PROCESS SIMULATION, MODELING & DESIGN FOR SOYBEAN OIL EXTRACTION USING LIQUID PROPANE

By

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CHAPTER 1

INTRODUCTION

1.1 Background Information & Problem Statement

The demand for vegetable oils has been increasing because of their use in areas involving chemical processing, specifically, bio-fuel manufacture and for direct consumption itself. With an increasing number of people shifting their diet from a non-vegetarian base to vegetarian, the demand for vegetable oil has risen. Solvent extraction and mechanical expulsion are the two most commonly employed processes for extracting oil from vegetables, seeds, flowers and other such sources which are rich in oil. The solvent extraction process is the more popular of the two methods and it is preferred over mechanical expulsion because the high quality of oil that is obtained, which requires minimal refining.

Environmental degradation is a major challenge facing humanity today along with finding alternate fossil fuels. Vegetable oils have the potential to solve this problem. As stated earlier, the increased interest in oil extraction from unconventional sources is due to this reason. Hence, it is critical to develop a process through which, vegetable oil extraction can be carried out efficiently and effectively.

Hexane has been the solvent conventionally used in this process. But, hexane is highly flammable and is also known to cause nervous damage to people exposed to it in sufficient quantities. Using hexane as solvent also results in a solvent loss of about 1 ~ 8 lit. / Metric ton of seeds processed [1]. All of these issues combined with the necessity of severe extraction (temperature and pressure) conditions and environmental concerns have resulted in renewed interests in using an alternative solvent for extraction. Typical solvents of interest are alcohols and supercritical fluids such as carbon dioxide. Alcohols require a high solvent to feed ratio, but solvent recovery becomes an issue as alcohols usually tend to form an azeotrope when mixed with water. Supercritical extraction using carbon dioxide has the disadvantage of the requirement of very high extractor pressures with magnitude in the range of around 20 ~ 35 MPa at 45 ^{o}C. This work explores the use of liquid propane as a solvent for extraction.

1.2 Literature Review

1.2.1 Extraction

Extraction is one of the most commonly employed unit operation for separating two or more compounds from a mixture. The process of separating a substance from a liquid mixture is termed as Liquid Liquid Extraction (LLE) and that for separating a substance from a solid material / mixture is called Solid Liquid Extraction. Solid-Liquid extraction or leaching as it is commonly referred to, is the separation of one or more components of a solid mixture by preferential absorption through contact with a liquid solvent.

The operation of leaching has been used in the metallurgical industry for ages. It is useful for extracting minerals and metals from their naturally occurring ore forms. Extraction is also used in for extracting materials from natural sources such as extraction of soybean oil, sugars, etc. Hot water is used as a solvent for extraction of sugar from sugar beets, while many organic solvents have been employed for extraction of vegetable oils. One of the most commonly encountered examples of extraction is the preparation of tea, where tea leaves are soaked in hot water to extract the essence in the leaves.

In addition to its conventional use, the term extraction is also used to refer to the mechanical extraction of a constituent from a solid phase. Mechanical extraction uses operations such as pressing and extrusion, which involve applying pressure on the solid to force out the desired constituent.

1.2.2 Operation Considerations

As in case of almost any process encountered in the chemical industry, solvent extraction has to be performed with a particular set of operating condition for maximum efficiency. The ease of extraction depends on two major factors:

1. Solid phase resistance or ease of solvent penetration into a solid
2. Solubility of the material to be extracted in the solvent

Solid phase resistance is a very important factor in extraction because, in most of the cases encountered the solute to be extracted doesn’t lie on the surface of the solid but is rather entrapped in pores and crevices. The greater the difficulty for the solvent to reach the solute, lower is the extraction efficiency. Various mechanical processing methods such as grinding, crushing, etc. are employed to reduce the size of the solid particle and thus decrease the solid phase resistance. Extraction of certain minerals such as gold or copper requires the ores to be ground and ball milled into smaller particles, before extraction can actually be carried out. Vegetable oil extractions from seeds require a different set of operations to be performed on the seed before it can undergo extraction. This process is called pretreatment and involves operations such as dehulling, grinding, flaking, etc.

Solubility, the other parameter that effects extraction, is defined as the ability of the solvent to dissolve a solute from a mixture (solid or liquid). It is reported in the units of amount of solute extracted per unit mass of solvent. Solubility is an intrinsic chemical property of the solvent and temperature is the parameter which influences it the most. Higher temperatures result in higher solubility. Hence, extraction processes are normally carried out at higher temperatures. Other ways to improve extraction efficiency include inducing better mixing, providing longer contact time between the solute and solvent, increased surface area and pressure variations.

1.2.3 Equipment

Extractors are available in different sizes and types. The type of extractor used depends up on a variety of factors such as the type of material extracted, the product quality needed, availability of funds, type of extraction to be carried out and the manner of contacting solvent with the solid. Thus broadly, extraction process can be classified into two types:

1. Batch Operation
2. Continuous Operation

Unsteady state extraction involves a batch or semi-batch operation. Here, a batch of solid is contacted with solvent, allowed to reach equilibrium and then separated (batch) or a batch of solid is contacted with a continuously flowing stream of solvent (semi-batch). The choice of the mode of operation depends on the desired production rate of the final product. Treybal [2] lists the different types of equipment that are frequently used in extraction processes. Some of the most widely used equipment being percolation tanks and agitated vessels.

Extraction processes can also be carried out in a continuous fashion by changing the process design considerations. Solid handling processes are always known to be more complicated to design and fabricate as compared to processes that work only with fluids because of the difficulty in transportation and handling of the solid material. Due to the process requirement to handle solids, continuous processes are less preferred as compared to batch operation. Some examples of steady state, continuous extractor operation include thickeners, DeSmet extractor, Bollman extractor, etc.

1.2.4 Solvent Characteristics

The characteristics an ideal solvent should possess as defined by Treybal [2] are,

1. availability
2. inflammability
3. non-toxicity
4. high solute solubility
5. high diffusion rate

Propane is one of the chemicals, which satisfies most of the characteristics required for a good solvent. The only disadvantage of using propane in comparison to hexane it requires a higher solvent to feed ratio. Table 1.1 compares the properties of propane and hexane and highlights important parameters such as the flash point, the boiling point, toxicological data, and fluidic properties which would suggest that propane could be a good substitute.

Parameter	n-Hexane	Propane
Density of liquid @ 60 F (lb / cu. ft.)	41.5	31.5
Vapor Pressure @ 70 F (Psia)	2.5	142
Boiling Point @ 1 atm (F)	156	-44
Flash Point (F)	-10	-156
Oil Solubility	Depends on temperature	Depends on temperature
Toxicological Limit Inhalation (ppm/hr), Oral (mg/kg)	Inhalation: 12000 Oral: 28700	Negligible
Explosion Limit (%)	1.2 ~ 7.7	2.1 ~ 9.5

Table 1.1 Solvent Properties [3]

1.2.5 Introduction to Soy

Soybean is one of the major food crops worldwide because of its favorable agronomic characteristics, high quality edible oil products, high quality animal feed meal, and it is available at reasonable prices [3]. Figure 1.1 shows the production of soybeans in various countries [4]. The use of soybean and soybean related products started around about the 1920’s in the United States, with less than 108,000 MT being produced in 1922. This has increased to about 80 million MT in 2001. Figure 1.1 gives a general idea of how the soybean oil production has grown in the past decade.

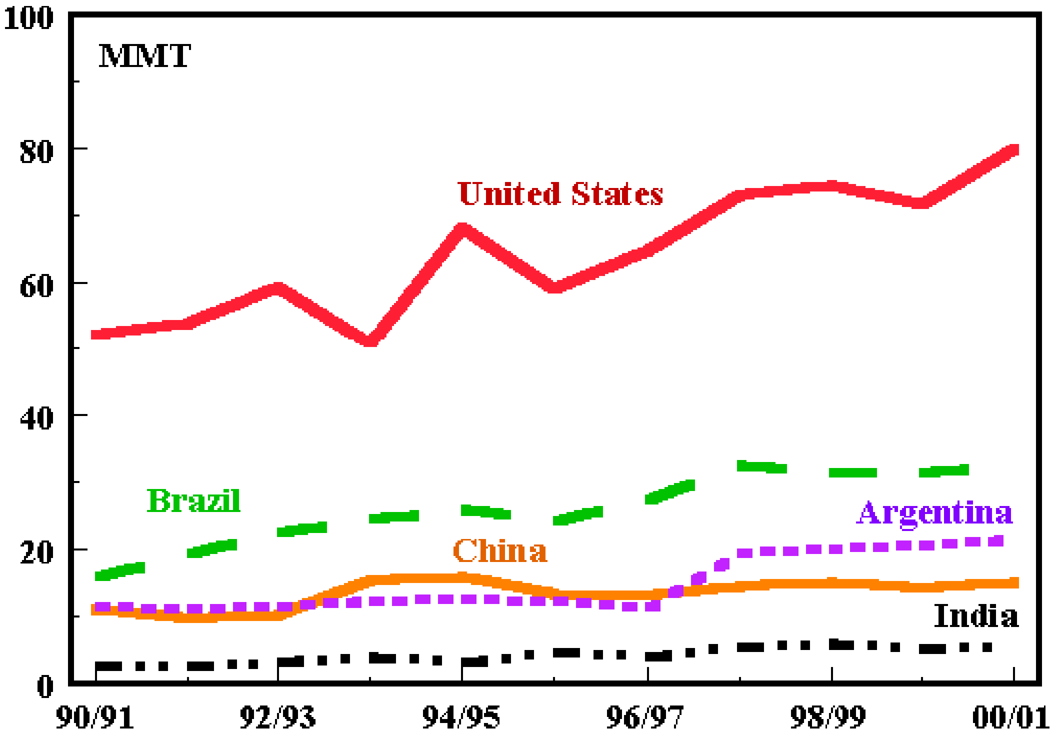

Figure 1.1 Soybean Production Revenues [4]

1.2.5.1 Soybean Composition

Commercial soybeans consists of about 20 % oil, with the rest constituting of proteins, carbohydrates, fatty acids, inorganics and minerals, amino acids, phospholipids, and sugar. The approximate composition of soy beans is summarized in Table 1.2 [3].

Component	Weight Percent
Moisture	11.0
Protein	37.9
Fat	17.8
Fiber	4.7
Ash	4.5

Table 1.2 Soybean Composition [3]

Carbohydrates [3]

Whole soybeans consist of about 35% carbohydrates, of which about 20 % is insoluble carbohydrate. Stachyose, raffinose, glucose and sucrose form the majority of the carbohydrates found in soybeans. Sugar (sucrose and glucose) is a major raw material used in the manufacture of ethanol. This high content of carbohydrates can also be put to use, by either extracting the sugars for edible use or for commercial chemical manufacture.

Fatty Acids [3]

Soybeans primarily consist of triglycerides and triglecerols, with linoleic, linolenic and oleic acids forming the majority. The other fatty acids present in soybeans and soybean oil are listed in Table 1.3 [3, 5].

COMPONENT	SOYBEAN (wt. %)	SOYBEAN OIL (wt. %)
Palmitic Acid	1.6	7 ~ 12
Stearic Acid	0.6	2 ~ 5.5
Oleic Acid	3.8	20 ~ 50
Linoleic Acid	8.7	35 ~ 60
Linolenic Acid	1.0	2 ~ 13
Myristic Acid	< 0.1	< 0.5

Table 1.3 Soybean oil fatty acid compositions [3]

Saturated fatty acids are the component that contribute to bodily fats in humans and hence are considered to be anti-nutritional when consumed. The low content of saturated fatty acids is what makes soybean oil popular as an edible oil.

Minerals & Inorganics [3]

Minerals form a very important part of the human diet and a person requires a minimum amount of minerals in his daily diet. Hence, the mineral content of soybeans is very important. Soybeans consist of about 2 % potassium, 0.5% sodium, 0.3 ~ 0.7% phosphorous with trace quantities of magnesium, calcium and iron.

Proteins [3]

Soybean meal is a very popular animal feed because of its high protein content. Proteins constitute about 40% of soybeans. Soy proteins consist of amino acids in varying compositions, trypsin inhibitors and haemagglutinins which are nutritionally important. Soy proteins are generally heat inactivated, which is as a major constraint when processing soy oil. Processing temperatures higher than 100 °F generally tend to depreciate the quality of the soy oil produced.

1.2.5.2 Physical Properties [3]

The physical properties of soybeans are a function of various parameters, which include climatic conditions during growth, oil composition, temperature and pressure, molecular weight, fatty acid chain length, etc. The physical properties of soybean are critical parameters which have to be considered when designing soy processing equipment and processes such as extractor, dryer, etc. The physical properties of soy oil are listed in Table 1.4 [3].

Property	Value
Specific Gravity at 25 °C	0.9175
Refractive Index, n_D^{25}	1.4728
Viscosity at 25 °C (cP)	50.09
Solidification Point (°C)	-10 ~ -16
Specific Heat at 19.7 °C (cal/g)	0.458
Heat of combustion (cal/g)	9478
Flash Point (°C)	328
Fire Point (°C)	363

Table 1.4 Physical Properties of Soybean Oil [3]

1.2.5.3 Uses [3]

Soybeans have been used by man from as early as 11th century B.C., when the Chinese started to use soy oil for edible purposes. Since then, the use of soy and its various constituents have found widespread use in the domestic as well as the industrial sectors. The primary use of soybeans includes edible oils, soaps, printing inks, explosives, etc.

One of the major applications of soy is the manufacture of wood adhesives from soy protein. Johnson, Laucks and Davidson obtained patents using soy meal and soy proteins to make adhesives and paints, though soy based glues lost out to petroleum based chemicals by the mid 1960's. However, with increasing petroleum costs and growing environmental concerns on petroleum based chemicals, the interest in soy based products has reignited. Other industrial uses of soy include manufacture of plastics. Henry Ford used soybeans and other soy allied materials to manufacture plastics, lubricants, window frames and other parts in his automobiles and trucks. Textile fibers, paper coating, firefighting foams, fertilizers, etc. are the other uses of soybeans.

1.2.5 Soy Extraction Process

The process of vegetable oil extraction consists of four main steps:

1. Feed Preparation
2. Extraction
3. Solvent and Oil Recovery
4. Oil Refining

There exist many different methods to carry out each of these steps depending on the type of extraction and the solvent used. A general block diagram for vegetable oil extraction is shown in Fig. 1.2.

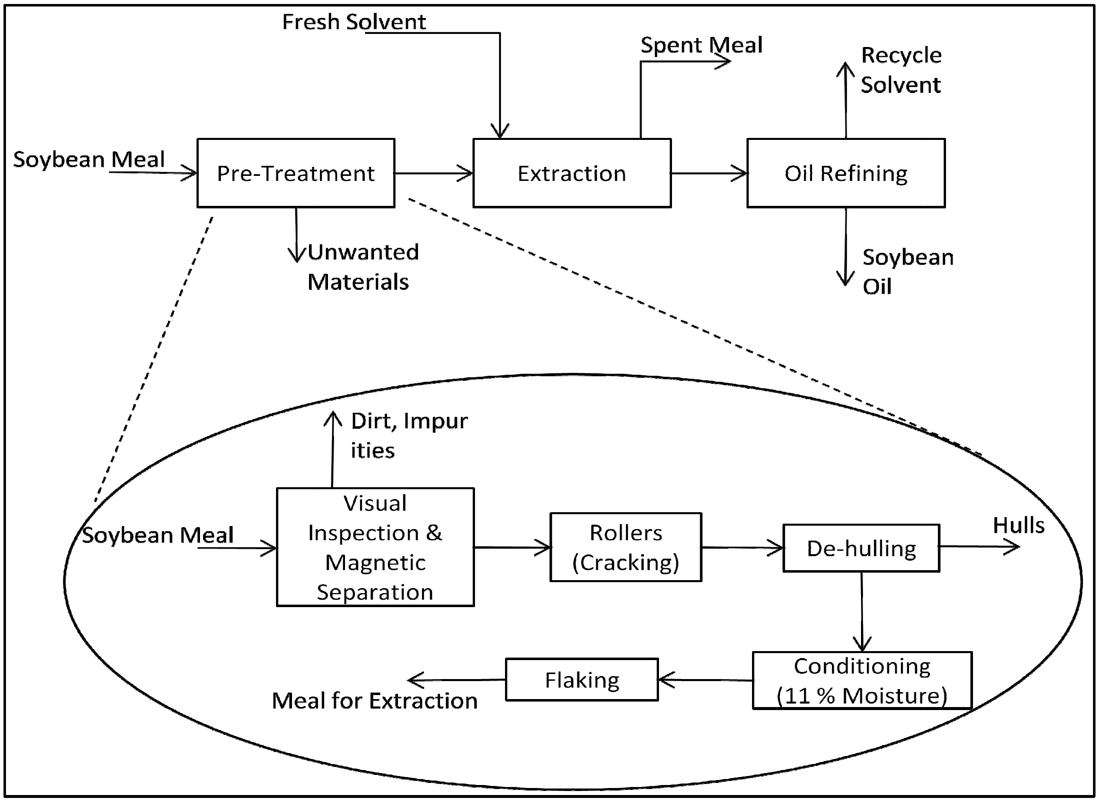

Figure 1.2 Vegetable Oil Processing Block Diagram

Feed preparation is one of the most important steps in oil extraction. As discussed earlier, this step is required to decrease the solid phase resistance of the system and thus allowing easy access of the solvent to the vegetable oil. The soybean seeds are first cleaned and dried to remove any macro impurities and reduce the moisture content of the seeds. The moisture in the soybean meal competes with the oil during extraction and thus reducing the overall efficiency of the process. Typically, soybeans are dried to a moisture content of less than 10 % before being sent for further processing [3]. This is followed by cracking, wherein the soybeans are passed through a set of rollers to cut and break the seeds into suitable sizes for further operations. The cracked soybeans are then sent to a dehulling unit, where the cotyledons ("rudimentary leaf of the embryo of the seed" [22]) are separated from the seed hulls. This is a very important step in the feed preparation as it removes one of the barriers (the hull) to solvent diffusion. The soybeans are then subjected to conditioning by heating at 161 ^{o}F in presence of steam or under water spray to bring back the moisture content of the stream to about 11 % to make the seeds easy to flake [3]. The soybeans are then flaked to obtain thicknesses in the range of 0.2 mm to 0.5 mm [3]. This is a very important step and all the extractors are designed based on the assumption of processing flaked meal. The flakes are then passed through an expander, which converts the flakes into collets (cone shaped sleeve [22]) and then the stream is sent for extraction.

The extraction process mainly consists of the extractor and the mode of extraction adopted i.e. continuous, batch or semi-batch. In continuous extractors, the solvent (in liquid phase) is contacted with a continuous stream of solid soybean meal flowing into the extractor; whereas, in a semi-batch process, a continuous stream of liquid solvent is contacted with a batch of meal. On the other hand, the batch process is carried out in a purely batch fashion, with no input or output streams present during the time of processing. When the solvent comes into contact with the solid meal, it first penetrates into the cake and then from the surface of the seed (flake), it diffuses into the pores. It then dissolves the oil and diffuses back to the surface and into the bulk flowing solvent stream. The solvent-oil mixture that exits the extractor is called the miscella. The miscella is then sent to the solvent–oil recovery system.

The solvent recovery system is the next step in the process and it consists of unit operations that separate the oil and solvent from the entering miscella. Evaporation, flash separation and distillation are the most commonly employed processes. The difference between the boiling points of the oil and solvent (usually hexane) is used as the driving force for separation. The separated solvent is then recycled back for reuse and the oil is sent for further refining.

There are not many literatures available on extraction of soybean oil from the seeds using propane as solvent; but there are some which discuss solvent extraction with respect to other kinds of oil seeds using a different variety of solvents, most commonly hexane. Most of the solvent extraction processes available in the literature vary in the type of solvents used, and the process conditions, required to obtain higher efficiency. Hexane has been commercially the most successful solvent employed in such a process and there is a large amount of data related to hexane related extraction, followed by extraction using supercritical carbon dioxide.

M. Barvi et al. (2002) [6], studied the extraction of sunflower oil using supercritical carbon dioxide and determined the optimized parameters necessary for maximum extraction to be achieved. The oil seeds were subjected to extraction using supercritical carbon dioxide at conditions of 280 bar and 40 ^{o}C for 10 mins. The extract phase was then subjected to a series of flash separations (pressure reducing as the extract moves downstream) to give an essentially solvent free oil. The extraction was about 60 % of the maximum attainable extraction; about 70 % with a considerable increase in operating time. Supercritical carbon dioxide can also be used to recover other constituents such as vitamins, isoflavones, sterols, etc. from oil bearing seeds [7]. This occurs at higher pressures and temperatures as compared to that necessary for fatty acid extraction [7].

Solvent extraction with supercritical carbon dioxide was found to be dependant on temperature and pressure of the extraction process. The extraction yields have been found to improve with increasing pressure due to an increase in solubility of various components in soybean, while it has been found to be inversely proportional to temperature [8]. This phenomenon of reduced oil extractability at increasing temperatures can be attributed to the thermal instability of soy oil and the denaturation of proteins which results in a decrease in the solubility at elevated temperatures [9].

N. Hassanen (1985) [1], F. Zhang (1995) [10] and M. Thobani (1995) [11] all did research on oil extraction as a part of their PhD thesises. Hassanen worked on semi-continuous extraction of oil seeds using multistage extractors. The study mainly dealt with cotton and soybean seed extraction using alcohol as solvent and also the application of sequential extractor models, with the extract / miscella from the first stage transferred to the second, then to the third and fourth and so on till the final stage. Higher yields were obtained while using iso-propanol as the solvent compared to using hexane with just 1.8 % by weight of oil remaining unextracted. Extraction using alcohols was also shown to result in a better quality extraction as compared to the traditional hexane extraction. Research also shows the ill effects of high moisture content in the fresh feed, which results in lower extraction yields. Isopropyl alcohol increased the oil recovery by 1.5 ~ 4.7 % from soybean flakes and provides an 4.8 ~ 13 % increase from broken soybeans as compared to that obtained while extracting with n-Hexane [12]. One common problem of using iso-propanol is its nature to form an azeotrope with water. Thus, the presence of water results in less solvent purity, which has to undergo rigorous separation before reuse.

Sparks et al. (2006) [8], also studied solvent extraction of rice bran using liquid propane. The extractions were carried out in a batch process and with pressure ranging from 0.62 ~ 0.76 MPa at temperatures of 45 ~ 65 ^{o}C. Maximum extraction yield attainable was about 14 % less than that attainable with hexane as solvent, indicating need for a higher solvent to meal ratio.

