Figure 5.9 showed that the increase in crosslinking resulted in an increase in the fiber density and an increase in the gel strength. Similarly, Fig. 5.6 showed an increase in the elastic and viscous moduli of the gels with an increase in the crosslinker concentration. These changes in the gel rheology with crosslinker concentration corresponded well with the AFM images of the gel. Figure 5.6 further shows that the increase in the viscous moduli was less distinct than the increase in the elastic moduli when the crosslinker concentrations were increased from 0.25 to 3.0 lb/Mgal. Figure 5.1 showed a very small difference in the viscosity of the gels prepared at 0.25 and 0.45 lb/Mgal, even though these gels had a very large difference in the sand suspension characteristics as evident from Figs. 5.2 and 5.4. Thus, the difference in the proppant transport characteristics was better described from the large difference in the elastic moduli than in the viscosities of these two gels. The borate-crosslinked gels characterized with their elastic moduli provided useful information about the crosslinked gel structure and about their capability to suspend and transport sand particles. The elastic modulus is hence a better property than viscosity to characterize and evaluate fracturing fluids for their suspension characteristics, and it should be further evaluated to establish a minimum criterion for satisfactorily transporting proppant through a fracture.

Comparison of the Oscillatory Measurements with Viscosity Data. The oscillatory measurements can be used to determine a complex viscosity of the gel. This viscosity can then be compared with the steady shear viscosity using the Cox Merz Rule.¹⁵⁴ This comparison might suggest a new viscosity criterion for satisfactory proppant transport through a fracture because the complex viscosity contains elastic moduli of the gel.

The complex viscosity values of the borate-crosslinked gels were determined using Equation 2.25 from the oscillatory data shown in Figure 5.6. These values were then compared to the steady shear viscosity measured at the initial time during the API viscosity test. The comparison is shown in Figure 5.10.

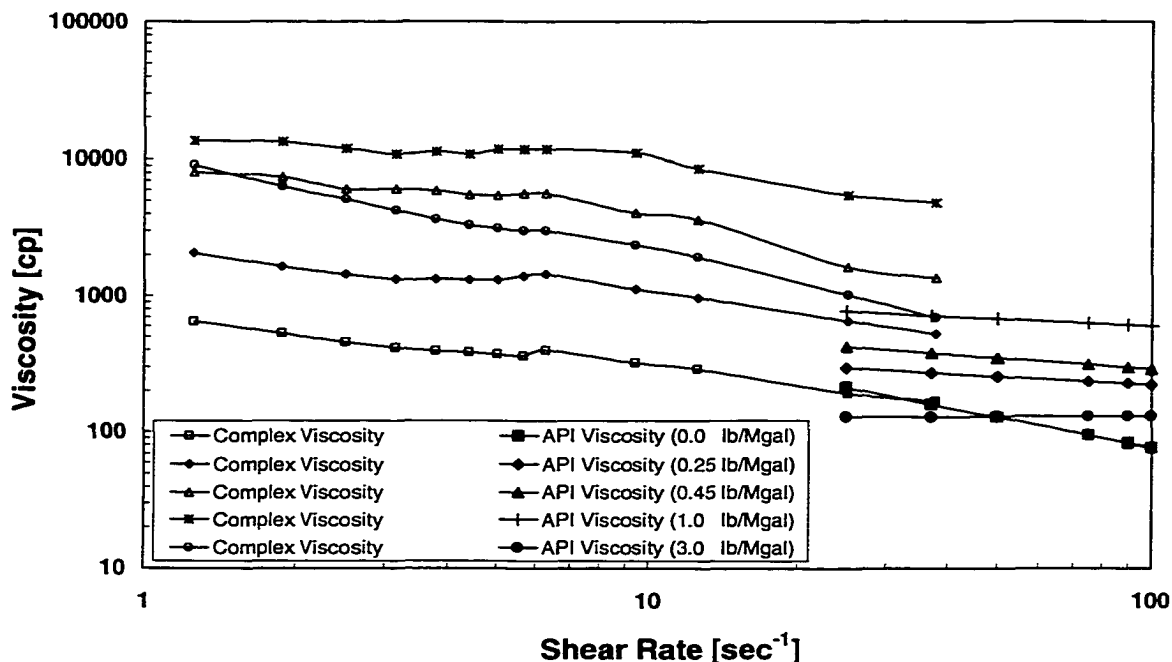


Figure 5.10—Comparison of Complex Viscosity and API Viscosity as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 11 (35 lb/Mgal Guar, 75°F)

Figure 5.10 shows an increase in complex viscosity and API viscosity values with an increase in the crosslinker concentration. The viscosities of the crosslinked gel prepared at 3 lb/Mgal were however lower than the other crosslinked gels, because of the phase separation observed in this gel. Figure 5.10 further points to a poor correlation between

the API and complex viscosities of all crosslinked gels. The non-crosslinked guar gel prepared at zero crosslinker concentration had similar viscosity values in the limited shear rate range where the oscillatory and the API viscosity data overlapped. Thus, the Cox Merz rule was applicable to the linear guar solution only and not to the borate-crosslinked guar gels. The non-applicability of this rule makes it difficult to provide a better minimum viscosity value for satisfactory proppant transport by extrapolating the complex viscosity data of the borate-crosslinked guar gel prepared at 0.45 lb/Mgal, which was minimum crosslinker concentration for successful suspension behavior.

5.4 CONCLUSIONS

The viscosity criterion currently used in the industry provides an insufficient method to evaluate crosslinked fluids for their proppant transport characteristics. The suspension capability of these gels was observed to be due to an increase in the fiber density across the gel. With an increase in the crosslinker concentration, the borate-crosslinked gels exhibited an increase in the density of the gel network as seen in the AFM images, and an increase in the elastic and viscous moduli as seen from the rheology characterization. Consequently, the capability of the gel to suspend and transport sand particles through the slot also increased. On comparing the gel characteristics from the AFM images and the rheological measurements, the elastic modulus seems to provide useful information about the suspension capabilities of borate-crosslinked gels. Therefore, the elastic moduli of crosslinked gels should be further evaluated to establish minimum rheology criteria for satisfactorily transporting proppant through a fracture.

CHAPTER 6

DEVELOPMENT OF RELATIONSHIP BETWEEN RHEOLOGY AND PROPPANT TRANSPORT

6.1 INTRODUCTION

The previous chapter described that the elastic moduli may provide a better description of the proppant transport behavior of borate-crosslinked gels. This observation was solely based on the evaluations performed at just one fluid pH. Any criterion based on the elastic moduli needs further evaluation and confirmation before accepting its application for the hydraulic fracturing stimulation.

Therefore, borate-crosslinked gels were prepared at three pHs: pH 9, 10 and 11, for further evaluating the elastic moduli based criterion at ambient temperature. The modulus was then further evaluated at elevated temperature and at higher slurry concentrations. These evaluations are described in this chapter.

The guar solutions were prepared and their pH adjusted; then at each pH, the solution was crosslinked with several crosslinker concentrations to prepare numerous borate-crosslinked guar gels. The crosslinker concentration was varied from zero to the values where the borate-crosslinked gel exhibited phase separation.^{117,123} The gels thus prepared were characterized in the laboratory for their viscoelastic properties and sand suspension capacities. Based on the laboratory results, a crosslinker concentration was selected at each pH for further evaluating the gel to transport 2 and 8 ppg slurries through the slot models. These evaluations were performed at ambient temperature, followed by one at an elevated temperature.

The results showed that the crosslinked guar gels exhibiting satisfactory proppant transport behavior could be prepared at each condition. It was observed that the fluids exhibiting satisfactory sand transport had similar elastic moduli at low frequencies. Therefore, the elastic modulus correlates better with the sand suspension and proppant transport capability of the crosslinked guar gels than viscosity.

6.2 RESULTS AND DISCUSSION

6.2.1 Evaluations at Ambient Temperature

Borate-Crosslinked 35 lb/Mgal Guar pH 9. The pH of the guar solution was first adjusted to 9. The solution was then crosslinked at crosslinker concentrations from 0 to 15 lb/Mgal.

The viscoelastic data of the borate-crosslinked guar gel at pH 9 are shown in Figure 6.1. The figure shows an increase in the elastic and viscous moduli with an increase in the crosslinker concentration. This increase in the elastic moduli was obtained because of an increase in the number of crosslinked sites formed in the polymer.

The response of the oscillatory measurements of polymers normally follow characteristic behavior with a slope of 2 for G' and unity for G'' curves at low frequencies. The response of the borate-crosslinked gels, however, did not exhibit this polymer characteristic behavior. The G' and G'' curves in Fig. 6.1 did not exhibit respectively the characteristic slope of 2 and 1 at low frequencies. This deviation could be due to a broad distribution of molecular weight in the gels.¹⁶⁵ The linear guar polymer itself has substantial polydispersity as discussed in Chapter 2. Some additional polydispersity might be introduced when the guar was crosslinked. The borate-

crosslinking is a bonding between the borate ions and the alcohol groups on the polymer; since the alcohol groups are in large number on a guar of 2.26 million molecular weight, the crosslink bonds could be randomly distributed along the polymer length. There can therefore be a broad distribution in the molecular weight between the crosslinks. In addition to the effect of polydispersity, the viscoelastic measurements would be influenced by an incomplete relaxation of crosslinks during the time scale of the low frequency measurements. These factors would have contributed to a deviation from the characteristic slopes in the oscillatory response of the borate-crosslinked guar gels.

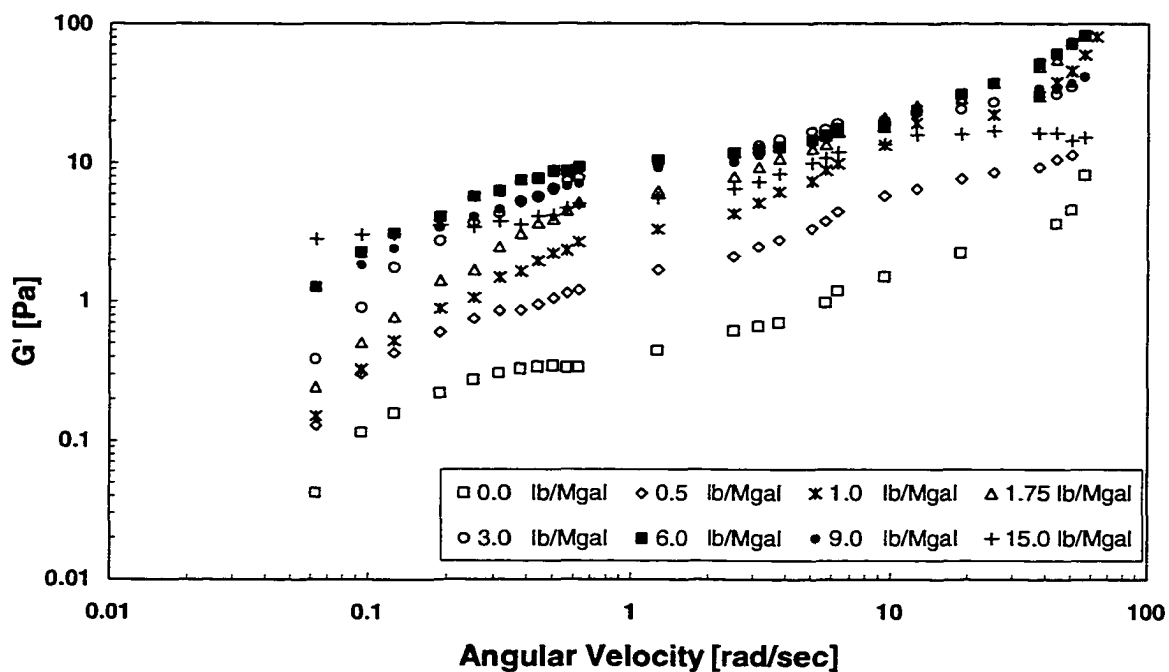


Figure 6.1(a)—Elastic Moduli as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 9 (35 lb/Mgal Guar, 75°F)

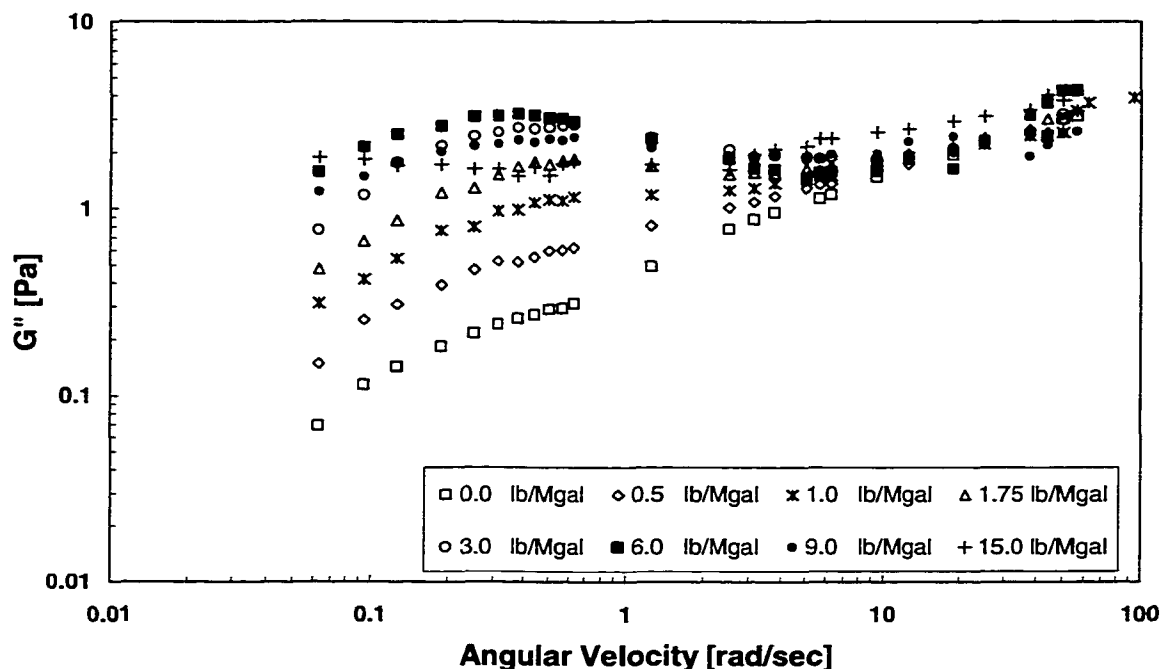


Figure 6.1(b)—Viscous Moduli as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 9 (35 lb/Mgal Guar, 75°F)

The frequency independent elastic modulus at high frequencies, which describes a network of crosslinks, was not observed in Fig. 6.1 (a). The absence of plateau moduli in the borate-crosslinked guar gel is explained through reversible nature of bonding in these gels. The bonds between the borate ions and polyols are not permanent as they break and reform with the applied shear.^{10,109,116} Because of the reversible crosslinks, a constant modulus would be exhibited at frequencies greater than the frequency for forming and breaking of the crosslink bonds.²¹⁶ Figure 6.1 (a) further shows that the gels prepared at crosslinker concentrations beyond 6 lb/Mgal had lower values of the elastic moduli at higher frequencies as compared to the gels having concentrations less than 6 lb/Mgal. This behavior indicated that at the higher frequencies, there was slippage in the

viscoelastic measurements of the gels having higher crosslinker concentrations. The slippage might come from a phase separation in the crosslinked gels. The gels having 9 and 15 lb/Mgal crosslinker were observed to have phase separated into a polymer mass and a watery phase when left unperturbed for 24 hr. This separation has been observed previously in borate-crosslinked gels and is ascribed to the over-crosslinking of guar polymer by the borate ions.¹⁰ Thus, the lower values of the elastic moduli for crosslinker concentrations above 6 lb/Mgal were due to the phase separation during the viscoelastic measurements.

Figure 6.1 (b) shows that at lower crosslinker concentrations G'' increased with an increase in frequency. However, at and beyond 1.75 lb/Mgal crosslinker concentration, G'' increased at low frequencies and then decreased, thereby exhibited a maximum. This maximum in the loss modulus was described as an indication of gel point in borate-crosslinked guar.³⁶ Power²¹⁷ used this gel point criterion to describe the proppant settling in crosslinked gels. He established the relationship between the gel point and the suspension characteristics using the settling velocity criterion described by de Kruijf et al.³⁵ This settling criterion proposed that for settling velocities less than 5 cm/min, the proppant transport was acceptable in a crosslinked fluid and for values less than 0.5 cm/min, the transport was perfect. To compare the settling velocity criteria with the gels prepared in the present study, the suspension settling velocities were measured in different crosslinked gels under conditions similar to that used to develop the criteria. These conditions were described in Section 3.3.3. Under these conditions, the settling velocities of 2 ppg slurry prepared in pH 9 gels are shown in Figure 6.2.

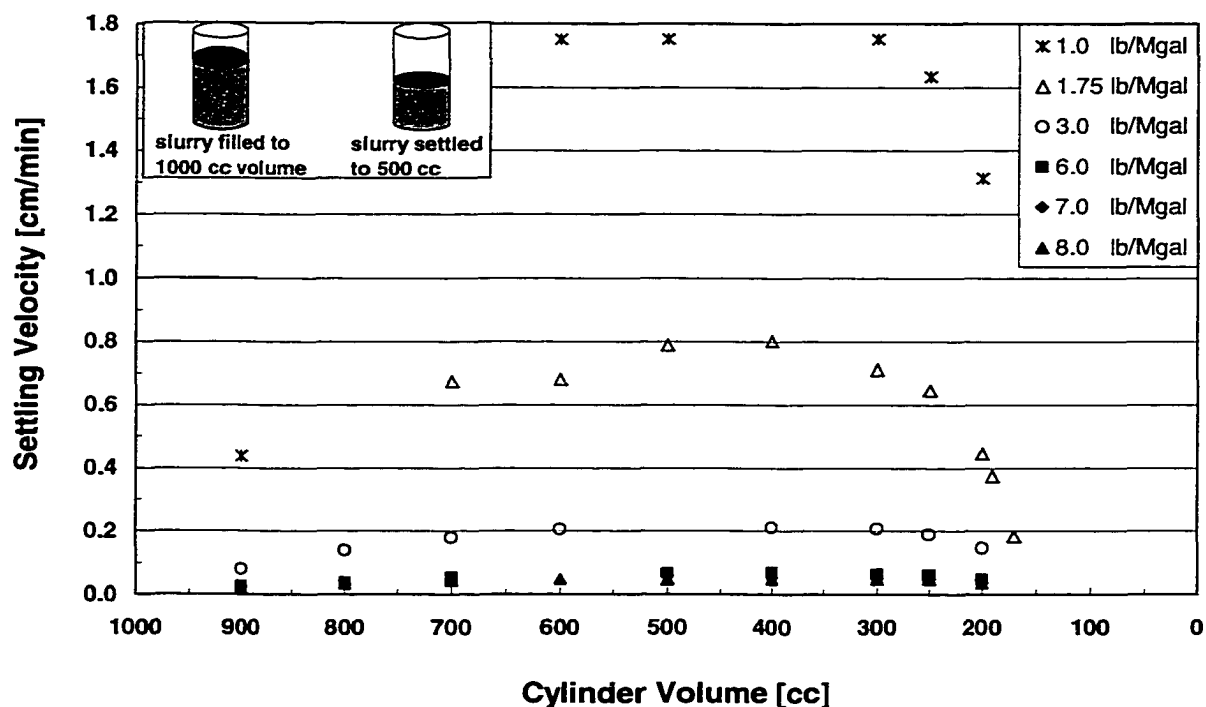


Figure 6.2—Suspension Settling Velocity of 2 ppg Slurry as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 9 (35 lb/Mgal Guar, 75°F)

Figure 6.2 shows a decrease in the settling velocities with an increase in the crosslinker concentration. It also shows that the suspension settling velocity was non-uniform across the height of the cylinder. The velocity increased as the interface moved down from the 1000 ml. mark (see inset Fig. 6.2), attained a maximum value in the cylinder middle, and then decreased as the interface settled into a bed. The height of the settled bed corresponded to the volume of sand in 2 ppg slurry. The reason for this non-uniform settling velocity is not well understood.⁵⁵ Though not evaluated in the present work, the non-uniform settling could be because of a competition between particle aggregation and hindered settling in the suspension. The initial increase in the settling velocity could be

from an increase in the sand concentration due to a redistribution of sand weight in a smaller volume of the crosslinked gel. The higher sand concentration could reduce the suspension capability of the gel and increase the settling velocity. As the sand continued to settle in the cylinder, the suspension capability of the gel could have been influenced by the movement of the crosslinked gels from the interstices between the sand particles. Since the gel movement from interstices is a slower process, the sand particles settled slowly and the velocity decreased. These observations are however not substantiated in the present study. A more comprehensive investigation is required to understand the non-uniform settling in viscoelastic fluids.

Figure 6.2 shows that the suspension settling velocities in the borate crosslinked gel having 1.75 lb/Mgal crosslinker were in the range of the values for acceptable proppant transport through a fracture.³⁵ This concentration also satisfied the gel point criteria,³⁶ as it exhibited a maximum in the loss modulus, as shown in Fig. 6.1 (b). This pH 9 gel thus satisfied the two available criteria.^{35,217} If these criteria are correct, the pH 9 gel prepared at 1.75 lb/Mgal should exhibit acceptable proppant transport behavior. Hence, this gel was evaluated in the slot models for its sand transport behavior.

Figure 6.3 shows the sand distribution in the HPS at 5 and 15 min when a 2 ppg sand slurry prepared in pH 9 gel having 1.75 lb/Mgal crosslinker was pumped through the slot model. The figure shows high sand concentrations in the bottom-half of the HPS and low concentration at the top. Clearly, sand particles settled during the slurry flow through the HPS. The sand accumulation at the bottom means that the suspensions flowing out of the HPS had a lower sand concentration than the input slurry of 2 ppg. These images depict that the gel would be unable to suspend and transport sand particles

through a fracture. This poor transport characteristic of the gel was observed despite it having satisfied both the gel point and the settling velocity criteria. The slurry test through the slot thus indicated that these two criteria were insufficient to describe the proppant transport capability of the viscoelastic guar gels.

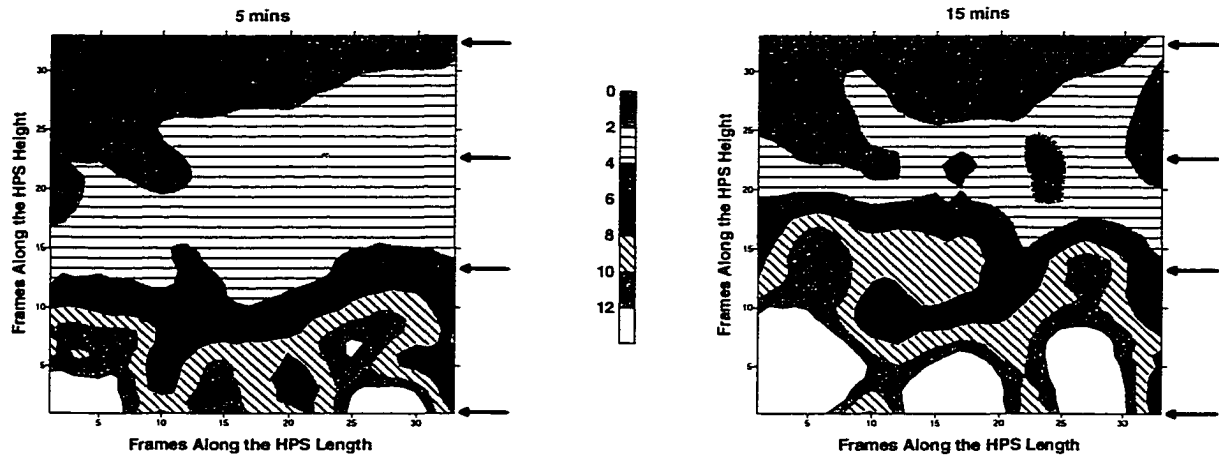


Figure 6.3—Proppant Concentration (ppg) in the HPS after 5 and 15 min of 2 ppg Slurry Transport with Borate-Crosslinked Guar, 1.75 lb/Mgal Crosslinker, pH 9 (35 lb/Mgal Guar, 75°F)

The sand transport capability of the borate-crosslinked guar gel would presumably be improved if a higher crosslinker concentration were used. This improvement is expected because with an increase in the crosslinker concentration, the suspension settling velocities decreased as shown in Figure 6.2. Possibly, the higher crosslinker would also improve the gel capability to transport proppant through a fracture. Therefore, a higher crosslinker concentration of 6 lb/Mgal was selected, and the new gel was evaluated in the slot model. Figure 6.4 shows the proppant transport behavior of this gel after 5 and 15

min. of slurry flow through the HPS. This figure shows a large improvement in the suspension capability of the gel as the sand was uniformly distributed in the slot. Unlike the previous gel, there was negligible settling, and no bed was observed at the bottom of the HPS. The figure does show some clusters of higher sand concentration, a phenomenon frequently observed in crosslinked guar.¹⁸⁰ These clusters being suspended in the gel would be transported through the slot with the slurry flow.

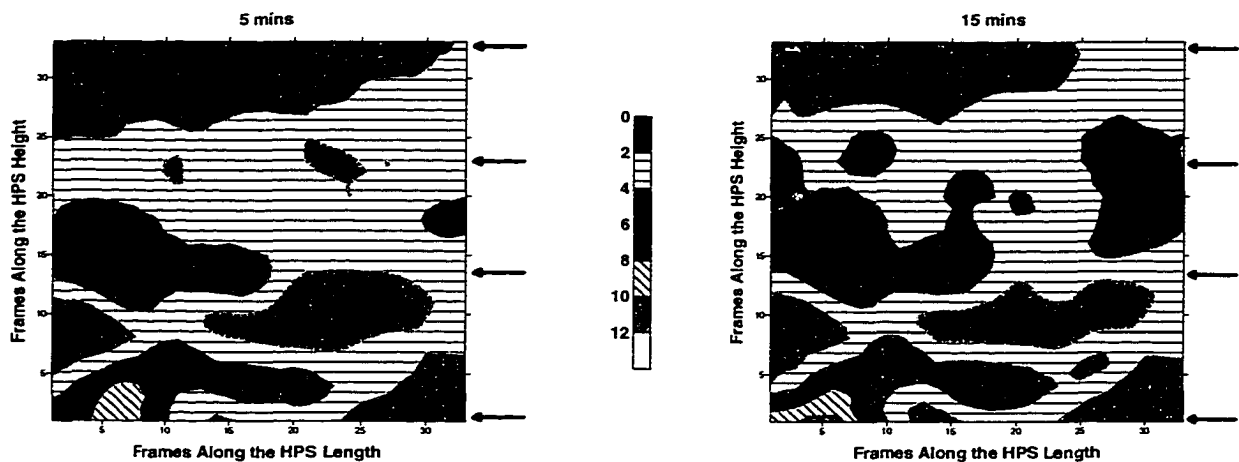


Figure 6.4—Proppant Concentration (ppg) in the HPS after 5 and 15 min of 2 ppg Slurry Transport with Borate-Crosslinked Guar, 6.0 lb/Mgal Crosslinker, pH 9 (35 lb/Mgal Guar, 75°F)

In addition to the flow through the HPS, the proppant transport behavior was also observed through the transparent slot and is shown in Figure 6.5. This figure depicts that there was no settling in the slot model. Thus, Figures 6.4 and 6.5 indicate that 6 lb/Mgal crosslinked gel would keep sand particles suspended and would satisfactorily transport proppant through the slot.



Figure 6.5—2 ppg Proppant Slurry Transport through Transparent Slot with Borate-Crosslinked Guar, 6.0 lb/Mgal Crosslinker, pH 9 (35 lb/Mgal Guar, 75°F)

The satisfactory proppant transport seen in Figs. 6.4 and 6.5 indicated that a higher crosslink density would be required than that in the borate-crosslinked guar gel selected using the available criteria. The question still remains regarding the rheological property that provides a better correlation to solids transport behavior of a fluid.

The higher crosslink density is again depicted in the higher elastic and viscous moduli of the gel prepared at 6 lb/Mgal than that at 1.75 lb/Mgal crosslinker concentration. Further test is therefore needed at other pH to compare these moduli to those in other gels exhibiting satisfactory proppant transport. Therefore, crosslinked guar gels were prepared at two other fluid pHs of 10 and 11.

Borate-crosslinked 35 lb/Mgal Guar pH 10.

Figure 6.6 shows the settling velocity data, and Figure 6.7 describes the viscoelastic properties of the borate-crosslinked guar prepared with different crosslinker concentrations at pH 10. As expected, Figure 6.6 displays a decrease in the settling velocities of 2 ppg slurry with an increase in the crosslinker concentration, and Fig. 6.7 demonstrates an increase in G' and G'' with an increase in the crosslinker concentration. These changes are similar to those seen in the gels prepared at pH 9, and are again due to an increase in the number of crosslinked sites formed in the guar at higher crosslinker concentration.

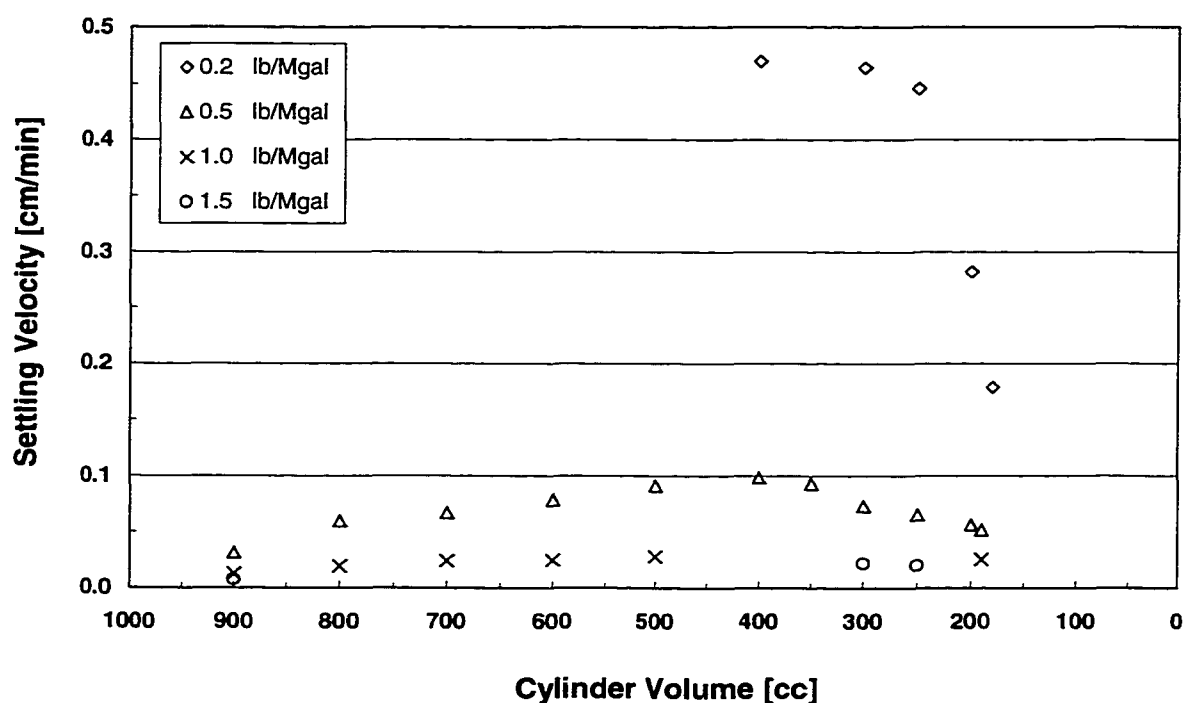
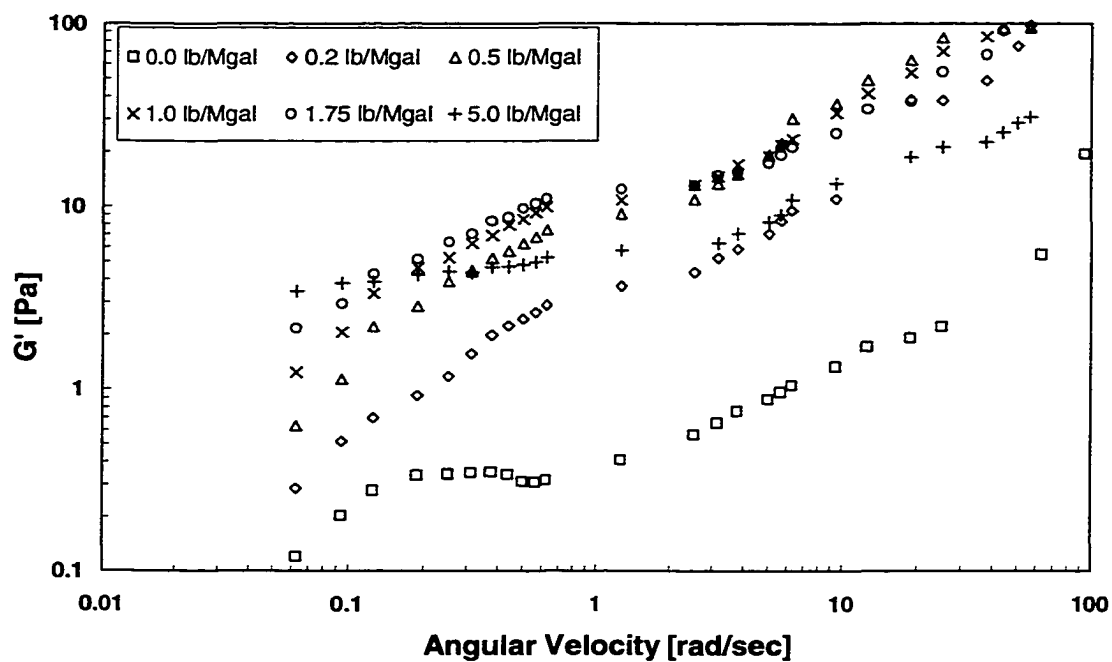
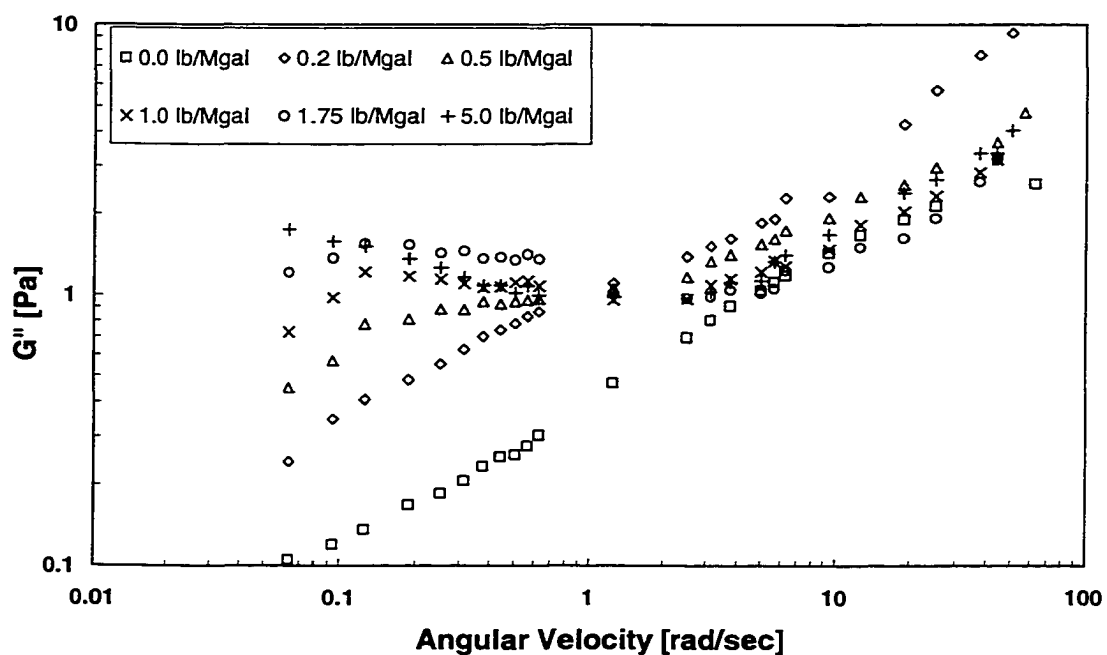


Figure 6.6—Suspension Settling Velocity of 2 ppg Slurry as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 10 (35 lb/Mgal Guar, 75°F)



(a) Elastic Moduli Curve



(b) Viscous Moduli Curve

Figure 6.7—Viscoelastic Measurements as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 10 (35 lb/Mgal Guar, 75°F)

The laboratory characterization of different fluids prepared at pH 10 was compared to that obtained with the crosslinked guar gel prepared at pH 9 with 6 lb/Mgal crosslinker. The comparison showed that pH 10 gel prepared with 0.5 lb/Mgal crosslinker concentration had similar suspension settling velocities to those observed in the pH 9 gel at 6 lb/Mgal crosslinker. These two formulations also had similar G' values at lower frequencies, but dissimilar G'' values. These similarities suggested that the pH 10 gel prepared with 0.5 lb/Mgal crosslinker might exhibit satisfactory proppant transport behavior. To confirm this hypothesis, a pH 10 gel was evaluated for its proppant transport capability through the slot models.

Figure 6.8 shows the proppant distribution in the HPS and Figure 6.9 depicts the slurry transport through the transparent slot. Figure 6.8 describes the sand distribution after 5 and 15 min of 2 ppg slurry flow through the HPS in pH 10 borate-crosslinked guar having 0.5 lb/Mgal crosslinker. The figure shows high sand concentration in the HPS bottom after 5 min of slurry flow, but a more uniform distribution of sand in the HPS at 15 min. It also depicts some high sand concentration clusters, but these suspended clusters being suspended would not affect the proppant transport behavior of this gel. Figure 6.9 also describes that this pH 10 gel successfully transported slurry through the slot model.

Figures 6.8 and 6.9 thus point that the pH 10 gel prepared with 0.5 lb/Mgal crosslinker satisfactorily transported sand and would provide an acceptable fluid formulation for fracturing.

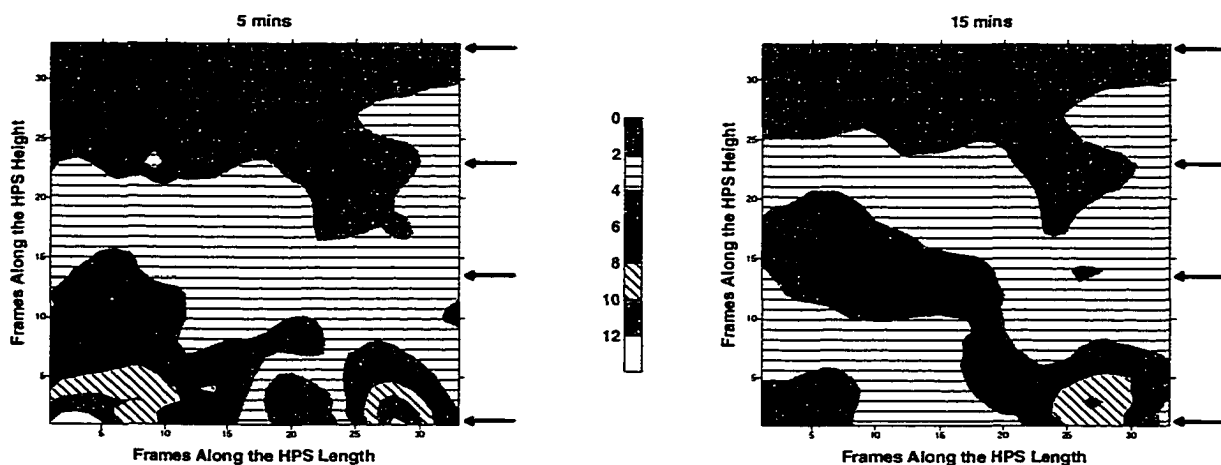


Figure 6.8—Proppant Concentration (ppg) in the HPS after 5 and 15 min of 2 ppg Slurry Transport with Borate-Crosslinked Guar, 0.5 lb/Mgal Crosslinker, pH 10 (35 lb/Mgal Guar, 75°F)

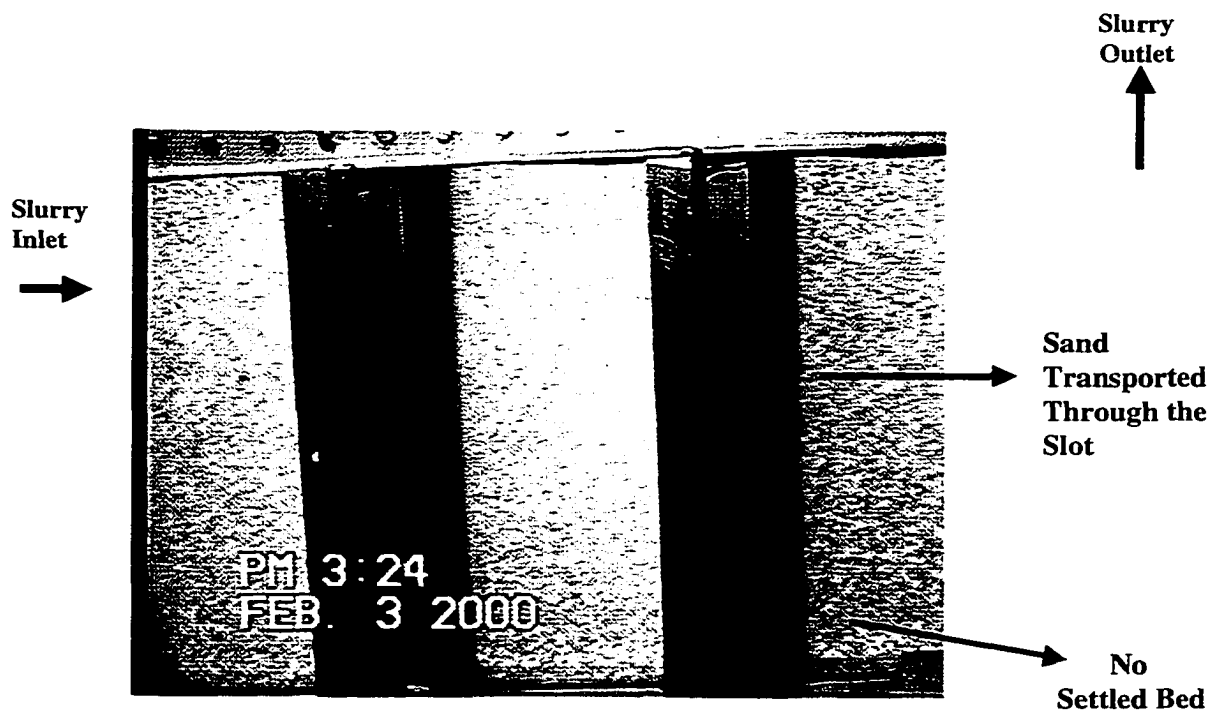


Figure 6.9—2 ppg Proppant Slurry Transport through Transparent Slot with Borate-Crosslinked Guar, 0.5 lb/Mgal Crosslinker, pH 10 (35 lb/Mgal Guar, 75°F)

Figure 6.9 depicts that a lower crosslinker concentration of 0.5 lb/Mgal was required in pH 10 gel to obtain almost similar proppant transport behavior to that observed in the pH 9 gel having 6 lb/Mgal crosslinker concentration. The lower crosslinker concentration in the pH 10 gel was due to a higher fraction of the borate ions in the fluid at this pH as compared to that in the pH 9 fluid. The actual pH in the fluid was about 9.8 and 8.8 respectively as the crosslinker solution reduced the gel pH slightly. For these pH values, Figure 2.3 indicates a borate fraction of 33% at pH 8.8 and 85% at pH 9.8. The lower borate fraction in pH 9 fluid means a higher crosslinker concentration would be required in this gel to exhibit similar characteristics to that in the pH 10 gel. The actual crosslinker concentration of 6 lb/Mgal was, however, much more than the value calculated from the borate fraction alone. This difference could possibly be because of the repulsive forces among the negatively charged borate ions, which would not allow ions to approach hydroxyl groups and establish crosslinked bonds. Consequently, a higher crosslinker concentration was required to achieve satisfactory performance with the pH 9 gel.

Figure 2.3 further suggests that a gel prepared at pH 11 might exhibit acceptable proppant transport at a crosslinker concentration close to 0.5 lb/Mgal because the borate ions fraction at this pH is 98%, which is close to that in the pH 10 gel.

Borate-crosslinked 35 lb/Mgal Guar pH 11. Therefore, 0.5 lb/Mgal crosslinker concentration was selected to evaluate suspension characteristics of borate-crosslinked guar gel at pH 11. This gel was, however, evaluated with an approach different from that used on the pH 9 and 10 gels.

Prior to flowing through the HPS, the pH 11 gel at 0.5 lb/Mgal was first evaluated in the transparent slot for its proppant transport behavior. The evaluation showed that the pH 11 gel having 0.5 lb/Mgal crosslinker exhibited satisfactory slurry transport, with sand being well distributed in the slot model without forming a settled sand bed. During the test, the crosslinker concentration was lowered to 0.45 lb/Mgal, and its the proppant transport behavior was again observed in the transparent slot model. At this concentration, the slurry was also satisfactorily transported through the slot, as was shown earlier in Figure 5.4. The crosslinker concentration was then further lowered to 0.40 lb/Mgal, and the gel was again evaluated in the slot. At this concentration, the sand particles began separating from the gel and started settling to the bottom of the slot, as shown in Figure 6.10. This figure depicts a diminishing capability of 0.4 lb/Mgal gel to successfully transport proppant through the slot. By first evaluating the pH 11 gel in the transparent slot, this test showed that 0.45 lb/Mgal concentration provided the minimum crosslinker concentration above which the pH 11 gel would exhibit acceptable slurry transport behavior.

After testing the gel in the transparent slot, the 2 ppg slurry in 0.45 lb/Mgal gel was pumped through the HPS to map the sand distribution for comparison with pH 9 and 10 gels. The proppant distribution through the HPS was shown earlier in Figures 5.5.

The suspension settling data of 2 ppg slurry in the pH 11 gels prepared at 0.45 lb/Mgal and other crosslinker concentrations are shown in Figure 6.11, and the viscoelastic data of the gels are shown in Figure 6.12. The data shown in Figs. 6.11 and 6.12 for the pH 11 gels are similar to those of the pH 9 and 10 gels.



Figure 6.10—2 ppg Proppant Slurry Transport through Transparent Slot with Borate-Crosslinked Guar, 0.40 lb/Mgal Crosslinker, pH 11 (35 lb/Mgal Guar, 75°F)

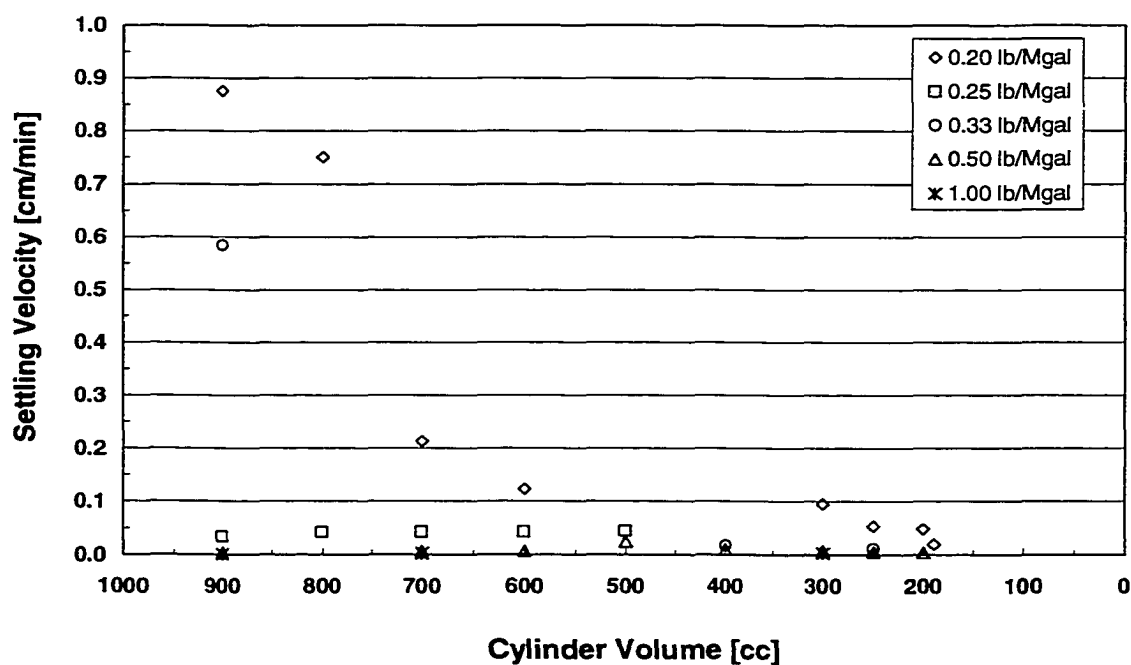
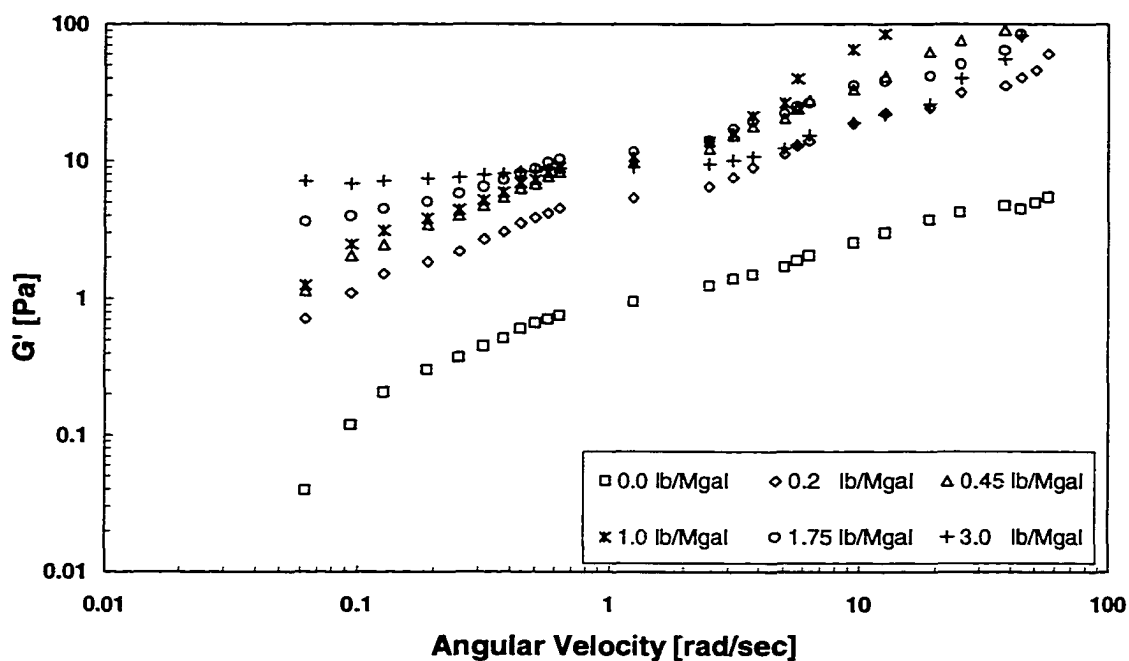
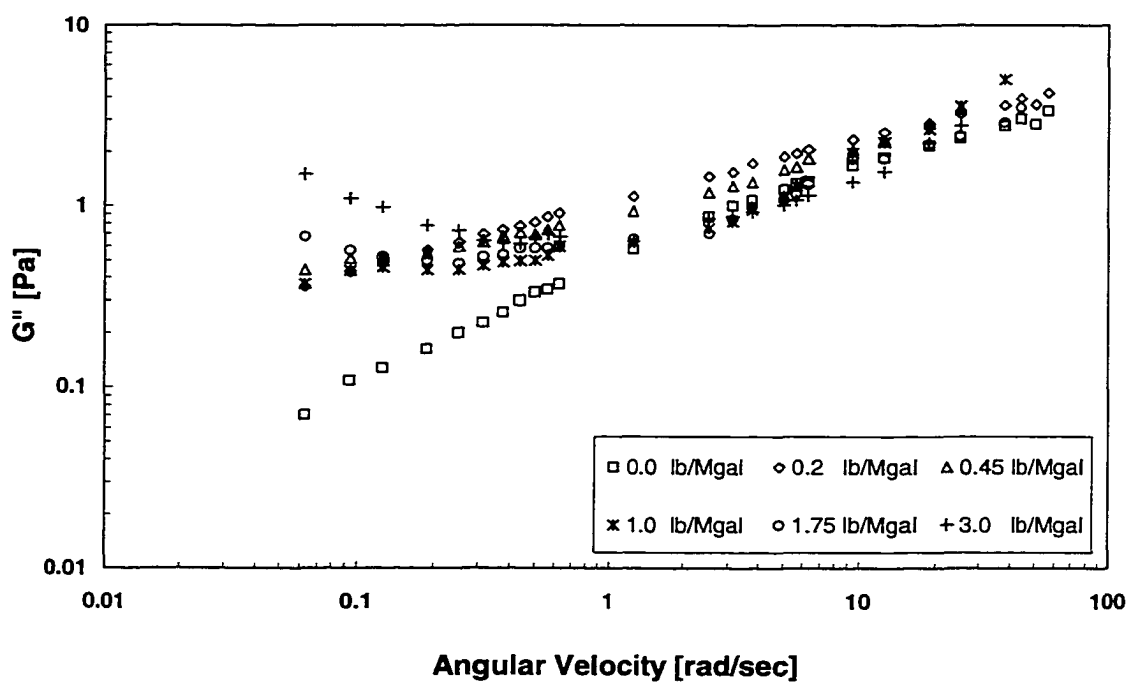


Figure 6.11—Suspension Settling Velocity of 2 ppg Slurry as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 11 (35 lb/Mgal Guar, 75°F)



(a) Elastic Moduli Curve

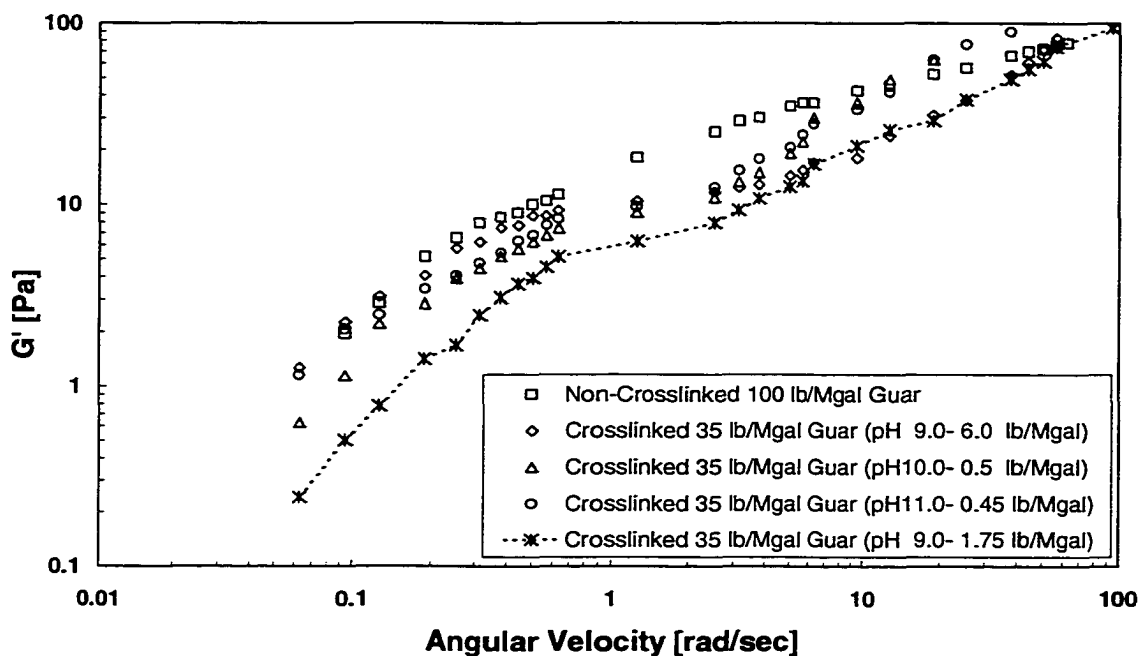


(b) Viscous Moduli Curve

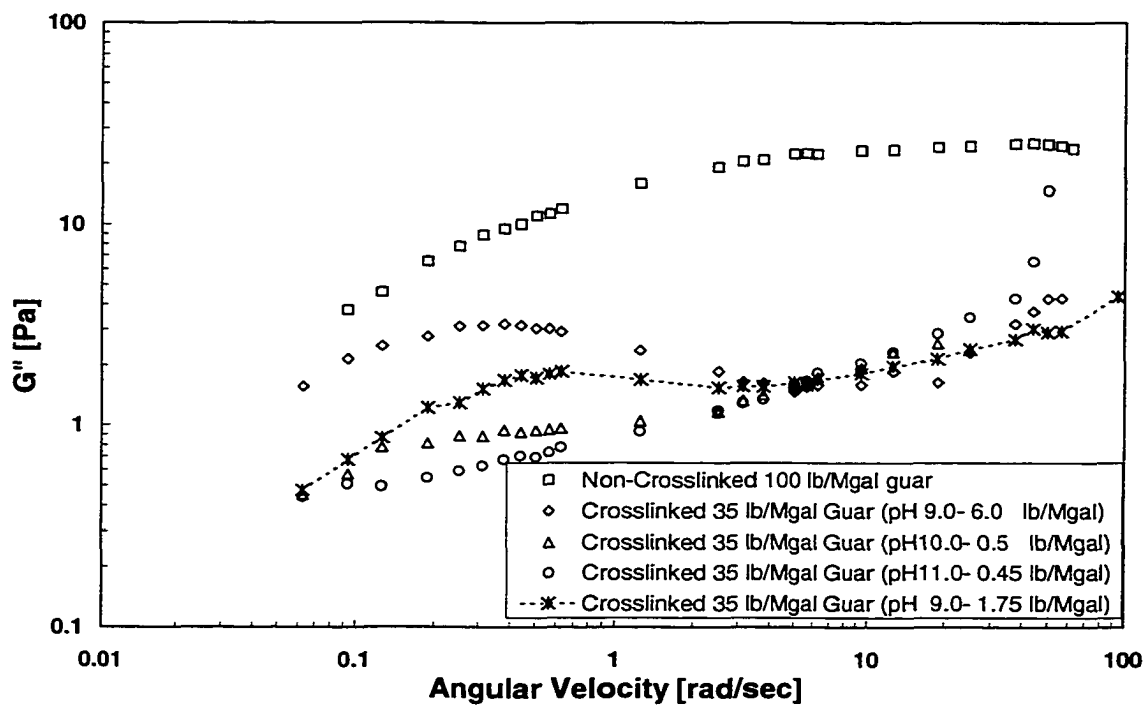
Figure 6.12—Viscoelastic Measurements as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 11 (35 lb/Mgal Guar, 75°F)

The viscoelastic data for the gels whose proppant transport behavior were shown earlier were combined in Figure 6.13. The figure shows that the crosslinked gels that exhibited satisfactory proppant transport had similar G' values throughout the frequency range sampled, particularly at the lower frequencies. These gels, however, had dissimilar G'' values, and hence had different viscosities. This difference implies that viscosity is a poor parameter to correlate with the sand transport characteristics of a viscoelastic fluid. This result further confirms why several researchers were unable to correlate the viscosity of borate-crosslinked gels with their particle suspension behavior. Figure 6.13 further shows the G'' values of the pH 9 crosslinked gel prepared at 1.75 lb/Mgal concentration were higher than that of the pH 10 and 11 crosslinked gels, even though its proppant transport behavior was poorer than that of the other gels. On the other hand, the poor performance of the pH 9 gel at 1.75 lb/Mgal was correctly depicted in its lower G' values. This result further confirmed that the elastic moduli better correlated the sand transport behavior of the crosslinked gels.