A number of patents have been obtained in the United States regarding solvent extraction processes for oil extraction from natural / vegetable sources. Franke obtained a US patent in 2001 for developing a multistage extraction process for vegetable oil extraction. The process consisted of a pressure vessel used as the extraction zone, which was operated in purely batch fashion. Liquid solvent was used for extraction, and the meal to be extracted was subjected to partial extraction with a known quantity of solvent. This miscella thus obtained was fractionated to separate the solvent from oil and the solvent is reused for extraction stage 2.

Propane when used in a narrow range of temperature and pressure conditions is known to selectively extract the triglycerides and fatty oils from the meal leaving behind unwanted byproducts of extraction such as phosphatides, sterols, etc. [13], as can be seen from the fact that propane is a major solvent used in separation / extraction of fatty acids and triglycerides from crude vegetable oil [14].

1.2.6 Process Modeling

Improving the process efficiency and finding an alternative solvent has always been of utmost importance in the study of vegetable oil extraction processes. There are numerous techniques using which the efficiency of the process can be improved and there are many compounds and combination of compounds that can be used in the process. Experimentation is not always the correct method for determining these optimum parameters. Experiments consume a large amount of time and also require a considerable amount of investment. Hence, it is necessary to have a better technique to implement such changes to a process and study the changes without having to conduct full scale experimentation – lab scale or on the actual unit. Process models are one way to accomplish this.

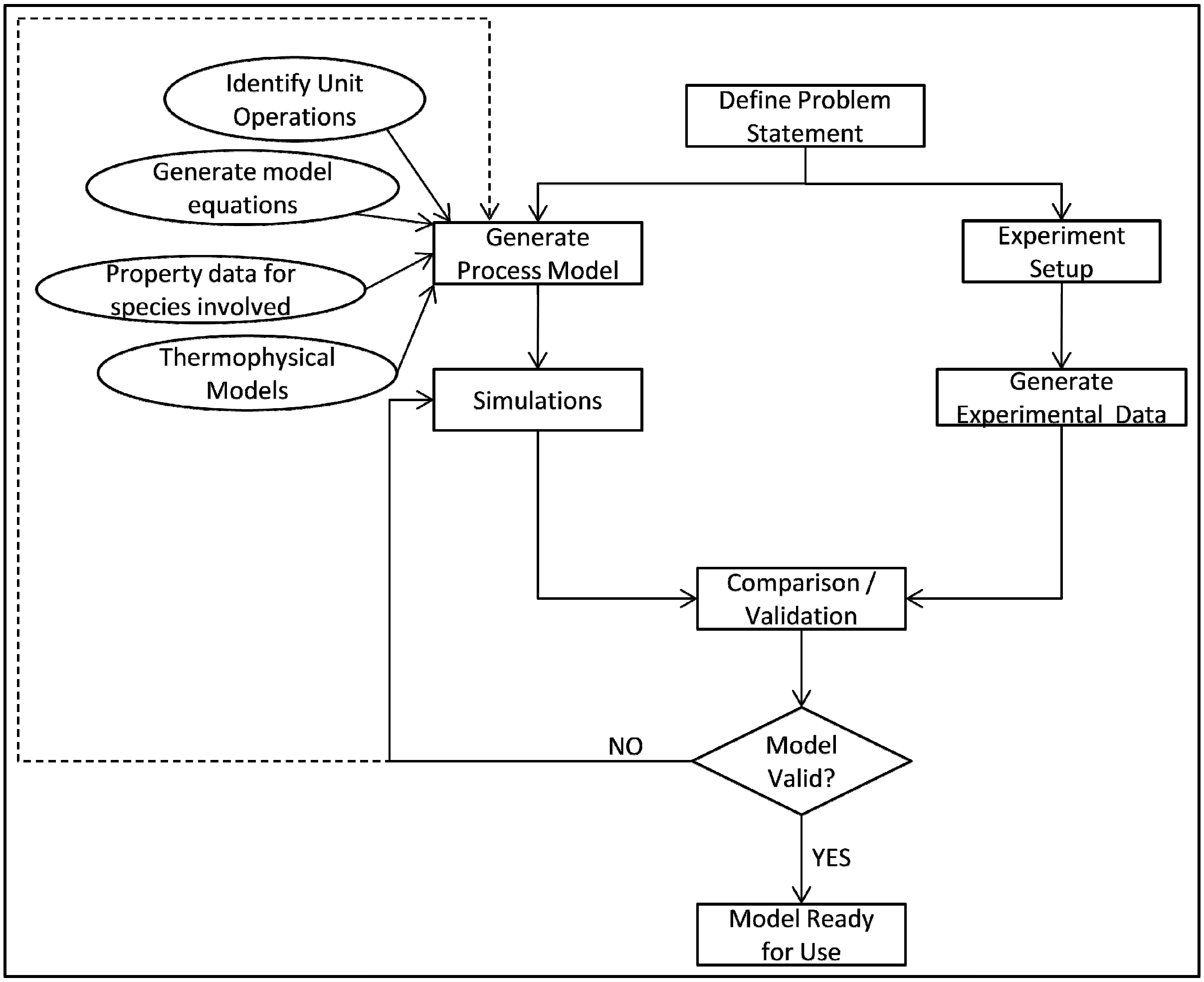

Figure 1.3 Flowchart to accomplish process modeling

Figure 1.3 shows the major steps involved in process modeling and simulation. The first phase is, ‘Identifying and defining the problem statement’. Here, the basic objective of the process model is formulated and the necessary questions the model has to answer are determined. This is followed by the ‘Model generation’ phase, where the actual model is put together. It could be a mathematical model for a single unit / process or it could be the model for the entire process system. A large amount of data is necessary for implementing this step. Data about the actual unit operations and process involved in the system, physical and chemical data for the various chemical compounds that are involved in the system and the thermophysical models that are necessary to predict the behavior of the species in different phase or as a mixture. This is also called as the thermodynamic modeling of the process. Once the model is ready with all the data fed in, the model is simulated i.e. an imitation of the model is run (mock up) using pre-developed computer simulation engines to give the resultant model output. This model result can be compared to experimental data previously generated and if the model fits, it can be sent for further implementation. Otherwise, the parameters of the model are changed and it is re-simulated till a valid model results.

Mathematical models form the basic block of any modeling problem. The governing equations for the system under investigation are generated and solved. The solution obtained can be compared with experimental data to check the validity of the model. Another kind of modeling problem is that resulting for the entire process system under consideration. Such kinds of models are necessary to simulate an entire process flowsheet and are used for validating or analyzing the feasibility for a particular process. These in general are referred to as process models, unless specified otherwise (Mathematical models, empirical models, etc.). Process models are a collection of pre-generated mathematical models for known unit operations or processes, which have to be collectively solved in order to generate a solution for the entire flowsheet. Various process simulators are available in the market which includes ASPEN, ChemCAD, HYSYS, PRO-II, etc. Predefined subroutines are available in each of these simulators, which can be used to create the necessary process flow diagram. The user needs to select the appropriate process units, chemical species involved, thermodynamic model, type of solving approach to be used (sequential or modular) and also needs to specify the necessary process streams and unit information such as temperature, pressure, composition, conversion (for reactors), etc.

Process models are created mainly for evaluation of a new process, to determine the economic feasibility, for process optimization and process modification. Little published literature exists on process modeling for soy oil extraction. Martinho et al. (2008) [15], generated a process model for soy oil extraction using hexane as the solvent and validated it with industrial data. The model was mainly generated to study the process and optimize operational parameters so as to obtain best possible solvent recovery. Soybean oil was modeled as a mixture of triglycerides, sterols, free fatty acids and tocopherols. Process models were created for extraction, solvent recovery, desolventization, mineral oil scrubber (MOS) and the water-solvent separation sections of the process. The process model was created to represent the most common process used in industry for solvent extraction. A sensitivity analysis of the results was carried out to determine the operating parameters that have the most notable effect on solvent recovery for the process. Few studies exist in the literatures that use propane as the solvent and hence, further investigation is required.

1.2.7 Thermodynamic Models

“Finding good value for inadequate or missing physical property parameters is the key to a successful simulation and this depends upon choosing the right estimation methods.” (E. Carlson, [16]). Selection of thermodynamic models is one of the most critical and important steps in process simulation. A model is only as good as the data that it is supplied with.

Hence, obtaining accurate physical and chemical properties and selecting the appropriate model for simulation is very important.

Properties of many chemical species can be found in the literature. But, not all of them follow a particular model as in; a model used for vapor estimation would not necessarily apply to liquids or solids. Application of such models could weaken the simulation results. Various thermodynamic model and property prediction models are available in process simulation software, some of which are presented in Table 1.4.

Some of the important factors effecting the selection of thermodynamic models are,

1. type of chemical species involved
2. region of interest (temperature and pressure)
3. mixing rules
4. data available

Thermodynamic Models – Equation of State	Thermodynamic Models – Activity Coefficient
Ideal Gas Law	Non Random Two Liquid (NRTL)
Henry’s Law	Van Laar
Lee Kesler (LK)	Pitzer
Redlich Kwong (RK)	Wilson
Soave Redlich Kwong (SRK)	Regular Solution
Predictive SRK	UNIFAQ
Peng Robinson (PR)	UNIQUAC
Benedict Webb Rubin (BWR)	Modified UNIQUAC
Statistical Associating Fluid Theory (SAFT)	

Table 1.4 Common thermodynamic models and equations in process simulation softwares

There are different types of chemicals that process industries deal with, organic as well as inorganic. The selection of the models is greatly effected by the property of the chemicals. Each of these chemicals has its own specific characteristic such as the polarity, ionic nature, and basic building block for the compound. There are innumerable chemicals and each one of them cannot be processed with a common set of assumptions. Each model has its own limitation. Thus, a thermodynamic model of a particular type is only applicable to a particular set of chemical compounds. Pressure and temperature conditions also affect the model selection criteria.

The process of model selection gets trickier when dealing with chemical mixtures. Chemicals alter their behavior in presence of other chemical(s). The presence of molecular forces of attraction, repulsion and others play a very important role in how a mixture behaves. One of the most common phenomena is that, a mixture never has a fixed boiling point. While dealing with mixtures, the most important data required is the equilibrium parameter such as vapor-liquid equilibrium, liquid-liquid equilibrium and solid-liquid equilibrium. E. Carlson [16] gives a list of figures which serve as a guide for simulation engineers for selection of thermodynamic models. These figures are presented in Appendix I.

CHAPTER 2

OBJECTIVES

2.1 Research Objectives

As can be seen from the literature review, not many models have been generated to simulate the process of soybean oil extraction, with the exception of a continuous steady state extraction unit using hexane as solvent [15]. Since propane use as a solvent has wide interest in the field of vegetable oil extraction, process models are necessary to check the feasibility of using propane in extraction units. Propane has been conventionally used for the extraction of oil components from crude oil, as it has a high selectivity for oil components as compared to the other compounds such as sterols, phosphatides, etc. present in vegetable seeds. Sparks et al. (2006) [8] studied the use of propane for extraction of rice bran oil. But, not much information is available in the literature regarding use of propane as a solvent for extraction of soybean oil. Hence, it is useful to determine the feasibility and economics of extracting soybean oil using propane.

The main intent of this study was to develop a process structure for obtaining high quality soybean oil from flaked soybean seeds. The following steps were identified as an outline of work required:

1. Study and analyze available literature regarding vegetable oil solvent extraction techniques, specifically using propane for extraction of soybean oil.
2. Temperature and pressure conditions play a critical role in extraction processes, and also affect the thermodynamic parameters and solubility characteristics of the solute. Hence, generate a model to predict the variations in extraction with extractor conditions.
3. Develop process diagrams, including the extractor and other down stream operations such as refining and solvent recirculation that are necessary for implementing the extraction process.
4. Generate process models to represent the system and perform steady state and dynamic simulations of the models to assess the variations and determine an optimum mode for extraction (steady or dynamic).
5. Test and validate the process models with experimentally obtained data using a 5 liter continuous propane extraction system located at the Hazardous Reaction Laboratories, Oklahoma State University.
6. Generate preliminary estimates for equipment and utility cost.

2.2 Scope of Work

This work deals with a process that uses liquid propane as solvent for extraction of soybean oil from flaked seed meal. The process developed would have a lower volatile emission and lower risk of toxic hazards. The process conditions are also optimized so that the process would result in maximum oil yield and higher quality oil as compared to the previous processes and at the same time reducing the capital investment and operating cost for the process. The report also gives a preliminary cost estimation analysis and an approximate figure for the capital investment that would be necessary to implement the process.

CHAPTER 3

PROCESS OUTLINE

The conventional process used for soy oil extraction has been discussed in the literature review section. It consists of a soybean meal pre-processing section, where the soybean seeds are subjected to various operations which make it suitable for solvent extraction. This is followed by the actual extraction process where the soybean meal is subjected to solvent treatment and finally the solvent-oil separation system necessary to refine the oil and recover solvent for the next cycle. This section deals with the processing units involved in the solvent extraction and subsequent sections including the oil refining methods necessary to get adequate oil purity.

For the purpose of initial assessment of the system and to have a feel of the operating parameters, batch and semi-batch operation modes were selected for investigation. The entire process is divided into 4 sub-sections for ease of analysis, each of which is detailed in the subsequent paragraphs. Also, alternative unit operations and/or processing methods have been analyzed for their applicability and feasibility.

3.1 Semi-Batch Processing

A semi batch process is a combination of batch processing and continuous processing. A semi batch process can have a continuous input stream without a continuous output and visa versa. It could also be a process which operates with one input (or output) in batch manner while the other one is in continuous manner.

The entire flowsheet starting with the solvent extraction step is analyzed for semi-batch operation. The system is divided into four sub-sections namely, solvent extraction, solvent recovery, oil processing and raffinate treatment.

3.1.1 Solvent Extraction

The solvent extraction stage consists of an agitated vessel type extractor also referred to as the DVT in this study. The extractor is a horizontal pressure vessel fitted with a plow agitator to ensure thorough mixing of the soybean meal and the solvent; thus providing improved process efficiency. The DVT is fitted with an external jacket, which is used for maintaining the extraction temperature at a constant value (as necessary). Temperature affects the solubility of oil components in the solvent, and hence it is very important to maintain a constant temperature for extraction. In addition to these accessories, the DVT is also equipped with a filter screen located at the solvent exit, to reduce or eliminate loss of solid meal along with the extract, which could lead to problems during the later stages. Figure 3.1 shows the flow diagram for this section.

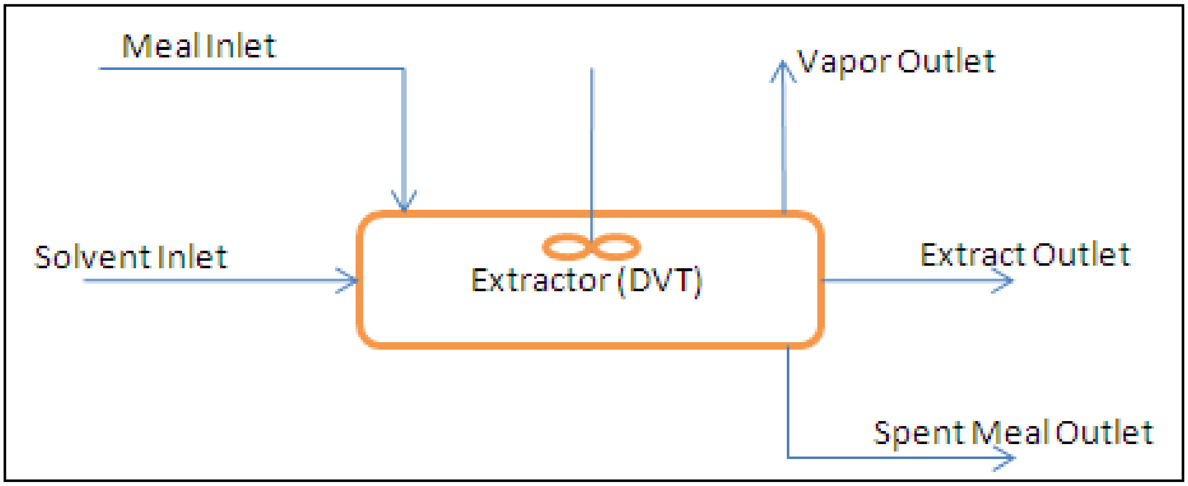

Figure 3.1 Extractor unit and flow streams

The extractor is initially filled with the pretreated and flaked soybean meal obtained from the previous section of the process. Once the extractor is filled, the DVT is subjected to vacuum to remove air from the system and eliminate the presence of oxygen in the extraction zone. Oxygen forms one of the sides of the fire triangle and hence, by eliminating it, chances of explosion can be reduced. The system is put under vacuum, to reduce its oxygen content (and also the non-compressible gases downstream) to lower than the minimum oxygen concentration (MOC). After this step, propane in gaseous form is passed into the DVT to increase the pressure to the desired level. At this time, liquid propane is pumped into the extractor and once it fills the system, the exit valve is opened and propane circulation step begins. The propane pumped into the system, extracts the oil from the seeds and exits through the solvent exit via the filter screen, and is sent to the solvent-oil separation section.

3.1.2 Solvent Recovery and Recycle

The purpose of the solvent recovery section of the process is to separate the extract, which is a mixture of soybean oil and propane and recover propane for reuse. The choice of processes to be employed for solvent recovery is based on the difference in the physical properties of soybean oil and propane. Differences in the boiling points are used as the driving force for separation. A schematic of this section is shown in figure 3.2.

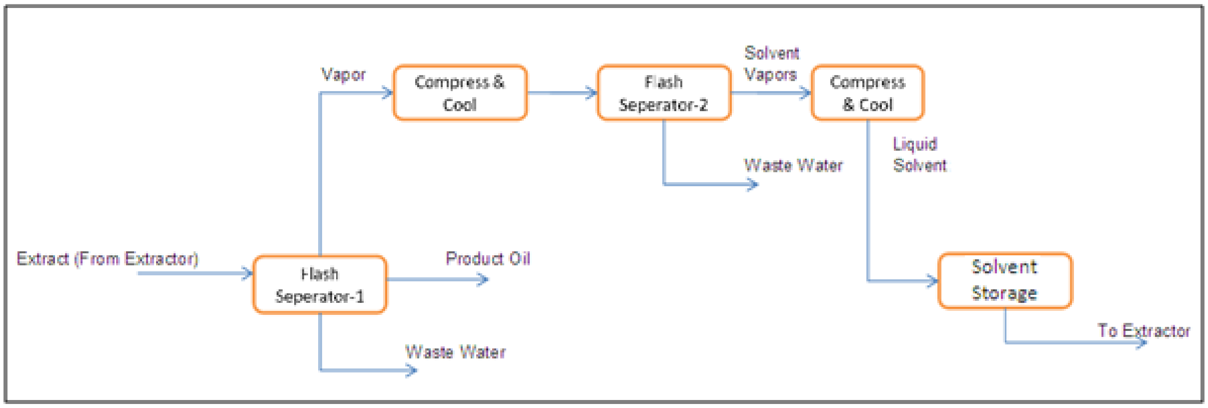

Figure 3.2 Flow diagram for solvent recovery system

This section employs the advantage of the low boiling point of propane to accomplish solvent recovery. The extract emerging from the extractor is heated to the required temperature and then flashed in a pressure vessel via a pressure relief valve. Here, the low boiling propane is separated from the higher boiling oil components, and thus a relatively pure oil stream (containing some propane) and a relatively pure gaseous propane stream are obtained.

The extract is predominantly a mixture of soybean oil components and propane, but it also contains traces of other contaminations, mainly moisture (water). The moisture present in the flaked soybean meal forms the source of this contamination, which competes with the oil components during extraction (though only a small amount is extracted as compared to the oil components) and flows to the solvent recovery section along with the extract. Hence, the streams obtained from the initial flash separation contain some water and consequently require further processing.

The water in the oil can be separated by taking advantage of the density difference and insolubility of oil in water and thus, water can be decanted out of the oil to leave a product stream mainly consisting soybean oil components with small quantities of propane. This stream is sent to the oil processing step to reduce the presence of propane to trace quantities. The gaseous propane exiting the initial flash also contains some amount of moisture and this moisture cannot be allowed to buildup. The buildup of moisture in the process has an adverse effect on the extraction efficiency and also increases the corrosion factors for the equipment. Hence, the gaseous propane stream is compressed to partially form a liquid, cooled to a required temperature and flashed again in a separate unit, to separate water as the liquid waste (bottoms) and a gaseous stream with high solvent propane fraction. This pure gaseous propane is then compressed, cooled and liquefied and is recycled back to the solvent holding tank (Figure 3.2).

3.1.3 Oil Processing

The oil that is obtained from the oil-solvent separation system still has some propane dissolved in it, which has to be removed. Also, apart from solvent, other impurities such as gums (lecithin’s), phosphatides, etc. are also known to be found in the extracted oil when using hexane extraction, and hence, hexane employing extraction processes require extra purification of the oil before it can be sent to the packaging and delivery section [3]. But, propane is known to be a very selective solvent and extracts only the major components of soy oil including the triglycerides and vitamins; while the other components of the soybean meal are extracted, only in traces. Thus, due to its high selectivity, soy oil obtained from propane extraction could be relatively pure and not need any further treatments.

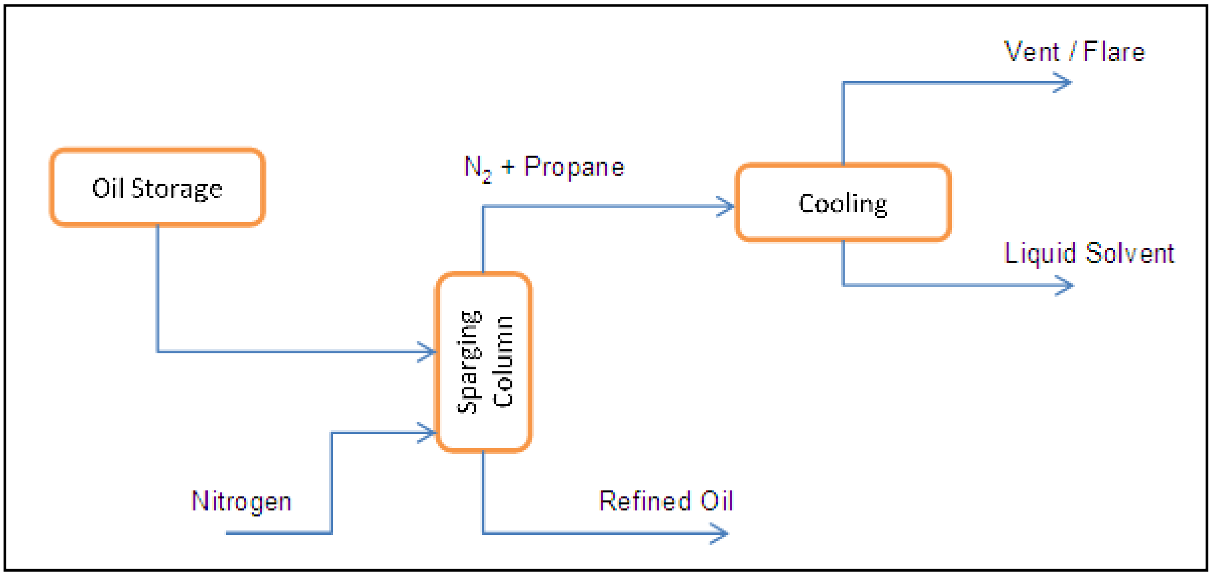

Figure 3.3 Process outline for the oil refining system

One additional processing unit is added to the extraction process, which is used to reduce the propane concentration to trace amounts. There are many possible separation techniques that can be employed. Flash separation, the process which was employed in the earlier step does not prove to be very useful, even at vastly different process condition. A distillation unit can be employed for this process, and can result in high purity oil, but requires a lot of energy input. Another disadvantage of using temperature as a driving force is the thermal degradation of oil when exposed to high temperatures. Other techniques which can be employed are membrane separation which uses the difference in molecular sizes of soy oil components and propane stripping or sparging. In this process, the operation of sparging is employed. The process consists of collecting the extracted soy oil in a pressure vessel, and the oil is continuously sparged with gaseous nitrogen for a sufficient amount of time so that the propane content in the oil is reduced to trace quantities (ppm or even ppb). Figure 3.3 shows a pictorial representation of this system. The propane nitrogen mixture is then cooled to partially condense the propane solvent, which is recycled back to the solvent collector. The nitrogen gas can be recycled for the next sparging cycle or can be vented to atmosphere or flared.

3.1.4 Raffinate Treatment

During extraction, solvent propane diffuses into the pores of the soybean flakes, dissolves the oil, and diffuses back to join the bulk flow. This void generated by the loss of oil from the pores is filled by the solvent, thereby resulting in a shift in the material balance for the system. Additionally, propane also tends to get entrained in the void spaces present in the soybean mass. This loss of solvent can be significant and it can result in significant differences in the economics of the process. Hence, it is very important to recover the solvent before the soybean meal is discharged. Another reason which dictates the necessity for this step is that, extracted soybean meal has a large content of nutrients, and can be used as animal fodder or human food. But before it can be sent for processing, propane has to be removed as it can be hazardous to living beings when consumed and can present a fire hazard.

The high volatility of propane is used as a tool to accomplish the desolventizing of the extracted soybean meal. Once the extraction step is completed, and the majority of liquid propane drained from the extractor, the liquid solvent exit is closed and vacuum is applied to the system through the vapor exit stream shown in Figure 3.1. This vacuum pressure results in gasification of solvent propane which is entrained in the pores of the soybean meal. This gaseous propane is compressed, cooled and recycled back to solvent storage. Nitrogen purging can also be used to remove propane. The soybean meal is then off loaded from the extractor for further processing.

3.2 Process Modifications & Alternatives

Many alternative technologies can be applied for each section of the process that have been discussed earlier, some of which have already been mentioned. From the four sections, the extraction section is the most important and improving the extraction zone can significantly improve the process yields. Two specific options concerned with the mode of operation for the extraction zone are detailed in this section.

3.2.1 Multiple Alternating Extractors Modification

Multiple extractors, operating in an alternating fashion can be used as a substitute for the proposed single extractor semi-batch operation. Figure 3.4 shows a schematic of operation when 2 alternating extractors are used. When one of the extractor’s is goes through the extraction step, the other is runs though the rest of the processing zones (raffinate processing, raffinate unloading, fresh meal loading) and visa versa.

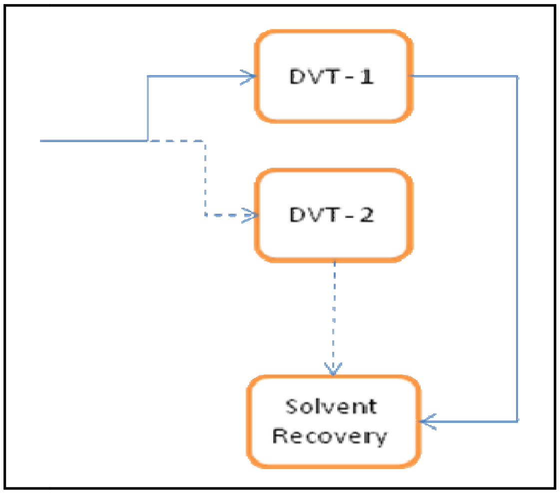

Figure 3.4 Schematic for multiple alternating extractor setup

Consider the operation for extractor 1. Initially, extractor 1 is filled with the fresh soybean meal and extractor 2 is not in operation. The extraction steps beings with circulation of solvent propane through extractor 1. During the time, extractor 2 is loaded with fresh meal, so that it can replace extractor 1 when it gets exhausted and proceeds to the other processing steps. This change of solvent circulation from extractor 1 to extractor 2 can be obtained using a switching mechanism with pipes and valves. During the time extractor 2 is being extracted, extractor 1 goes through the rest of the processing, namely – raffinate processing, raffinate unloading and fresh meal loading and is ready for the next batch to be extracted. When extractor 2 completes its extraction, extractor 1 enters the extraction step and thus, a virtually continuous product stream can be obtained, unlike the semi-batch process where, product is obtained only during the extraction and drain step and only the extractor is under some form of continuous processing while the other equipment lay dormant.

3.2.2 Multiple Continuous Extractors Alternative

The concept of continuous operation of multiple extractors is similar to the alternating extractor concept. But in this case, a continuous and steady product generation rate can be achieved, which is not possible with alternating extractors. In the earlier concept, with the extractors operating in rotations, continuous production is achievable, but not at a steady production rate. The amount of product oil extracted decreases as extraction time increases and reduces to a minimum at the end of the extraction time. Hence, the concept of continuous extractor operation is introduced to overcome this problem of the earlier operations.

This system consists of multiple extractors operating in series (placed one after the other on the same process line), with one extractor in spare. Figure 3.5 shows the general idea of such a system.

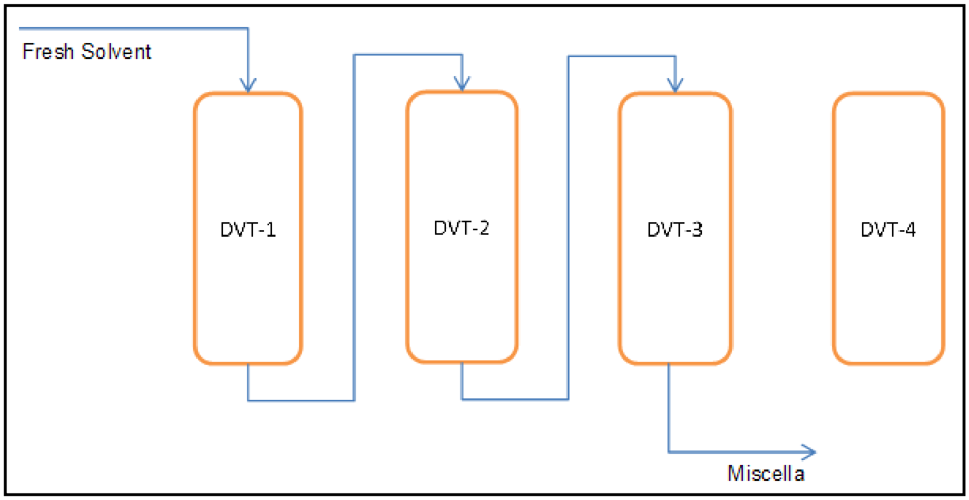

Figure 3.5 Stage 1 for the multiple continuous extractor setup

Consider the operation of such a system. The process initially starts with all the four extractors being loaded with the fresh meal to be extracted and just the first 3 extractors are placed in the process with the fourth one being offline. As extraction proceeds, after a specific duration of time, the meal in DVT-1 would be spent and cannot be extracted any further. At this moment, DVT-1 is detached from process and DVT-4 is inserted into the process such that miscella from DVT-3 flows into the freshly loaded extractor and the extraction operation continues. Figure 3.6 illustrates this scheme.

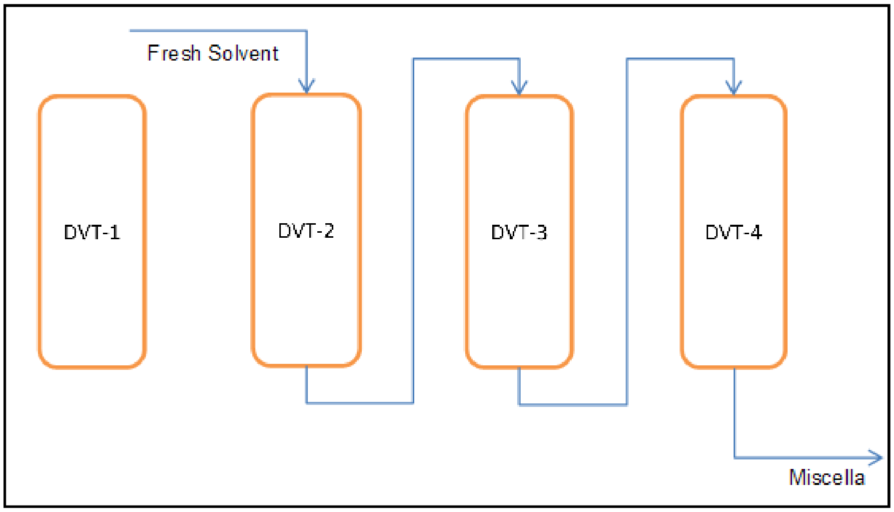

Figure 3.6 Multiple continuous extractor setup – Stage 2

Meanwhile, DVT-1 which was detached undergoes the remaining operations and is made ready with the next batch of fresh meal loaded into it. By this time, DVT-2 gets exhausted and is removed from the series, with freshly loaded DVT-1 placed at the end, after DVT-4 as shown in Figure 3.7.

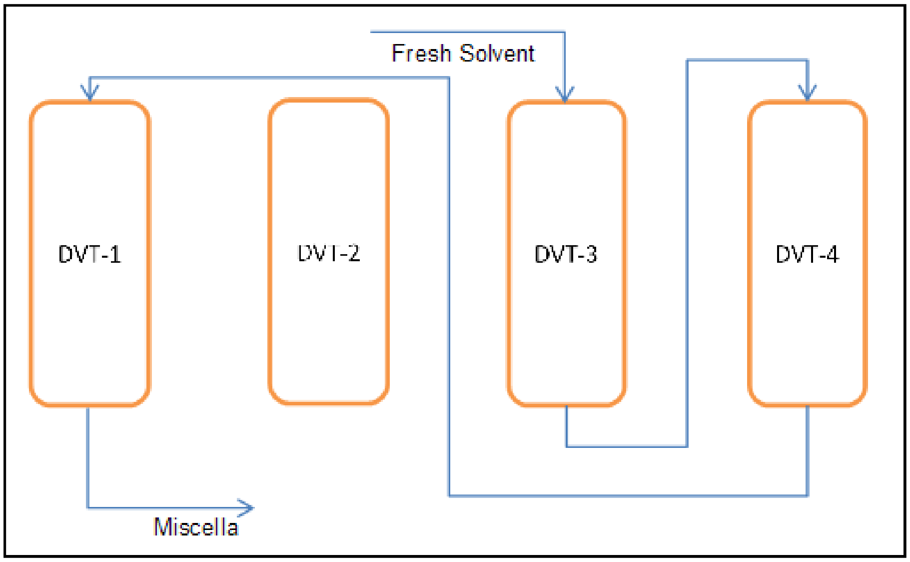

Figure 3.7 Multiple continuous extractor setup – Stage 3

DVT-2 undergoes the same post- and pre- extractions operations as DVT-1, when DVT-3 gets exhausted and is removed from the series; DVT-2 is placed at the end of the series after DVT-1. This is shown in Figure 3.8.

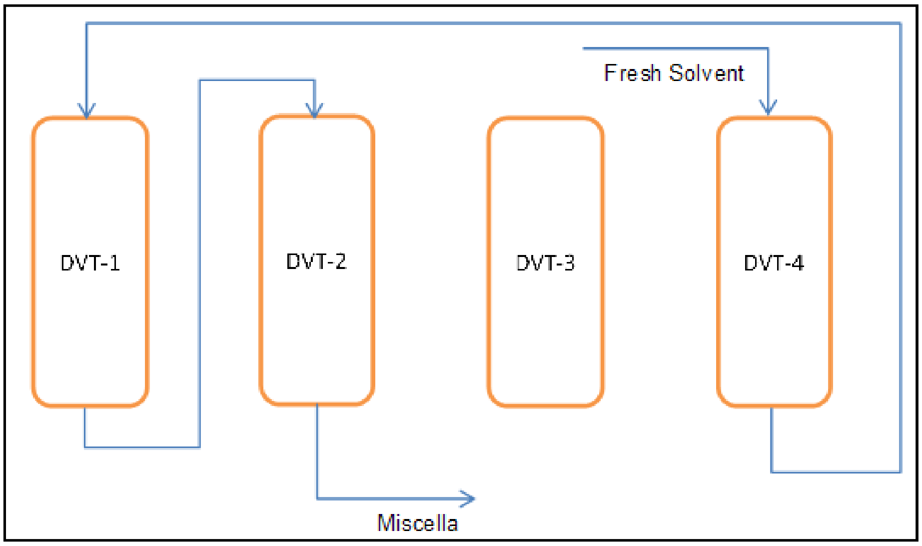

Figure 3.8 Multiple continuous extractor setup – Stage 4

This forms one complete cycle of extraction, and this continues for each run. One fresh extractor replacing a spent extractor and the process can be made to give a steady production rate. The choice of the total number of extraction vessels is dependent on the time required to empty and refill off line vessels and also on the cost of each additional vessel. A minimum of three process vessels is required for this type of continuous extraction.

CHAPTER 4

EXPERIMENTAL METHODS

4.1 Experiment Objectives

The fundamental aim of the study is to provide an efficient and economically viable process for the extraction of soybean oil using compressed liquid propane as the solvent. Other goals of the study are to obtain an estimate of optimum processing conditions for extraction and scale up the present lab-scale experiment to a pilot plant. The experimental setup and procedures used were designed so as to evaluate the following:

1. Determine soybean oil extraction capabilities of propane.
2. Experimentally simulate a semi-continuous process for oil extraction at ambient temperatures.
3. Investigate the effect of various process parameters on extraction yields.
4. Determine solvent losses along with the oil as well as the raffinate.

All of the experiments were performed at the 5 - liter extractor unit located at the Hazardous Reaction Laboratories, Oklahoma State University and were conducted by Dr. Maness (Professor – Department of Agriculture, OSU) and his co-workers.

4.2 Materials

Industrial grade propane (99.9 % purity) without mercaptan was obtained from Stillwater Steel / Air Gas LLP. The propane was obtained as compressed liquid in 100 Lbs. cylinders. Nitrogen required for sparging and raffinate treatment systems was also obtained from the same source. Various different variety of soybean were used for extraction, some being dehulled and others not, to determine the effect of dehulling on the overall extraction efficiency. The sources of soybean meal are confidential and hence are not mentioned in this report.

Soybean meal for the experimentation was obtained ready for direct extraction, with some of the pre-processing steps required before extraction being already carried out at the source companies. The meal obtained was already cracked, dehulled and flaked / crushed (run through a set of drum dryer).

4.3 Experimental Setup

The experimental setup used for this system consisted of a set of four pressure vessels to carryout extraction, oil-solvent separation; solvent-water separation and solvent recycle collection respectively. The material of construction for each of the four pressure vessels was stainless steel Type 304, with a pressure rating of 300 psig for the extraction vessel and solvent collection vessel. Table 4.1 gives the dimensions and ratings for each of the vessels.

Vessel	Material	Pressure Rating (psig)	Internal Diameter (inches)	Length (inches)
Solvent Collector	304 SS	300	6	36
Extractor	304 SS	300	4	24
Oil-Solvent Separator	304 SS	300	4	30
Solvent-Water Separator	304 SS	300	4	30

Table 4.1 Experimental Unit specifications (Manufactured by Eden Labs) from Dr. Maness [25]

The Oil-Solvent separator is a flash unit, fitted with a three way relief valve to flash the miscella before it enters into the vessel. This vessel is also fitted with a sample hold up cap at the bottom, to collect the separated extract (oil). Figure 4.1 shows a schematic for the experimental apparatus and a more detailed PID is given in Appendix – III a.

A vacuum pump, driven using compressed air is used for maintaining vacuum on the system and also to pull out solvent, compress and recycle it back to the solvent collector. Ball valves have also been used at various locations (Fig. 4.1) to control and manipulate the flow of solvent to the extraction column. Pressure gauges and temperature gauges were hooked on to each of the units to monitor the temperature and pressure; which was controlled by flowing hot / cold water though the jackets provided for each vessel. Also, a flow meter was attached in the exit line for the extraction unit, to monitor the miscella / solvent flow rate thought the system.

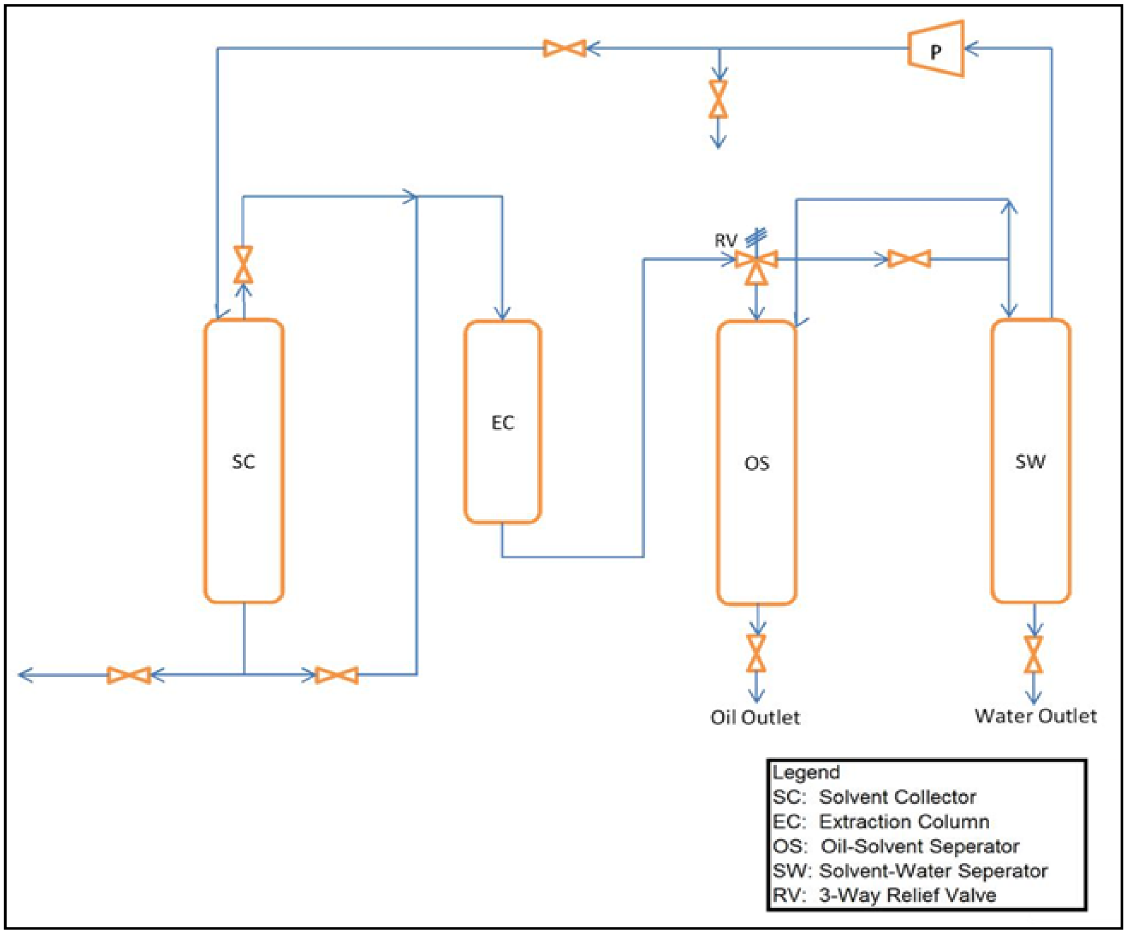

Figure 4.1 Flow diagram for the experimental setup

4.3.1 Soybean Pre-treatment Process

As pointed out earlier, the soybean obtained for experiments were not always cracked. Even whole beans were obtained. In case when whole beans were provided by the suppliers, the beans were first cracked in a hammer mill to form smaller particles and the cracked beans were later passed through a drum dryer, to simulate flaking of the beans. The flaked beans are then used for extraction. Also, there were cases where the soybean obtained were cracked, but still had considerable amount of larger size particles. Hence, these too had to be sent through a mild round of cracking to reduce the particle size.

4.3.2 Semi-Continuous Extraction Process

Initially, adequate amount of solvent is loaded into the solvent collector vessel from the source propane cylinder and the known quantity (2 kg / batch) of pre-treated soybean meal is filled in cotton bags and place into the extraction unit. Once the raw materials are placed into the system, vacuum was applied on the entire system (excluding the solvent collector), to remove any air (oxygen) present in the process, as it could lead to increased chances for explosion and also create non-compressible gases in the recovered solvent. Propane circulation was started and the flow rate of the extract was adjusted to 0.1 gpm H_2O. The liquid solvent flows through the extraction vessel and extracts the oil from the soybean meal. The exit stream / miscella / extract was flashed through a 3-way relief valve and passed on to the first of the flash units or the oil-solvent separator; where oil was obtained as the bottom product, as gaseous solvent was passed on to the downstream flash unit which acts as additional volume for the flash. The solvent was separated and the gaseous solvent obtained was compressed and recycled back to the solvent collector. The extraction was carried out for 60 mins and after extraction, the solvent inlet to the extraction vessel was closed and vacuum was applied to the system (as earlier), to drain all the solvent from the extraction vessel and remove most residual solvent entrained along with the soybean particles.

The temperature of each of the unit is controlled using water jackets, through which either hot or cold liquid can be passed. The product oil obtained from the first flash unit is collected in stainless steel cap attached at the bottom of the unit. Appendix – III b lists the detailed operational procedure for handling the experimental apparatus.

4.3.3 Sparging Setup

The product oil collected from the experimental setup contains some quantity of propane still dissolved in it. It is important to know the effects of sparging and designing this particular unit to complete the extraction process.

The sparging unit used for experimentation consisted of a cylindrical vessel, with a cap, a liquid exit at the bottom and inlets and exits for gas used for sparging (Figure 4.2). Oil from the 3 ~ 4 extraction batches (1 to 2 lit.) are placed in the unit and nitrogen gas under controlled process conditions (temperature and pressure) is sparged through the system. Nitrogen is purged through the system for a finite amount of time, depending on the batch size of oil available for sparging.