Previously, the gel point and the settling velocity criteria available in the literature were shown to be poor predictors of the fluid's ability to satisfactorily transport proppant. Another criteria deserves mention: de Kruijf et al.³⁵ proposed a minimum elastic modulus of 2 Pa at 5 rad/sec to design a fracturing fluid which would exhibit marginally acceptable sand suspension behavior. All samples shown in Fig. 6.13 satisfied this criterion, and yet all did not exhibit acceptable slurry transport. The figure points that if the minimum elastic moduli of the crosslinked guar gels vary from approximately 1 Pa at 0.1 rad/sec to 10 Pa at 1.0 rad/sec, then the fluid would successfully transport sand particles through a fracture.



(a) Elastic Moduli Curve



(b) Viscous Moduli Curve

Figure 6.13—Comparison of the Viscoelastic Measurements of non-Crosslinked and Borate-Crosslinked Guar Gels prepared at different pHs, 75°F

Non-Crosslinked Guar Solution. At a sufficiently higher polymer concentration, a non-crosslinked guar can also show adequate sand transport capabilities. Hence, if our hypothesis is correct, then the elastic moduli criteria can be used to identify the polymer concentration at which a linear guar solution would be able to transport sand through a slot. By comparing the viscoelastic data for non-crosslinked and crosslinked gels, a 100 lb/Mgal guar solution was found to have similar elastic modulus at lower frequencies to that of the crosslinked gel, as shown in Fig. 6.13. Because of the similar elastic moduli, this non-crosslinked guar solution should also exhibit satisfactory slurry transport.

Figure 6.14 shows the sand distribution in the HPS and Figure 6.15 presents the proppant slurry transport through the transparent slot. Figure 6.14 describes the distribution after 5 and 15 min. of a 2 ppg slurry flow in 100 lb/Mgal guar solution. Some settling was seen in the HPS after 5 min., but the settled sand was carried away with the fluid when the slurry flow was continued, as seen in the 15 min. image.

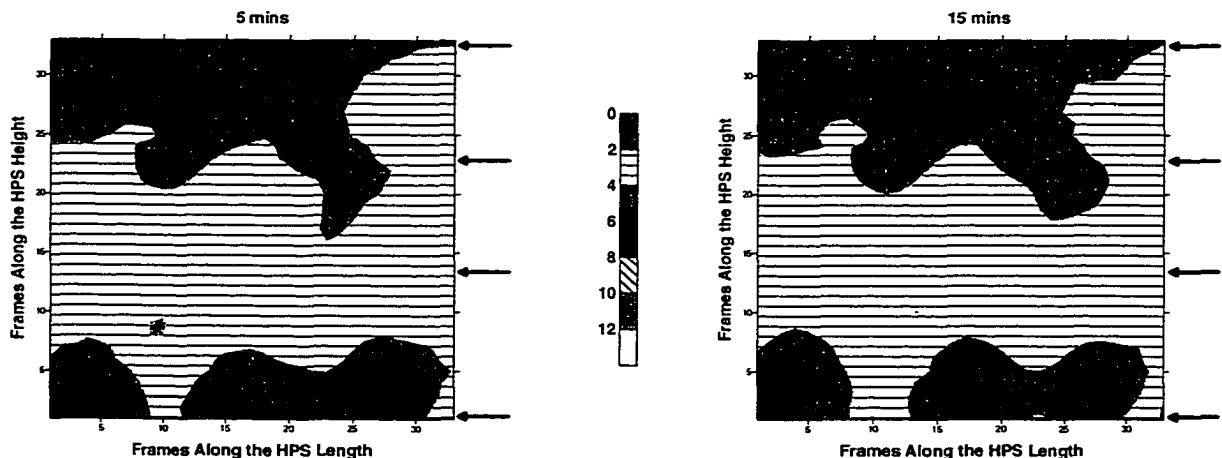


Figure 6.14—Proppant Concentration (ppg) in the HPS after 5 and 15 min of 2 ppg Slurry Transport with non-Crosslinked 100 lb/Mgal Guar, 75°F



**Figure 6.15—2 ppg Proppant Slurry Transport through Transparent Slot with
non-Crosslinked 100 lb/Mgal Guar, 75°F**

Figures 6.14 and 6.15 show that the proppant distribution in the linear guar was similar to that in the crosslinked gels. No settled bed formed during the slurry flow through the transparent slot. Therefore, 100 lb/Mgal guar solution was able to satisfactorily transport sand through the slot models.

This result implies that the entanglement couplings in a polymer describe the proppant transport capability of a polymeric fluid. These couplings are better revealed in the elastic modulus of crosslinked guar gels and non-crosslinked guar solutions. Hence the elastic modulus provides a better method to correlate the laboratory characteristics of a fluid to its proppant transport capability.

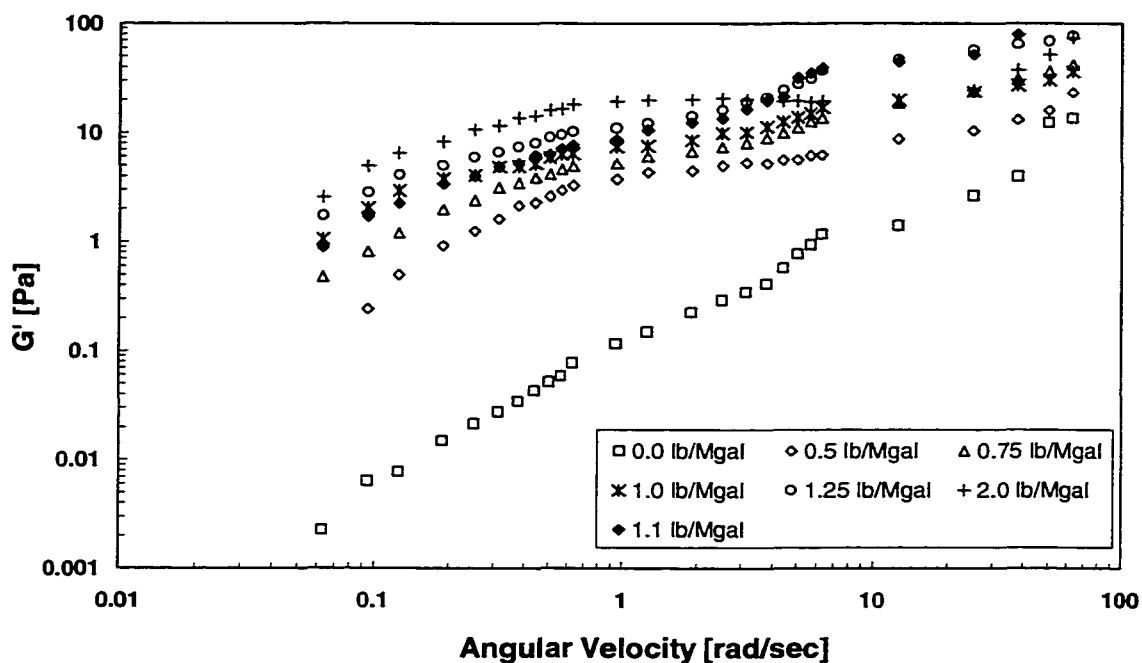
6.2.2 Effect of Elevated Temperature

The elastic modulus criterion described in Section 6.2.1 was developed from the fluid evaluation at ambient temperature. The same criteria should be applicable at elevated temperature. This is confirmed below.

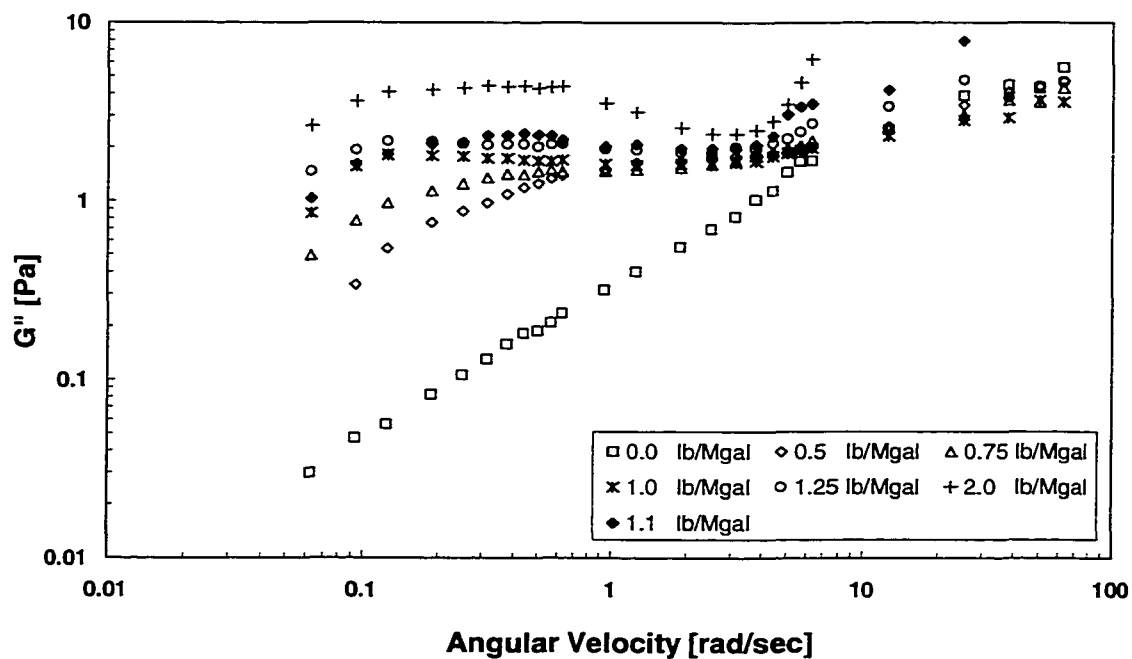
Section 2.2 discussed that the guar polymer degrades at high temperature, so a higher polymer concentration would be required to evaluate the relationship of the elastic moduli with proppant transport behavior. Also, a higher fluid pH would be required to achieve similar borate ions to that at the ambient temperature, as depicted in Figure 2.3. Hence, the elevated temperature evaluations were performed in linear 40 lb/Mgal guar solutions at pH 11.5. These solutions were then crosslinked at different crosslinker concentrations to identify the crosslinker concentration that had similar elastic moduli as that of the gels, which exhibited acceptable proppant transport at ambient temperature.

Borate-Crosslinked 40 lb/Mgal Guar pH 11.5 Figure 6.16 describes the viscoelastic properties and Figure 6.17 presents the suspension settling velocities of the borate-crosslinked 40 lb/Mgal guar gels, pH 11.5, prepared at several crosslinker concentrations at 130°F.

Figure 6.16 displays an increase in the elastic and viscous moduli with an increase in the crosslinker concentration. The viscoelastic behavior of these gels at 130°F is similar to that seen earlier in the samples analyzed at the ambient temperature.



(a) Elastic Moduli Curve



(b) Viscous Moduli Curve

Figure 6.16—Viscoelastic Measurements as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 11.5 (40 lb/Mgal Guar, 130°F)

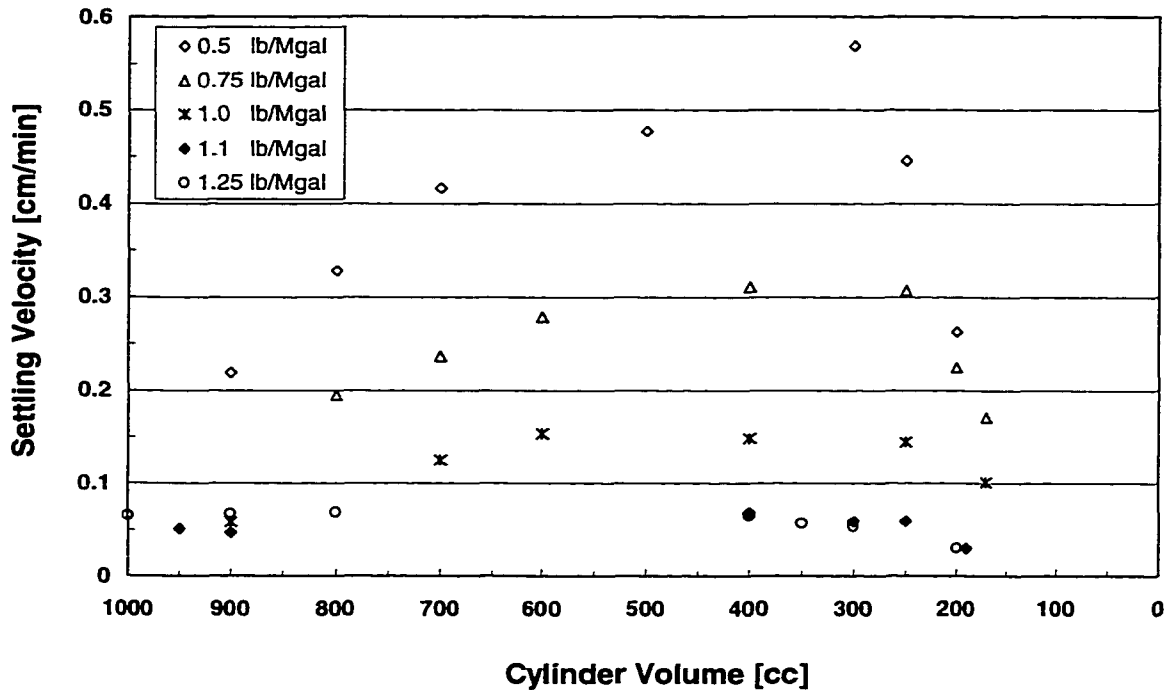


Figure 6.17—Suspension Settling Velocity of 2 ppg Slurry as a Function of Crosslinker Concentration in Borate-Crosslinked Guar, pH 11.5 (40 lb/Mgal Guar, 130°F)

The elastic moduli of the different gels shown in Figure 6.16 were compared with those of the gels in Figure 6.13. The comparison showed that the moduli of the pH 11.5 gel prepared at 1 lb/Mgal were slightly lower and those of the gel at 1.25 lb/Mgal were slightly higher than those of the gels exhibiting acceptable proppant transport. Therefore, the crosslinked gels prepared from 1.0 to 1.25 lb/Mgal were further evaluated in the large-scale facility for identifying the critical crosslinker concentration above which the proppant transport behavior of the crosslinked would be satisfactory at 130°F.

A 2 ppg slurry was prepared in borate-crosslinked 40 lb/Mgal guar gel at pH 11.5 and its proppant transport behavior was observed by changing the crosslinker concentration from 1.25 to 1 lb/Mgal during the experiments. The gel prepared at 1.25

lb/Mgal concentration satisfactorily transported proppant slurry through the transparent slot without exhibiting particle settling. The crosslinker rate was then reduced from 1.25 to 1.1 lb/Mgal, and the slurry flow through the slot was continued. At this lower rate also, the proppant distribution was uniform throughout the slot, as shown in Figure 6.18. The crosslinker rate was further reduced to 1.0 lb/Mgal and the slurry flow continued. The proppant slurry in this gel flowed non-uniformly across the slot height. The slurry was moving slowly near the top and bottom boundaries as compared to that flowing in the middle section of the slot. The slow moving slurry in the slot implied that the gel capability to transport proppant through the slot had begun to decrease at 1.0 lb/Mgal. Therefore, the crosslinker concentration of 1.1 lb/Mgal was the critical value, above which the crosslinked gel would satisfactorily transport proppant through a fracture.

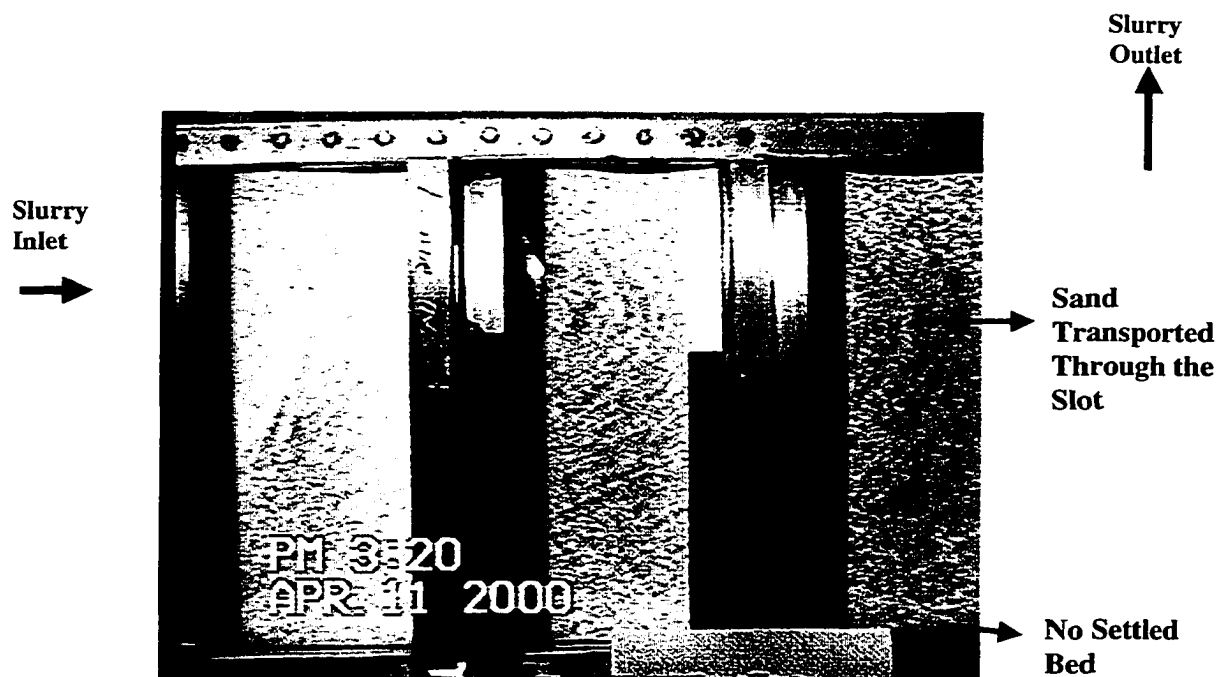


Figure 6.18—2 ppg Proppant Slurry Transport through Transparent Slot with Borate-Crosslinked Guar, 1.1 lb/Mgal Crosslinker, pH 11.5 (40 lb/Mgal Guar 130°F)

The 2 ppg slurry prepared in borate-crosslinked 40 lb/Mgal guar gel crosslinked at 1.1 lb/Mgal concentration was then diverted through the HPS. The proppant distribution in the HPS is shown in Figure 6.19. The figure presents a uniform concentration throughout the HPS with few high sand concentration regions. These concentrated sand regions being suspended in the slurry were transported with the slurry flow as seen in the 15 min. image.

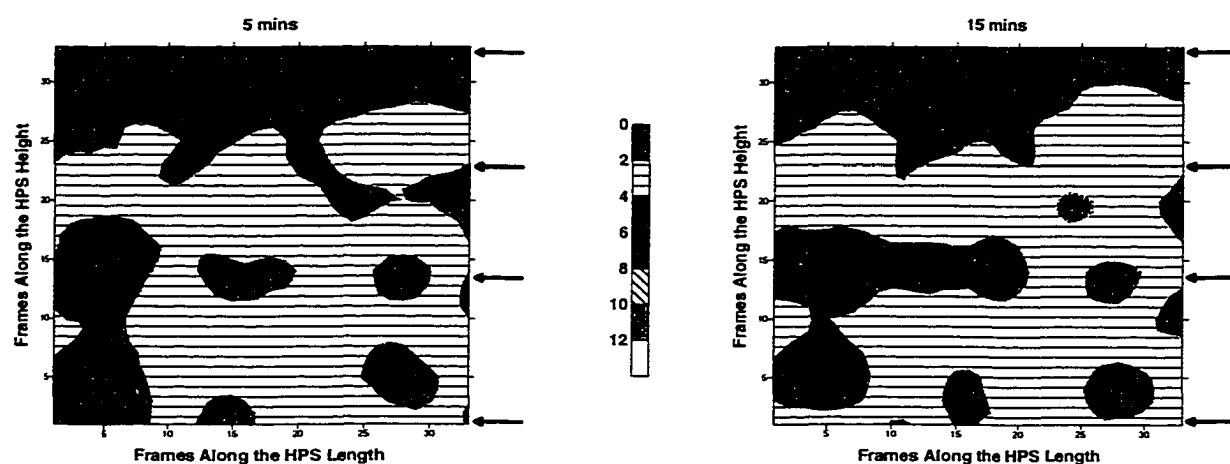


Figure 6.19—Proppant Concentration (ppg) in the HPS after 5 and 15 min of 2 ppg Slurry Transport with Borate-Crosslinked Guar, 1.1 lb/Mgal Crosslinker, pH 11.5 (40 lb/Mgal Guar, 130°F)

Figures 6.18 and 6.19 depict that the gel prepared at 1.1 lb/Mgal crosslinker concentration would satisfactorily transport proppant at 130°F. The satisfactory proppant transport through the slot with this gel, and a good comparison of its elastic moduli at 130°F with those of the gels prepared at ambient temperature further confirms the correlation of the elastic moduli with the proppant transport through a fracture.

6.2.3 Effect of Sand Concentration

Till now, the crosslinked gels were evaluated for their capability to transport proppant slurry prepared at 2 ppg. The fracturing treatments are, however, performed at sand concentrations varying from 2 to 10 ppg, so the fracturing fluid must be designed to transport higher concentration sand slurries. The relationship between the fluid rheology and its sand transport capability, which was developed from studying 2 ppg slurry only, should be validated with higher concentration slurries. Therefore, a sand concentration of 8 ppg was selected to prepare slurries in borate-crosslinked gels at pH 9 and 10. These slurries were then studied for their proppant transport behavior in the transparent slot and HPS.

Borate-Crosslinked 35 lb/Mgal Guar pH 9. A 35 lb/Mgal guar solution was prepared at pH 9 and crosslinked with 6 lb/Mgal concentration to evaluate its capability to transport 8 ppg slurry through the slot models. This concentration was selected because the corresponding pH 9 gel transported 2 ppg slurry through the models.

The borate-crosslinked gel at pH 9 and 6 lb/Mgal crosslinker exhibited again an acceptable performance as the sand particles were uniformly distributed across the slot cross-section. Because of this satisfactory performance, the crosslinker concentration was reduced to 4 lb/Mgal to identify the critical crosslinker concentration above which the gel would transport the proppant slurry. At 4 lb/Mgal crosslinker concentration, the proppant particles began to separate from the fluid and layers of diverse sand concentration became visible in the slot. When the crosslinker concentration was further reduced to 3.5 lb/Mgal, the sand began to separate from the gel and settle to the bottom of

the transparent slot. The diminishing capability of the gels prepared at 3.5 and 4 lb/Mgal concentrations depicted their unsatisfactory performance to carry 8 ppg slurry.

The crosslinker concentration was then increased to 5 lb/Mgal and slurry transport through the slot was again observed. The gel exhibited satisfactory proppant distribution throughout the transparent slot, as shown in Figure 6.20. This experiment indicated that the crosslinker concentration of 5 lb/Mgal provided the minimum concentration for this guar gel to transport 8 ppg slurry through the fracture. Hence the 8 ppg slurry prepared in this gel was pumped through the HPS and the sand distribution obtained. Figure 6.21 depicts the sand concentration in the HPS at 5 and 15 min of slurry flow through the HPS. The figure again shows that the sand concentration was uniform with the sand concentration in the HPS varying from 8 to 12 ppg.