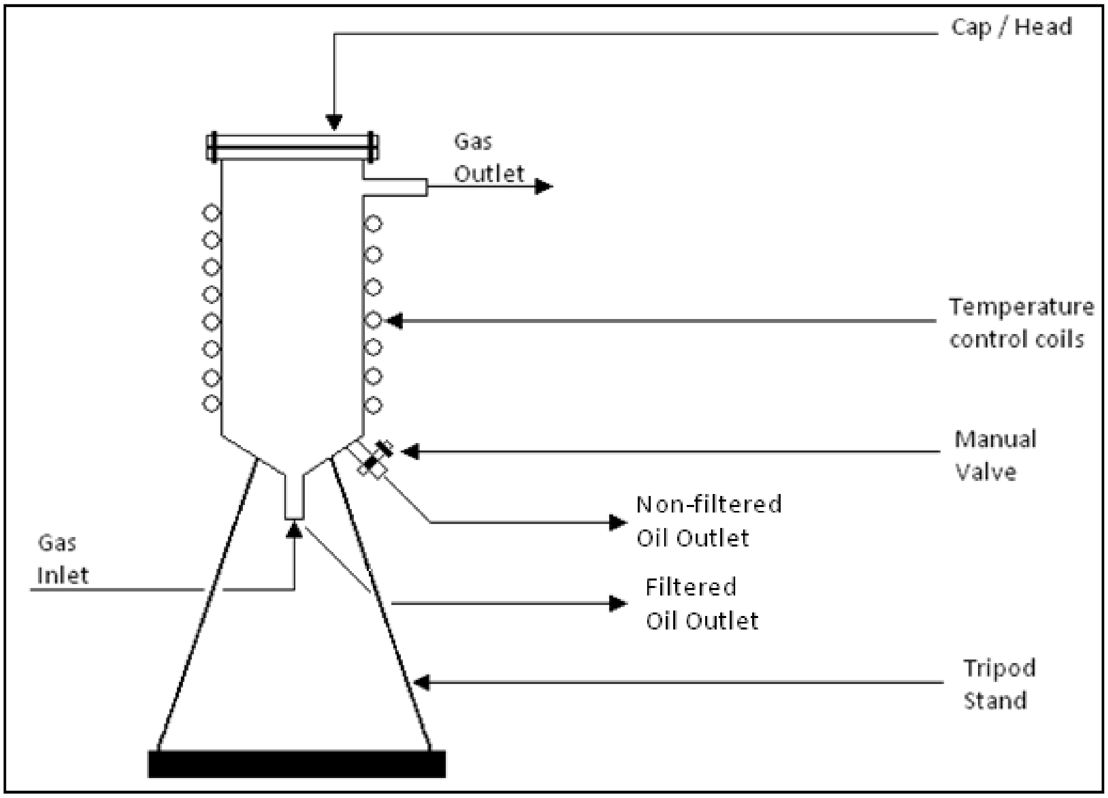

Figure 4.2 Schematic of the sparging unit

4.4 Experimental Parameters

The extraction rate and yield of the oil obtained depend on various process parameters, which include the soybean particle size, the type of pre-treatment methods used, extraction temperature, pressure and solvent flow rate. These parameters affect the extraction rate and the oil extracted from the soybean; the process also consists of the separation units, which play an important role in obtaining pure solvent for recycling; which again indirectly affects the extraction rate. Hence, process conditions of the separation units are also crucial. The extraction process in this experiment was carried out such that high solubility of oil in propane is obtained and also keeping in mind that temperatures higher than 100 ^{o}F result in possible de-naturation of the meal proteins and accelerated ageing of the extracted oil. The range of process conditions applied for experimentation for each unit is specified in Table 4.2.

Unit	Batch Size (lbs)	Pressure (psig)	Temperature (^{o}F)
Solvent Collector	-	130 ~ 265	60 ~ 130
Extractor	2	210 ~ 265	65 ~ 90
Oil-Solvent Separator	-	50 ~ 90	40 ~ 85
Solvent-Water Separator	-	50 ~ 110	50 ~ 85

Table 4.2 Experimental operating conditions from Dr. Maness [25]

The final product oil obtained from the sparging unit is then packed in a jar and vacuum is applied using a manual vacuum generator. The jar is placed under vacuum for a definite amount of time to inhibit oxygen exposure to the oil, which is then sealed and sent for quality check.

Experimental Setup Images

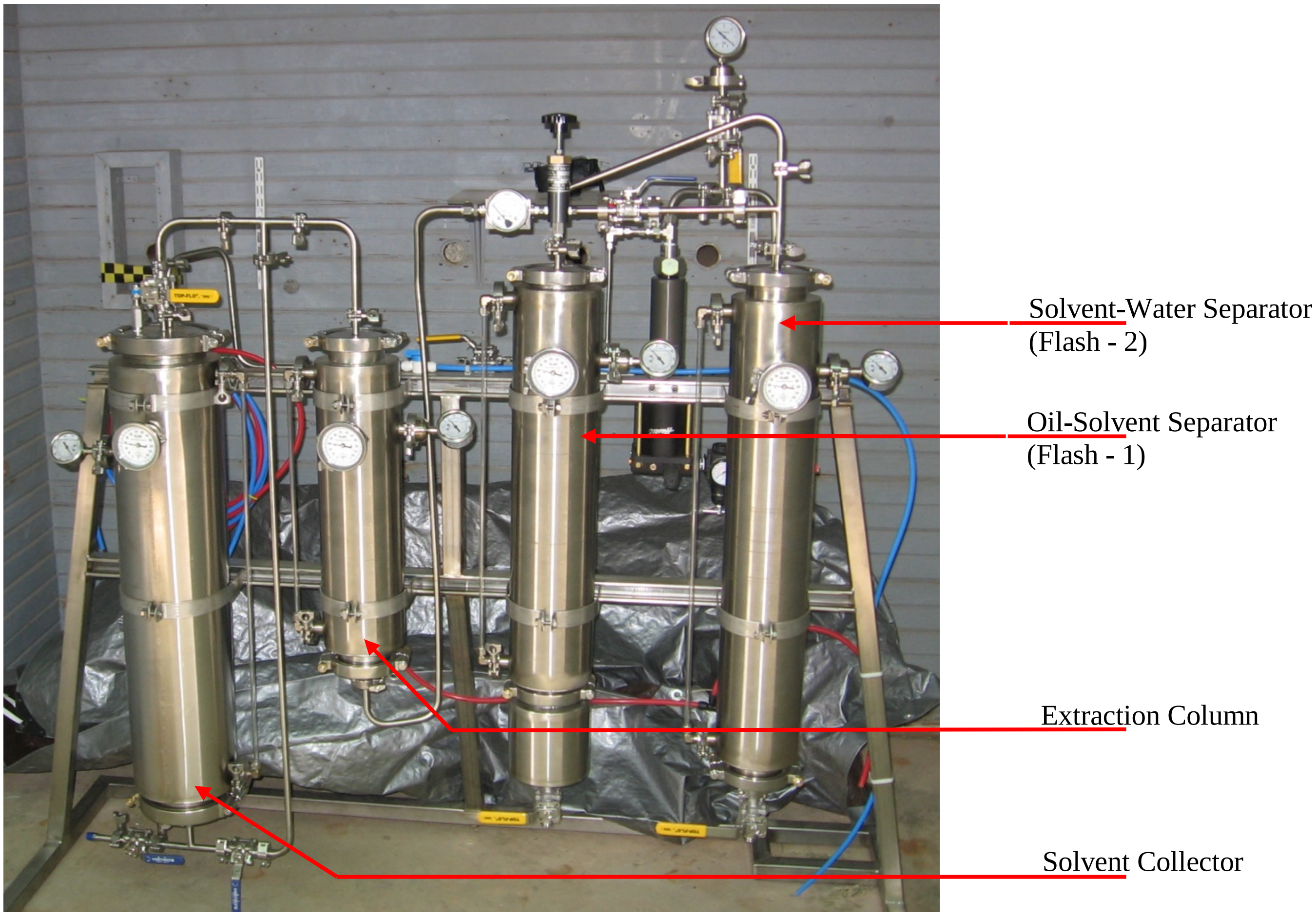

Figure 4.3 Complete lab scale extraction unit

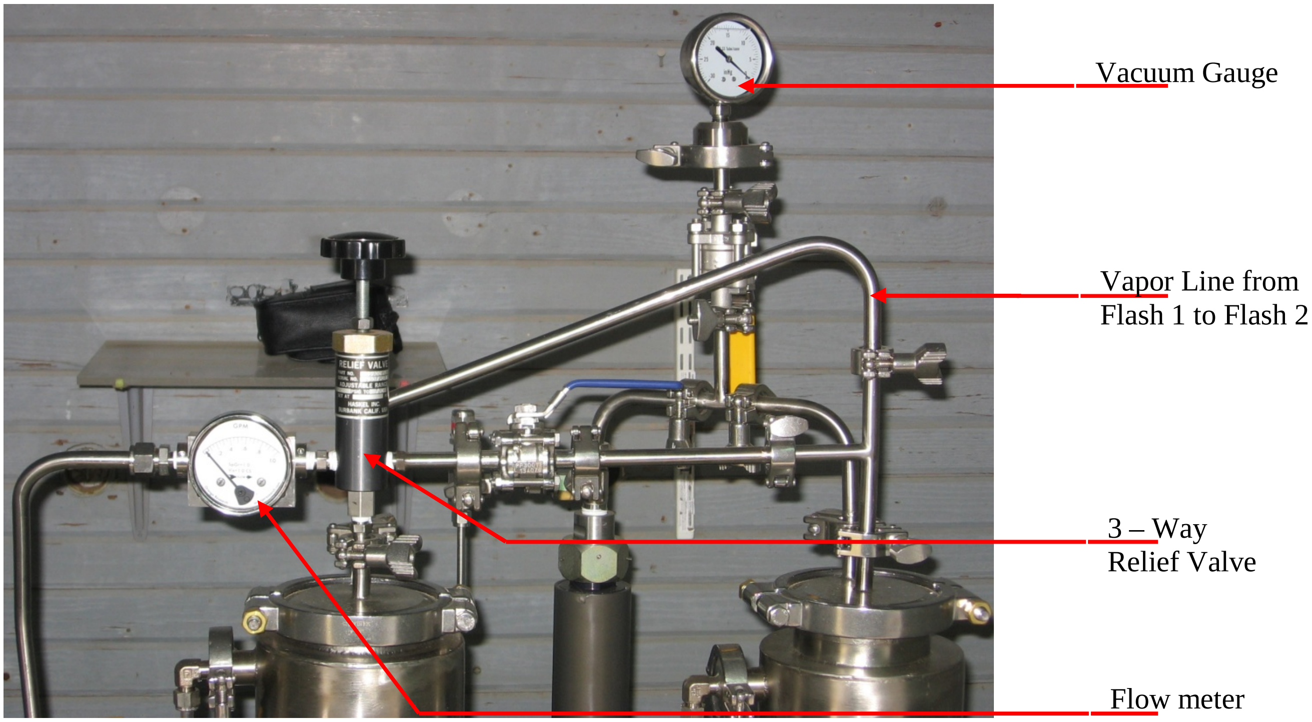

Figure 4.4 Enlarged view of separation train streams (inlet & outlet)

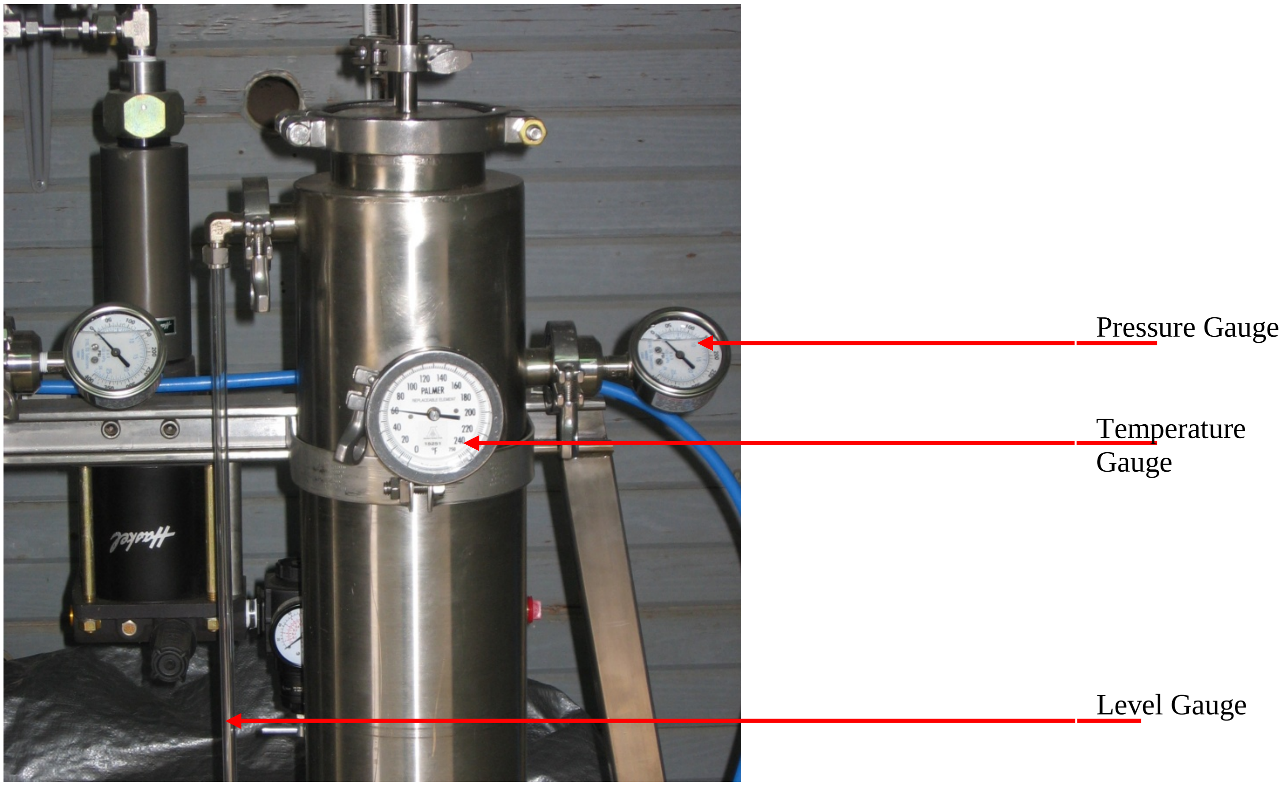

Figure 4.5 Enlarged view of pressure and temperature measurement units

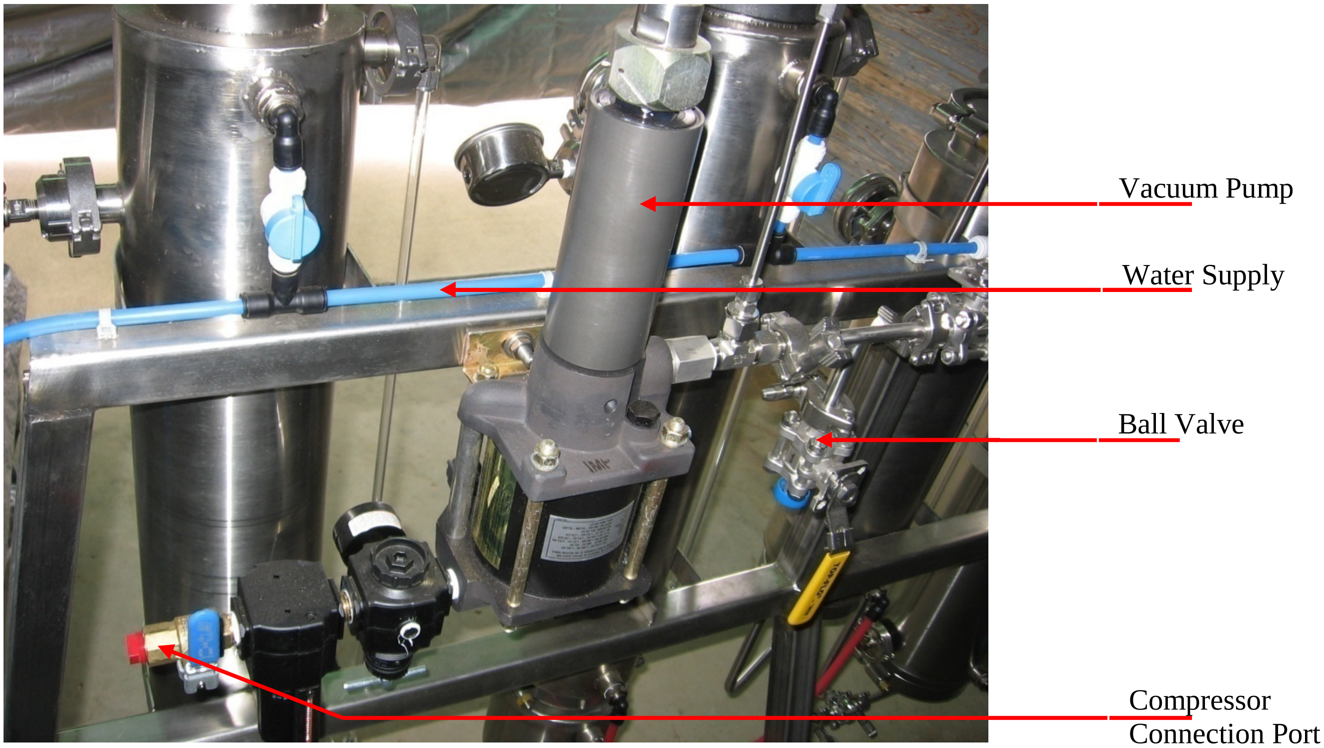

Figure 4.6 Recycle line and Vacuum pump unit

CHAPTER 5

EXTRACTOR & OTHER MODELS

The process simulation software ChemCAD v5.6 was used for modeling and simulation purposes. As discussed in the process outline, the process can be partitioned into different sections, and models are necessary to simulate each of these sections. The modeling technique used is to model the units using modules of predefined equipment types in the software and/or as a combination of predefined modules, where specific modules do not exist.

5.1 Extractor Simulation Models

The extractor is the most important and the most difficult section to be modeled. As seen in the process flow diagram (Figure 3.1), the extractor consists of five streams – the solvent inlet, meal inlet, extract outlet, raffinate outlet and vapor line. The extractor consists of two physical operations of which one is the extraction operation while the other is the filter operation that prevents the meal from exiting along with the extract (solvent-oil exit).

The extractor unit operation is one of the most difficult processes to be modeled because of its necessity to handle solid materials (soybean meal and the raffinate). This coupled with the inherent semi-batch nature of the process makes modeling such units even more challenging.

Two basic types of process models were generated to get good approximations for the extractor operation; the steady state simulation model and the dynamic simulation model. More emphasis was put on the dynamic models as it represents the actual process more accurately (physical sense).

The different models generated are listed in Table 5.1. The dynamic vessel module was the most frequently employed of the unit operation modules available in ChemCAD. Apart from the dynamic vessel module, the solid washed module and mixer module were tested to model the extraction process. Various combinations of dynamic vessel, mixer, solid washer along with pressure filters and separation screens were also employed. In addition to this, user defined modules were also created to control the process stream flows and to make the process behave in a semi-continuous manner. Each of the models listed above in Table 5.1 are described in the following pages.

Different Extractor Simulation Models

1. Models for Steady State Simulation
2. Models for Dynamic Simulation
   2.1 Only Liquid Models – Dynamic Vessel / Mixer Module to simulate LLE.
   2.2 Models Handling Solids
      2.2.1 Solid Washer Models and Recycle
      2.2.2 Dynamic Vessel Models and Recycle
         2.2.2.1 With Solid Screen
         2.2.2.2 With Pressure Filter

Table 5.1 Various Extractor Simulation Models Created

5.1.1 Models for Steady State Simulation

An initial attempt to model the process was carried out in form of a continuous, steady state simulation model handling just fluids. Two models types of steady state models were proposed:

1. Extractor model using Dynamic vessel or Mixer module
2. Extractor model using Solid Washer module

The process basically consisted of the extractor unit (mixer type), which was followed by a series of flash separation units. Fresh solvent was mixed with recycle solvent from the separation train and sent to the extractor, which had a separate inlet for the soybean meal. As the process was created to handle systems containing only fluids, the soybean meal was represented as a mixture of soybean oil components and water (moisture in the meal). The resultant mixture parameters were calculated using simple material and energy balances (by the simulator). The mixture obtained was then sent to the flash separation units, which were used to simulate the Oil-solvent and Solvent-Water separators. The separated solvent was recycled back to be mixed with fresh solvent for reuse. Figure 5.1 shows the simulation diagram for this particular system model.

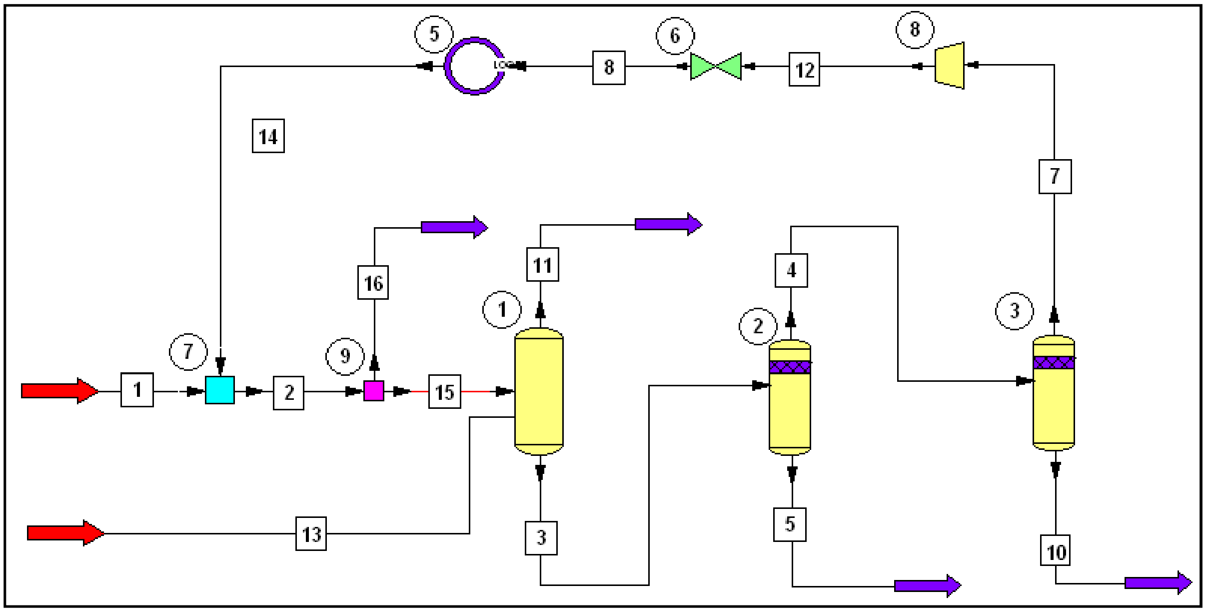

Figure 5.1 Steady state model employing mixer and dynamic vessel

In the process simulation setup shown above, the divider unit (Unit 9) is used to keep the solvent inlet to the extractor constant at required rate, while the loop solver (Unit 5) on the recycle line is used as the convergence solver, necessary for the recycle stream values to converge. Streams 1 and 13 are the input streams for solvent propane and soybean meal respectively; while streams 5 and 10 represent the output streams for product soybean oil and waste waster respectively.