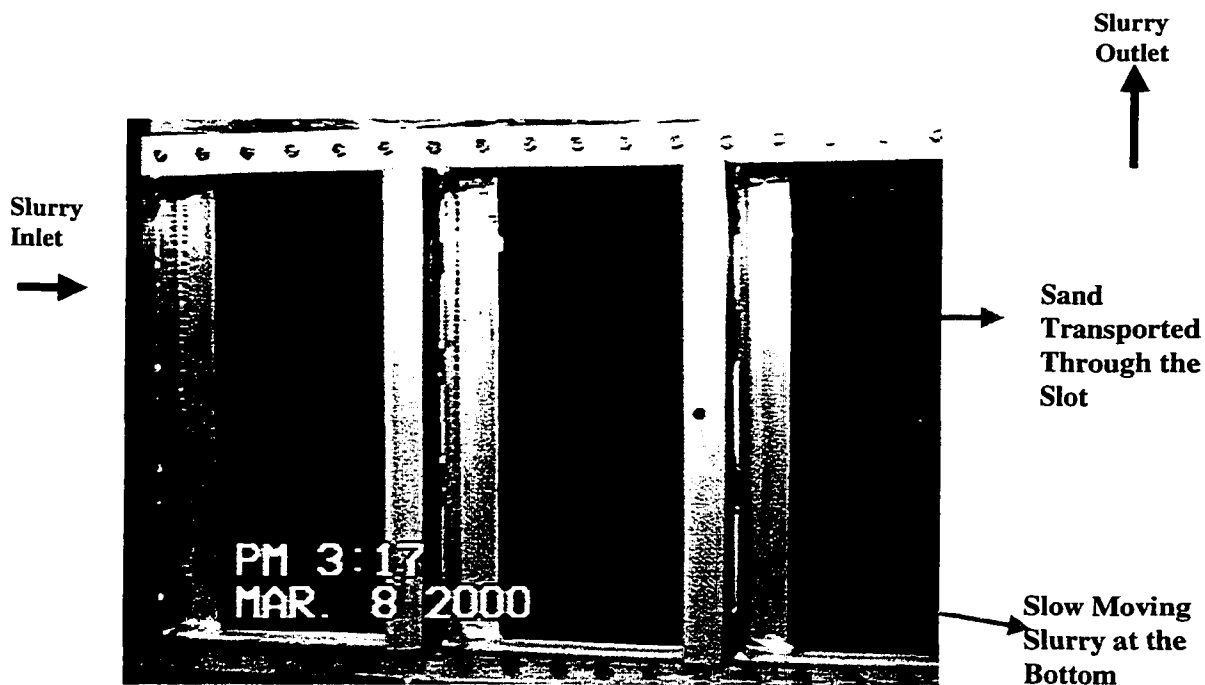


Figure 6.20—8 ppg Proppant Slurry Transport through Transparent Slot with Borate-Crosslinked Guar, 5 lb/Mgal Crosslinker, pH 9.0 (35 lb/Mgal Guar, 75°F)

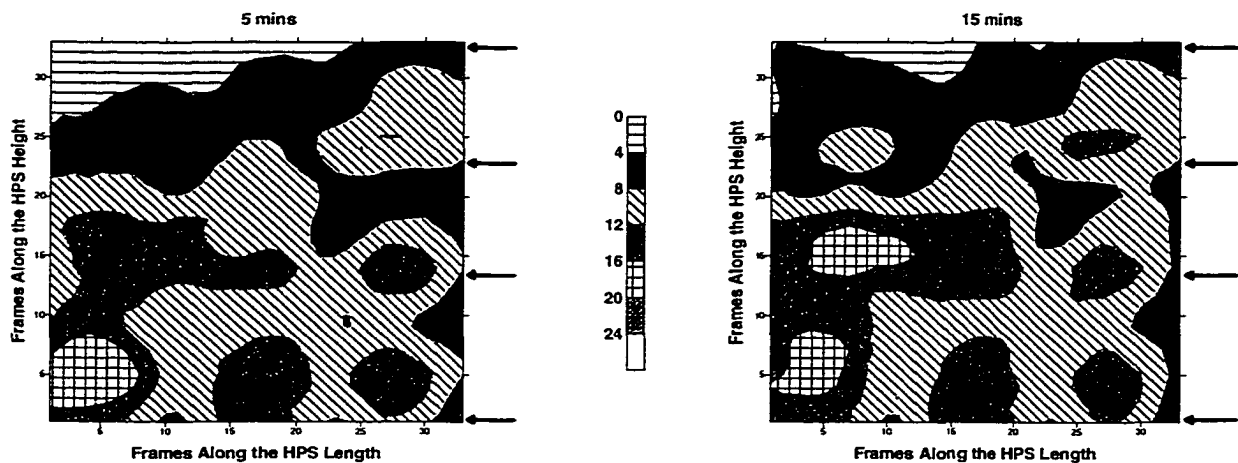


Figure 6.21—Proppant Concentration (ppg) in the HPS after 5 and 15 min of 8 ppg Slurry Transport with Borate-Crosslinked Guar, 5 lb/Mgal Crosslinker, pH 9.0 (35 lb/Mgal Guar, 75°F)

Borate-Crosslinked 35 lb/Mgal Guar pH 10. In another test, 8 ppg slurry was prepared in borate-crosslinked 35 lb/Mgal guar gel at a pH of 10. Section 6.2.1 described that a crosslinker concentration of 0.5 lb/Mgal was sufficient to transport 2 ppg slurry with pH 10 gel, so this concentration might also transport 8 ppg slurry through the slot.

The crosslinked gel at 0.5 lb/Mgal was able to transport 8 ppg slurry through the slot without forming any settled bed in the transparent slot. The slurry flow was however not uniform across the slot height. The slurry flowed slowly in the bottom section of the slot, which indicated that this gel had difficulty keeping the sand in suspension. Therefore, the crosslinker concentration was increased to 0.55 lb/Mgal and slurry transport through the transparent slot was again observed. At this crosslinker concentration, the slurry flow was still non-uniform with slower moving slurry at the slot bottom occupying less region than that seen in the gel prepared at 0.5 lb/Mgal. The proppant transport behavior of the gel improved when the crosslinker concentration was

increased from 0.5 to 0.55 lb/Mgal, but the behavior could improve further. The crosslinker concentration was hence increased to 0.6 lb/Mgal and slurry transport through the transparent slot observed. Figure 6.22 shows the proppant transport through the transparent slot at 0.6 lb/Mgal concentration. The figure shows that the slurry transport was uniform throughout the slot indicating that this concentration was satisfactorily transporting proppant through the fracture. The gel was then diverted through the HPS to obtain a digital image of the sand distribution in the slot model. Figure 6.23 shows the proppant distribution in the HPS at 5 and 15 min of 8 ppg slurry flow with borate-crosslinked guar gel at pH 10. The figure shows the sand distribution to be similar to that seen in Figure 6.21 with pH 9 gel.

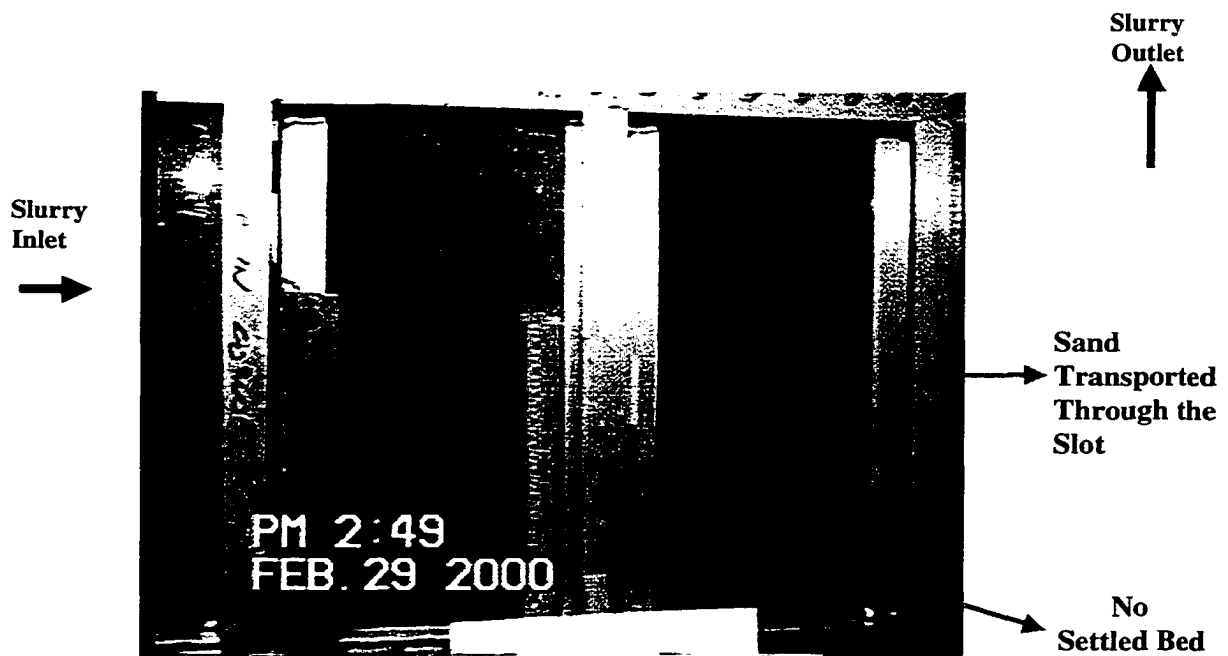


Figure 6.22—8 ppg Proppant Slurry Transport through Transparent Slot with Borate-Crosslinked Guar, 0.6 lb/Mgal Crosslinker, pH 10.0 (35 lb/Mgal Guar, 75°F)

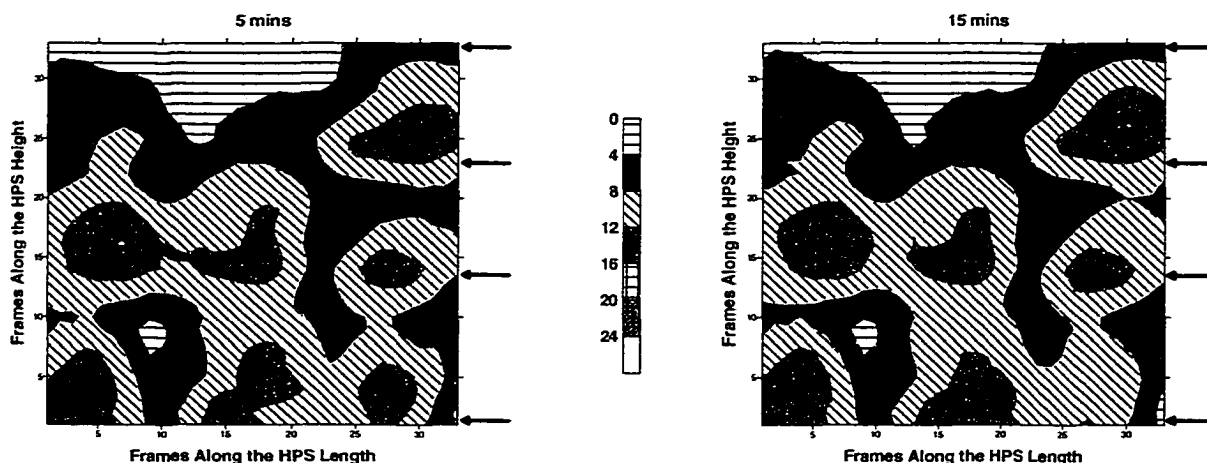


Figure 6.22—Proppant Concentration (ppg) in the HPS after 5 and 15 min of 8 ppg Slurry Transport with Borate-Crosslinked Guar, 0.6 lb/Mgal Crosslinker, pH 10.0 (35 lb/Mgal Guar, 75°F)

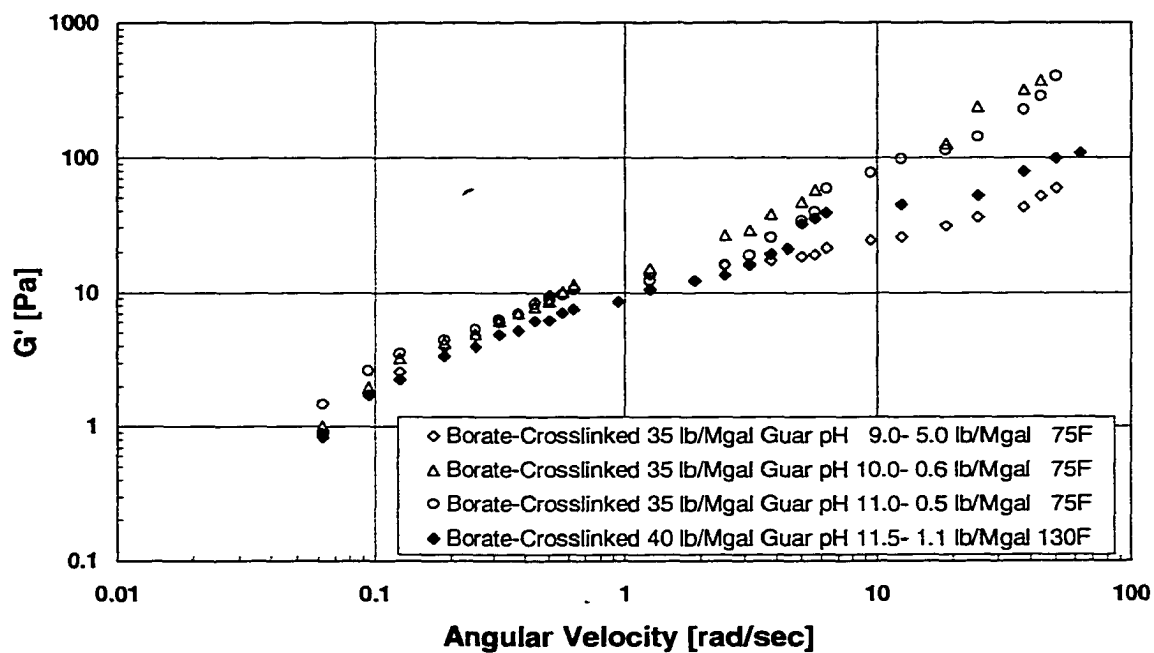
The proppant transport behavior of the borate-crosslinked guar gel prepared at pH 10 showed that whereas a crosslinker concentration of 0.5 lb/Mgal was sufficient to transport a 2 ppg slurry, a higher crosslinker concentration of 0.6 lb/Mgal was required to transport 8 ppg slurry. The higher crosslinker concentration indicates that a higher crosslink density was required to transport higher concentration slurry. This further implies that higher elastic moduli or higher gel strength was required to transport slurry prepared at 8 ppg than that at 2 ppg.

Section 6.2.1 described that the pH 10 gel prepared with 0.5 lb/Mgal crosslinker and pH 11 gel at 0.45 lb/Mgal concentration exhibited satisfactory proppant transport. This observation was confirmed from Figure 2.3, which displayed that these two gels had similar borate ions concentration. Similarly, the satisfactory transport of 8 ppg slurry with 0.6 lb/Mgal concentration in the pH 10 gel would be exhibited in the pH 11 gel at 0.5 lb/Mgal crosslinker concentration. This similarity was also observed in the identical

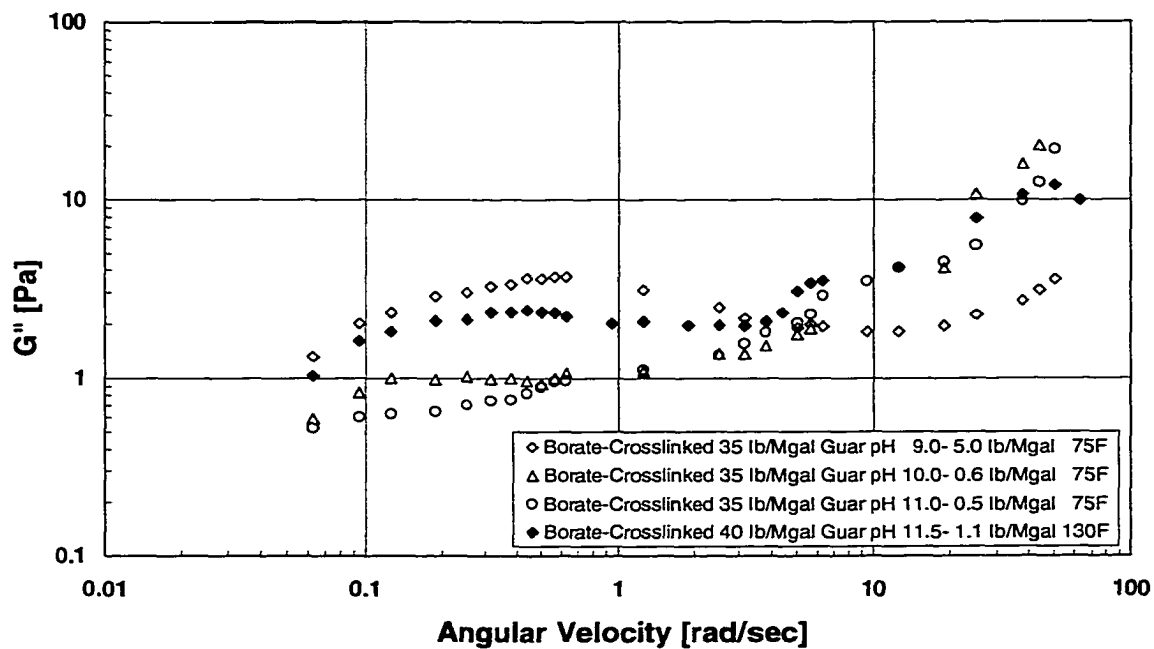
elastic moduli of these gels. Thus, pH 11 borate-crosslinked guar gel prepared at 0.5 lb/Mgal crosslinker concentration would transport 8 ppg slurry through a fracture.

Comparison of the Measured Rheology. The viscoelastic data of the crosslinked gels that satisfactorily transported 2 and 8 ppg slurry at ambient and elevated temperatures were compared. The comparison is shown in Figure 6.24. The figure shows that these gels had similar elastic moduli at lower frequency regions, but dissimilar viscous moduli in the entire frequency range. These gels also had dissimilar API viscosity values, as shown in Figure 6.25. This figure demonstrates that though these gels had similar proppant transport characteristics, they did not have similar viscosities.

The comparison of the rheological properties highlights the poor correlation of fluid viscosity to describe proppant transport behavior. Figure 6.24, on the other hand, depicts that the elastic modulus of these gels were similar, so this modulus describes a better correlation between the fluid characteristics and their sand transport behavior. The gels described in this chapter provided the minimum crosslinker concentration at each pH to satisfactorily transport proppant through a fracture. Beyond this crosslinker concentration, the proppant transport capability of the borate-crosslinked gels would improve. Therefore, the elastic moduli of these gels with the critical crosslinker describe the minimum moduli required to satisfactorily transport proppant through a fracture. The elastic moduli of Figure 6.24 therefore provide the minimum rheological property criteria that can be used by the industry to evaluate the performance of fracturing fluids prior to their use in a stimulation treatment.



(a) Elastic Moduli Curve



(b) Viscous Moduli Curve

Figure 6.24—Comparison of Elastic and Viscous Moduli of Borate-Crosslinked Guar Gels prepared at different pHs

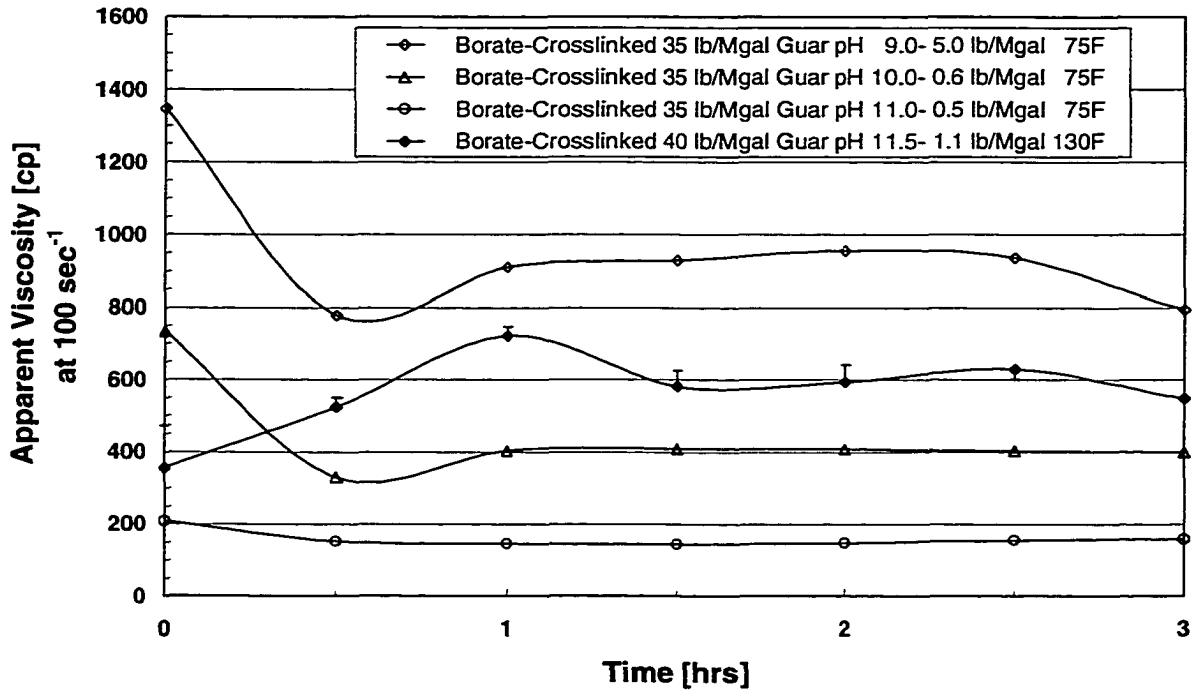


Figure 6.25—Comparison of API Viscosity of Borate-Crosslinked Guar Gels prepared at different pHs

The elastic moduli of the borate-crosslinked gels that satisfactorily transported proppant through the fracture models are given in Table 6.1. The table depicts that these gels have similar elastic moduli at low frequencies. Hence, these moduli were used to calculate average moduli, which were then fitted with an empirical equation given by

$$G' = 0.5 + 20\omega - 8\omega^2 \dots\dots\dots (6.1)$$

where G' is in Pa, and ω is in rad/sec varying from 0.1 to 1 rad/sec.

The elastic moduli given in Table 6.1 and Eq. 6.1 provide the minimum moduli required to satisfactorily transport proppant through a fracture. Hence, these values can be used by the industry to compare and evaluate the crosslinked fracturing fluids for their capability to transport proppant through a fracture. The fluids having elastic moduli

greater than those given in Table 6.1 should satisfactorily transport proppant. Similarly, Eq. 6.1 can be incorporated into fracturing simulators. The minimum elastic moduli given by Eq. 6.1 can be compared to the elastic moduli of several fluids prior to their use in a fracturing treatment and thus, evaluate and select the correct fluid formulation for the treatment.

**Table 6.1-Elastic Moduli of Borate-Crosslinked Guar Gels
Exhibiting Satisfactory Proppant Transport**

Angular Velocity [rad/s]	Elastic Modulus [Pa] of Borate-Crosslinked Guar Gels				Average Minimum Elastic Moduli [Pa]
	35 lb/Mgal Guar, pH 9 5.0 lb/Mgal 75°F	35 lb/Mgal Guar, pH 10 0.6 lb/Mgal 75°F	35 lb/Mgal Guar, pH 11 0.5 lb/Mgal 75°F	40 lb/Mgal Guar, pH 11.5 1.1 lb/Mgal 130°F	
0.063	0.84	1.02	1.48	0.89	1.1
0.094	1.79	1.97	2.59	1.71	2.0
0.126	2.54	3.26	3.53	2.24	2.9
0.188	3.98	4.22	4.39	3.37	4.0
0.251	4.81	4.92	5.28	3.96	4.7
0.314	6.03	6.15	6.30	4.87	5.8
0.377	6.92	6.99	6.92	5.15	6.5
0.440	8.47	7.82	8.16	6.11	7.6
0.503	9.51	8.63	8.86	6.21	8.3
0.565	9.93	10.14	9.65	7.03	9.2
0.628	10.79	11.45	10.50	7.55	10.1
1.257	13.58	15.19	12.19	10.48	12.9

6.3 CONCLUSIONS

The crosslinked guar gels exhibited an increase in the viscous and elastic moduli with an increase in the crosslinker concentration. A critical crosslinker concentration was observed at each pH where the borate-crosslinked guar gel satisfactorily transported slurry through the fracture models. This concentration was a function of fluid pH,

temperature and slurry concentration. The gels prepared with these concentrations exhibited dissimilar API viscosity and viscous moduli. They, however, had similar elastic moduli in the rubbery flow region, i.e. at frequencies below 1 rad/sec. These results imply that the elastic modulus provides a better method than viscosity to correlate the rheological characteristics of a fluid to its proppant transport behavior.

CHAPTER 7

CORRELATION VALIDATION WITH ZIRCONATE-CROSSLINKED GEL

7.1 INTRODUCTION

The results presented in the previous chapters were developed from the study performed on borate-crosslinked guar gel system. After borate-crosslinked gel, zirconate-crosslinked gel is the second most widely used fracturing fluid system.^{17,85,218} The zirconate-crosslinked gels have been successfully used to stimulate oil and gas production around the world.^{2,6,62,125,219} Some examples of the use of these gels are to stimulate gas production from 500 to 1700 MSCFD in Wilcox formation in Webb county in Texas, and to enhance production up to 3500 MSCFD in the Wasatch formation in Duchesne and Uintah counties in Utah.¹²⁵ Therefore, it would be useful to the industry if the results presented on the borate-crosslinked gels are applicable to the other crosslinked gels such as zirconate-crosslinked gels.

It is pointed out in Chapter 6 that the fluid elasticity better correlates with the proppant transport behavior of a borate-crosslinked gel than the fluid viscosity. The same might be true for zirconate-crosslinked gels; the proppant transport behavior of the zirconate-crosslinked gels cannot be explained with viscosity alone. Dawson¹²⁵ observed that a zirconate-crosslinked formulation having 322 cp viscosity exhibited good proppant suspension, whereas another formulation having a higher viscosity of 521 cp exhibited only fair suspension behavior. It is possible that this anomalous behavior can be explained from the elastic moduli of these formulations. Thus, if the elastic moduli

criteria developed for borate-crosslinked gel is indeed valid for zirconate-crosslinked gels, then it would enhance the applicability of the proposed criteria and benefit the industry. This evaluation would also increase our confidence in the elastic moduli criteria. Therefore, the elastic moduli criteria must be confirmed and validated on zirconate-crosslinked gels.

Differences between Zirconate and Borate-Crosslinking. Zirconate-crosslinked gels are different from borate-crosslinked gels. This difference arises mostly from the crosslinking mechanism in the two crosslinked gel systems.

The borate-crosslinking is mostly the association of hydroxyl groups on guar polymer with borate ions in the crosslinker solution. The zirconate-crosslinking is considered to take place through a number of associations. Unlike borate-crosslinking, the hydroxyl groups on guar polymer do not directly bond with the zirconium ions, they bond with the hydroxyl groups bonded to the zirconium crosslinker.¹⁶ The zirconate crosslinking can also take place through covalent bonding of the crosslinker with carboxylate group on the carboxyalkyl derivatives of guar. This mechanism suggests that not just the hydroxyl group but the carboxyl group can also participate in establishing zirconate-crosslinked gels. Thirdly, the zirconate-crosslinking can also occur from an interaction of guar chain with the surface of a colloidal zirconium particle, and not just between the isolated crosslinker species and the guar polymer.⁸⁴ The multiple mechanisms for crosslinking between the zirconate crosslinker and the guar polymer imply that the zirconate-crosslinking is substantially different from the borate-crosslinking.

Furthermore, since the crosslinking with zirconium involves both covalent and hydrogen bonding, the implication of shear on these bonds are also different from that in borate-crosslinking. The bonds established in zirconate-crosslinking take longer time to reheal as compared to those in borate-crosslinked gels, where the bonds rapidly reheal and reform. Because of the longer period for rehealing, the bonding in the zirconate-crosslinked gels is considered irreversible whereas that in the borate-crosslinked gels is reversible.^{10,143}

The fundamental difference between the borate and the zirconate-crosslinked gels implies that it is important to understand the relationship of individual gel system to its proppant transport behavior. Therefore, the elastic moduli criteria developed from the borate-crosslinked gels should be validated with the zirconate-crosslinked gels.