The solid washer extractor model replaces the dynamic vessel (Unit 1) in the above model with a solid washer. The solid washer is closer to physically representing the extractor operation than the earlier model, as it handles 4 streams as compared to the 3 streams for the earlier model. Also, the solid washer as the name suggests is specifically used when working with solids. In this case, the soybean meal is modeled as a solid with entrained oil, with a dummy chemical species being created on ChemCAD with the average properties of soybean meal.

The soybean meal consists of a mixture of the pseudo-component soybean along with the soybean oil components and water (moisture). Figure 5.2 shows the process simulation model using the solid washer module. The solid soybean mixture enters the unit along with a simultaneous input of liquid propane solvent. The exit liquid stream consists of a mixture of soybean oil components, water and solvent propane, while the solid exit consists of the raffinate depleted in soybean oil along with some entrained propane. The calculations are simple material and energy balances based on the process parameters entered for the solid washer. The important parameters being mixing efficiency, L-S ratio, temperature, solid loss fraction. In addition to these, the pressure drop has to be specified. Figure 5.3 shows the specifications dialogue box for the solid washer operation.

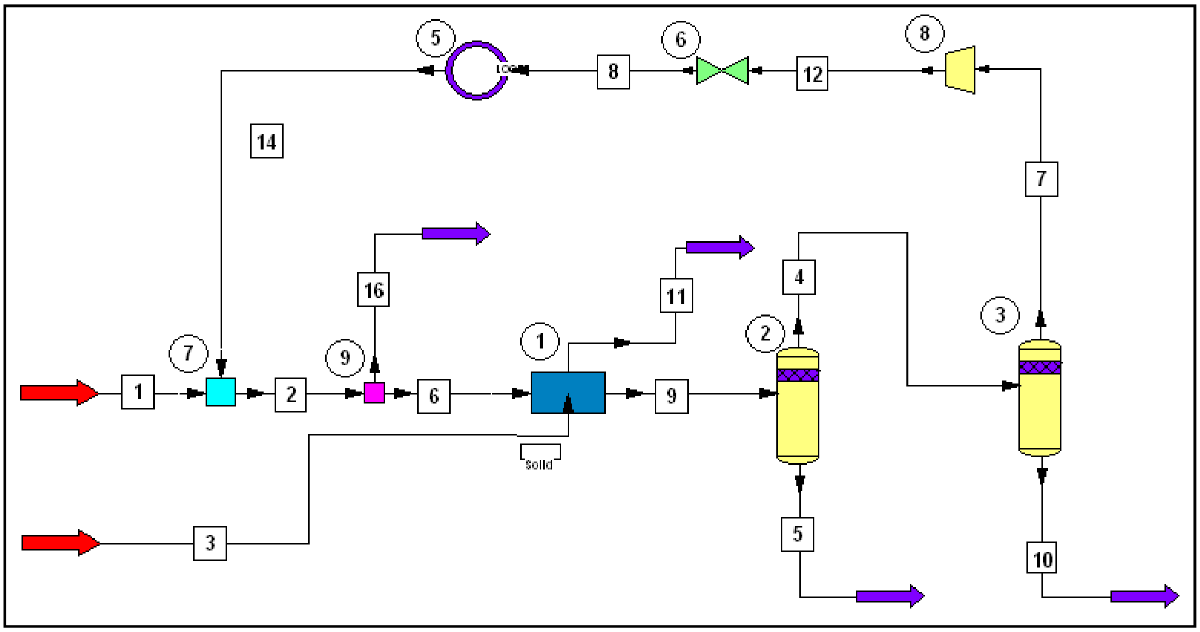

Figure 5.2 Steady state model employing solid washer

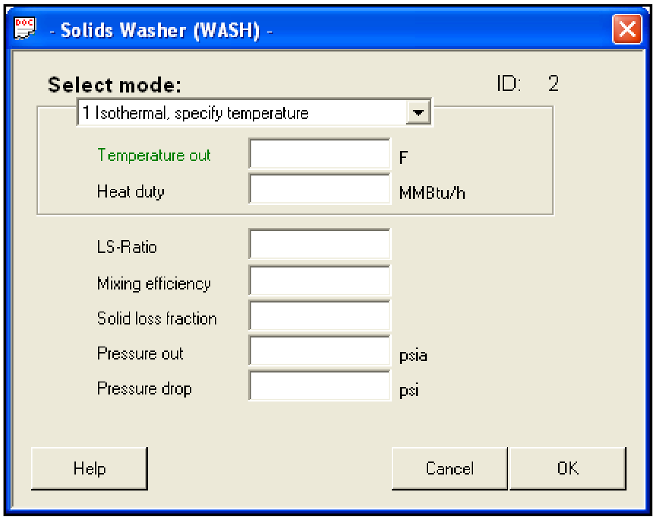

Figure 5.3 Solid washer Specification dialogue box

Raffinate treatment to remove the entrained solvent is not a part of this model as that constitutes a dynamic / unsteady state situation. This particular simulation method has some limitations. The pros and cons for the steady state simulation are detailed below.

The model can predict an approximate value for the amount of oil extractable from the mixture, but this would be just a rough approximate as the model doesn't physically represent the actual process. The model is simple and takes little time to execute. However, there are a number of disadvantages to this model:

1. The model can only predict approximate results.
2. The model does not take equilibrium considerations into account. Its just mixing and separation.
3. The model does not do well to show differences obtained due to varying extraction parameters such as temperature, pressure, amount of extract and the solvent flow rate.

5.1.2 Dynamic Simulation Model

The steady state models do not represent the actual physical process. The inherently semi-continuous nature of the process makes it necessary to develop a dynamic model to better represent the process. Many different dynamic state models were generated, which have been listed in Table 5.1.

The dynamic simulation models are almost the same as the steady state models, with the only difference being that the simulation for this case are performed at unsteady state instead of steady state. Also, the process being a semi-batch process, the dynamic simulation modules in ChemCAD cannot be used directly, but have to be modified with additional user defined unit operations modules to achieve the final model.

5.1.2.1 Only Liquid Models Simulating LLE

This model is similar to the dynamic vessel / mixer model from the steady state simulations. The soybean meal is again represented as a mixture of soybean oil components and water (moisture) all in the liquid phase. But, instead of the soybean being fed in a continuous fashion as in the steady state model, the feed is in form of a batch charge and it is placed in the extractor (similar to the actual physical system).

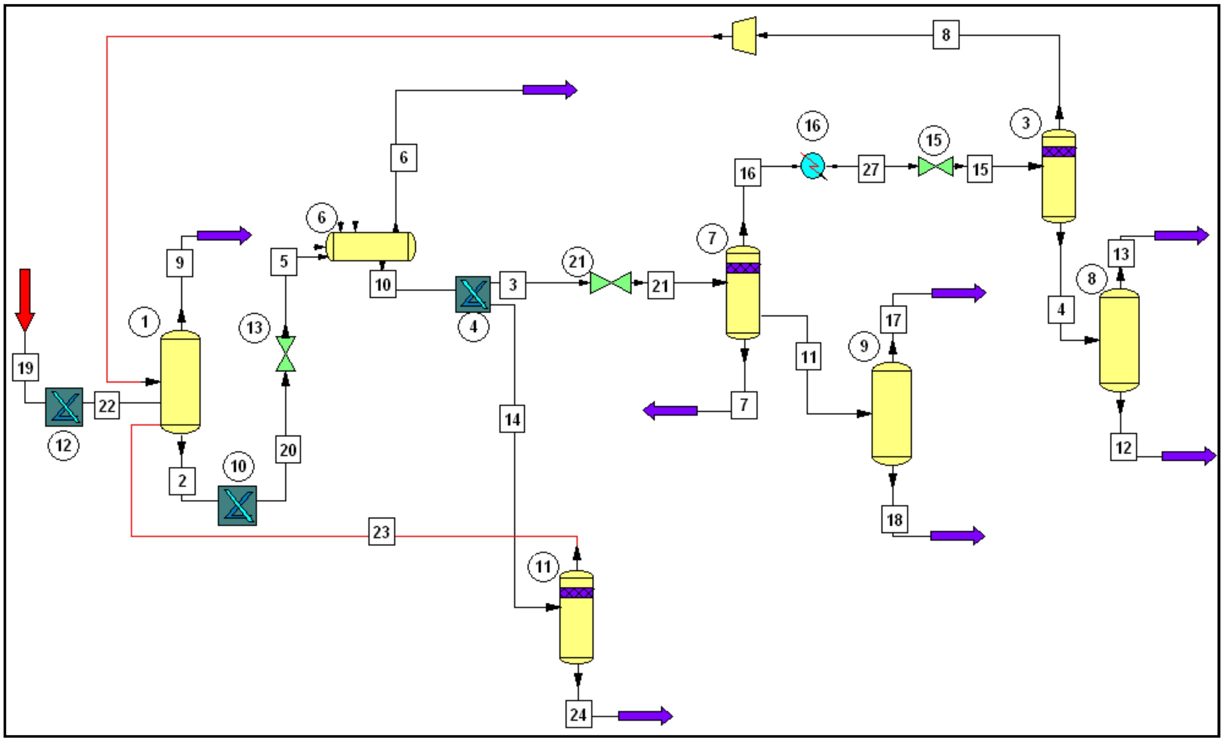

Figure 5.4 Dynamic model simulating process as LLE

Figure 5.4 shows the simulation model used. The extractor is represented by the horizontal dynamic vessel (Unit 6). This particular model also incorporates the raffinate treatment segment of the process. The desolventizer used in the actual extraction plants is modeled using a flash unit. The residual content of the extractor remaining after the end of the extraction period is sent to the desolventizer flash unit (Unit 11). The later stages of model remain the same. The user defined modules – Unit 12, 10 and 4 are programmed on VBA – Excel using the ChemCAD –Excel interface. They are modeled to act as valves, which switch off and switch on as needed. The details of operation time for each of the used defined units are provided in Table 5.2. Units 8 and 9 are dynamic vessels, though here, they are used to represent storage tanks. Unit 9 is necessary to determine the amount of water separated from the process and solvent lost along with this waste water. The recycle solvent obtained from the solvent-water separator is compressed to liquid state and sent back to the solvent collector.

User Defined Unit	Open / Start Time (mins)	Close / End Time (mins)	Note
12	0	20	Not necessary for every cycle
10	20	80	Operates during the extraction period
4	20	80	Operates during the extraction period

Table 5.2 Operation time for vessels on the dynamic, LLE simulating model

Though this simulation model is closer to the manner of operation of the actual unit, it still deviates from the actual because of its inability to include solids. The positives from this model are that,

1. It simulates the process in dynamic, semi-continuous manner similar to the working of the actual process.
2. Takes the thermodynamic equilibrium conditions into account – extraction rate changes with temperature and pressure.

Though, the model has some negatives:

1. Does not include solids
2. Does not show the differences in extraction rate with varying solvent flow rates.

5.1.2.2 Models Handling Solids

All the solid handling models were based on the basic structure of the dynamic liquid models, with some additional processing steps and changes. The dynamic vessel and the solid washer modules were the chief operation used to model the extractor. Additional units were also used in combination with these key modules to better approximate the working of the extractor routine. The combinations used are listed in Table 5.1.

To achieve better resemblance of the physical working of the actual process, solids are necessary to be included in the simulation models. ChemCAD provides options to include solid components in the simulation. The user can select solid components using the '*pick solids*' option available on ChemCAD. The component selected as solid remains solid throughout the simulation and is not included in the thermodynamic calculations such as vapor pressure determination. Also in case where multiphase (Vapor-Liquid-Liquid-Solid) equilibrium exists, the solid component always flows out with the stream containing the heavier of the two liquid phases present.

5.1.2.2.1 Solid Washer and Recycle

The solid washer model is built on the base constructed in the previous dynamic model. The dynamic vessel extractor is replaced by the solid washer module. Unlike the dynamic vessel module, the solid washer module does not have an option to enter a batch charge and requires continuous inlet streams to process. Hence, a recycle operation is introduced for the solid exit stream. Figure 5.5 shows the details of this simulation model.

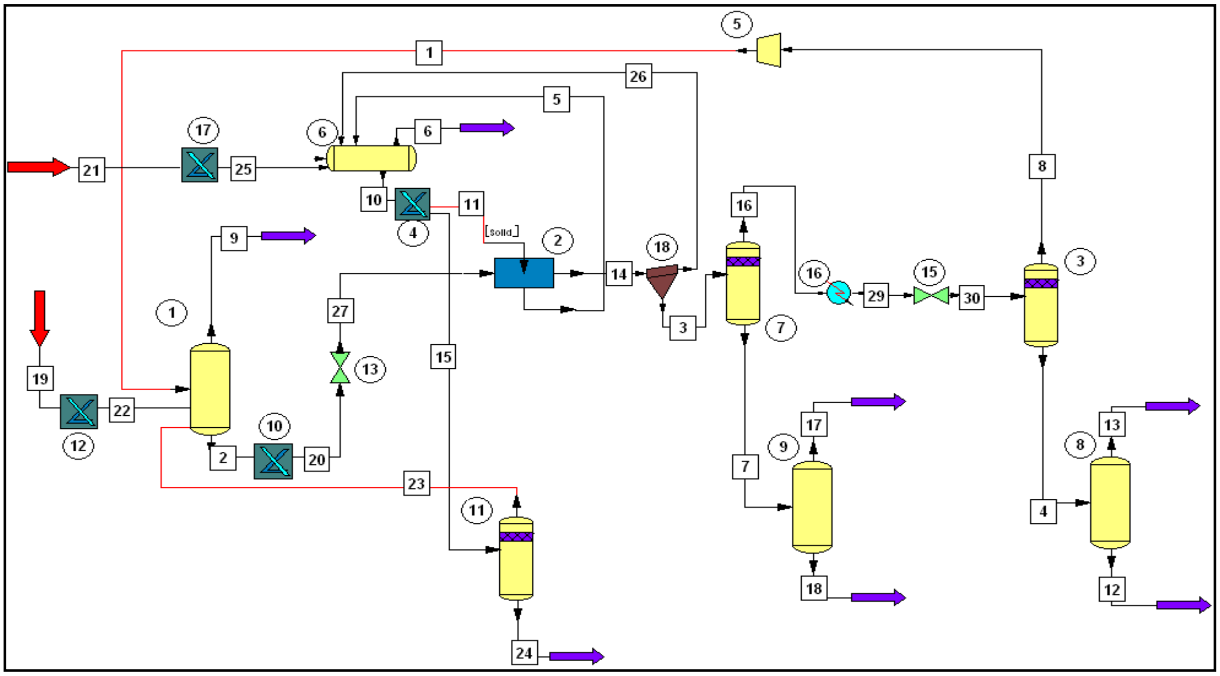

Figure 5.5 Dynamic model employing solid washer with recycle stream

The extractor unit consists of the dynamic vessel (Unit 6), user defined unit (Unit 4), the solid washer (Unit 2) and the solid screen separator (Unit 18). The dynamic vessel just acts as a hold up tank and switch connector valve. It is used to hold the unextracted solid components back to the solid washer. The contents of the dynamic vessel are immediately recycled back to the solid washer for extraction; thus maintaining a continuous solid input stream. The solid washer is the unit where the extraction process occurs. It consists of a liquid inlet stream through which solvent propane flows into the system, partially extracts the entering solid stream and exits through stream 14. The simulation is performed assuming that a portion of solids will be lost along with the solvent and hence, a solid screen separator is placed in the setup, to remove these solid particles. The partially extracted solid component exits the solid washer through stream 5, which is recycled to the dynamic vessel.

The rest of the processing model is kept the same, with no major changes. This model also includes the raffinate processing segment of the process; but does not include the solvent draining stage. The unextracted solids remaining in the dynamic vessel at the end of the extraction period are sent to the raffinate treatment flash unit through the user defined unit 4. The user defined units remain the same with no major changes made in the program routine.

The model improves on the previous dynamic model by including solid components in the simulation; but it still possesses some deficiencies:

1. The model cannot predict the variations in extraction yield with changing extraction process conditions – Temperature, Pressure and flow rate.
2. Though the model includes solids, the solids are not operated in batch fashion, but rather a semi-continuous manner.

The model does well to simulate solvent entrainment in the raffinate.

5.1.2.2.2 Dynamic Vessel and Recycle

As seen for case of the solid washer model, the introduction of solids to the simulations necessitates the use of solid processing unit operations in the model. The solid separation screen was used in the previous case. Two different combinations of units along with the dynamic vessel were explored. These being the solid separator screen and the pressure filter.

For this particular model, the dynamic vessel was used to model the extractor along with the solid processing unit operations. Unlike the solid washer module, dynamic vessel does not separate the solid and liquid streams at the outlet, and hence a single outlet stream is obtained. This stream consists of a mixture of solid component, soybean oil components, water (moisture) and solvent propane and has to processed / filtered to obtain a solid free liquid stream. Figure 5.6 shows the dynamic vessel model with solid screen.

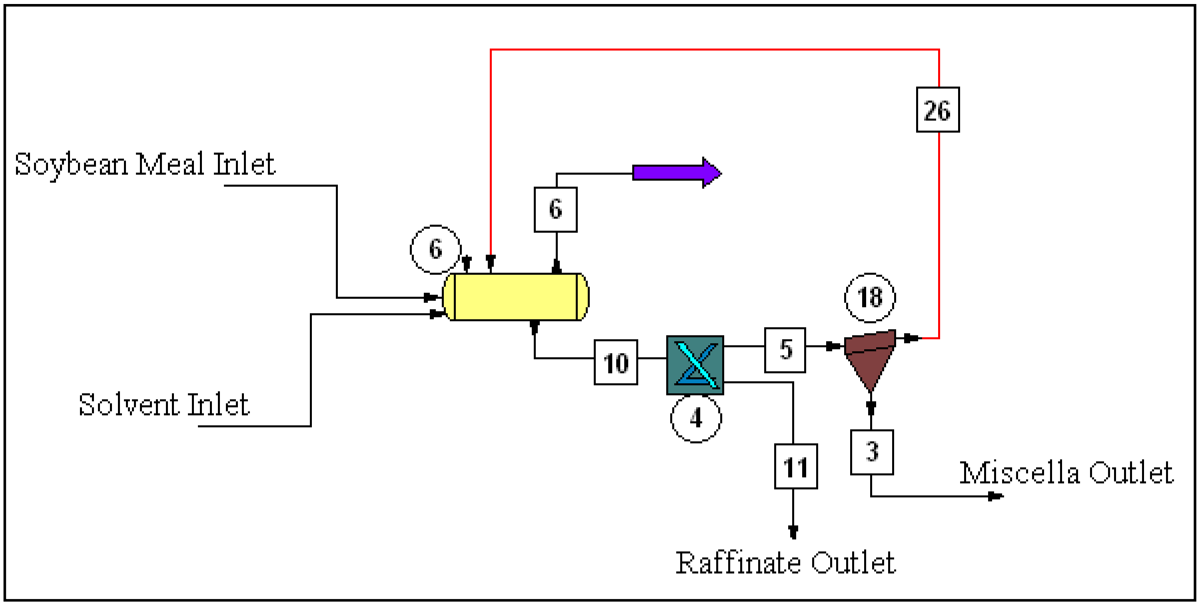

Figure 5.6 Dynamic model employing dynamic vessel module with solid screen and recycle

This particular model faces a similar problem as the solid washer model. Though the dynamic vessel does have the batch charge option, the presence of large quantities of solids in the outlet stream makes its necessary to filter these solids and recycle them. Thus, make the solid phase semi-continuous instead of the required batch mode. On the other hand, the solid screen that is used separates the solid based on particle size and separation efficiency and does not include liquid entrainment in separated solids. The remainder of the model is the same as the earlier simulations.

The actual extraction unit carries out the extraction and also has a filter screen attached to it. Thus, the process also performs filter action. To simulate this working, extractor can be modeled as a combination of a pressure vessel, where extraction occurs followed by a pressure filter as shown in Figure 5.7.

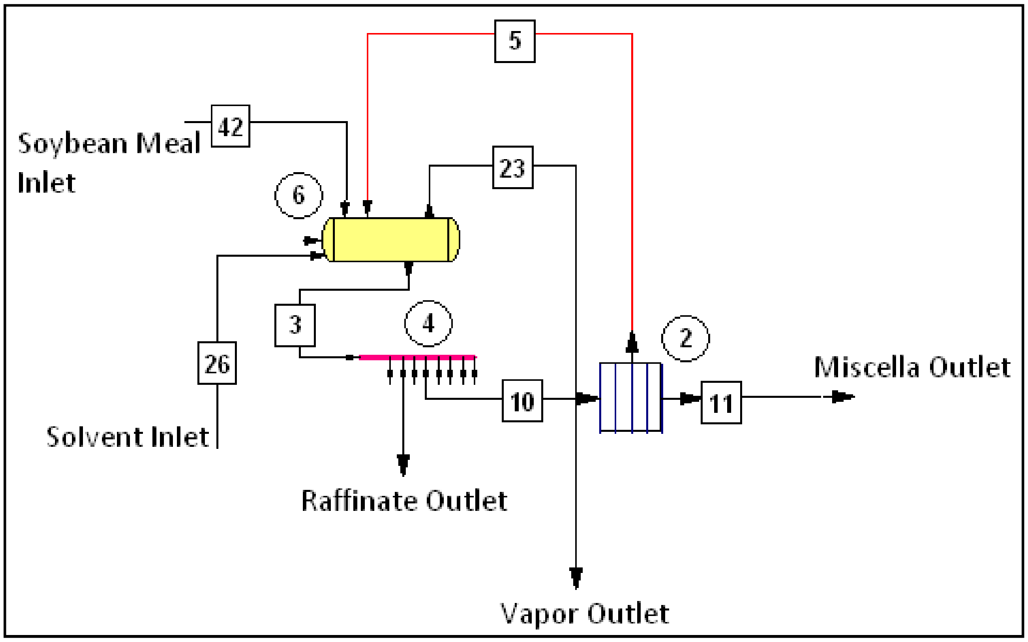

Figure 5.7 Dynamic model employing dynamic vessel and pressure filter modules

The pressure filter module takes into account the liquid entrainment and pressure drop in the system while performing the simulation. This gives a much better approximation of the actual unit as compared to the earlier models.