7.2 EXPERIMENTAL PROCEDURE

Fluid Preparation. The fluid formulation for preparing zirconate-crosslinked gel was taken from the mixing instructions given in a SPE paper by Prud'homme.²²⁰

A hydroxypropyl guar solution (HPG) was first prepared in Norman city tap water, using the similar procedure described in Section 3.2 for preparing a guar solution. Here, the solution was prepared at a concentration of 40 lb/Mgal using HPG polymer (Halliburton, Duncan, OK, Part No. 70.15331, Batch H9611-671-C, 11/96).

The HPG solution was allowed to hydrate for one hour, after which the solution viscosity was measured using Model 35 Fann viscometer to confirm the quality of the prepared solution. To the HPG solution, sodium carbonate powder (OCI Chemical Corp. Shelton, CT, Batch No. 0251-1996; TIN 217 CB) was added at a concentration of 5

lb/Mgal of the solution to buffer the HPG solution. The solution was then mixed for 30 minutes to dissolve the carbonate powder.

After buffering the HPG solution, triethanol amine TEA (Equistar Chemicals, Houston, TX, CAS No. 102-71-6, 97% Trihydroxyethylamine) was added as a delaying agent for zirconate-crosslinking of HPG. The solution was further mixed and its pH was checked. Prud'homme suggested the solution would be at pH 9.5. However, the solution prepared in the present study was observed to have pH less than 9.5. So, a sodium hydroxide solution had to be added to raise the HPG solution pH to 9.5. The HPG solution thus prepared was ready for crosslinking with zirconium crosslinker.

TEA Concentration. The TEA concentration in the HPG solution prepared above depends on the crosslinker concentration used and the delay period required in the crosslinking reaction.

Prud'homme²²⁰ recommended adding TEA at a concentration of 0.1 gal/Mgal of the HPG solution. His experimental setup was however different from that used in the present study, as he had formulated the solution for evaluation only in the laboratory and not for field-scale testing, which would be performed in our study. The TEA concentration, therefore, had to be adapted to delay crosslinking during the high shear-conditioning environment used here.

In addition to the TEA concentration, the concentration of zirconium crosslinker required adjustments. Prud'homme²²⁰ described use of 10% zirconium lactate solution as the crosslinker solution added at a concentration of 2.25 gal/Mgal of the HPG solution. He did not evaluate the zirconate-crosslinked gel prepared in his work for proppant

transport behavior. For the proppant transport evaluation, a lower or a higher crosslinker concentration might be required. This concentration had to be identified using the elastic moduli criteria developed in the previous chapters. Furthermore, the crosslinker used in the present research was an aqueous sodium zirconium lactate (Trade Name Tyzor 217, DuPont); this solution though similar to that used by Prud'homme might have different zirconium activity. Hence again, the crosslinker concentration would have to be adjusted to this crosslinker solution. The zirconate-crosslinker concentration would then determine the quantity of the delay agent required. The concentrations of TEA and crosslinker solutions were therefore inter-related. These concentrations were identified by performing preliminary laboratory and large-scale testing.

In addition to identifying the concentrations of the chemicals, the experimental procedures for shear-conditioning and heating the zirconate-crosslinked gels were modified for the proppant transport evaluations performed in this research. This modification also required preliminary testing of the formulations in the laboratory.

Preliminary Testing. The preliminary large-scale test were performed to evaluate the delayed crosslinking, which was monitored through pressure drop across the coiled tubing.

The test was performed by pumping zirconate-crosslinked HPG gels prepared at different TEA and zirconate crosslinker concentrations through 3000 ft length of 1.5 in. coiled tubing. The TEA concentrations were adjusted by mixing it in the HPG solution, and the crosslinker concentration was controlled through injection rate of the crosslinker solution. The resultant pressure drop of the zirconate-crosslinked HPG gel was measured

across the coiled tubing. The gel was also physically checked for crosslinking. The TEA concentration was maintained at two values, 0.15 and 0.20 gal/Mgal, and the crosslinker concentration was varied from 2.5 to 3.5 gal/Mgal.

Figure 7.1 shows the pressure drop across the 3000 ft coiled tubing and percentage increase in the pressure drop of the zirconate-crosslinked gel over that of the non-crosslinked HPG solution. The figure illustrates that the pressure drop decreased with an increase in the TEA concentration or with a decrease in the crosslinker concentration.

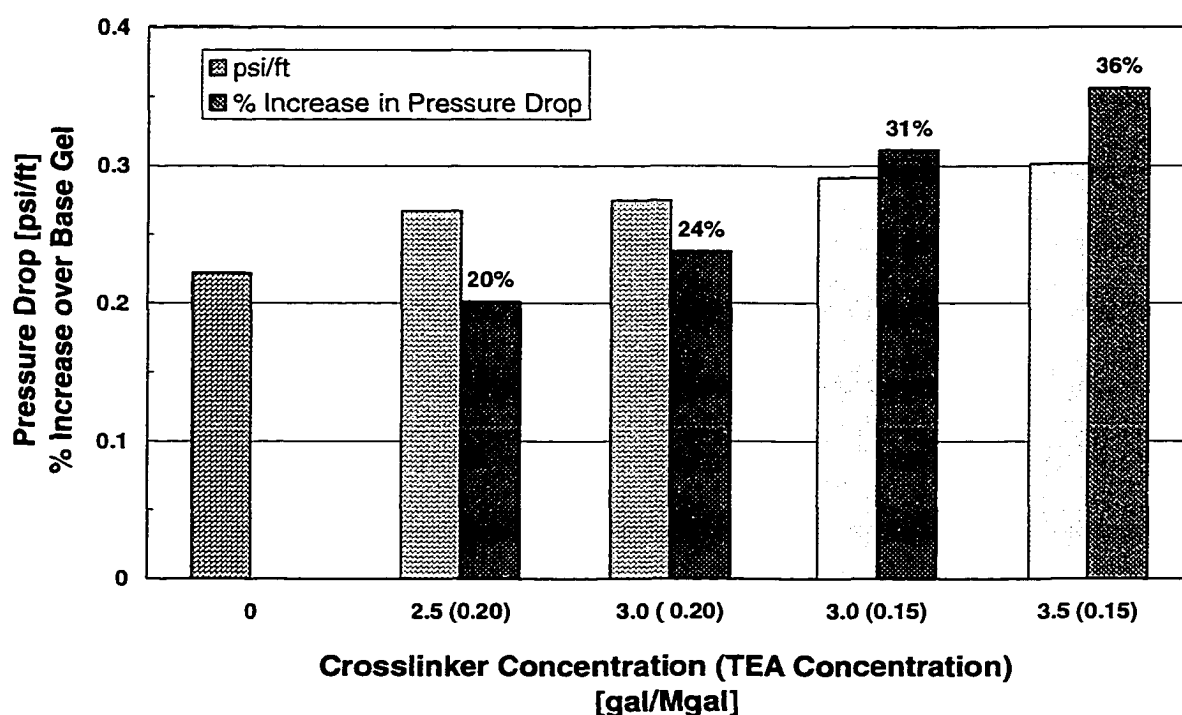


Figure 7.1—Effect of Zirconium Crosslinker and TEA Concentration on Pressure Drop of Zirconate-Crosslinked HPG

The results in Figure 7.1 indicate that the delay agent was behaving in accordance to its function. Its action however was not sufficient to prevent the gel from getting

crosslinked because the pressure drop increased to 36% over that of the non-crosslinked gel. The gels were also observed to be choppy having segregated gel domains, even when the crosslinker concentration was reduced to 2.5 gal/Mgal. Moreover, they did not form a single gel mass, characteristic of a crosslinked gel, even after heating them to 150°F. The choppiness of these gels suggested that the gels were either overcrosslinked or excessively sheared in the shear-conditioning system. Therefore, either the crosslinker concentration was to be reduced or the TEA concentration needed to be increased.

Besides indicating adjustments in the concentrations, this test pointed that the laboratory heating procedure for zirconate-crosslinked gels required modifications. Zirconate-crosslinked gels reheal over a longer period than that taken by borate-crosslinked gels, so they might be irreversibly damaged after being subjected to high shear-conditioning. Furthermore, heating enhances zirconate-crosslinking of the HPG solutions, so these gels could not be subjected to high shear after raising their temperature. So, the laboratory heating procedure used for the borate-crosslinked gels, which are reversibly crosslinked, could not be used for heating the zirconate-crosslinked gels. This meant that after adding zirconium crosslinker to the HPG solution, the solution should be shear preconditioned in the blender and then heated to the formation temperature. Also, the heating period in the laboratory should be representative of the few minutes of heating in the heat exchanger, used in the field-scale testing.

Trials on the available heating apparatus suggested that heating in a microwave oven would simulate the short duration of heating of zirconate-crosslinked gels from ambient to the reservoir conditions. Therefore, after shear-conditioning one liter of the zirconate-crosslinked gel, 250 cc of the gel was transferred to a microwave oven (Model

Spacesaver by Tappan, 750 Watts capacity) for heating. The oven was able to heat the gel to 150°F in just under 2 min.

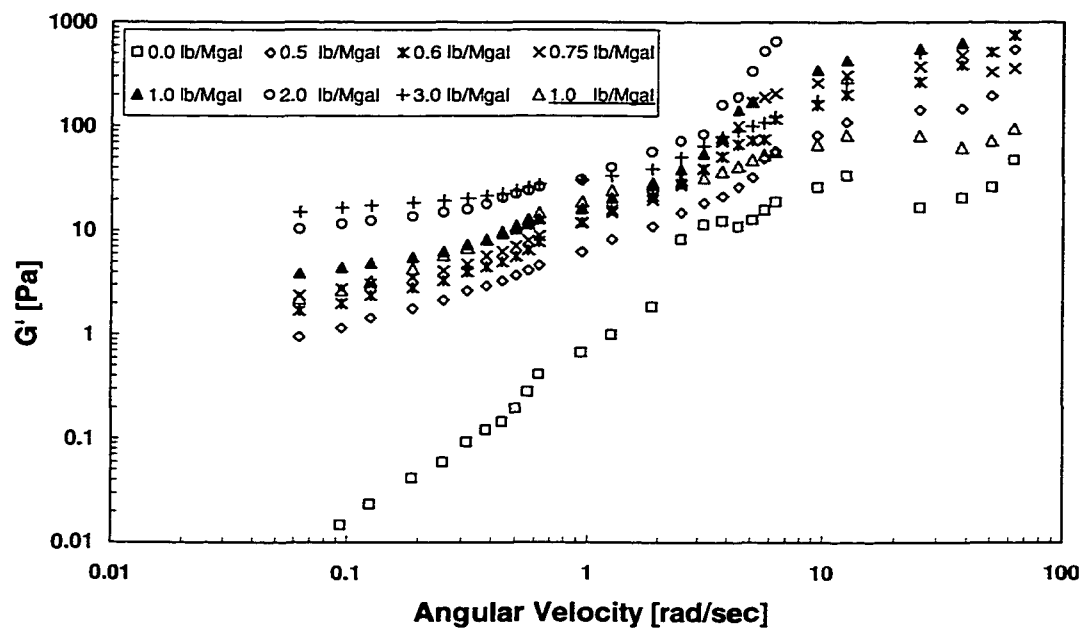
Laboratory Preparation Procedure for Crosslinked Gels. From the preliminary large-scale tests, following procedure was adopted for the zirconate-crosslinked gels.

A 40 lb/Mgal HPG solution was first hydrated and then, the sodium carbonate powder was added at a concentration of 5 lb/Mgal. One liter of this solution was transferred to the Waring blender and the TEA solution was added at a concentration of 0.15 lb/Mgal. This mixture was stirred in the blender, where the solution pH was adjusted to 9.5 and a desired amount of the zirconium crosslinker solution added. The HPG solution containing buffer, delay agent and crosslinker was then shear conditioned in the blender at 1000 sec^{-1} for four min. After shear conditioning the gel, 250 cc of the sheared sample was transferred to the microwave for heating to 150°F. Meanwhile, the cone and plate fixture of the Bohlin rheometer was preheated to the desired temperature for at least 30 min. After heating the zirconate-crosslinked gel sample in the microwave for approximately 100-110 sec, a few cc of the sample was loaded to the rheometer for the viscoelastic measurements.

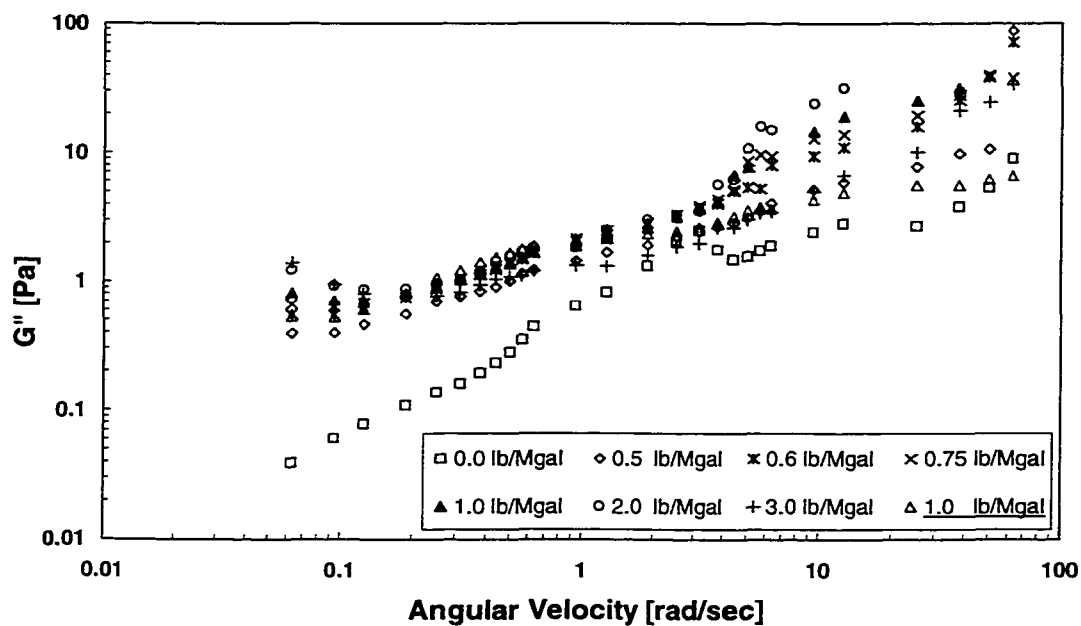
This procedure was used to prepare and characterize the zirconate-crosslinked HPG gels at several crosslinker concentrations.

7.3 RESULTS AND DISCUSSION

Viscoelastic Measurements. Figure 7.2 presents the elastic and viscous moduli of the zirconate-crosslinked HPG gels prepared at several crosslinker concentrations.



(a) Elastic Moduli Curve



(b) Viscous Moduli Curve

Figure 7.2—Viscoelastic Measurements as a Function of Crosslinker Concentration in Zirconate-Crosslinked HPG, pH 9.5 (40 lb/Mgal HPG, 150°F)

(Underlined Legend represents the Sample Sheared in Field-Scale Facility)

The viscoelastic response of the zirconate-crosslinked gel was similar to that of the borate-crosslinked gels described in the last chapter. The elastic and viscous moduli of these gels increased with an increase in the crosslinker concentration. The increase in the moduli could be attributed to an increase in the number of crosslinked sites formed on the HPG polymer when the crosslinker concentration was increased. The figure also shows that the crosslinked gel prepared at 3 gal/Mgal had lower moduli at higher frequencies as compared to those of the other gels. This behavior indicated presence of wall slippage during the viscoelastic measurements of this gel, due to it being over-crosslinked.

The viscoelastic data of the zirconate-crosslinked gels were examined against the data for the borate-crosslinked gels that exhibited satisfactory proppant transport behavior. This comparison pointed that the elastic moduli of the zirconate gels prepared at 0.6 lb/Mgal and higher crosslinker concentrations were of similar order as those in the borate-crosslinked gels. Therefore, the viscoelastic data of the zirconate-crosslinked gels prepared at 0.75 and 1 lb/Mgal were plotted against those of the borate-crosslinked gels.

Figure 7.3 compares the elastic moduli of the zirconate-crosslinked HPG gel with the minimum elastic moduli determined from evaluations on borate-crosslinked guar gels, which were presented in Figure 6.24, Table 6.1 and Eq. 6.1. The figure displays that the zirconate-crosslinked HPG gel prepared at 0.75 gal/Mgal had lower elastic moduli and the gel prepared at 1 gal/Mgal had higher elastic moduli than the data of the borate-crosslinked guar gels which exhibited satisfactory proppant transport. This result pointed that the zirconate-crosslinked HPG gel prepared at 1 gal/Mgal should be able to satisfactorily transport proppant through a fracture.

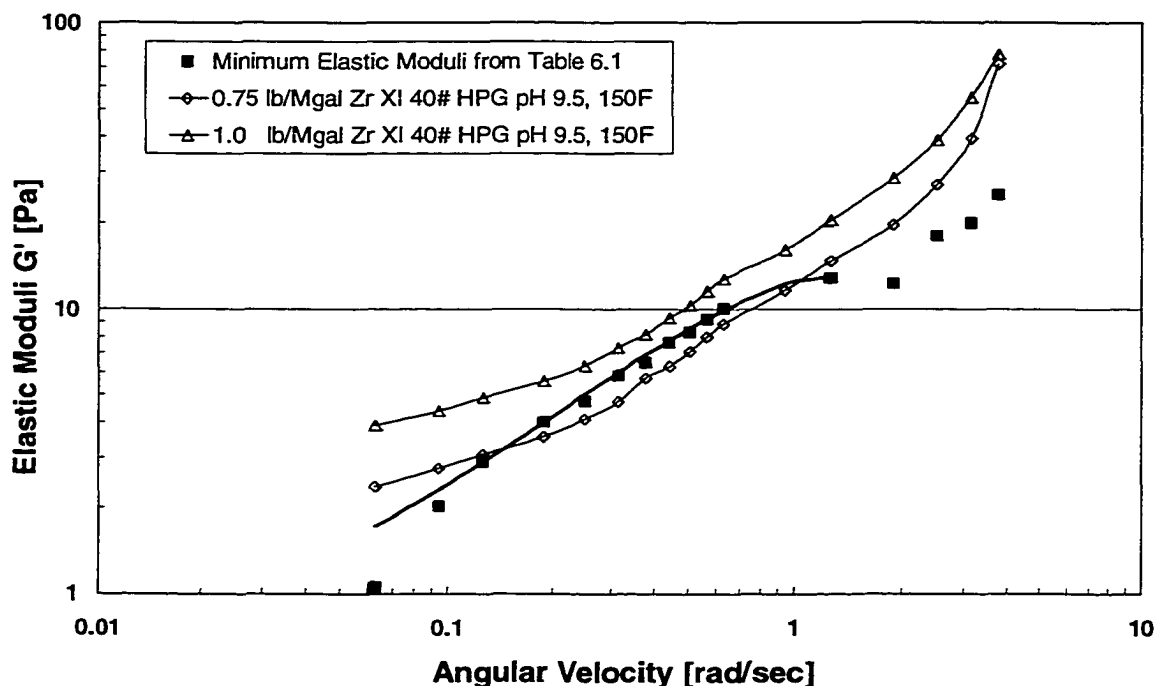


Figure 7.3—Comparison of Elastic Moduli of Zirconate-Crosslinked Gels with the Minimum Elastic Moduli Determined with Borate-Crosslinked Gels

Evaluation of the TEA concentration. Before evaluating the performance of the zirconate-crosslinked gel for proppant transport, two additional preliminary tests were performed to confirm the delaying action of 0.15 lb/Mgal TEA concentration on the crosslinked gel prepared at a zirconium concentration of 1 gal/Mgal. These two tests included a laboratory rheology test for viscoelastic response and a large-scale test for pressure drop across the coiled tubing.

The rheology test was performed to observe drop in the elastic moduli of the crosslinked gels when the TEA concentration was reduced from 0.15 to 0.1 gal/Mgal. The lower TEA concentration could be necessitated because a higher concentration of the delaying agent might delay the crosslinking of the HPG solution even during its flow

through the heat exchanger. This prolonged crosslinking would cause sand to settle in the low shear environment of the exchanger. Because of this undesirable possibility, viscoelastic measurements were made on the zirconate crosslinked HPG gels prepared at 0.6 and 1.0 gal/Mgal crosslinker concentration, and delayed with 0.1 and 0.15 gal/Mgal TEA concentration. The results of these measurements are presented in Figure 7.4.

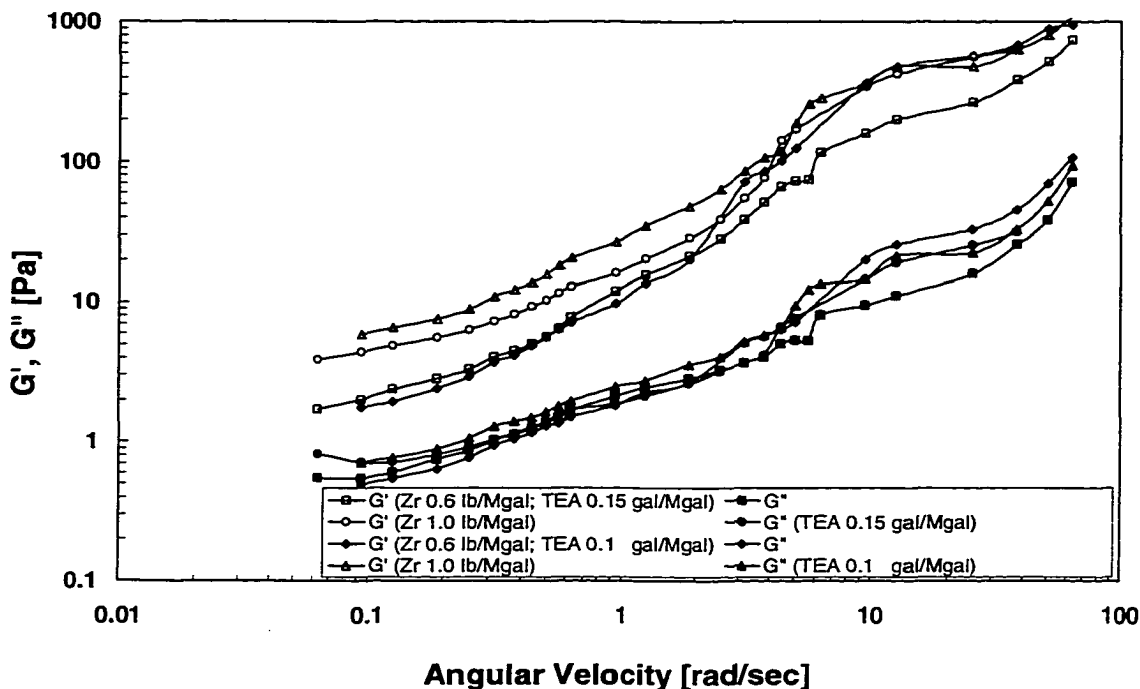


Figure 7.4—Effect of TEA Concentration on the Viscoelastic Measurements of Zirconate-Crosslinked HPG Gels, 150°F

Figure 7.4 shows that there was not much difference in the viscoelastic response of the 0.6 gal/Mgal gel delayed with 0.1 and 0.15 gal/Mgal TEA concentration. The elastic moduli of the 1 lb/Mgal gel were higher when delayed at 0.1 than at 0.15 gal/Mgal TEA concentration. The higher elastic moduli at lower TEA concentration pointed to a higher crosslinking from an insufficient delaying, which might harm the crosslinked gel during

the high shear environment. Thus, the TEA concentration of 0.15 gal/Mgal would be sufficient to delay the zirconate-crosslinking.

The delaying action of the TEA was further confirmed by measuring the pressure drop across the coiled tubing for flow of several zirconate-crosslinked gels delayed with TEA concentration of 0.15 lb/Mgal. Figure 7.5 illustrates the pressure drop per ft of coiled tubing and the percentage increase in the pressure drop over that of the non-crosslinked HPG solution. The crosslinked gel prepared at 1 lb/Mgal exhibited a 14% increase in the pressure drop. This increase was not much and was not different from the increase in the pressure drop observed even when the crosslinker concentration was reduced to 0.3 gal/Mgal. The figure thus points that the zirconate-crosslinked gel at 1 lb/Mgal was sufficiently delayed.

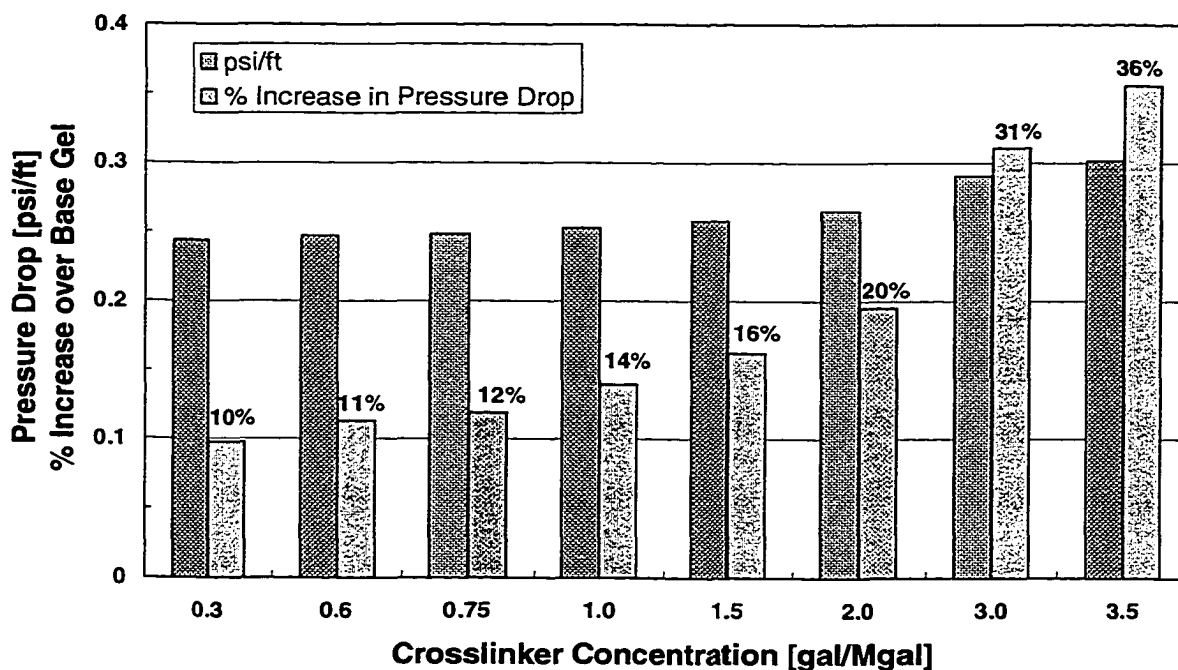


Figure 7.5—Effect of Zirconium Crosslinker Concentration on the Pressure Drop of Zirconate-Crosslinked HPG Gels, 75°F

The pressure drop measurements confirmed that TEA concentration of 0.15 lb/Mgal would delay the zirconate-crosslinking of the HPG solution during the shearing in the coiled tubing. The viscoelastic measurements had earlier indicated that the zirconate-crosslinked HPG gel prepared at 1 lb/Mgal crosslinker concentration should be able to satisfactorily transport proppant through a fracture. Thus, this gel was evaluated for its proppant transport capability using the field-scale facility.

Proppant Transport Evaluation.

Figure 7.6 presents the proppant transport behavior of 4 ppg slurry in zirconate crosslinked 40 lb/Mgal HPG gel having 1 gal/Mgal crosslinker concentration and at 150°F. The figure demonstrates that the sand was uniformly distributed in the transparent slot and that there was no settling in the model. Thus, the gel was able to successfully transport slurry through the slot model.