5.2 Sparger Model

Steady state models as well as unsteady state type models were created for the sparging unit. The main intention behind simulating this unit was to determine the amount of propane lost during sparging. For modeling this kind of system, the input used was the product obtained from the extraction process. Since only composition was the crucial element for this system, just a flash unit was used. A mixture of propane and soybean oil was flashed and the heavier product obtained was used as the input for the sparger model (both steady and dynamic models).

5.2.1 Steady State Model

The simulation model used for this process is shown in Figure 5.8. Propane and soybean oil were mixed and flashed to obtain the required composition of propane soybean oil mixture, which was then mixed with nitrogen gas and flashed to obtain the final refined oil. Single flash as were as multiple flashes were examined.

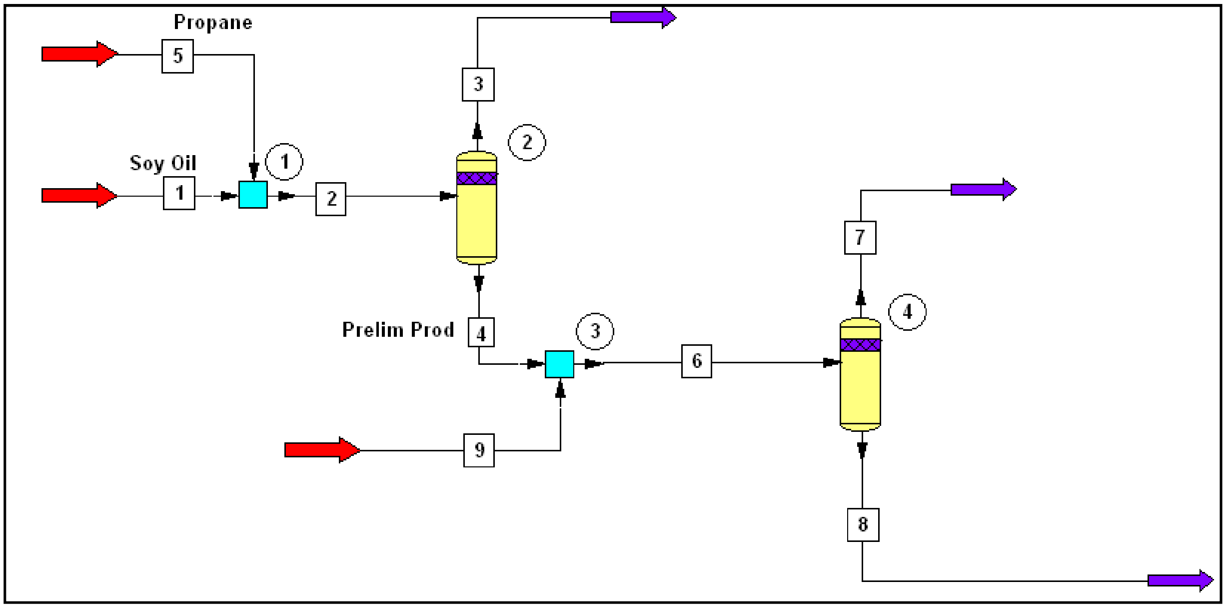

Figure 5.8 Steady state sparger simulation model

5.2.2 Dynamic Model

The dynamic process consisted of a similar arrangement to that of the steady state process, the only difference being that the preliminary product was first collected from the process and sent to a sparging vessel (Unit - 4) operated at 65 ^{o}F and 15 psia (1 atm) wherein, nitrogen was bubbled through the system. The process flow diagram for the system is shown in Figure 5.9.

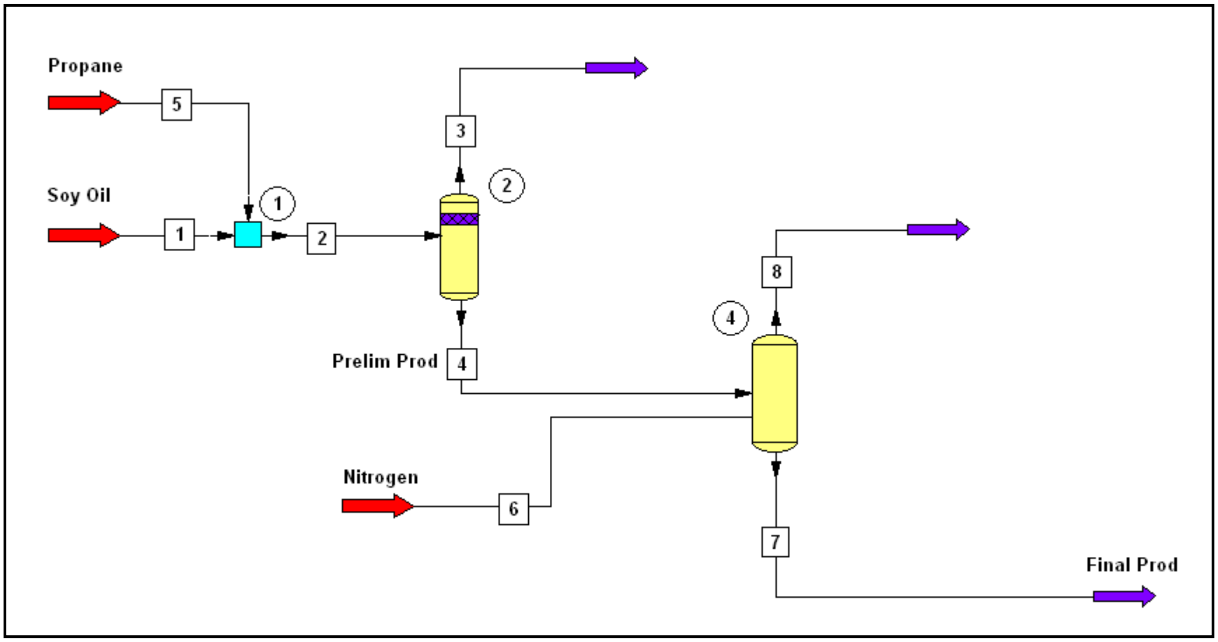

Figure 5.9 Dynamic sparger simulation model

The process conditions before the sparger vessel remains the same as in the steady state process. The extraction was carried out to obtain about 11.9 lb of preliminary product which was sent to the sparger unit.

5.3 Vacuum Pump Models

Vacuum pump effect is a major part of the simulation model for the process. Even though it is not evident (Figure 5.7), vacuum pump model was incorporated in the '*Dynamic Vessel and Recycle Models*'. A similar approach as used earlier was followed, with steady state and dynamic simulations both being created for this purpose.

The steady state model was formed from using multiple flash units in series, with the pressure decreasing (Equation 5.1) as the process proceeds downstream.

$$P_2 = P_1 \exp(-ck)$$

This model wasn't found to be very effective as it did not adequately represent the process and hence, two dynamic models were proposed. The first model was similar to the steady state model, with dynamic vessels replacing the multiple flash units. Each of the unit operates for a fixed amount of time, before its contents are transferred to the next vessel, which operates at a lower pressure as determined by the decay law.

The first model was inefficient for implementation. Hence, a newer model using a user defined unit to simulate the pressure reduction operation was generated. The unit responsible for pressure reduction in the dynamic vessel is installed upstream of the unit, unlike a conventional vacuum pump which is installed downstream of the dynamic vessel (Figure 5.10). Unit operation 3 is the user added module, which works such that the pressure in Dynamic Vessel (Unit 4) decreases with time, thus simulating the action of a vacuum pump.

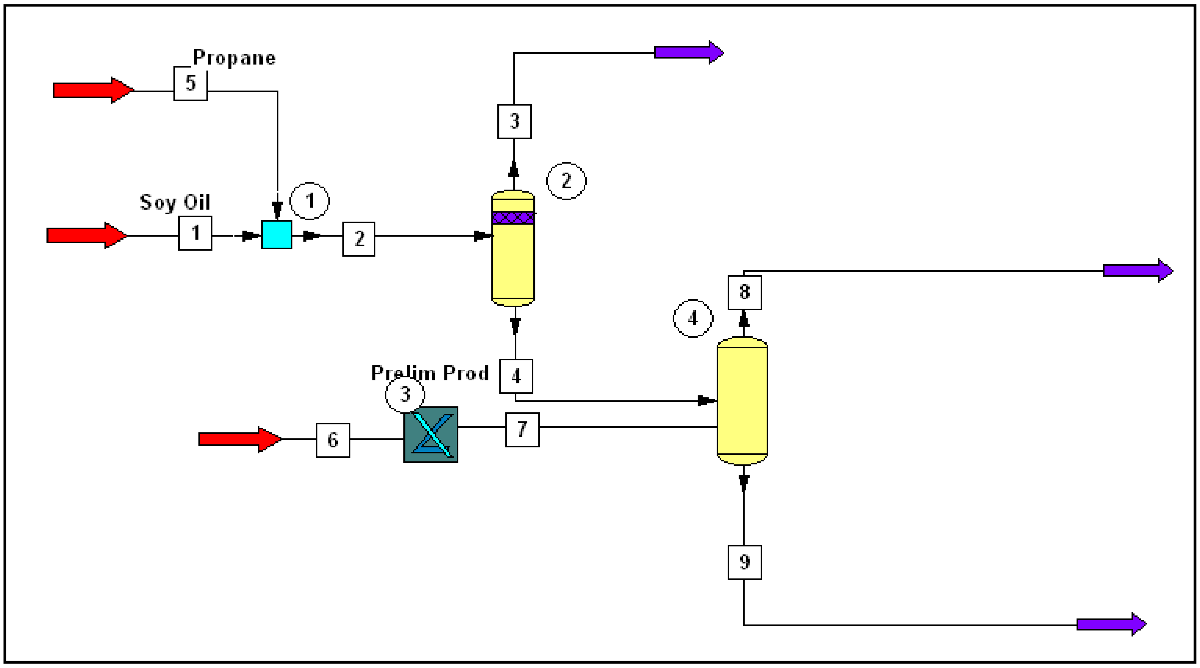

Figure 5.10 Dynamic vacuum pump simulation model

5.4 Model Calibration

The dynamic vessel and recycle model approaches the working of the actual extraction process and the experiment. Hence, this model is chosen to be studied further. The extractor unit in this model is represented by a combination of the dynamic vessel and the pressure filter. The presence of these two unit operations introduce a set of operating parameters to be specified in before the simulations can be run. Of all the parameters, the important ones being -

1. For Pressure Filter: Pressure drop, moisture fraction, particle size and porosity.
2. For Dynamic Vessel: Vessel dimensions, Liquid outlet flow rate

The liquid outlet flow rate of the dynamic and the moisture fraction for the pressure filter are the most important parameters for modeling this system.

Though the model approaches the working of the actual setup, it cannot generate good solutions if the parameters specified are incorrect. Hence, it is very important that correct values of these parameters are used during simulation.

The dynamic vessel is simulated with just a single outlet, which made up of both the liquid as well as the solid components (Section 5.1.2.2). Hence, dynamic vessel liquid flow rate is determined based on the amount of solid meal being extracted and the solvent circulation rate. Depending on the solvent circulation rate, an average of 30 to 40 % (weight) of solids is present in the liquid exit stream. Hence, to determine the flow rate for the entire stream, a good estimate for the liquid flow can be given by:

$$DynamicVesselLiquidFlowRate = \frac{SolventCirculationRate}{0.4}$$

The other important parameter is the moisture fraction. The soybean meal is modeled as a mixture of soybean oil components and water (moisture) both in liquid phase entrained in a solid pseudo-component used to represent soybean particles. The oil content of the soybean meal is assumed to be 20% (weight) along with 10% (weight) moisture. This contributes to 30% (weight) of the soybean meal with the remaining 70% (weight) for the pseudo-component. The soybean oil and liquid in the real world scenario is not actually entrained on a solid matrix, but is present inside the solid particle. Due to this reason, to get a good approximate during extraction, the minimum moisture content for the pressure filter cake needs to be 30% (weight) so that no extracted oil is obtained if there is no solvent flow to the extractor. In addition to this, as liquid solvent flows through the system, it gets entrained in the solid matrix. This has to be taken into consideration while specifying the liquid retention capacity for the pressure filter.

The model is unable to predict temperature variations in extraction. But, accurate predictions can be obtained by changing the liquid retention (moisture fraction parameter) with temperature. For this, a relation between the liquid retention and extraction temperature is necessary. This has been done by calibrating the moisture fraction parameter to the extractor temperature and the results for the calibration are presented below (Table 5.3):

Extractor Temperature (^oF)	Simulation Yield (lb)	Liquid Retention (wt. fr.)
70	0.4378	0.57
72	0.3709	0.54
75	0.4616	0.52
78	0.4617	0.48
80	0.5793	0.42
81	0.495	0.42

Table 5.3 Calibration curve data set

Figure 5.11 gives a plot of liquid retention v/s extractor temperature. This particular set of data can be regressed and fitted into an exponential curve given as:

$$y = 3.9477e^{-0.027x}$$

Where, y = Liquid retention

x = Extractor Temperature (^oF)

The R-square value obtained using this equation is found to be about 0.96.

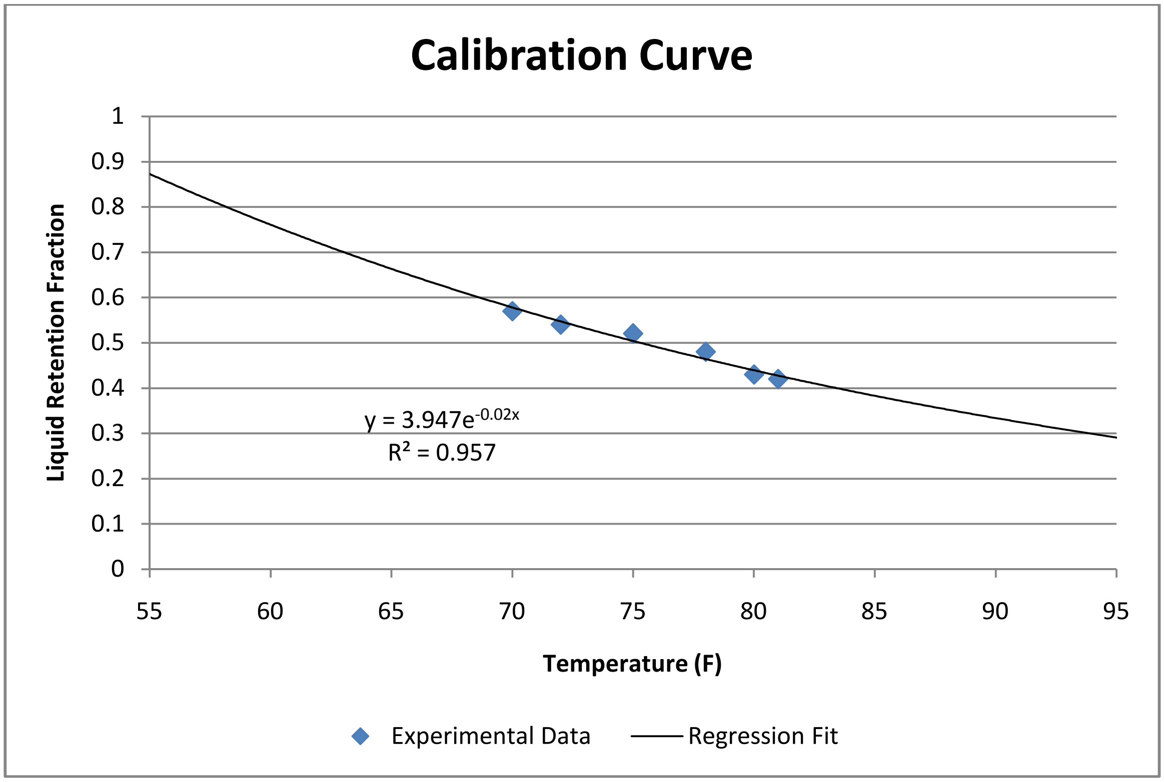

Figure 5.11 Liquid retention calibration curve

CHAPTER 6

RESULTS & DISCUSSION

6.1 Experiment Results

The experimental setup and procedure discussed in Chapter 4 were used to conduct multiple runs using flaked soybean meal to determine the extent of extraction obtained and the effects of various parameters on the extraction process. Table 6.1 gives a summary of the extraction process conditions and the extraction yields obtained from the various runs performed.

Run	Meal Extracted (lb)	Extractor Temperature (^oF)	Extractor Pressure (psia)	Extraction Time (mins)	Extract Yield (lb)
1	2.8725	68	205	60	0.326
2	2.06	70	213	60	0.3775
3	1.577	72	245	60	0.3315
4	1.8845	78	255	60	0.3965
5	2.2895	75	240	60	0.4565
6	1.992	81	245	60	0.412
7	2.612	80	225	60	0.462
8	1.9965	75	225	60	0.387
9	2.1285	86	220	20	0.2775
10	2.1535	72	220	40	0.2845
11	2.119	83	230	20	0.349

Table 6.1 Experimental data summary from Dr. Maness [25]

Even though the temperature and pressure parameter for each of the equipment were controlled using water jackets, some variations do occur due to the increase and decrease in solvent flow through the system. Hence, the average values of the process parameters have been used for analysis and simulation purposes. From an observation of the results summary, extraction temperature, extraction pressure and the amount of fresh meal loaded in the extractor seem to be the parameters that affect the extraction yield significantly. The separation process parameters are also extremely important for the system as they signify the extent of oil purification obtainable.

6.1.2 Effects of Temperature

The experiments were carried out in a very narrow range of temperatures, because of the sensitivity of the extraction process to temperatures. Lower temperatures result in lower yields and higher temperatures lead to thermal degradation of the oil. Extraction was carried out for temperatures ranging from 67 ^{o}F to 83 ^{o}F. Figure 6.1 shows the variations in extraction yield with temperature. Since, the extract obtained from the experiment would be a mixture of oil and propane, the actual percentage yield has been calculated by determining the propane content of the mixture from ChemCAD simulations using the flash vessels described in models discussed earlier. The results of which have been tabulated in Table 6.2.

Run	Extractor Temperature (^{o}F)	Yield (lb)	Propane content (wt. %)	% Yield
2	70	0.3775	030	64.14
6	81	0.412	23.2	79.42
8	75	0.387	23.3	74.34

Table 6.2 Effects of temperature on extraction from Dr. Maness [25]

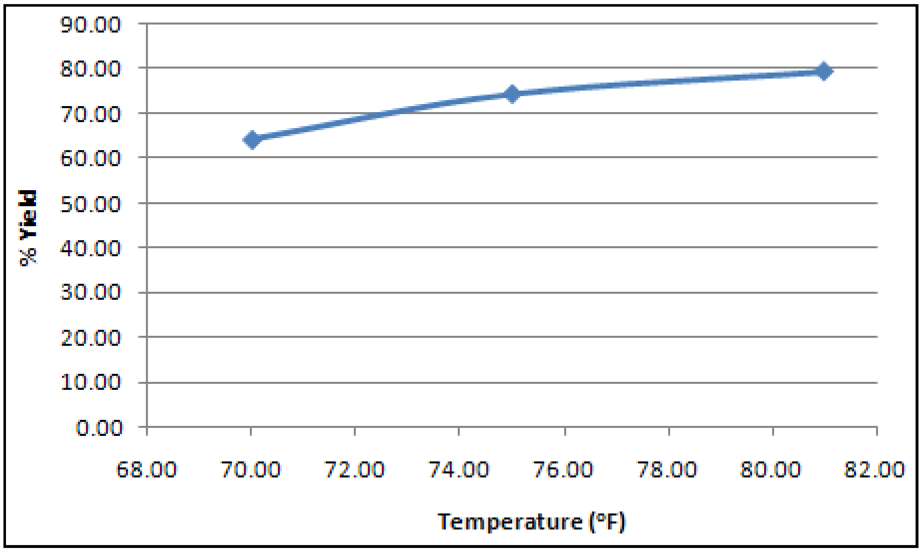

Figure 6.1 Effects of temperature on extraction

It can be seen that increasing the extractor temperature increases the extraction yield. This observation is consistent with the theory of extraction processes, which dictates that increased temperatures result in higher extraction rates and thus higher yields. Hence, higher the extractor temperature better is the process yield. But, there exists an upper limit to the temperature range which is determined by the stability of the oil at that temperature and other economic considerations.

6.1.2 Effects of Pressure

The results indicate that extraction yield does not depend a large deal on the extractor pressure. A comparison of runs can be used to verify this. Table 6.3 gives the comparison of two sets of runs with similar extractor temperature and meal extracted.

	Set 1		Set 2	
Run	5	8	4	6
Pressure	240	210	255	245
% Yield	77	76	81	79

Table 6.3 Effects of pressure changes on extraction from Dr. Maness [25]

The table gives the percent yield against extractor pressure for two sets of comparisons. The first set compares results from runs 5 and 8. The temperature of extraction for both of the runs was 75 ^{o}F and the extraction yields obtained were very much close. A similar set of result was observed with set 2. A larger deviation in the yields was observed for set 2 and this can be attributed to the variations in temperature and material used. Temperature for run 4 was 78 ^{o}F; while for run 6 were 81 ^{o}F.

Though pressure doesn't have a very significant effect on extraction yield, it plays a very important role in the process. The pressure for the system has to be adjusted so as to ensure that the solvent is sufficiently dense to form a stable liquid phase. This criterion sets the lower limit for the pressure for the process, while the higher limit is set depending on the economics for the process. Higher the pressure greater is the energy consumed to maintain that pressure and higher pressures also increases the equipment costs.

6.1.3 Other Observations

In addition to the extractor process parameters, there exist other variables that affect the extraction yields. This set of variables which play an important role in the process includes the solvent flow rate through the system, the external conditions, the separation train parameters and mixing effect.

There were no experiments performed with variable solvent flow rate. A fixed value of 0.1 gpm H_2O was used for all the runs and hence, this particular section of the process cannot be quantified. On the other hand, the external condition includes that of the atmospheric temperature and pressure. Though there is no direct effect of this variable on the process, it was observed that during colder weather, higher amount of energy was necessary to run the process as compared to when running the experiment in warmer conditions. However, no specific data had been recorded for these parameters.

The remaining two important parameters are the mixing effect and the separation train parameters. The mixing effect plays a major role in the extraction yield obtainable. Since, the extractor used for experimentation was a simple pressure vessel with no built-in mixer, loading the extractor with larger amount of meal reduces the miscibility and diffusivity of the solvent within the meal and thereby reduces the extraction yield. Table 6.4 gives the details of variations of the extraction yield for each of the runs along with temperature and amount of meal processed.