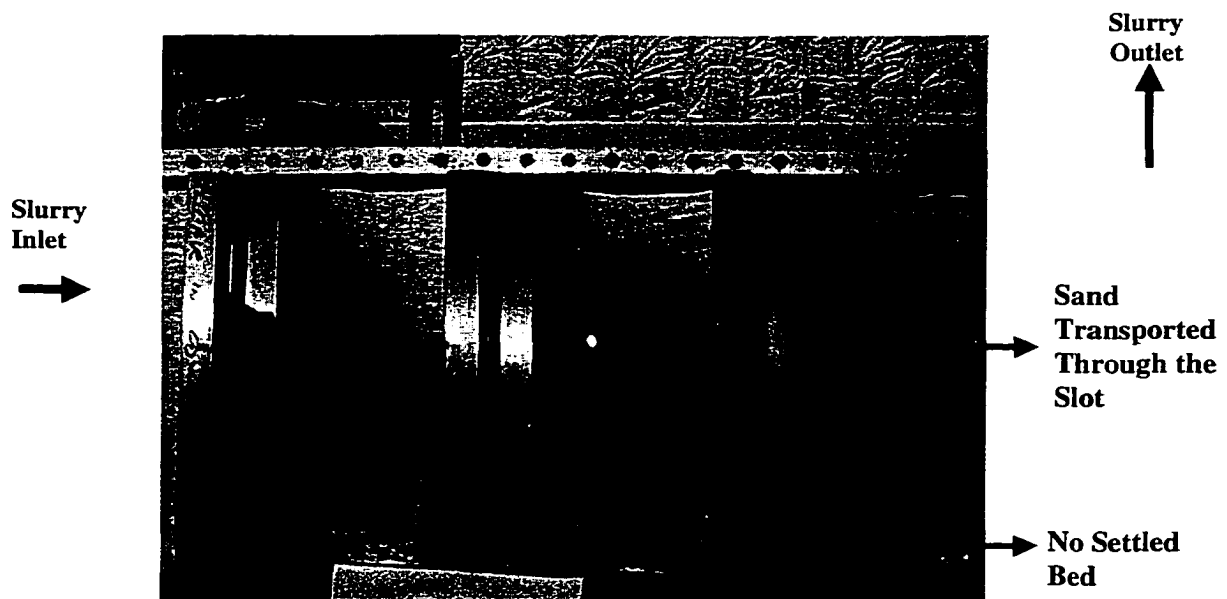


Figure 7.6—4 ppg Proppant Slurry Transport through Transparent Slot with Zirconate-Crosslinked HPG, 1 lb/Mgal Crosslinker, pH 9.5 (40 lb/Mgal HPG 150°F)

A similar response was observed in the HPS. Figure 7.7 describes the proppant distribution after 5 and 20 min of 4 ppg slurry flow through the HPS at 150°F. This figure also demonstrates a satisfactory proppant transport behavior of this gel.

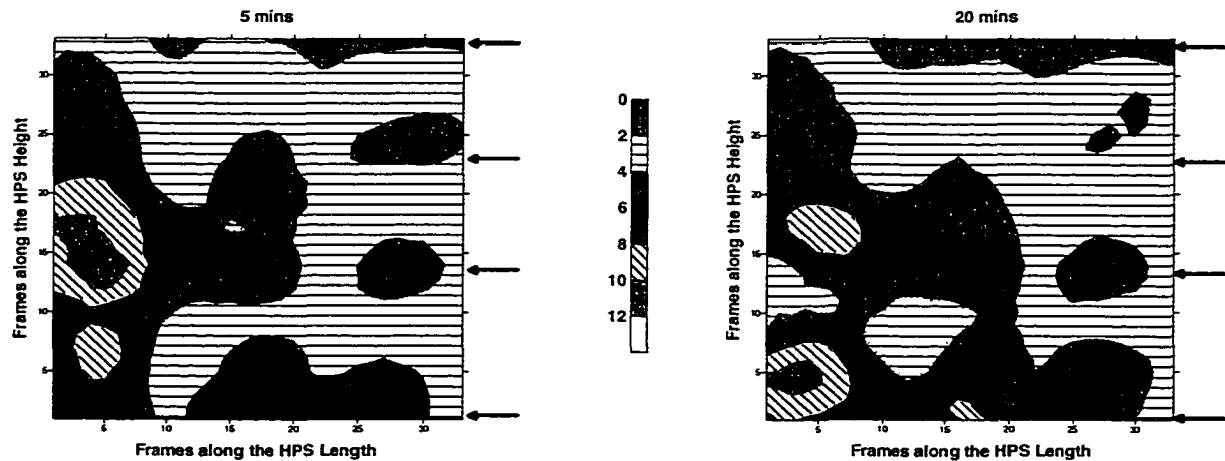


Figure 7.7—Proppant Concentration (ppg) in the HPS after 5 and 20 min of 4 ppg Slurry Transport with Zirconate-Crosslinked HPG, 1 lb/Mgal Crosslinker, pH 9.5 (40 lb/Mgal HPG 150°F)

During the proppant transport evaluation using the field-scale facility, a sample of the non-slurried zirconate-crosslinked HPG gel was captured to analyze its viscoelastic characteristics. It was taken from the test flow loop, just before the gel flowed through the slot models. This sample thus represented the zirconate-crosslinked gel entering a fracture. The viscoelastic response of this sample, prepared in the field-scale test facility, was compared to that of the samples prepared in the laboratory. This comparison is shown in Figure 7.2. The comparison presents an identical response in the gels prepared on two different scales. The similarity of the viscoelastic response of these gels implied

that the procedure used to prepare and heat zirconate-crosslinked gels in the laboratory well represented those on the field-scale test facility.

The successful slurry transport with the zirconate-crosslinked gel prepared at 1 gal/Mgal confirmed and validated the elastic moduli criteria developed in this investigation. Therefore, this criterion was developed from the borate-crosslinked gels and, validated with the zirconate-crosslinked gels.

7.4 FLUID EVALUATION PROCEDURE FOR THE INDUSTRY

The methodology presented in this study can be used by the industry to characterize fracturing fluids in the laboratory for proppant transport behavior prior to their use in field. This methodology is summarized below in a step-wise procedure.

1. Prepare the base fracturing fluid according to its mixing procedure.
2. Add delay agents and buffers to the prepared solution.
3. Transfer one liter of the solution to a Waring blender, which has a tachometer to monitor blender speed or shear rate.
4. Meanwhile, preheat the rheometer and the measuring fixture to the test temperature; set the test parameters for the oscillatory measurement
5. Add the desired amount of crosslinker solution to the base fluid in the blender while mixing the fluid.
6. Continue mixing, and shear condition the crosslinked gel in the blender at the shear rate and shearing duration corresponding to the conditions in the wellbore.

7. After shear conditioning the gel, transfer 250 cc of the crosslinked gel to a beaker and heat it in a microwave oven to the test temperature; the heating time would depend on the wattage of the oven.
8. Transfer the heated gel to the rheometer and perform oscillatory measurements.
9. Compare the measured elastic moduli with those of the gels exhibiting satisfactory proppant transport and establish formulation for fracturing treatment.

CHAPTER 8

IMPLICATIONS FOR SETTLING IN NON-NEWTONIAN FLUIDS

8.1 INTRODUCTION

Experimental data on single particle settling in viscoelastic fluids are traditionally described by a non-dimensional drag correction factor as a function of Weissenberg number.^{46,48,50,51} In the creeping flow region, the drag factor X_e is defined as

$$X_e = \frac{C_d Re_p}{24} = \frac{d_p^2 (\rho_p - \rho) g}{18 \mu_o u_\infty} \dots\dots\dots (8.1)$$

where u_∞ is the single particle settling velocity in an infinite medium, and C_d is drag coefficient. The drag factor X_e is 1.0 for a single particle settling in a viscous Newtonian fluid. In viscoelastic fluids, the drag factor has been observed to be more than as well as less than one.

Though Eq. 8.1 describes the settling of single particle, it can be used to define a similar concept for suspension settling also. If the suspension settling in the crosslinked guar gels was in the creeping flow region, the experimental data for the suspension settling in these gels can be used to determine the drag factor and compare the calculated values with that for the suspension settling in Newtonian fluids. Any deviation of the experimentally determined values from the Newtonian fluid data can then describe the influence of viscoelastic properties on suspension settling in crosslinked fracturing fluid.

Shear-Induced Particle Migration. Section 2.4.1 discussed that particles transported in shear-thinning fluids migrated towards higher shear rate regions; whereas

in viscoelastic fluids, they migrated in opposite direction from high shear to low shear rate regions.¹⁹² This migration has also been shown to be significant across the cross-section in plane Poiseuille flow.^{221,222} If the particles, when flowing as slurries, migrate in slots then the static settling velocity in crosslinked guar gels would not correlate with the slurry transport in a fracture. In such conditions, the suspension settling characterization performed under static fluid conditions would not be relevant for the proppant transport study of fracturing fluids.

The particle migration was not studied in the present research. Tehrani²²³ observed that there was no particle migration in the slurries prepared in the borate-crosslinked gels having fluid pH above 8.2. Since the crosslinked gels in the present research were always prepared at pH values above pH 8.2, the shear-induced migration was probably insignificant here. Tehrani²²³ further concluded that the borate-crosslinked gels having relaxation time of over one second did not display particle migration. The relaxation times of the borate-crosslinked gels prepared were also found to evaluate if particle migration was significant in this research. These values are presented in the following sections.

8.2 SUSPENSION SETTLING IN NON-NEWTONIAN FLUIDS

Equation 8.1 is valid in creeping flow region, so a similar correlation for the suspension settling should be valid in creeping flow. Hence, the flow regime during suspension settling in the borate-crosslinked gels prepared in this research was first determined.

The suspension settling lies in the creeping flow region for particles Reynolds numbers less than 0.2. The particles Reynolds number Re_p for a suspension prepared in a

non-Newtonian fluid is defined as,⁵⁵

$$Re_p = \frac{\rho u_{suspension} d_p}{\mu_o} \dots\dots\dots (8.2)$$

where ρ is the density of the fluid; the density of a crosslinked guar is that of water.

The suspension settling data presented in Chapter 6 showed that the suspension settling velocity decreased with an increase in the crosslinker concentration. The settling velocity in the crosslinked gel will similarly be less than that in the non-crosslinked gel. Equation 8.2 shows that a fluid having lower settling velocity will have a lower particle Reynolds number. Furthermore, the viscosities of the crosslinked gel are higher than that of the non-crosslinked gels; so the particle Reynolds number of the crosslinked gel will be lower than that of the non-crosslinked gel. Therefore, if the suspension settling in non-crosslinked guar solution was in creeping flow region, the same would be true for the crosslinked gels.

The viscosity and suspension settling velocity data of non-crosslinked 35 lb/Mgal guar solution were presented in Table 4.10. From these data and using Eq. 8.2, the particles Reynolds number was calculated to be 2×10^{-3} for 2 ppg slurry settling in 35 lb/Mgal guar solution. This number shows that particles settling in the guar solution was in the creeping flow region, so the suspension settling in all borate-crosslinked 35 lb/Mgal guar gels was also in the creeping flow region.

For suspensions settling in Newtonian fluids, the suspension settling velocity is related to the single particle settling velocity. One widely used correlation is the Richardson and Zaki equation;²²⁴ this and other correlations are summarized in Table 8.1. These correlations relate the suspension settling velocity and single particle velocity to

the concentration of particles in the suspension, with a correction for the wall effects on suspension settling. The correlations are of the form

$$\frac{u_{suspension}}{u_{\infty}} = f\left(\frac{d_p}{D}, (1-\chi)\right) \dots\dots\dots (8.3)$$

where D is the diameter of the cylinder in which suspension settling is observed.

Table 8.1-- Correlations for Suspension Settling under Creeping Flow ($Re_p < 0.2$)

Ref.	$\frac{u_{suspension}}{u_{\infty}} = f\left(\frac{d_p}{D}, (1-\chi)\right)$	$Xe = \frac{I}{f\left(\frac{d_p}{D}, (1-\chi)\right)}$ at $f(0.0104, 0.9167)$
224	$(1-\chi)^{\left(4.65+19.5\frac{d_p}{D}\right)}$	1.53
225	$(1-\chi)^2 10^{-1.82\chi}$	1.69
226	$\frac{(1-\chi)^2}{\left(1+\chi^{1/3}\right) \exp\left\{\frac{5\chi}{3(1-\chi)}\right\}}$	1.99
227 228	$\left(\frac{I}{1+2.35\left(\frac{d_p}{D}\right)}\right) (1-\chi)^{\frac{(5.09+0.2839 Re_p^{0.877})}{(1+0.104 Re_p^{0.877})}}$	1.60
229	$\left(1-1.15\left(\frac{d_p}{D}\right)^{0.6}\right) (1-\chi)^{4.8}$	1.64
230	$0.8(1-\chi)^{4.7}$ for $\chi > 5\%$	1.88
		Average $Xe = 1.72$

Substituting Eq. 8.3 into Eq. 8.1 yielded

$$X_e = \frac{C_d Re_p}{24} = \frac{d_p^2 (\rho_p - \rho) g}{18 \mu_o} \frac{1}{u_{suspension}} f\left(\frac{d_p}{D}, (1 - \chi)\right). \quad (8.4)$$

Since the value of X_e is one for single particle settling in a Newtonian fluid, Eq. 8.4 was reduced to the following equation for suspensions settling in viscous Newtonian fluids

$$\frac{d_p^2 (\rho_p - \rho) g}{18 \mu_o} \frac{1}{u_{suspension}} = \frac{1}{f\left(\frac{d_p}{D}, (1 - \chi)\right)} \dots\dots\dots (8.5)$$

Thus, for suspension settling in a fluid, Eq. 8.5 was written as

$$X_e = \frac{d_p^2 (\rho_p - \rho) g}{18 \mu_o} \frac{1}{u_{suspension}} \dots\dots\dots (8.6)$$

where $X_e = \frac{1}{f\left(\frac{d_p}{D}, (1 - \chi)\right)}$ for suspension settling in Newtonian fluids. Any deviation

from this Newtonian correlation was due to the viscoelastic and non-Newtonian characteristics of the suspending fluid.

The available correlations, shown in Table 8.1, can be used to estimate the drag factor for suspension settling in Newtonian fluid. Using different correlations, the values of X_e were calculated for the experimental conditions of this study: slurry volume fraction, χ , of 0.083 and diameter ratio, d_p/D , of 0.0104. The calculated values are shown in Table 8.1. The table shows the drag factor was in a range from 1.5 to 2, with an average value of 1.72 for suspension settling in a Newtonian fluid under the experimental conditions used in this research.

The drag factor can also be determined from the experimentally measured suspension settling velocities in the crosslinked guar gels prepared at pH 9, 10, and 11,

crosslinked at ambient temperature and pH 11.5 at 130°F. It can then be plotted against the Weissenberg number.

The Weissenberg number is defined as a product of a characteristic time of the fluid and a characteristic strain rate in the flow.¹⁵⁴ The characteristic time of the fluid λ can be determined using the viscoelastic data,^{55,114,154} and the strain rate from Eq. 4.9. Thus, the Weissenberg number is defined as

$$We = \lambda \dot{\gamma} = \lim_{w \rightarrow 0} \left(\frac{G'}{wG''} \right) \frac{u_{suspension}}{d_p(1 - \chi)} \dots\dots\dots (8.7)$$

For the borate-crosslinked gels, the characteristic time was determined using the viscoelastic data at a frequency of 0.09425 rad/sec (approximately 0.1 rad/sec), and the characteristic strain was calculated from the settling data obtained in the glass cylinder. The calculated values of the Weissenberg number are shown in Table 8.2 for the crosslinked gels prepared at the different fluid pHs.

For the drag factor, Eq. 8.6 was slightly modified. In the equation, the fluid viscosity was replaced with the complex viscosity, which was calculated from the viscoelastic data of the crosslinked gels at 0.1 rad/sec. Thus, Eq. 8.6 was modified as

$$Xe = \frac{d_p^2(\rho_p - \rho)g}{18\mu^*} \frac{l}{u_{suspension}} \dots\dots\dots (8.8)$$

Equation 8.8 was then used to determine the drag correction factor for different crosslinked gels. The calculated values are reported in Table 8.2, and are plotted as a function of the Weissenberg number in Figure 8.1. The figure also shows the drag factor for the suspension settling in the Newtonian fluid.

Table 8.2--Drag Factor of the Suspension Settling in Borate-Crosslinked Guar

Crosslinker Concentration [lb/Mgal]	G'	G''	μ^*	λ [sec]	$u_{\text{suspension}}$ [cm/min]	We Eq. 8.7	Xe Eq. 8.8
	[Pa]	[Pa]	[Pa sec]				
at 0.094 rad/sec							
Borate-Crosslinked 35 lb/Mgal Guar, pH 9, 75°F							
0.2	0.18	0.16	2.60	12.04	7.58	26.55	0.11
0.5	0.30	0.26	4.22	12.6	4.14	15.16	0.12
1.0	0.32	0.42	5.64	8.12	1.02	2.41	0.37
1.5	0.58	0.69	9.56	9.04	0.80	2.11	0.28
1.75	0.51	0.67	8.93	7.97	0.49	1.14	0.48
3.0	0.90	1.18	15.74	8.06	0.21	0.50	0.63
5.0	1.79	2.02	28.64	9.42	0.09	0.24	0.84
6.0	2.26	2.14	33.00	11.22	0.07	0.225	0.93
7.0	2.01	1.98	29.89	10.77	0.06	0.18	1.22
8.0	2.00	1.87	29.08	11.35	0.05	0.16	1.54
Borate-Crosslinked 35 lb/Mgal Guar, pH 10, 75°F							
0.2	0.52	0.35	6.63	15.79	0.47	2.16	0.68
0.5	1.14	0.57	13.51	21.22	0.098	0.61	1.59
1.0	2.06	0.97	24.17	22.43	0.028	0.18	3.11
1.5	3.34	1.36	38.27	26	0.022	0.17	2.50
Borate-Crosslinked 35 lb/Mgal Guar, pH 11, 75°F							
0.2	1.10	0.43	12.52	26.96	0.875	6.86	0.19
0.25	0.75	0.32	8.71	24.72	0.044	0.32	5.50
0.5	2.59	0.60	28.24	45.82	0.024	0.32	3.11
1.0	2.49	0.44	26.82	60.56	0.004	0.0705	19.65
Borate-Crosslinked 40 lb/Mgal Guar, pH 11.5, 130°F							
0.5	0.24	0.34	4.42	7.53	0.569	1.25	0.84
0.75	0.81	0.78	11.92	11.09	0.31	1.0	0.57
1.0	2.03	1.56	27.22	13.79	0.153	0.61	0.51
1.1	1.71	1.61	24.91	11.22	0.068	0.22	1.24
1.25	2.82	1.94	36.34	15.49	0.069	0.31	0.84

Table 8.2 illustrates that for each pH, as the crosslinker concentration was increased, the Weissenberg number decreased, whereas the drag factor increased. The decrease in the Weissenberg number could imply a decrease in the elastic characteristic of the fluid when the crosslinker concentration was increased. However, the elastic moduli data shown in Figs. 6.1, 6.7, and 6.11 point an increase in the moduli with an increase in the crosslinker

concentration. The decrease in We with the crosslinker concentration was due to several orders of magnitude decrease in the suspension settling velocity which offset the increase in the elastic modulus, as shown in Table 8.2. Since the effect of the decrease in the suspension velocity overwhelmed the increase in the elastic moduli with an increase in the crosslinker concentration, the Weissenberg number, as defined in Eq. 8.5, is not a correct predictor of the elastic characteristics of the gel.

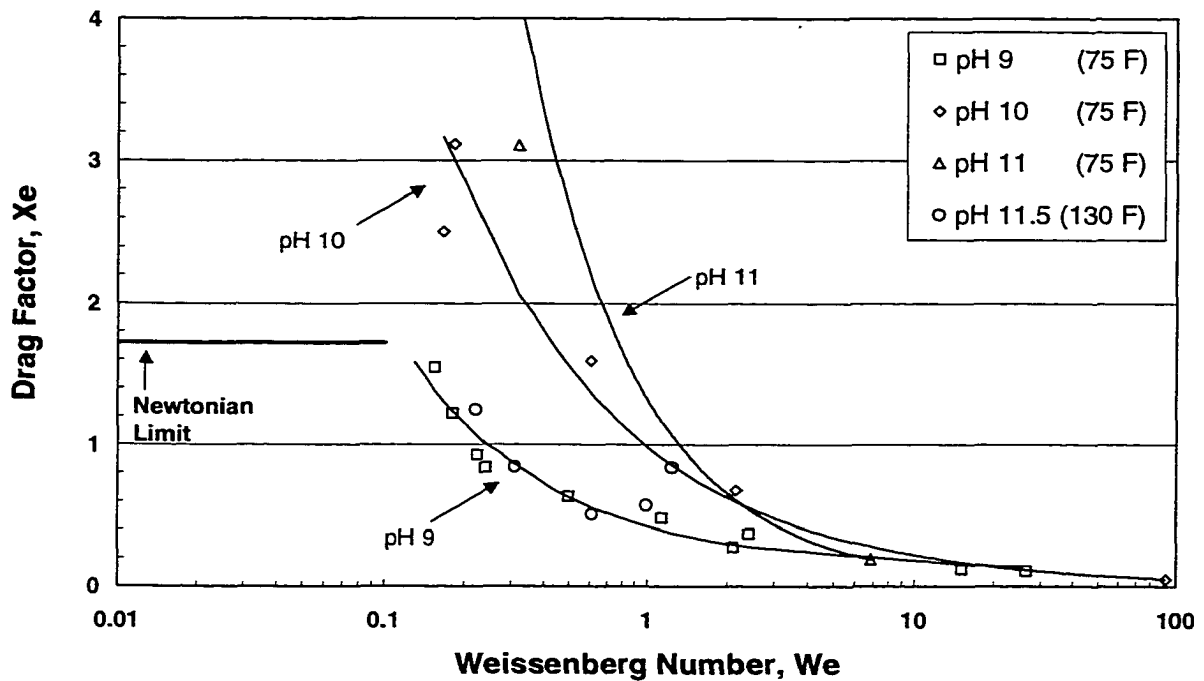


Figure 8.1—Dimensionless Drag Factor as a Function of Weissenberg Number at a slurry volume fraction, χ , of 0.083 and diameter ratio, d_p/D , of 0.0104

As mentioned earlier, Tehrani²²³ observed no particle migration in borate-crosslinked gels prepared above fluid pH 8.2. He also concluded that such gels had relaxation time of over one second. Table 8.2 shows the characteristic time was more than one second for all crosslinked gels prepared in this study. Therefore, the proppant particles probably did

not migrate during the slurry flow through the slot models.

Figure 8.1 displays a decrease in the drag factor with an increase in the Weissenberg number. It suggests that at high We , the drag on the suspensions settling in the crosslinked gels was less than that in a viscous Newtonian fluid. The lower drag means there might have been aggregative sedimentation in the suspensions settling in the crosslinked gels prepared at lower crosslinker concentration.^{54,195} This aggregative sedimentation is similar to the high sand concentration clusters observed in Figs. 6.4 and 6.8 during the slurry flow through the slots.

Figure 8.1 further illustrates that the drag factor did not approach the limiting value at low We in Newtonian fluids. However, the drag factors were higher at low We values, in the gels prepared at higher crosslinker concentrations. This higher drag on the suspensions was due to a higher density of the crosslinked networks formed in the gels at higher crosslinker concentrations. Since the higher crosslinked density would impart more elasticity to the gel, the influence of fluid elasticity was exhibited in a higher drag factor, but was not described with a lower We . The Weissenberg number was therefore unable to correctly describe the characteristic behavior of the crosslinked gels, and is not sufficient to describe the suspension settling in the viscoelastic fluids.^{55,196}

Figure 8.1 suggests that at a given We , the drag correction factor was not similar in the crosslinked gels prepared at all pHs. It shows that the pH 9 gel at 75°F and pH 11.5 gel at 130°F had lower X_e than the other two gels. Similarly, Table 8.2 shows that the characteristic time λ of these gels was lower than that of the pH 10 and 11 gels. These differences suggested that the crosslinked networks established in the gels were dissimilar which contributed to different drag on the suspensions settling in them.

Solomon and Muller⁵¹ observed different drag factors for single particle settling in three Newtonian elastic fluids, and they described this difference to dissimilarities in the molecular weight and extensibility of the fluids. They studied the drag in these fluids by changing the polymer and solvent characteristics of the fluid. In our study, the solvent and the base polymer were kept identical, but the crosslinking between the polymer chains was changed. The drag factors were different in our study because the dissimilar crosslinked gels caused the molecular weight between the crosslinks and possibly the chain conformation to differ.

The results presented in this study imply that the molecular architecture of crosslinked gels plays an important role in the suspension behavior of these gels. Further work is needed to better understand the difference in the crosslinked networks formed in the gels prepared at different pHs.

CHAPTER 9

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

9.1 CONCLUSIONS

1. The crosslinked gels exhibiting satisfactory proppant transport behavior had dissimilar viscosity and viscous moduli. These gels, however, had similar elastic moduli at low frequencies. The elastic modulus is, therefore, a better property than viscosity to correlate the rheological characteristics of a fluid to proppant transport through a fracture.
2. The suspension capability of crosslinked gels was observed to be due to an increase in the fiber density across these gels. With an increase in the crosslinker concentration, the borate-crosslinked gels exhibited an increase in the density of the gel network as seen in the AFM images, and an increase in the elastic and viscous moduli as seen from the rheology characterization. Consequently, the capability of the gel to suspend and transport sand particles through slot models improved.
3. "The gel point criterion" describing the maximum in viscous modulus and "The settling velocity criteria" describing the elastic modulus are observed to be insufficient to describe proppant transport characteristics of borate-crosslinked guar gels.
4. Crosslinked guar gels exhibit a critical crosslinker concentration above which the gels satisfactorily transport proppant through a fracture. This concentration is a function of the fluid pH, temperature, and slurry concentration.

5. Similarly, there is a critical settling concentration of guar above which the settling velocity of suspension in the non-crosslinked guar solution decreases considerably with an increase in the polymer concentration. This critical concentration is analogous to the overlap concentration of a polymeric solution.
6. This study proposes a minimum elastic moduli criterion for satisfactory proppant transport through a fracture. This criterion was developed from rheological characterization of borate-crosslinked guar gels at ambient and elevated temperatures. It was then validated with characterization of zirconate-crosslinked gels at conditions different from those used in its development. The criterion was, however, evaluated at fracture shear rate of 20 sec^{-1} in the HPS and 185 sec^{-1} in the transparent slot, and at Reynolds numbers, based on the viscosity of linear 35 lb/Mgal guar, of 4 in the HPS and 50 in the transparent slot models.
7. This study also proposes a step-by-step procedure to evaluate a fracturing fluid for its capability to transport proppant through a fracture.
8. The rheological criterion proposed in this investigation was developed from comprehensive evaluations performed in the laboratory and on a field-scale test facility. This criterion is based on the elastic moduli evaluated at reservoir conditions and provides the minimum value required for satisfactory proppant transport. It is given in a tabular form in Table 6.1 and an empirical equation in Eq. 6.1. The criterion can be used by the industry to develop and evaluate new and existing fluids at test conditions prior to their use in stimulation treatments, and to demonstrate the effectiveness of these fluid formulations for fracturing application.

9.2 RECOMMENDATIONS FOR FUTURE WORK

1. The present investigation was performed using guar based polymeric systems because guar is the most popular fracturing fluid formulation used today. Recently, surfactant based fracturing fluids were introduced in the industry; so it would be useful to evaluate the elastic modulus criteria proposed in this study on the surfactant based fluids.
2. The elastic moduli criterion presented in this study is based on the evaluations made using a fracture model. This model is a simplification of a real fracture. Further evaluation of the proposed criterion on actual fracturing treatments would help to validate its application to the industry.
3. Chapter 8 showed that the dimensionless drag factor of the crosslinked gels prepared at different pHs was not similar and that the Weissenberg number was unable to correctly depict the characteristic behavior of the crosslinked gels. Further work is needed to better understand the difference in the crosslinked networks formed in the gels prepared at different pHs.
4. Chapter 5 described the Atomic Force Microscopy (AFM) measurements on the dried samples of the crosslinked guar. The dried samples do not correctly depict the actual structure of the crosslinked gel under fracture flow conditions. An improved method of visualization of the structure formed in these fluids under the realistic field conditions would help to further understand their suspension characteristics.
5. The proppant settling data presented in Chapter 6 showed non-uniform settling velocities under static fluid conditions. The suspension settling velocity first

increased and then decreased. A more comprehensive investigation is required to understand this non-uniform settling in viscoelastic fluids.

6. Since the elastic modulus is observed to be a better indicator of the proppant transport capability of a fracturing fluid, the fluid elasticity should be incorporated into the fracture simulator. This will help to provide a more accurate prediction of the fracture geometry formed during a fracturing treatment.

CHAPTER 10

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APPENDIX A

VISUAL BASIC MACROS

A.1. Macros to Analyze Intensity Data from Nine Cameras of the Vision System

A.1.1 Macro Processes Light Intensity Data from the LEDs on the HPS Vision System for Sand Concentration Calculation

Sub Macro_1_Intensity_Concentration_for_All_Cameras_Macro()

' Intensity_Concentration_Macro Macro

' Macro takes data from each camera and opens the camera data file,

' Picks up and averages data from two frames: one from every 10th sec frame and other from the row after the 10th sec frame

' Calculates average intensity values for each frame and sand concentration values for each camera

' Requires that I enter only the TEST DATE in mmdd format

' Macro 1. Copies the vision data for each camera and pastes on a new workbook where

' all data from all cameras is stored as "vision mmdd 2000 all cameras.xls" file

' 2. Plots average intensity and sand concentration values from each column of the HPS cameras

Application.ScreenUpdating = False

' Creates a new workbook and names it as "vision mmdd 2000 all cameras.xls" file

' Saves the created workbook in above name at u:\test99-00\proppant transport

Workbooks.Add

' Enters the test date

testdate = InputBox("enter the test date in 'mmdd' format", "test date")

directory1 = "U:\Test99-00\proppant transport\"

bookallcameras = "vision " & testdate & " 2000 all cameras.xls"

AllCamerasData = directory1 & bookallcameras

ActiveWorkbook.SaveAs FileName:=AllCamerasData, FileFormat:=xlNormal

' Enters the test date and goes to the sub directory where the vision raw data is stored under testing date folder

directory2 = "test" & testdate & " 2000"

directory = directory1 + directory2

' Selects each camera one by one; opens the camera data file,

' Picks up and averages data from two frames: one from every 10th sec frame and other from the row after the 10th sec frame

' Calculates the average intensity values for each frame and sand concentrations for each camera

For i = 1 To 9

ChDir directory

BookOpen = "CAM" & i & ".DAT"

bookclose = "Camera " & i & ".xls"

Workbooks.OpenText FileName:=BookOpen, Origin:=xlWindows, StartRow:=1, _

DataType:=xlDelimited, TextQualifier:=xlDoubleQuote, Tab:=True, Comma:=True, Space:=False

```

'       There are two sheets in each camera file; one is designated by "CAM" and it contains the raw data
'       from the vision system; other is designated by "Camera" and it contains selected raw data from
'       every ten seconds

n = 1      'Frame count
m = 6      'First row in the "Camera" sheet
b = 2      'first column of the vision data in CAM sheet
c = 122    'Last column of the vision data in CAM sheet
d = 125    'Column that contains the intensity values averaged from 121 fibers of each camera

'       Selects the CAM sheet and rename it so that a common name can be used for macro operation

ActiveSheet.Select
    With Selection
        ActiveSheet.Name = "Sheet1"
    End With

'       Adds a blank sheet for managing data from every 10th sec frame and gives it a common name for
macro operation

Sheets.Add
ActiveSheet.Select
ActiveSheet.Name = "Sheet new"

'       Fun begins, the macro selects every 10th sec vision data

For a = 6 To 50000 Step 27 'Number of the source row in the row data
    If IsEmpty(Sheets("Sheet1").cells(a, 2).Value) Then GoTo 20 'checks for vision data
    Sheets("Sheet new").cells(m, 1).Value = n

'       As there is a fluctuation in vision data analyzed by vision system;
'       Averaging the data for each fiber with two consecutive frames

    For j = 2 To 122
        apone = a + 1
        Sheets("Sheet new").cells(m, j).Value = (Sheets("Sheet1").cells(a, j).Value +_
            Sheets("Sheet1").cells(apone, j).Value) / 2
    Next j

'       Average the intensity values from 121 fibers of each camera
    Sheets("Sheet new").cells(m, d).Select
    ActiveCell.FormulaR1C1 = "=average(RC[-123]:RC[-3])"
        m = m + 1      'moves to the next row in 'Camera Sheet
        n = n + 27      'moves to the next frame
Next a                'repeats the process for all data of the selected camera

20

'       Inserts a column in "camera" sheet for placing time for each 10 sec on the data

Columns("A:A").Select
Selection.Insert Shift:=xlToRight
Range("A5").Select
Selection.Font.Bold = True
ActiveCell.FormulaR1C1 = "Time (sec)"
Columns("A:A").ColumnWidth = 10.14

```

```

Range("A6").Select
ActiveCell.FormulaR1C1 = "0"
Range(cells(7, 1), cells(m, 1)).Value = "=τ[-1]c+10"

```

Writes and manages the average intensities & sand concentration for each frame and all fibers

```

Range("DV4").Select
Selection.Font.Bold = True
ActiveCell.FormulaR1C1 = "Average Intensity"
Columns("DV:DV").ColumnWidth = 17.43

```

```

Range("DW4").Select
ActiveCell.FormulaR1C1 = "X"
Selection.Font.Bold = True
With Selection
    .HorizontalAlignment = xlCenter
End With

```

```

Range("DW2").Select
Selection.Font.Bold = True
With Selection
    .HorizontalAlignment = xlCenter
End With
ActiveCell.FormulaR1C1 = "Io"
With ActiveCell.Characters(Start:=1, Length:=1).Font
    .Name = "Arial"
    .FontStyle = "Bold"
    .Size = 10
End With
With ActiveCell.Characters(Start:=2, Length:=1).Font
    .Name = "Arial"
    .FontStyle = "Bold"
    .Size = 10
    .Subscript = True
End With

```

```

Range("DX2").Select
ActiveCell.FormulaR1C1 = "=R[4]C[-2]"      'initial intensity value is vision data at 0 sec
Range("DX4").Select
ActiveCell.FormulaR1C1 = "Sand Concentration"
Selection.Font.Bold = True
Range("DX5").Select
ActiveCell.FormulaR1C1 = "lb/gal"
mminusone = m - 1

```

Calculates normalized intensity and then sand concentration using Hejjo et al.²⁰² equation

```

Range(cells(6, 127), cells(mminusone, 127)).Value = "=1-RC[-1]/R2C128"

Range(cells(6, 128), cells(mminusone, 128)).Value = _
    "=58.937*(RC[-1]^3)-51.451*(RC[-1]^2)+16.971*(RC[-1])-0.00772"

Sheets("Sheet new").Range("A1:E3").Value = Sheets("Sheet1").Range("A1:E3").Value
Range("B5").Value = "Frame"
Range("B5").Font.Bold = True

```

```

Range(cells(6, 1), cells(mminusone, 130)).NumberFormat = "0.00"
Range(cells(6, 1), cells(mminusone, 130)).HorizontalAlignment = xlCenter

'
    Renames the files with corresponding cameras
    Sheets("Sheet1").Name = "CAM" & i
    Sheets("Sheet new").Name = "Camera " & i

    bookclose = "Camera " & i & ".xls"

'
    Saves the vision data file for each camera as Excel file

    directorybookclose = directory & "\" & bookclose
    ActiveWorkbook.SaveAs FileName:=directorybookclose, FileFormat:=xlNormal
    cameradatasheet = "Camera " & i

'
    Copies the "camera" sheet from individual camera file to "all" camera file
Workbooks(bookclose).Sheets(cameradatasheet).Copy After:=Workbooks(bookallcameras).Sheets(i)

'
    Closes the camera data file as Excel file
Workbooks(bookclose).Close
'
    Saves the file containing data from "all" cameras
Workbooks(bookallcameras).Save

Next i      'goes to the next camera file

Workbooks(bookallcameras).Activate
'*****

'
    Plots average intensity and sand concentration values from each column of cameras

    m = m - 1      ' defines this way so that we have the m data rows only
'
    Defines the range so that the end row can be defined with a variable

Dim rone, rtwo, rthree As Range
Set rone = Range(cells(6, 1), cells(m, 1))      ' for time values
Set rtwo = Range(cells(6, 126), cells(m, 126))  ' for intensity values
Set rthree = Range(cells(6, 128), cells(m, 128)) ' for sand concentrations

Charts.Add
    ActiveChart.ChartType = xlXYScatter
    ActiveChart.SetSourceData Source:=Sheets("Camera 1").Application.Union(rone, rtwo), _
        PlotBy:=xlColumns
    ActiveChart.Location (xlLocationAsNewSheet)
    ActiveChart.Name = "int 1,4,7"      'Names the chart after the camera numbers

With ActiveChart
    .HasTitle = True
    .ChartTitle.Characters.Text = _
        "Light Intensity in the HPS during Proppant Transport of 2 ppg Slurry in Borate_
        -Crosslinked 35lb Guar/Mgal, pH 9.0, 6 ml/l ambient"
    .Axes(xlCategory, xlPrimary).HasTitle = True
    .Axes(xlCategory, xlPrimary).AxisTitle.Characters.Text = "Time [sec]"
    .Axes(xlValue, xlPrimary).HasTitle = True
    .Axes(xlValue, xlPrimary).AxisTitle.Characters.Text = "Average Light Intensity"
End With

```

```

ActiveChart.ChartTitle.Select      ' Formats the chart title
    With Selection.Font
        .Name = "Arial"
        .Size = 16
    End With

ActiveChart.Axes(xlValue).Select    'Formats the Y AXIS and its title
    With Selection.TickLabels.Font
        .Name = "Arial"
        .Size = 14
        Bold = True
    End With
Selection.TickLabels.NumberFormat = "General"
    With Selection.Border
        Weight = xlHairline
        LineStyle = xlAutomatic
    End With
    With Selection
        .MajorTickMark = xlInside
        .MinorTickMark = xlInside
        .TickLabelPosition = xlNextToAxis
    End With
ActiveChart.Axes(xlValue).AxisTitle.Select
    With Selection.Font
        .Name = "Arial"
        .Size = 18
        .Bold = True
    End With
    Selection.Left = 4
    Selection.Top = 127
ActiveChart.Axes(xlCategory).Select 'Formats the X AXIS and its title
    With Selection.TickLabels.Font
        .Name = "Arial"
        .Size = 14
        .Bold = True
    End With
Selection.TickLabels.NumberFormat = "General"
    With Selection.Border
        .Weight = xlHairline
        .LineStyle = xlAutomatic
    End With
    With Selection
        .MajorTickMark = xlInside
        .MinorTickMark = xlInside
        .TickLabelPosition = xlNextToAxis
    End With
ActiveChart.Axes(xlCategory).AxisTitle.Select
    With Selection.Font
        .Name = "Arial"
        .Size = 18
        .Bold = True
    End With

ActiveChart.PlotArea.Select        'Formats the plot area
    Selection.Top = 67
    Selection.Height = 352

```

```

        With Selection.Border
            .ColorIndex = 1
            .Weight = xlThin
            .LineStyle = xlContinuous
        End With
        Selection.Interior.ColorIndex = xlNone

ActiveChart.Legend.Select      'Formats the legend
    Selection.Left = 472
    Selection.Top = 101
    Selection.Font.Name = "Arial"
    Selection.Font.Size = 14

'        Formats the chart 1 series for the intensities from Camera 1

ActiveChart.SeriesCollection(1).XValues = "='Camera 1'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(1).Values = "='Camera 1'!R6C126:R" & m & "C126"
ActiveChart.SeriesCollection(1).Name = "Camera 1"
ActiveChart.SeriesCollection(1).Select
    With Selection.Border
        .Weight = xlHairline
        .LineStyle = xlNone
    End With
    With Selection
        .MarkerBackgroundColorIndex = xlNone
        .MarkerForegroundColorIndex = 1
        .MarkerStyle = xlSquare
        .MarkerSize = 5
    End With
'        Adds new series from Camera 4 and formats it in the chart

ActiveChart.SeriesCollection.Add Source:=Sheets("Camera 4").Application.Union(rone, rtwo), _
    Rowcol:=xlColumns, CategoryLabels:=True, Replace:=False
ActiveChart.SeriesCollection(2).XValues = "='Camera 4'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(2).Values = "='Camera 4'!R6C126:R" & m & "C126"
ActiveChart.SeriesCollection(2).Name = "Camera 4"
ActiveChart.SeriesCollection(2).Select
    With Selection.Border
        .Weight = xlHairline
        .LineStyle = xlNone
    End With
    With Selection
        .MarkerBackgroundColorIndex = xlNone
        .MarkerForegroundColorIndex = 3
        .MarkerStyle = xlDiamond
        .MarkerSize = 5
    End With

'        Adds new series from Camera 7 and formats it in the chart
ActiveChart.SeriesCollection.Add Source:=Sheets("Camera 7").Application.Union(rone, rtwo), _
    Rowcol:=xlColumns, CategoryLabels:=True, Replace:=False

ActiveChart.SeriesCollection(3).XValues = "='Camera 7'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(3).Values = "='Camera 7'!R6C126:R" & m & "C126"
ActiveChart.SeriesCollection(3).Name = "Camera 7"

```

```

ActiveChart.SeriesCollection(3).Select
    With Selection.Border
        .Weight = xlHairline
        .LineStyle = xlNone
    End With
    With Selection
        .MarkerBackgroundColorIndex = xlNone
        .MarkerForegroundColorIndex = 5
        .MarkerStyle = xlTriangle
        .MarkerSize = 5
    End With

'      Copies the chart and enters the series for plotting the intensities from Cameras 2,5,8
Sheets("int 1,4,7").Copy After:=Sheets(1)
ActiveSheet.Name = "int 2,5,8"

ActiveChart.ChartArea.Select
ActiveChart.SeriesCollection(1).XValues = "='Camera 2'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(1).Values = "='Camera 2'!R6C126:R" & m & "C126"
ActiveChart.SeriesCollection(1).Name = "Camera 2"

ActiveChart.SeriesCollection(2).XValues = "='Camera 5'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(2).Values = "='Camera 5'!R6C126:R" & m & "C126"
ActiveChart.SeriesCollection(2).Name = "Camera 5"

ActiveChart.SeriesCollection(3).XValues = "='Camera 8'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(3).Values = "='Camera 8'!R6C126:R" & m & "C126"
ActiveChart.SeriesCollection(3).Name = "Camera 8"

'      Copies the chart and enters the series for plotting the intensities from Cameras 3,6,9
Sheets("int 2,5,8").Copy After:=Sheets(2)
ActiveSheet.Name = "int 3,6,9"

ActiveChart.ChartArea.Select
ActiveChart.SeriesCollection(1).XValues = "='Camera 3'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(1).Values = "='Camera 3'!R6C126:R" & m & "C126"
ActiveChart.SeriesCollection(1).Name = "Camera 3"

ActiveChart.SeriesCollection(2).XValues = "='Camera 6'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(2).Values = "='Camera 6'!R6C126:R" & m & "C126"
ActiveChart.SeriesCollection(2).Name = "Camera 6"

ActiveChart.SeriesCollection(3).XValues = "='Camera 9'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(3).Values = "='Camera 9'!R6C126:R" & m & "C126"
ActiveChart.SeriesCollection(3).Name = "Camera 9"

'      Copies the chart and enters series for plotting the sand concentration from Cameras 3,6,9
Sheets("int 3,6,9").Copy After:=Sheets(3)
ActiveSheet.Name = "sand 3,6,9"

'      Reformats the chart title and y axis for the sand concentration graphs
ActiveChart.ChartTitle.Select
    Selection.Characters.Text = _
        "Sand Concentration in the HPS during Proppant Transport of 2 ppg Slurry in Borate_
        -crosslinked 35lb Guar/Mgal,pH 9.0, 6 ml/l, ambient"
ActiveChart.Axes(xlValue).AxisTitle.Select

```

```

        Selection.Characters.Text = "Average Sand Concentration [lb/gal]"
ActiveChart.PlotArea.Select
        Selection.Left = 64

'        Enters the series for plotting the intensities from Cameras 3,6,9
ActiveChart.ChartArea.Select
ActiveChart.SeriesCollection(1).XValues = "=" & Camera 3"!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(1).Values = "=" & Camera 3"!R6C128:R" & m & "C128"
ActiveChart.SeriesCollection(1).Name = "Camera 3"

ActiveChart.SeriesCollection(2).XValues = "=" & Camera 6"!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(2).Values = "=" & Camera 6"!R6C128:R" & m & "C128"
ActiveChart.SeriesCollection(2).Name = "Camera 6"

ActiveChart.SeriesCollection(3).XValues = "=" & Camera 9"!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(3).Values = "=" & Camera 9"!R6C128:R" & m & "C128"
ActiveChart.SeriesCollection(3).Name = "Camera 9"

'        Reformats the y and x axis of the graph so that sand concentration from camera 1 or 2 or 3
'        is at the top and from camera 7 or 8 or 9 is at the bottom of the chart
ActiveChart.Axes(xlValue).Select
        With ActiveChart.Axes(xlValue)
            .MinimumScale = 0
            .MaximumScale = 8
            .MinorUnit = 1
            .MajorUnit = 2
            .Crosses = xlCustom
            .CrossesAt = 8
            .ReversePlotOrder = True
            .ScaleType = xlLinear
        End With
ActiveChart.Axes(xlCategory).Select
        With Selection
            .MajorTickMark = xlOutside
            .MinorTickMark = xlOutside
            .TickLabelPosition = xlHigh
        End With
ActiveChart.Axes(xlCategory).AxisTitle.Select
        Selection.Left = 284
        Selection.Top = 430
ActiveChart.Legend.Select
        Selection.Left = 534
        Selection.Top = 85

'        Enters the series for plotting the sand concentration from Cameras 2,5,8
Sheets("sand 3,6,9").Copy Before:=Sheets(4)
ActiveSheet.Name = "sand 2,5,8"

ActiveChart.ChartArea.Select
ActiveChart.SeriesCollection(1).XValues = "=" & Camera 2"!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(1).Values = "=" & Camera 2"!R6C128:R" & m & "C128"
ActiveChart.SeriesCollection(1).Name = "Camera 2"

ActiveChart.SeriesCollection(2).XValues = "=" & Camera 5"!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(2).Values = "=" & Camera 5"!R6C128:R" & m & "C128"
ActiveChart.SeriesCollection(2).Name = "Camera 5"

```



```

ActiveChart.SeriesCollection(3).XValues = "='Camera 8'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(3).Values = "='Camera 8'!R6C128:R" & m & "C128"
ActiveChart.SeriesCollection(3).Name = "Camera 8"
'      Enters the series for plotting the sand concentration from Cameras 2,5,8
Sheets("sand 2,5,8").Copy Before:=Sheets(4)
ActiveSheet.Name = "sand 1,4,7"

```

```

ActiveChart.ChartArea.Select
ActiveChart.SeriesCollection(1).XValues = "='Camera 1'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(1).Values = "='Camera 1'!R6C128:R" & m & "C128"
ActiveChart.SeriesCollection(1).Name = "Camera 1"

```

```

ActiveChart.SeriesCollection(2).XValues = "='Camera 4'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(2).Values = "='Camera 4'!R6C128:R" & m & "C128"
ActiveChart.SeriesCollection(2).Name = "Camera 4"

```

```

ActiveChart.SeriesCollection(3).XValues = "='Camera 7'!R6C1:R" & m & "C1"
ActiveChart.SeriesCollection(3).Values = "='Camera 7'!R6C128:R" & m & "C128"
ActiveChart.SeriesCollection(3).Name = "Camera 7"

```

```

ActiveWorkbook.Save

```

End Sub

A.1.2 Macro Converts Intensity Data from the Vision System into Sand Contour Plotting Format

```

Sub Macro_2_data_in_contour_plot_format()

```

```

'
'      Data_in_contour_plot_format Macro
'      Macro takes data from the format processed and recorded by the vision computer and converts it
'      into new format for ease of contour plotting
'
'      Macro recorded 1/28/00 by Naval Goel
'      Macro 1. Arranges intensity data from vision computer format to a format which is easy to
'              generate contour plot
'              2. Calculates sand concentration from intensity data for desired time steps
'      Requires that I open the file which contains intensity data for all nine cameras also requires that
'      I supply the required time stage through a dialog box and allows only 10 time steps

```

```

Application.ScreenUpdating = False

```

```

j = 0      'for time stages

```

```

'      Initial time in sec for base intensity values

```

```

Itime = InputBox("Please Enter the time in sec for initial intensity calculations", "Initial Time")

```

```

'      Ascribes arrays for 9 cameras, 11 time stages, row 11 to 43 for intensities and row 55 to 87 for
'      sand concentration

```

```

Dim RIntensity(11 To 43, 440, 11, 9), SandConc(55 To 87, 440, 11, 9)

```

```

'      Inserts a sheet to store the vision data
Sheets.Add
ActiveSheet.Select

```

```

ActiveSheet.Name = "Contour Data"

' Describes the headings for the data

Sheets("Contour Data").Select
Rows("1:1").Select
    Selection.Font.Bold = True
    Selection.Font.Size = 12
' Highlights first row (cameras 1,2,and 3) and third row (cameras 7,8 and 9) of sand concentrations
Rows("55:65").Select
    Selection.Interior.ColorIndex = 46
Rows("77:87").Select
    Selection.Interior.ColorIndex = 4
' Highlights second row of the cameras 4,5,6 for the intensity values
Rows("22:32").Select
    Selection.Interior.ColorIndex = 48
' Performs Calculation for each time stage

1    NewTime = j * 44 + 1
    Sheets("Contour Data").cells(1, NewTime).Value = "time = " & Itime & " sec"
    Sheets("Contour Data").cells(5, NewTime).Value = "Intensity at " & Itime & " sec"
    Sheets("Contour Data").cells(53, NewTime).Value = "Sand Concentration (lb/gal) at " & Itime & _
        " sec"

    CameraRow = 6 + Itime / 10
'    CameraRow and CameraColumn describe camera sheets cells
'    ContourRow and ContourColumn describe the Contour Data sheets cells

'    Performs calculations for camera 1,2 and 3

For i = 1 To 3
    Datasheet = "Camera " & I
    CameraColumn = 3
    For ContourRow = 11 To 21
        ContourColumn = 1 + (i - 1) * 11 + j * 44

'        Copies the intensity values to a variable and then copy this variable to the contour data sheet
'        Defining with a Variable helps to perform sand concentration calculations

5    RIntensity(ContourRow, ContourColumn, j, i) = Sheets(Datasheet).cells(CameraRow, _
        CameraColumn).Value
    Sheets("Contour Data").cells(ContourRow, ContourColumn).Value = RIntensity(ContourRow, _
        ContourColumn, j, i)
    ContourColumnInitial = ContourColumn - j * 44 'denotes base intensities
    ContourRowSand = ContourRow + 44 'for sand concentration calculations

'    Normalizes the intensity values at any time t with that at initial time

    NormalizedIntensity = 1 - (RIntensity(ContourRow, ContourColumn, j, i)) / _
        (RIntensity(ContourRow, ContourColumnInitial, 0, i))
    SandConc(ContourRowSand, ContourColumn, j, i) = 58.937 * (NormalizedIntensity ^ 3) _
        - 51.451 * (NormalizedIntensity ^ 2) + 16.971 * NormalizedIntensity - 0.00772
    Sheets("Contour Data").cells(ContourRowSand, ContourColumn).Value = _
        SandConc(ContourRowSand, ContourColumn, j, i)

    CameraColumn = CameraColumn + 1
    ContourColumn = ContourColumn + 1

```

```

ContourColumnLast = 1 + i * 11 + j * 44
If ContourColumn = ContourColumnLast Then GoTo 9
' To copy only 11 columns and then move to next row

If CameraColumn = 124 Then GoTo 10 'as there are only 121 fibers for each camera
GoTo 5

9 Next ContourRow 'next row of fibers in the same camera
10 Next i 'Next camera
' Performs calculations for cameras 4,5 and 6

For i = 4 To 6
  Datasheet = "Camera " & i
  CameraColumn = 3
  IMinusThree = i - 3
  For ContourRow = 22 To 32
    ContourColumn = 1 + (IMinusThree - 1) * 11 + j * 44
    ' To enter data for cameras 4 to 6 under cameras 1 to 3

    ' Copies the intensity values to a variable and then copy this variable to the contour data sheet
    ' Defining with a Variable helps to perform sand concentration calculations

15 RIntensity(ContourRow, ContourColumn, j, i) = Sheets(Datasheet).cells(CameraRow, _
    CameraColumn).Value
    Sheets("Contour Data").cells(ContourRow, ContourColumn).Value = RIntensity(ContourRow, _
    ContourColumn, j, i)
    ContourColumnInitial = ContourColumn - j * 44 'denotes base intensities
    ContourRowSand = ContourRow + 44 'for sand concentration calculations

    NormalizedIntensity = 1 - (RIntensity(ContourRow, ContourColumn, j, i)) / _
    (RIntensity(ContourRow, ContourColumnInitial, 0, i))

    SandConc(ContourRowSand, ContourColumn, j, i) = 58.937 * (NormalizedIntensity ^ 3) _
    - 51.451 * (NormalizedIntensity ^ 2) + 16.971 * NormalizedIntensity - 0.00772
    Sheets("Contour Data").cells(ContourRowSand, ContourColumn).Value = _
    SandConc(ContourRowSand, ContourColumn, j, i)

    CameraColumn = CameraColumn + 1
    ContourColumn = ContourColumn + 1

    ContourColumnLast = 1 + IMinusThree * 11 + j * 44

    If ContourColumn = ContourColumnLast Then GoTo 19
    ' To copy only 11 columns and then move to next row

    If CameraColumn = 124 Then GoTo 20 'As there are only 121 fibers for each camera

    GoTo 15

19 Next ContourRow 'Next row of fibers for the same camera
20 Next i 'Next camera
' Performs calculations for cameras 7, 8 and 9

```

```

    Datasheet = "Camera " & i
    CameraColumn = 3
    IMinusSix = i - 6

For ContourRow = 33 To 43
    ContourColumn = 1 + (IMinusSix - 1) * 11 + j * 44
    ' To enter data for cameras 7 to 9 under cameras 1 to 3

    ' Copies the intensity values to a variable and then copy this variable to the contour data sheet
    ' Defining with a Variable helps to perform sand concentration calculations

25    RIntensity(ContourRow, ContourColumn, j, i) = Sheets(Datasheet).cells(CameraRow, _
                                                CameraColumn).Value
    Sheets("Contour Data").cells(ContourRow, ContourColumn).Value = RIntensity(ContourRow, _
                                                ContourColumn, j, i)

    ContourColumnInitial = ContourColumn - j * 44    'Denotes base intensities
    ContourRowSand = ContourRow + 44    'For sand concentration calculations

    NormalizedIntensity = 1 - (RIntensity(ContourRow, ContourColumn, j, i)) / _
        (RIntensity(ContourRow, ContourColumnInitial, 0, i))
    SandConc(ContourRowSand, ContourColumn, j, i) = 58.937 * (NormalizedIntensity ^ 3) _
        - 51.451 * (NormalizedIntensity ^ 2) + 16.971 * NormalizedIntensity - 0.00772

    Sheets("Contour Data").cells(ContourRowSand, ContourColumn).Value = _
        SandConc(ContourRowSand, ContourColumn, j, i)

    CameraColumn = CameraColumn + 1
    ContourColumn = ContourColumn + 1

    ContourColumnLast = 1 + IMinusSix * 11 + j * 44

    If ContourColumn = ContourColumnLast Then GoTo 29
    ' To copy only 11 columns and then move to next row

    If CameraColumn = 124 Then GoTo 30    'As there are only 121 fibers for each camera

    GoTo 25

29    Next ContourRow    'Next row of fibers for the same camera
30    Next i    'Next camera

    ' Identifies cameras 2,5 and 8
    MiddleColumn = 12 + j * 44
    MiddleColumnplusTen = MiddleColumn + 10

    Range(cells("11", MiddleColumn), cells("43", MiddleColumnplusTen)).Select
    ' Identifies intensity values
        Selection.Font.Bold = True
        Selection.Font.ColorIndex = 5

    Range(cells("55", MiddleColumn), cells("87", MiddleColumnplusTen)).Select
    ' Identifies sand concentration values
        Selection.Font.Bold = True
        Selection.Font.ColorIndex = 5