Run	Temperature (^oF)	Meal Processed (lb)	% Yield
1	68	2.8725	42.57
2	70	2.06	64.14
3	72	1.577	76.73
4	78	1.8845	80.11
5	75	2.2895	76.96
6	81	1.992	79.42
7	80	2.612	70.75
8	75	1.9965	74.74

Table 6.4 Effects of meal loading on extraction from Dr. Maness [25]

Comparison of runs 2 and 3 shows a steep decline in the percent yield obtained with a very small decrease in temperature. This can be attributed to the presence of higher amount of meal in the extractor for processing in run 2 as compared to run 3. Another data that clearly indicates the effect of higher loading is run 1. The extractor temperature for run 1 is 68 ^oF, but the achievable extraction was just about 44 %, which is very low as compared to that obtained in runs 2 and 3, which are at a slightly higher temperature. Another result which strongly suggests the effect mixing can have on the extraction yield is an comparison on runs 6 and 7, where a 1 ^oF rise in temperature results in about 10 % increase in extraction yield, but on a closer look, this increase in yield can be attributed to the decreased loading of the extraction, which provided better mixing of the meal and solvent. An analysis of the extracted / spent meal indicated that, the experimental runs with higher loading resulted in spent meals with blocks of the meal which did not come in contact with the solvent at all i.e. channeling occurs in case when extractor is loaded to or near full capacity. This problem of channeling can be reduced or eliminated by introducing a mixing element in the extractor. Thus, allowing the extractor to be loaded to full capacity for each run.

6.2 Simulation Results

The various simulation models formulated have been discussed in the earlier sections. Out of these, the “Dynamic Vessel with Pressure Filter and Recycle Model” is found to represent the experimental process well and hence the actual system. So, this model was chosen for further analysis.

All of the models that were formulated consist of multiple parameters that need to be adjusted according to the process conditions to generate viable results. This list of parameters includes the solvent holder and extractor sizes, the temperature and pressure conditions for each of the equipment involved and other process parameters related to each of the equipment models. It is very important that the values entered for these parameters are accurate, correct and in accordance to the process.

The model consists of process units which are operated at constant temperature and pressure conditions; but some fluctuations exist when running the experimental unit. Hence, data for temperature and pressure obtained from the experiments are converted to average values, which are then used in the simulation model. A sample of the detailed experimental observations including the time fluctuations in temperature and pressure for each of the four process units is presented in appendix V.

The other process parameters were calibrated based on the model and experiment characteristics so that an accurate estimate can be obtained for the process. The various parameters of importance are already discussed in the model calibration section of the report.

From these, the solvent collector outlet flow, the extractor outlet flow and the pressure filter moisture content are the most important. Calibration for each of the experimental results was carried out by using the trial and error method of simulating the model multiple times to get an accurate fit. The simulation results for the experimental runs are tabulated below (Table 6.5). The moisture fraction data listed in the table is value used for the moisture fraction parameter for the pressure filter. On the other hand, Table 6.6 shows the comparison and percent deviation of the simulation model results from the experimental results. The assumption that goes while making this comparison is that the propane content of the extract obtained experimentally is equal to that obtained from simulation. This assumption is necessary because, composition analysis of the extract was not performed to determine the exact experimental composition.

Run	Extractor Conditions (°F, psia)	Flash-1 Conditions (°F, psia)	Flash-2 Conditions (°F, psia)	Moisture Fraction	% Yield
1	68 220	48 63	52 70	0.61	55.92
2	67 230	65 88	63 100	0.59	79.50
3	72 260	71 92	70 108	0.55	92.30
4	78 270	83 103	76 122	0.49	92.62
5	75 255	69 83	61 94	0.53	83.51
6	81 260	75 91	72 99	0.42	95.42
7	80 240	77 87	75 93	0.43	88.71
8	75 240	74 90	71 100	0.53	87.63

Table 6.5 Simulation run summary mimicking experimental runs

Run	Propane Content (Wt. fr.)	% Yield		% Deviation
		Experiment	Simulation	
1	0.25	42.57	55.92	23.87
2	0.30	64.14	79.50	19.32
3	0.27	76.73	92.30	16.87
4	0.24	80.11	92.62	13.51
5	0.23	76.96	83.51	7.84
6	0.23	79.42	95.42	16.77
7	0.20	70.75	88.71	20.25
8	0.23	74.74	87.63	14.71

Table 6.6 Percent deviation of simulation runs from experimental results

This table gives the comparison between the percent yields for the experiment results and the results obtained by simulation of calibrated model. It also shows the percent deviation of the model results from the experimental results. The model calibration has been performed such that it matches the experimental data as closely as possible while at the same time gives logical estimates for process conditions for which experiments have not been carried out. This coupled with the manual errors that exist during experimentation, result in some deviations in the model predictions, which goes to as much as 23%. Based on this calibration data and other parameter values, simulations have been run to test the dependence of extraction yield and extract quality on equipment temperatures and pressures and the solvent flow rate.

6.2.1 Extraction Yield and Extraction Temperature

It is well known that temperature plays a very important role in extraction processes. This has been confirmed by the experimental results, which show that extraction yield increases with increasing temperatures. The simulation model calibrated based on the experiments were run several times to determine the variations of extraction yield with temperature. Figure 6.2 gives the extract accumulation with respect to time at 81 ^{o}F (Run 6).

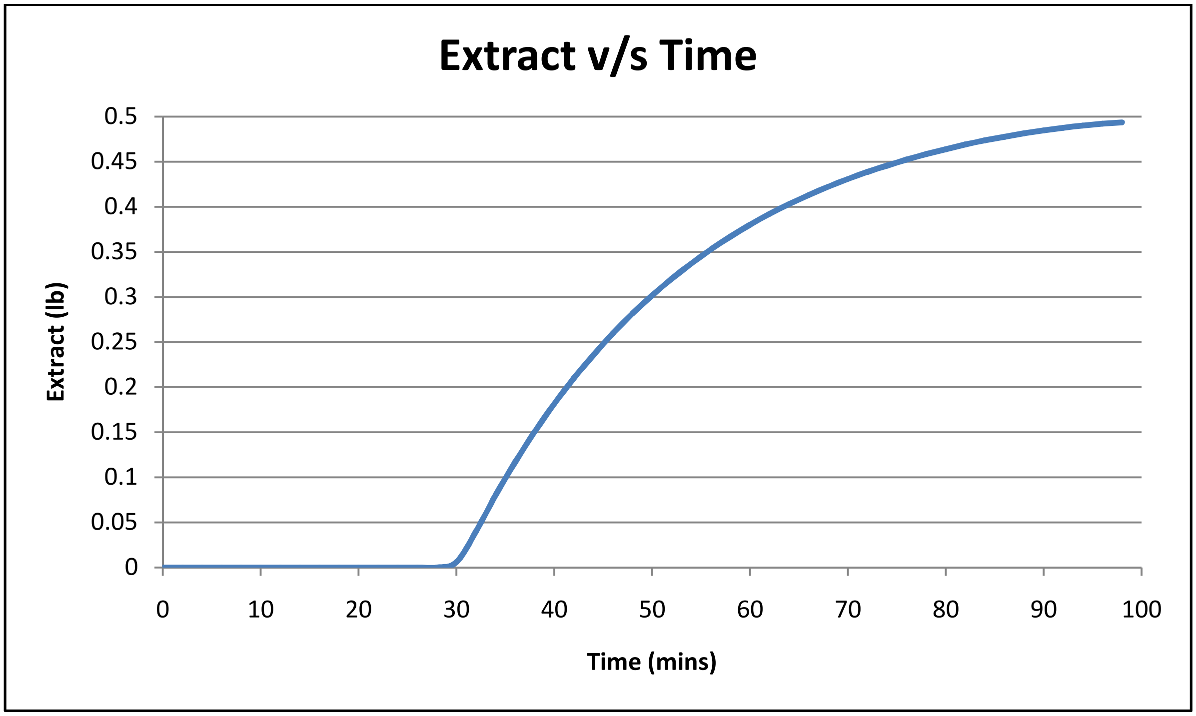

Figure 6.2 Variations in extraction yield with increasing time

This figure shows the extract obtained as the extraction time increases. The initial part of the plot is a straight line passing along the ‘time’ axis, which indicated that no extract is obtained during that period of the process. This period includes the fresh meal loading to the system and the propane pumping to fill the extractor, the process steps that occur before solvent circulation begins. The solvent circulation stage begins 30 mins after the start of the fresh processing and accumulation of extract also starts by this time as shown (Figure 6.2). The process follows a path somewhat parabolic in nature as it levels off to the maximum amount of oil that can be extracted at the specified conditions.

From the experimental data, it is already known that extraction yield increases with temperature. This phenomena is shown in Figure 6.3. the figure depicts the extraction yield (weight) as a function of time for 4 different values of temperature. The extractor pressure, the seperator temperature and pressure conditions, solvent flowrate and the amount of meal processed have been held constant to obtain data which gives the extraction yield dependence on temperature. The other process parameters for these runs were: Solvent flow rate of 6 lb/hr, Flash-1 at 50 psia and 80 ^{o}F, Flash-2 at 80 psia and 60 ^{o}F and meal processed equal to 2 lb (per cycle).

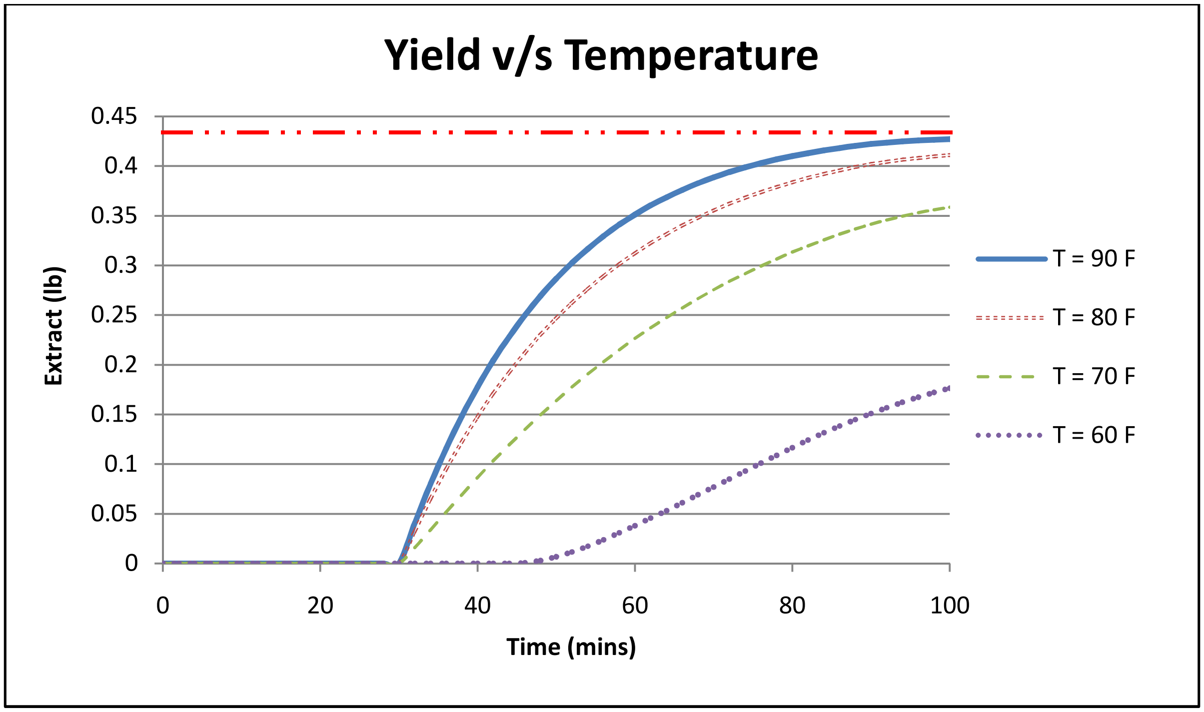

Figure 6.3 Effects of extractor temperature on process yield

The figure shows the extraction curves for the process carried out at four different temperatures. As expected, the total extraction yield is maximum for the highest temperature value investigated (90 ^{o}F), and decreased as temperature decreased, with the minimum yield obtained for extraction at 60 ^{o}F. In addition to the extraction yield, the path followed by the extraction curves indicates that decreasing the extraction temperature results in lower extraction rates; which is confirmed as the extraction curves tend to have a more flatter regime at lower temperatures. To more clearly express the variance of extraction yield with temperature, extraction yield is plotted as a function of temperature in Figure 6.4. This figure confirms that extraction yield and rates are both dependent on extraction temperature.At lower temperatures, the extraction yield increases very quickly, but this rate of increase reduces to zero gradually as the extraction yield approaches its maximum attainable value.

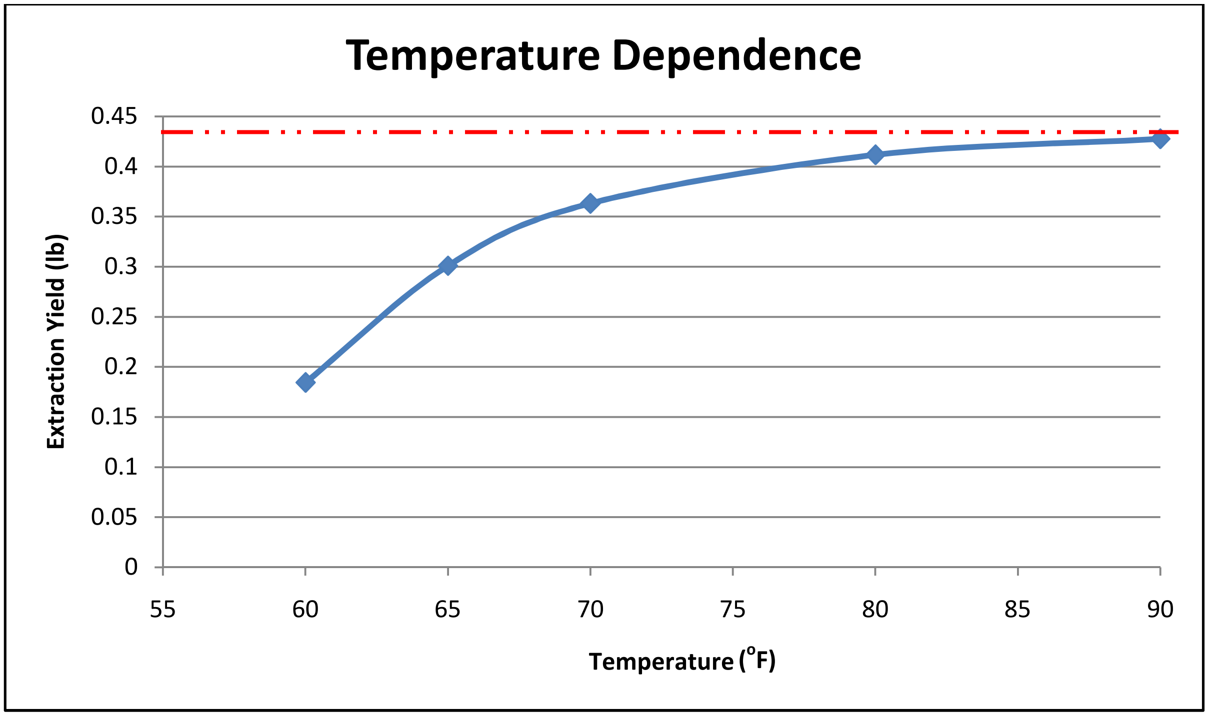

Figure 6.4 Variations in extraction yield with changing extractor temperature

Though extraction yield is directly proportional to temperature, there exists a upper limit to the allowable temperature. This limit is decided by the stability of the product oil at higher temperature. It is known that soybean oil is stable in temperatures upto 100 ^{o}F at atmospheric pressure. Hence, this value of temperature (100 ^{o}F) acts as the upper limit for the current process.

On the otherhand, extraction yield is not dependent on extractor pressure as much as it depends on temperature. Very large changes in pressures might be required to get appreciable changes in the extraction yield. This particular observation obtained from the experiments has been confirmed by the simulation results.

6.2.2 Extract Quality and Separation Train Parameters

Extract quality is a very important parameter. One of the objectives of the project was to study the extent of processing necessary to the miscella obtained from the extractor. Two separation units (flash columns) are used for the processing purpose, the first one being used as the Solvent-Oil separator, while the next being used as the Water-Solvent separator. The main aim of these separators is to achieve the best separation possible i.e. lowest amount of solvent propane content of the final extract, with minimum loss of oil and also minimum contamination and loss of the solvent. To obtain the best possible purity, higher temperatures are required in flash-1. Hence, simulation runs have been carried out for this maximum temperature limit, which is take as 90 ^{o}F. Table 6.7 gives the variations in total oil extract obtained at various Solvent-Oil separator pressures, at 90 ^{o}F.

Pressure (psia)	Extract (lb)	Propane Content (wt. %)	Oil in Extract (lb)
20	0.38	2.39	0.37
30	0.41	3.70	0.39
40	0.41	5.10	0.39
45	0.42	5.93	0.39
50	0.42	6.77	0.39
60	0.43	8.59	0.39

Table 6.7 Variation of extract oil content with changing Oil-Solvent separator pressure

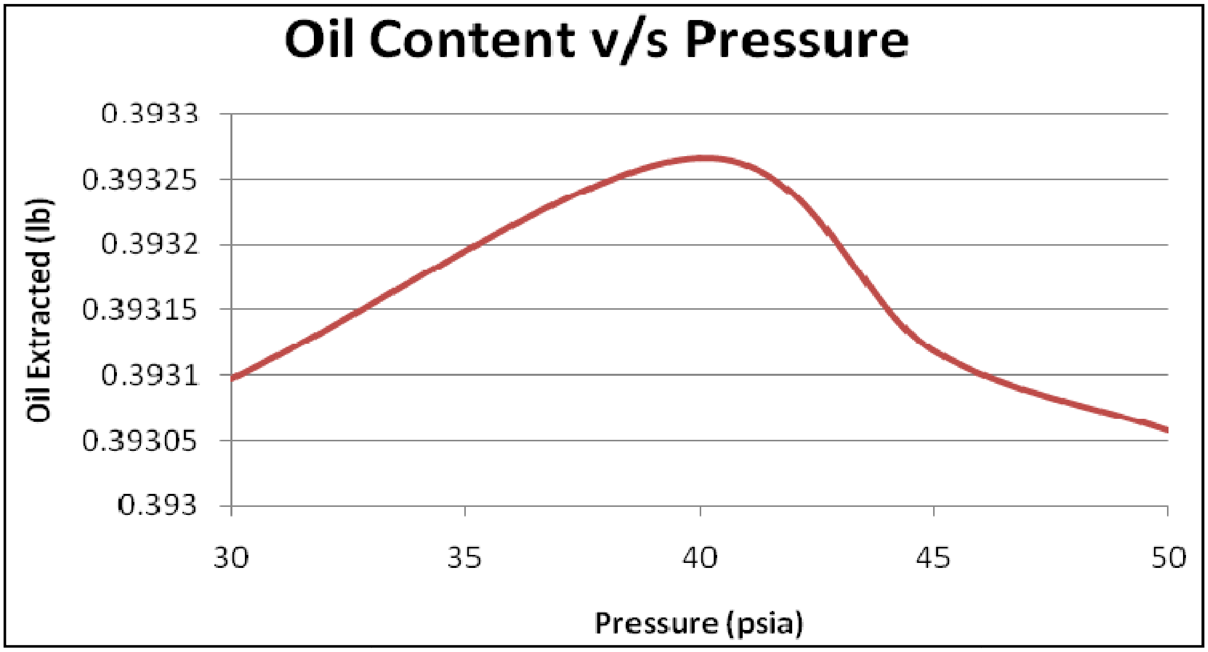

Figure 6.5 Variation of extract oil content with changing Oil-Solvent separator pressure (90 ^{o}F)

The above table lists the extract obtained, the propane content of the extract and the amount of oil present in the extract. The best value of pressure would be the value where maximum oil content in obtained with minimum propane content. To determine this, a plot of oil extracted against pressure is generated (Figure 6.5). From the plot, it can be seen that, the maximum oil content is obtained when the pressure is around 41 psia at 90 ^{o}F. This is the best possible value of pressure for the SW separator at 90 ^{o}F. Similar simulations at 80 ^{o}F resulted in lower extractions. Hence, the best conditions to operate flash-1 would be at 90 ^{o}F and 41 psia.

Pressure (psia)	Extract (lb)	Propane Content (Wt. %)	Propane Loss (lb)	Solvent Purity (wt. % propane)
30	0.012	0.0001	1.15E-8	99.81
40	0.015	0.0040	5.97E-7	99.84
50	0.018	0.0130	2.34E-6	99.86
65	0.020	0.0200	3.82E-6	99.88
80	0.021	0.0200	4.20E-6	99.89
90	0.022	0.0260	5.50E-6	99.90

Table 6.8 Variations in solvent purity with changing Solvent-Water separator pressure

Similar analysis has been carried out for the Water-Solvent separator and the data is presented in Table 6.8. Temperature of 60 ^{o}F was chosen for the purpose of simulation. The propane loss was analyzed for different values of pressure and the best possible value was chosen. From the tabulated data, it can be seen that, decreasing the unit pressure results in lower solvent loss, but at the same time also decreases the solvent purity. The constraint for the process is to have a solvent purity of greater than 99 % propane, which is not violated. Also, this solvent purity is going to decrease after each run. Hence, after a two to three of runs, the solvent purity would drop to about 98 %, which would result in inefficient extraction. Hence, it is advisable to use a pressure, where the solvent purity does not decrease below 99.85 % for the first run. Hence, optimum pressure would be about 60 to 70 psia at 60 ^{o}F for the Water-solvent separator.

6.2.3 Extraction Yield and Solvent Flow rate

Apart from the process temperature and pressure parameters, extraction yield is also highly dependent on the solvent flow rate. Figure 6.6 shows the extraction curves obtained for different values of solvent flow rates.