```

```

' Asks for data from more time stages

j = j + 1
Response = MsgBox("Do you want to enter another time? This is your no. " & j & _
    " time stage", vbYesNo)
If Response = vbYes Then
    Itime = InputBox("Enter time in sec. This is your no. " & j & _
        " time stage. Total stages allowed are 5")
    GoTo 1
End If

' Formats Sand Concentration values to two decimal places

Rows("55:87").Select
Selection.NumberFormat = "0.00"
    With Selection.HorizontalAlignment = xlCenter
    End With

' Clears the colors filled in those cells that are in between the data cells
j = 0

40 ClearColumn = 34 + j * 44
    ClearColumnplusTen = ClearColumn + 10

    Range(Columns(ClearColumn), Columns(ClearColumnplusTen)).Select
        Selection.Interior.ColorIndex = xlNone
        j = j + 1
    If j < 5 Then GoTo 40

End Sub

```

A.1.3 Macro Equalizes the Intensity and Sand Concentration Data from Bad LEDS with Data of Eight LEDS Around Each Bad LED

```

Sub Macro_3_Zeros_Equalizes_Negative_Value_Cells_in_contour_data()
'
' Total number of columns
m = 256
' Number of times the sand concentration is less than zero
n = 0

Application.ScreenUpdating = False

' Sets the negative sand concentrations to zero before equalizing them

For i = 55 To 87
    For j = 1 To m
        If cells(i, j).Value < 0 Then cells(i, j).Value = 0
    Next j
Next i

For i = 55 To 87
' Number for identifying beforeTheFirst & afterTheLast column with value
k = 1

```

```

        kminone = k - 1
        For j = 1 To m
            If IsEmpty(cells(i, j).Value) Then GoTo 10
            Cells that are before and after, and below and above the EDGE cells of the fibers are defined here
            '
            Takes care of the "before and after empty-row" cells
            cells(54, j).Value = cells(55, j).Value
            cells(88, j).Value = cells(87, j).Value
            '
            Takes care of the "after empty-columns" cells
            LastJ = 33 * k + 11 * kminone
            nextToLastJ = 33 * k + 11 * kminone + 1
            cells(i, nextToLastJ).Value = cells(i, LastJ).Value
            '
            Takes care of the "before empty-columns" cells
            FirstJ = 44 * k + 1
            beforeTheFirstJ = 44 * k
            cells(i, beforeTheFirstJ).Value = cells(i, FirstJ).Value

            iminone = i - 1
            iplusone = i + 1
            jminone = j - 1

            If jminone = 0 Then jminone = j
            jplusone = j + 1
            whatis = cells(i, j).Value
            If whatis <= 0 Then
                '
                If the cells have a negative value then it is set equal to an average of the eight cells around it
                a = cells(iminone, jminone).Value
                b = cells(i, jminone).Value
                c = cells(iplusone, jminone).Value
                d = cells(iminone, j).Value
                e = cells(iplusone, j).Value
                f = cells(iminone, jplusone).Value
                g = cells(i, jplusone).Value
                h = cells(iplusone, jplusone).Value

                cells(i, j).Value = (a + b + c + d + e + f + g + h) / 8
                n = n + 1
            End If
        Next j
10    Next i

    n = n - 1089
    cells(53, 6).Value = "number of sand concentrations values < 0 are"
    cells(53, 11).Value = n
    '
    Clears the contents of the "before and after, above and below" cells
    Rows("54:54").ClearContents
    Rows("88:88").ClearContents

    m = 0
11    ClearColumn = 34 + m * 44
    ClearColumnplusTen = ClearColumn + 10
    Range(Columns(ClearColumn), Columns(ClearColumnplusTen)).Select
    Selection.ClearContents
    m = m + 1
    If m < 5 Then GoTo 11
End Sub

```

A.1.4 Macro Converts Sand Concentration Data in Surfer Software Compatible Format

Sub Macro_6_data_in_surfer_software_format()

Application.ScreenUpdating = False

```
m = 33
n = 33
mplusone = m + 1
nplusone = n + 1
```

Dim alpha(34, 34), beta(34, 34), gamma(34, 34)

ActiveSheet.Name = "contour datasheet"

For dataRow = 2 To mplusone

For dataColumn = 2 To nplusone

alpha(dataRow, dataColumn) = Sheets("contour datasheet").cells(dataRow, 1).Value

beta(dataRow, dataColumn) = Sheets("contour datasheet").cells(1, dataColumn).Value

gamma(dataRow, dataColumn) = Sheets("contour datasheet")._cells(dataRow, dataColumn).Value

Next dataColumn

Next dataRow

Sheets.Add

ActiveSheet.Name = "surfersheet"

For dataRow = 1 To m

For dataColumn = 1 To n

targetrow = (dataRow - 1) * m + dataColumn

rowplusone = dataRow + 1

columnplusone = dataColumn + 1

Sheets("surfersheet").cells(targetrow, 1).Value = beta(rowplusone, columnplusone)

Sheets("surfersheet").cells(targetrow, 2).Value = alpha(rowplusone, columnplusone)

Sheets("surfersheet").cells(targetrow, 3).Value = gamma(rowplusone, columnplusone)

Next dataColumn

Next dataRow

End Sub

A.1.5 Macro Generates Sand Concentration Contours Using Surfer Software

Sub Main

' See Page 535 of Reference 231 to understand this program

' This Macro is for a sand concentration level of 2 ppg

Dim SurferApp, Plot, ContourMapFrame, ContourMap As Object

Dim InFile, GridFile, BaseName As String

Set SurferApp=CreateObject("Surfer.Application")

SurferApp.Visible=True

```

InFile=GetFilePath ("","DAT;TXT;CSV;XLS",CurDir(),"Select data file",0)
    If InFile="" Then End

    BaseName=InFile
    ExtStart=InStrRev(InFile,".")
    If ExtStart>1 Then BaseName=Left(InFile,ExtStart-1)

    GridFile=BaseName+".grd"
    SurferApp.GridData DataFile:=InFile, NumCols:=33, NumRows:=33, xMin:=1, xMax:=33, _
        yMin:=1, yMax:=33, Algorithm:=srfInverseDistance, DupMethod:=srfDupNone, _
        ShowReport:=False, AnisotropyRatio:=1, AnisotropyAngle:=0, _
        IDPower:=1, IDSmoothing:=1, OutGrid:=GridFile

    Set Plot=SurferApp.Documents.Add(srfDocPlot)
    Set ContourMapFrame=Plot.Shapes.AddContourMap(GridFile)

    SurferApp.ScreenUpdating=False

    Set ContourMap=ContourMapFrame.Overlays(1)
        ContourMap.FillContours          = True
        ContourMap.ShowColorScale        = True
        ContourMap.ColorScale.LabelFont.Size = 18
        ContourMap.ColorScale.LabelFont.Bold = True
        ContourMap.ColorScale.LabelFont.Face = "Arial"
        ContourMap.Levels.LoadFile(SurferApp.Path+"\Colors20percent.lvl")

    '      Formats the axis title
    For Each axis In ContourMapFrame.Axes
        If axis.AxisType=srfATLeft Then
            axis.Title          = "Frames along the HPS Height"
            axis.TitleFont.Size = 18
            axis.TitleFont.Bold = True
            axis.TitleFont.Face = "Arial"
        End If

        If axis.AxisType=srfATBottom Then
            axis.Title          = "Frames along the HPS Length"
            axis.TitleFont.Size = 18
            axis.TitleFont.Bold = True
            axis.TitleFont.Face = "Arial"
        End If
    Next

    '      Inserts a text object to indicate time on the chart
    Dim Text As Object

    Set Text = Plot.Shapes.AddText(x:=4, y:=9, Text:=" mins")
        Text.Font.Size = 18
        Text.Font.Bold = True
        Text.Font.Face = "Arial"
        Text.Font.Color = srfColorBlue

    SurferApp.ScreenUpdating=True
    Plot.SaveAs(BaseName+".srf")

End Sub

```


A.2. Macros for Oscillation Data Analysis

A.2.1 Master Macro Processes Viscoelastic Data Obtained from Bohlin Rheometer

Sub Bohlin_Oscillation_Master_Macro()

```
,  
,      Bohlin_Oscillation_Master_Macro Macro  
,      Imports, saves, and plots the data from the oscillatory measurements on Bohlin  
,  
,
```

ChDir "U:\BOHLIN\MACROS"

Workbooks.Open filename:="ocMACRO1.XLS"

Application.ScreenUpdating = False

10

```
Book1 = InputBox("Enter the workbook to open like oc06136a", "Open Bohlin RawData Files")  
Book11 = "a:\\" + Book1 + ".oct"  
Workbooks.OpenText filename:=Book11, Origin:=xlWindows, StartRow:=1,_  
    DataType:=xlDelimited, TextQualifier:= _  
    xlDoubleQuote, ConsecutiveDelimiter:=True, Tab:=True, Space:=True
```

ChDir "U:\BOHLIN\"

```
Book2 = InputBox("Enter the directory route Guar\Linear", "Store Bohlin RawData Files")  
Book22 = "u:\bohlin\" + Book2 + "\"
```

ChDir Book22

```
Book33 = Book1 + ".xls"
```

ActiveWorkbook.SaveAs filename:=Book33, FileFormat:=xlNormal

Windows(Book33).Activate

```
Mac = "ocMACRO1.XLS!Bohlin_oscillation_data_plot_macro"
```

Application.Run Macro:=Mac

ActiveWorkbook.Save

```
Msg = "Do you want to open more files ?"  
Style = vbYesNo  
Title = "Open Bohlin RawData Files"  
Response = MsgBox(Msg, Style, Title)
```

```
If Response = vbYes Then GoTo 10
```

End Sub

A.2.2 Macro Analyses and Plots the Viscoelastic Data: OCMacro1.xls

```
'      Bohlin_oscillation_data_plot_macro Macro
'      Rearranges and plots the oscillatory measurement data
'
```

```
Public LastRow As Integer
Public LastRow1 As Integer
```

Sub Bohlin_oscillation_data_plot_macro()

```
ActiveSheet.Select
    With Selection
        ActiveSheet.Name = "Sheet1"
    End With

    Rows("1:1").Select
        Selection.Insert Shift:=xlDown
    Range("E2:G2").Select
        Selection.Cut Destination:=Range("E1:G1")
    Range("E1:G1").Select
        Selection.Font.Bold = True
        With Selection.Font
            .Name = "Arial"
            .Size = 12
        End With
    Range("B3").Select
        Selection.Cut Destination:=Range("F3")
    Range("F3").Select
        Selection.Font.Bold = True
    Range("D6").Select
        Selection.Font.Bold = True
    Columns("B:B").ColumnWidth = 9.43
    Columns("E:E").ColumnWidth = 9

    RowNo = 15
    RowNo1 = 16
10    Do Until IsEmpty(Cells(RowNo, 2).Value)
        RowNo = RowNo + 1
        RowNo1 = RowNo + 1
    Loop

    If IsEmpty(Cells(RowNo1, 2)) Then GoTo 20
        RowNo = RowNo1
    GoTo 10

20    LastRow = RowNo - 1

    Range("a12").Select
        ActiveCell.FormulaR1C1 = "Angular Velocity"
    Range("a13").Select
        ActiveCell.FormulaR1C1 = "rad/sec"
    Range("a15").Select
        ActiveCell.FormulaR1C1 = "=2*PI()*RC[+3]"
    Selection.AutoFill Destination:=Range("a15:a" & LastRow), Type:=xlFillDefault
```

```

        Range("a15:a" & LastRow).Select
        Range("E15:I" & LastRow).Select
Dim r1, r2 As Range

Set r1 = Range("a15:a" & LastRow)
Set r2 = Range("E15:I" & LastRow)

        Range("a15:j" & LastRow).Select
        Selection.NumberFormat = "General"
Charts.Add

ActiveChart.ChartWizard Source:=Sheets("sheet1").Application.Union(r1, r2), _
        Gallery:=xlXYScatter, Format:=2, PlotBy:= _
        xlColumns, CategoryLabels:=1, SeriesLabels:=0, HasLegend:=1
ActiveChart.PlotArea.Select
    With Selection.Border
        .ColorIndex = 16
        .Weight = xlThin
        .LineStyle = xlContinuous
    End With
    Selection.Interior.ColorIndex = xlNone

ActiveChart.SeriesCollection(1).Select
    With Selection.Border
        .ColorIndex = 4
        .Weight = xlThin
        .LineStyle = xlContinuous
    End With
    With Selection
        .MarkerBackgroundColorIndex = 4
        .MarkerForegroundColorIndex = 4
        .MarkerStyle = xlDiamond
        .Smooth = False
    End With

ActiveChart.SeriesCollection(1).AxisGroup = 2
    With ActiveChart.SeriesCollection(1)
        .Name = """"Phase Angle""""
    End With

ActiveChart.SeriesCollection(2).Select
    With Selection.Border
        .ColorIndex = 9
        .Weight = xlThin
        .LineStyle = xlContinuous
    End With
    With Selection
        .MarkerBackgroundColorIndex = 2
        .MarkerForegroundColorIndex = 9
        .MarkerStyle = xlSquare
        .Smooth = False
    End With
    With ActiveChart.SeriesCollection(2)
        .Name = """"Viscosity""""
    End With

```

```

ActiveChart.SeriesCollection(3).Select
    With Selection.Border
        .ColorIndex = 1
        .Weight = xlThin
        .LineStyle = xlContinuous
    End With
    With Selection
        .MarkerBackgroundColorIndex = 1
        .MarkerForegroundColorIndex = 1
        .MarkerStyle = xlSquare
        .Smooth = False
    End With
    With ActiveChart.SeriesCollection(3)
        .Name = ""G*""
    End With

ActiveChart.SeriesCollection(4).Select
    With Selection.Border
        .ColorIndex = 3
        .Weight = xlMedium
        .LineStyle = xlContinuous
    End With
    With Selection
        .MarkerBackgroundColorIndex = 2
        .MarkerForegroundColorIndex = 3
        .MarkerStyle = xlCircle
        .Smooth = False
    End With
    With ActiveChart.SeriesCollection(4)
        .Name = ""G""
    End With

ActiveChart.SeriesCollection(5).Select
    With Selection.Border
        .ColorIndex = 5
        .Weight = xlMedium
        .LineStyle = xlContinuous
    End With
    With Selection
        .MarkerBackgroundColorIndex = 2
        .MarkerForegroundColorIndex = 5
        .MarkerStyle = xlTriangle
        .Smooth = False
    End With
    With ActiveChart.SeriesCollection(5)
        .Name = ""G""
    End With
ActiveChart.ChartArea.Select

ActiveChart.ChartGroups(1).SeriesCollection(1).PlotOrder = 4
    With ActiveChart
        .HasTitle = True
        .Axes(xlCategory, xlPrimary).HasTitle = True
        .Axes(xlValue, xlPrimary).HasTitle = True
        .Axes(xlValue, xlSecondary).HasTitle = True
    End With

```

```

ActiveChart.ChartTitle.Select
    With Selection.Font
        .Name = "Arial"
        .FontStyle = "Bold"
        .Size = 28
    End With
    Selection.Characters.Text = "LABORATORY RHEOLOGY: BOHLIN Zirconate-crosslinked_
        40 lb HPG/Mgal, pH 9.5, 66oC, . ml/l crosslinker"

    With Selection.Characters(Start:=1, Length:=29).Font
        .Name = "Arial"
        .FontStyle = "Bold"
        .Size = 28
    End With
    With Selection.Characters(Start:=30, Length:=73).Font
        .Name = "Arial"
        .FontStyle = "Bold"
        .Size = 18
    End With
    With Selection.Characters(Start:=74, Length:=1).Font
        .Name = "Arial"
        .FontStyle = "Bold"
        .Size = 18
        .Superscript = True
    End With
    With Selection.Characters(Start:=75, Length:=1).Font
        .Name = "Arial"
        .FontStyle = "Bold"
        .Size = 18
        .Superscript = False
    End With
ActiveChart.ChartTitle.Select
    Selection.Left = 93
    Selection.Top = 3

ActiveChart.Axes(xlCategory).AxisTitle.Select
    Selection.Characters.Text = "Angular Velocity [rad/sec]"
    With Selection.Font
        .Name = "Arial"
        .FontStyle = "Bold"
        .Size = 18
    End With
ActiveChart.Axes(xlValue, xlPrimary).AxisTitle.Select
    Selection.Characters.Text = "G*, G', G''' [Pa], Viscosity [Pas]"
    With Selection.Font
        .Name = "Arial"
        .FontStyle = "Bold"
        .Size = 18
    End With
ActiveChart.Axes(xlValue, xlSecondary).AxisTitle.Select
    Selection.Characters.Text = "Phase Angle [Degrees]"
    With Selection.Font
        .Name = "Arial"
        .FontStyle = "Bold"
        .Size = 18
    End With

```

```

ActiveChart.Axes(xlValue, xlPrimary).Select
    With Selection.Border
        .Weight = xlHairline
        .LineStyle = xlAutomatic
    End With
    With Selection
        .MajorTickMark = xlInside
        .MinorTickMark = xlInside
        .TickLabelPosition = xlNextToAxis
    End With
    With ActiveChart.Axes(xlValue, xlPrimary)
        .MinimumScaleIsAuto = True
        .MaximumScaleIsAuto = True
        .MinorUnitIsAuto = True
        .MajorUnitIsAuto = True
        .CrossesAt = 0.01
        .ScaleType = True
    End With
    With Selection.TickLabels.Font
        .Name = "Arial"
        .FontStyle = "Bold"
        .Size = 14
    End With

ActiveChart.Axes(xlValue, xlSecondary).Select
    With Selection.Border
        .Weight = xlHairline
        .LineStyle = xlAutomatic
    End With
    With Selection
        .MajorTickMark = xlInside
        .MinorTickMark = xlNone
        .TickLabelPosition = xlNextToAxis
    End With
    With ActiveChart.Axes(xlValue, xlSecondary)
        .MinimumScaleIsAuto = True
        .MaximumScaleIsAuto = True
        .MinorUnitIsAuto = True
        .MajorUnitIsAuto = True
        .CrossesAt = 0
    End With
    With Selection.TickLabels.Font
        .Name = "Arial"
        .FontStyle = "Bold"
        .Size = 14
    End With

ActiveChart.Axes(xlCategory).Select
    With Selection.Border
        .Weight = xlHairline
        .LineStyle = xlAutomatic
    End With
    With Selection
        .MajorTickMark = xlInside
        .MinorTickMark = xlInside
        .TickLabelPosition = xlNextToAxis
    End With

```

```

        With ActiveChart.Axes(xlCategory)
            .MinimumScaleIsAuto = True
            .MaximumScaleIsAuto = True
            .MinorUnitIsAuto = True
            .MajorUnitIsAuto = True
            .CrossesAt = 0.01
            .ScaleType = True
        End With
        With Selection.TickLabels.Font
            .Name = "Arial"
            .FontStyle = "Bold"
            .Size = 14
        End With
ActiveChart.Legend.Select
        With Selection.Font
            .Name = "Arial"
            .Size = 14
        End With
        Selection.Position = xlBottom
ActiveChart.Legend.Select
        Selection.Left = 164
        Selection.Top = 440

ActiveChart.PlotArea.Select
        Selection.Left = 60
        Selection.Top = 69
        Selection.Height = 334
        Selection.Width = 538
ActiveChart.PlotArea.Select
        With Selection.Border
            .ColorIndex = 1
            .Weight = xlThin
            .LineStyle = xlContinuous
        End With
        Selection.Interior.ColorIndex = xlNone

        With ActiveChart.PageSetup
            .CenterHeader = " "
            .CenterFooter = ""
            .LeftMargin = Application.InchesToPoints(0.75)
            .RightMargin = Application.InchesToPoints(0.75)
            .ChartSize = xlFullPage
            .CenterHorizontally = True
            .CenterVertically = True
        End With

ActiveChart.Axes(xlCategory).AxisTitle.Select
        Selection.Left = 226
        Selection.Top = 403
ActiveChart.Axes(xlValue, xlPrimary).AxisTitle.Select
        Selection.Left = 4
        Selection.Top = 168
ActiveChart.Axes(xlValue, xlSecondary).AxisTitle.Select
        Selection.Left = 610
        Selection.Top = 160

```

```

ActiveChart.ChartArea.Select
    With ActiveChart.TextBoxes.Add(337, 230, 387, 246)
        .Select
        .AutoSize = True
        .Formula = "'=sheet1'!$E$4"
    End With
    With Selection.Font
        .Name = "Arial"
        .Size = 12
    End With
ActiveChart.DrawingObjects("Text 1").Select
    Selection.Left = 613
    Selection.Top = 440

Sheets("Chart1").Select
Sheets("Chart1").Name = "rheogram"
Sheets("sheet1").Select
Sheets("sheet1").Name = "oscillation rheology"

Range("I8").Select
    ActiveCell.FormulaR1C1 = "'Shear Strain = 0.05 '"
Sheets("rheogram").Select
    With ActiveChart.TextBoxes.Add(338, 257, 418, 273)
        .Select
        .AutoSize = True
        .Formula = "'=oscillation rheology'!$I$8"
    End With
    With Selection.Font
        .Name = "Arial"
        .Size = 12
    End With
ActiveChart.DrawingObjects("Text 2").Select
    Selection.Left = 1
    Selection.Top = 440

End Sub

```


APPENDIX B

PROCEDURE FOR TWO-DIMENSIONAL FRACTURING DESIGN

The following section describes the procedure used for fracture design using the two dimensional GdK model.¹⁴⁶

The setting velocity, v_t , of a single particle in the fracturing fluid was determined from

$$v_t = \frac{(2n+1)d_p}{108n} \left[\frac{(\rho_p - \rho)d_p}{72k} \right]^{1/n} \dots\dots\dots (B.1.1)$$

where d_p is mean diameter of the proppant particle and for a 20/40 mesh sand, it is 0.0272 in.; ρ_p and ρ are particle and fluid density and are equal to 165 and 62.8 lb/ft³ respectively. The single particle settling velocity was then corrected for the effect of other particles in proppant slurry, and settling time for the proppant particles was determined as¹⁴⁶

$$t_{\text{settling}} = \frac{h_f}{60F_{hr}v_t} \dots\dots\dots (B.1.2)$$

where F_{hr} is the correction factor for hindered settling. For the slurry concentration used in Section 4.2, F_{hr} is 0.45. In Eq. B.1.2, h_f is fracture height, and for the GdK model, it is constant and is assumed equal to the formation height. The settling time was then compared with the pumping time of the proppant-laden slurry, given by

$$t_{\text{proppant}} = \frac{V_{\text{total}} - V_{\text{pad}}}{42Q_t} \dots\dots\dots (B.1.3)$$

If the settling time was less than the pump time, the slurry settled down and the fracturing treatment was unsuccessful, else the dimensionless parameters for the GdK model were determined to design the fracture. The parameters defined in the model are

$$K_s = \frac{7.48C\sqrt{t}}{V_{sp}} \dots\dots\dots (B.1.4)$$

$$K_u = \frac{12C\sqrt{t}}{w_w} \dots\dots\dots (B.1.5)$$

$$K_L = \frac{Ch_f L_f}{5.615Q\sqrt{t}} \dots\dots\dots (B.1.6)$$

$$K_{uL} = 2.286 * 10^{-5} \left(1 - \nu^2\right) \left[\frac{Q}{h_f C^2} \right]^3 \frac{\mu}{Et} \dots\dots\dots (B.1.7)$$

where C is fracturing fluid loss coefficient in $\text{ft}/\text{min}^{1/2}$, t is pumping time in min., V_{sp} is spurt loss in gal/ft^2 , w_w is fracture width at wellbore in in., h_f is average fracture height in ft, L is fracture half-length in ft, Q is surface injection rate in bbl/min, E is Young's Modulus of elasticity in psi, ν is dimensionless Poisson's ratio, and μ is fracturing fluid viscosity in cp. Using these dimensionless parameters and the fracturing parameters listed in Table 4.1, the fracture width and half-length were determined. These values were calculated for conditions after pumping the pad volume and the total fluid volume.

Then, the fracture dimensions were checked for fluid efficiency, which is defined as the percent of fluid remaining in the fracture after fluid loss. The efficiency of the pad, EFF_{pad} , and the total job, EFF_{total} , are given by

$$EFF_{pad} = \frac{98h_f(L_{f\ pad})(w_{w\ pad})}{V_{pad}} \dots\dots\dots (B.1.8)$$

and
$$EFF_{total} = \frac{98h_f(L_{f\ total})(w_{w\ total})}{V_{total}} \dots\dots\dots (B.1.9)$$

Finally, the propped fracture half-length, L_{fp} , was determined as

$$L_{fp} = \frac{0.0051[EFF_{pad} + EFF_{total}](V_{total} - V_{pad})}{h_f w_{w\ total}} \dots\dots\dots (B.1.10)$$

Since there would be some settling during the fracturing treatment, a proppant bed would be formed. So the velocity of the fluid flowing over the bed would increase and the actual propped fracture height would change. Therefore, an equilibrium velocity-width term $(v_w)_{eq}$ and an equilibrium fluid velocity, v_{eq} , was determined as follows

$$(v_w)_{eq} = \frac{v_t}{0.041 \left[\frac{\rho v_t d_p}{\mu} \sqrt{\frac{4r_H}{d_p}} \right]^{1/2}} \dots\dots\dots (B.1.11)$$

$$\text{and } v_{eq} = \frac{\rho_p \bar{w} [(v_w)_{eq}]^2}{2.993 \mu} \dots\dots\dots (B.1.12)$$

where r_H is the hydraulic radius and is equal to average width $\bar{w}$, given by $\bar{w} = \frac{\pi}{4} w_w$.

The fluid viscosity, μ , was evaluated at 170 sec^{-1} shear rate. Equation B.1.12 is applicable for laminar flow only; so the flow regime in the fracture was checked from the Reynolds number, defined in field units as

$$\frac{\gamma_{fluid} Q/2}{\mu h_f} < 0.384 \dots\dots\dots (B.1.13)$$

From v_{eq} , equilibrium bed height, h_{eq} , was calculated as

$$h_{eq} = \frac{0.562 Q}{w v_{eq}} \dots\dots\dots (B.1.14)$$

Thus, the propped fracture height, h_f' , was determined as $h_f' = h_f - h_{eq}$.

Next, the production increase from the fracturing treatment was determined. The final proppant concentration in the fracture, $\bar{c}_p$, was determined by

$$\bar{c}_p = \frac{\text{weight of proppant}}{\text{propped area}} = \frac{\text{proppant concentration} [V_{fluid} - V_{pad}]}{2 h_f' L_{fp}} \dots\dots\dots (B.1.15)$$

The fracture flow capacity, FC , in md-ft, was determined at the closure stress and the proppant concentration in the fracture.¹⁴⁶

Finally, the production increase obtained from the stimulated treatment was determined from the productivity curves.¹⁴⁶ The curves are drawn for various values of

$\frac{h_f'}{H}$ and $\frac{L_{fp}}{r_e}$ ratios, and have the following x-axis

$$x - axis = \frac{c_r}{2} \frac{h_f'}{H} \ln \left(\frac{r_e}{r_w} \right) \dots\dots\dots (B.1.16)$$

where, $c_r = \frac{FC}{\pi k L_{fp}}$. From the known values, y-axis values were read from the

production increase curves. Finally, the ratio of productivity of the fractured formation, J_f , to that of the unstimulated formation, J_o , was calculated by

$$\frac{J_f}{J_o} = (y - axis\ value) \frac{\ln\left(\frac{r_e}{r_w}\right)}{6.215} \dots\dots\dots (B.1.13)$$

The procedure described above was used to determine the productivity increase obtained as a function of the fracturing fluid viscosity. The results obtained were discussed in Section 4.2.