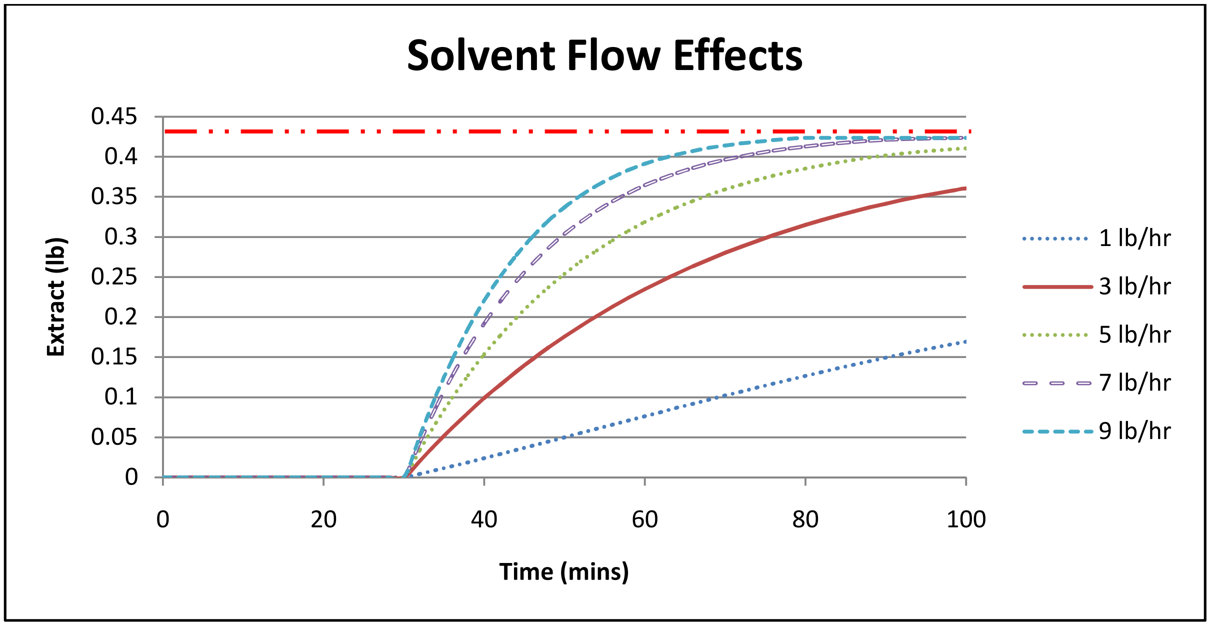

Figure 6.6 Effect of solvent flow rate on Extraction yield

The plot shows how extraction yield varies with variations in solvent flow rate. Higher solvent flow rates result in higher extraction rates thus leading to faster extraction; while lower solvent rates result in a more flatter extraction profile. Thus, increasing the solvent flow rate increases the total yield for the system. Table 6.9 presents the data for the extraction yield and solvent loss as a function of solvent flow rate.

Solvent Flow Rate (lb/hr)	Extraction yield (%)	Solvent Loss (%)
1	40.85	0.168
3	85.15	0.298
5	96.86	0.334
7	100.00	0.347
9	100.00	0.361

Table 6.9 Variations in Solvent loss for changing solvent flow rate

From the table, it can also be seen that higher solvent flow rates lead to increased loss of solvent. Hence, very high flow rates could be uneconomical. To achieve total extraction (100 % yield), a minimum flow rate about of 7 lb/hr would be necessary, with the other process parameters as defined in the previous sections. At this flow rate the total solvent loss is 0.0290 lb, which comes out to be around 0.35 %.

6.3 Sparger Simulation Results

Of the two sparger models discussed in section 5.2, the dynamic model (5.2.2) follows the actual system appropriately and hence, was chosen for further analysis. Figure 6.7 shows the process model for the dynamic simulation sparger (same as Figure 5.9).

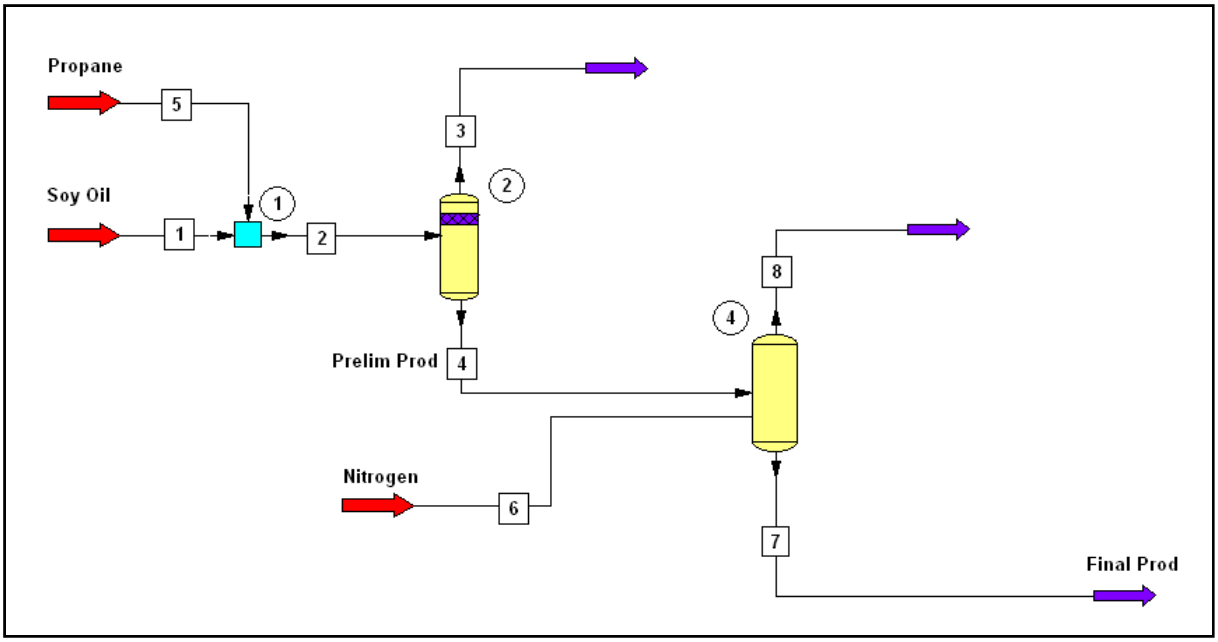

Figure 6.7 Dynamic sparger simulation model

In this system, the flash separator (Unit 2) is used to model the extraction process, while the dynamic vessel module (Unit 4), models the sparger. Unit 2 is set to operate at 65 ^{o}F and 29 psia and Unit 4 operates at 65 ^{o}F and 15 psia. The extraction process model (flash separator) supplies the sparger with 11.9 lb of miscella (soy oil – propane mixture), with an initial propane content of 0.02299 weight fraction; which is then subjected to multiple runs with different nitrogen flow rates. The effects of varying nitrogen flow rates on extract purity are tabulated below (Table 6.10).

Nitrogen (lb/hr)	Propane Content (wt. %)
0	2.30
0.1	2.25
0.2	2.21
0.5	2.09
1	1.91
2	1.63
5	1.12
10	0.70

Table 6.10 Effect of nitrogen flow on final product purity

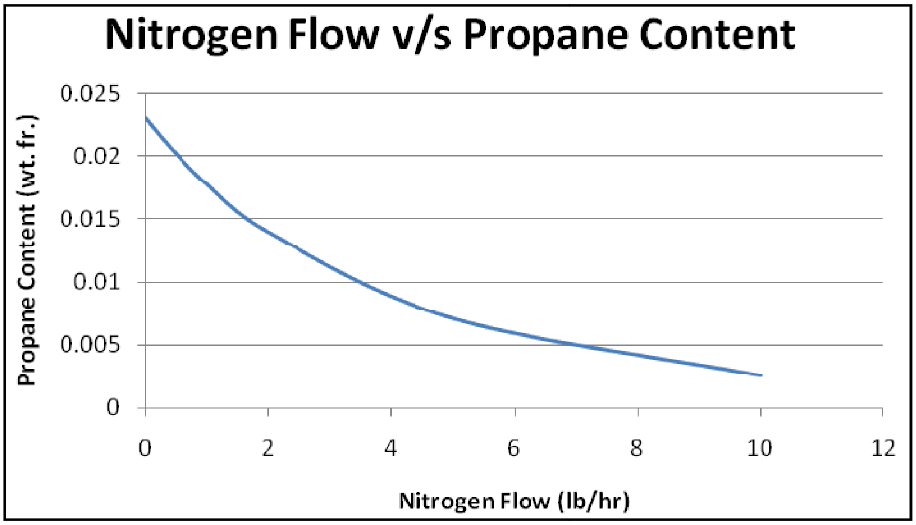

Figure 6.8 Effect of nitrogen flow on final product purity

The plot as well as the table gives the variations of extract propane content as a function of nitrogen flow rate through the sparger. From Figure 6.8, it can be seen that increasing the nitrogen flow results in higher reduction in propane and thus facilitates a purer product.

On a similar note, product purity can also be represented as a function of time (Table 6.11 and Figure 6.9). This representation is particularly useful for determining the sparging time necessary for obtain the specified purity limits.

Time (mins)	Propane Content (Wt. fr.)
0	2.30
5	2.06
10	1.80
15	1.58
30	1.09
50	0.67
75	0.37
100	0.21

Table 6.11 Effect of sparging time on final product purity

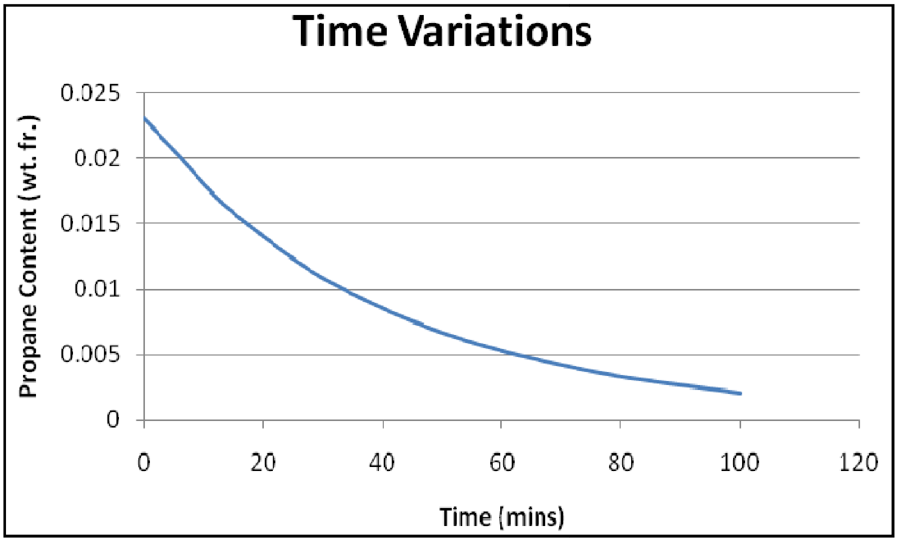

Figure 6.9 Effect of sparging time on final product purity

The above simulations were carried out at a fixed sparger temperature of 65 °F. Temperature plays an important role in the initial refining of the miscella (Oil-Solvent separator) to give relatively purer oil, which is then subjected to sparging. Hence, temperature would be an important factor even during the sparging process. Various simulations were carried out with the sparger temperature in the range 65 ~ 85 °F and the results are given in Table 6.12.

Nitrogen (lb / hr)	Propane Content (wt. %) @T (°F)				
	65	**70**	**75**	**80**	**85**
0	2.30	2.13	1.98	1.84	1.72
0.1	2.25	2.07	1.93	1.79	1.67
0.2	2.21	2.02	1.88	1.75	1.63
0.5	2.09	1.87	1.79	1.61	1.51
1	1.91	1.65	1.53	1.42	1.32
2	1.63	1.29	1.19	1.11	1.03
5	1.12	0.65	0.60	0.55	0.51
10	0.70	0.23	0.21	0.19	0.17

Table 6.12 Effect of sparging temperature on product purity

Table 6.12 gives the data for propane content of the extract for various nitrogen flow rates at 5 different sparger temperature in the range 65 ~ 85 ^{o}F. This tabulated data represented as plots in Figure 6.10.

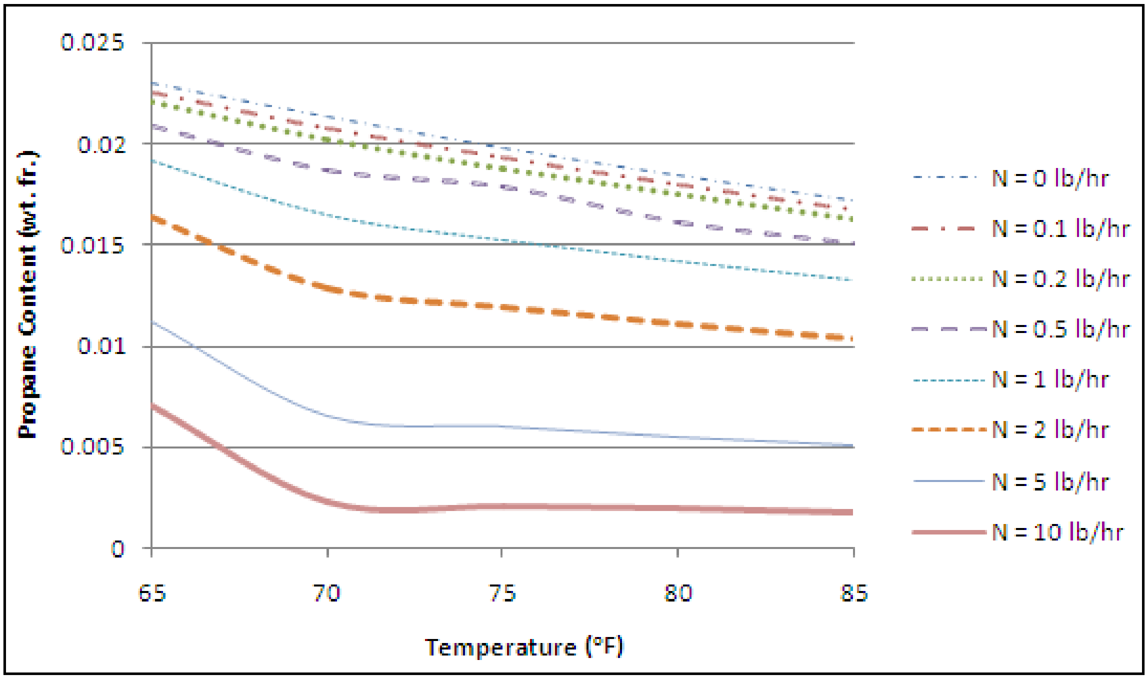

Figure 6.10a Effect of sparging temperature on product purity

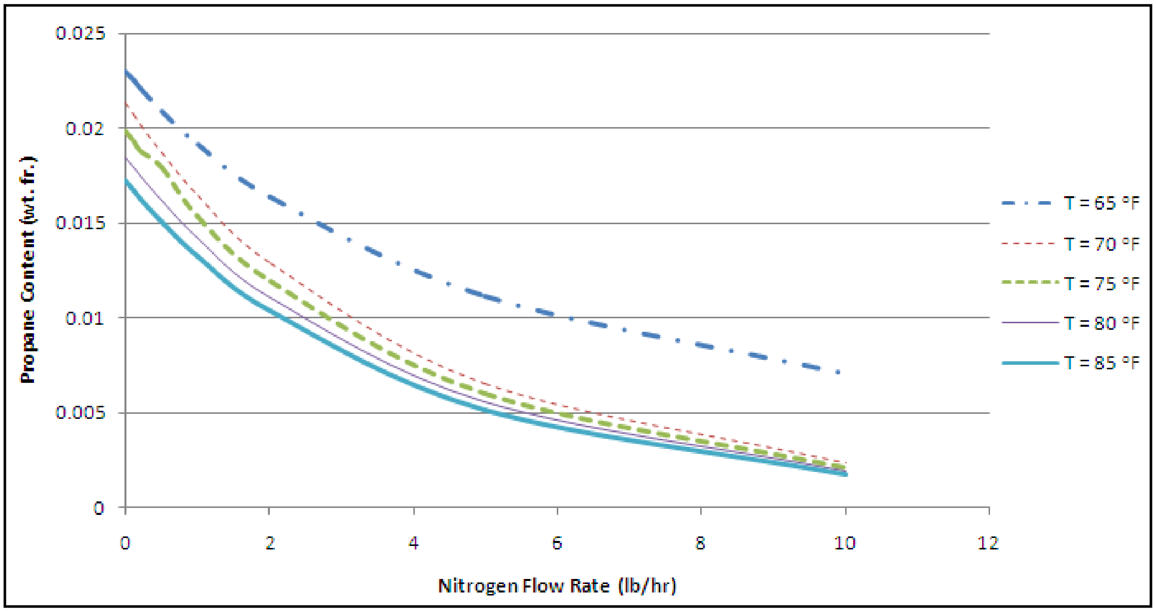

Figure 6.10b Effect of nitrogen flow on product purity at varying temperatures

The two plots (6.10 a & b) displayed above give the variations in extract purity (propane content of extract) as a function of temperature and nitrogen flow rate respectively. It can be seen that, higher temperature and higher nitrogen flow is suitable to obtain a highly pure product. But, there exists an upper limit to these values. The upper limit for temperature is decided by the oil stability at elevated temperatures, while that of the nitrogen flow rate is based on the oil loss along with the exit nitrogen gas. Very high flow velocities for nitrogen could result in entrainment, and thus loss of product, which is undesirable.

CHAPTER 7

130 Liter Extractor (DVT – 130)

7.1 Single Extractor Model Parameters & Estimates

Designing an extraction system using a 130 liter extractor unit is one of the major objectives of this research. The basic extraction process for separating soy oil from the flaked soybean seeds using propane as the solvent has already been detailed in the previous sections and the process flow diagrams for the same are given in Appendix II. The necessary model parameters and process estimates (using the model) are detailed in this section.

7.1.1 Batch Scheduling

The extraction process is made up of multiple steps that have to be performed in a sequence to achieve the required yield. Table 7.1 lists the batch scheduling and the required input and circulation flow rates for the extraction of one 130 liter flaked soybean batch.

	Steps	Time		Flow Rate (lb/hr)	DVT Pressure (psia)
		mins	hrs		
1	Load Fresh Meal to DVT Load Solvent	20	0.33	335 415	
2	Seal and Vacuum	10	0.17		to 11 psia
3	Pump solvent to DVT	6	0.10	401	
4	Solvent circulation	54	0.90	201	
5	Drain Extractor	16	0.27		
6	Dry Raffinate	14	0.23		to 20 psia
7	Unload DVT	10	0.17	400	
8	Optional Cleaning	30	0.50		
	Total	160	2.67		

Table 7.1 Batch operation summary and scheduling from Dr. Bowser [26]

The extractor scheduling time were decided based on an educated guess to obtain a balance between minimum cycle time and equipment ratings necessary to achieve the indicated values. The entire batch can be completed in about 2.7 hrs, including the cleaning operation and 2.2 hrs without cleaning. The flaked soybean meal is loaded into the extraction column and the solvent into the solvent collector. Solvent loading step is not needed to be performed for every batch as the solvent used for extraction is compressed and recycled back to the collector with minimum loss.

Once the extractor is loaded with fresh meal, it is subjected to a vacuum, to reduce the pressure in the system to about 11 psia (565 mmHg). The DVT is then pressurized with propane vapors, following which the solvent circulation begins. The DVT consists of a thermal jacket to control the temperature at a desired level and, it also consists of an agitator to ensure through contact between solvent and the soybean meal. The solvent-oil mixture exiting the extractor is continuously removed and sent to the oil refining flash units, where the oil is separated from the miscella and the solvent is recycled back to the solvent collector for reuse. In addition to this, the residual propane present in the extracted oil is removed by sparging the extract with the inert nitrogen gas and the gas mixture separated is burnt (flared) to remove propane from the exiting mixture.

Once sufficient oil has been removed or the extraction rate decreases such that no significant changes occur to the oil yield, propane circulation is stopped and the extractor is drained to remove the propane entrained in the meal. This step is followed by the raffinate drying process, to remove any residual solvent that remains in the meal. This is accomplished by reducing the extractor pressure to vaporize propane, which separates from the meal. This propane is compressed and recycled back to the solvent collector for later use.

7.1.2 Model Parameters

The flow parameters for important streams have already been summarized in Table 7.1. Apart from the flow values, other important model parameters include those of the extractor (DVT) and the oil refining flash units.

The extractor, which is represented by a combination of a dynamic vessel and a pressure filter, has the most important of the parameters, which quantify the resultant extraction rates. Based on the analysis of the model, the extractor temperature of 80 ^{o}F was decided to be used, to maximize the extraction rate, while not exceeding the oil degradation temperature.

The dynamic vessel also needs a value for the outlet flow, which was set at two and half times the value of solvent flow rate to take into account the solids that exit the extractor along with the miscella. The exit miscella flows to the pressure filter, which separates the miscella from the solids. The moisture (liquid) content for this unit was estimated based on the model calibration curve presented in the earlier section, which comes out to be about 42% (wt.); this adjusted to include the mixing effect that would be available in the actual unit and set at about 39%. And the pressure drop for the filter unit is set to 120 psi.

For the oil refining section which consists of two flash units, the temperature and pressure conditions were decided based on the results obtained in the previous section. The primary flash (Solvent-Oil Separator) was operated at 70 ^oF and 10 psia, with the decanter operating at 50 ^oF and 15 psia. While, the secondary (Water-Solvent separator) operates at 40 ^oF and 10 psia. All the model parameters are listed in Table 7.2.

Equipment	Parameters
Extractor (Dynamic Vessel)	T = 80 ^oF, P = 200 psia
	Outlet Flow = 2.5 * Solvent circulation Rate
Pressure Filter	$\Delta P = 120\ ^oF$
	Moisture Fraction = 0.39
Oil-Solvent Separator	T = 70 ^oF, P = 10 psia
Decanter	T = 50 ^oF, P = 15 psia
Solvent-Water Separator	T = 40 ^oF, P = 10 psia

Table 7.2 Process operating parameters

7.1.3 Estimated Results

The assumptions that go into the simulation model include the various density values; soybean meal composition, etc. and these are listed in Appendix VI. The extraction process simulation was carried out according to the scheduling given in Table 7.1, for a single batch to determine the amount of soy oil extracted from each batch for the conditions listed in Table 7.2. Other process related details are summarized in Table 7.3.

CYCLE DETAILS	
Max # of cycles / day	11
Time per cycle (mins)	130
Soy beans processed (lb)	112
Solvent Used (lb)	138.33
Solvent Circulation Rate (lb / hr)	201
Oil in feed (each cycle) - lb	22.33
Moisture in feed (each cycle) - lb	11.17

Table 7.3 Cycle details for batch processing from Dr. Bowser [26]

The total soy oil extraction achieved from the system at the specified process conditions comes to be about 19.34 lb, with traces of propane and water present in it. This amounts to an extraction yield of about 85 %. This can be increased to 91 %; by increasing the solvent circulation rate to 245 lb/hr (total solvent volume used is about two and half times the volume of soybean processed per hour). Table 7.4 summarizes all the details involved with the estimates.

Product Obtained (lb)	19.34	Product Obtained (lb)	20.60
Composition		Composition	
Propane	0.0118	Propane	0.0118
Water	0.0042	Water	0.0042
Soy Oil	0.984	Soy Oil	0.984
Soy Oil in product (lb)	19.03	Soy Oil in product (lb)	20.27
% Yield Achieved	85.23	% Yield Achieved	90.78
% Solvent Loss	0.36	% Solvent Loss	0.37

Table 7.4 Extraction results and estimates

The variations in process variables and parameters with time as the process proceeds to completion are listed in Figure 7.1.

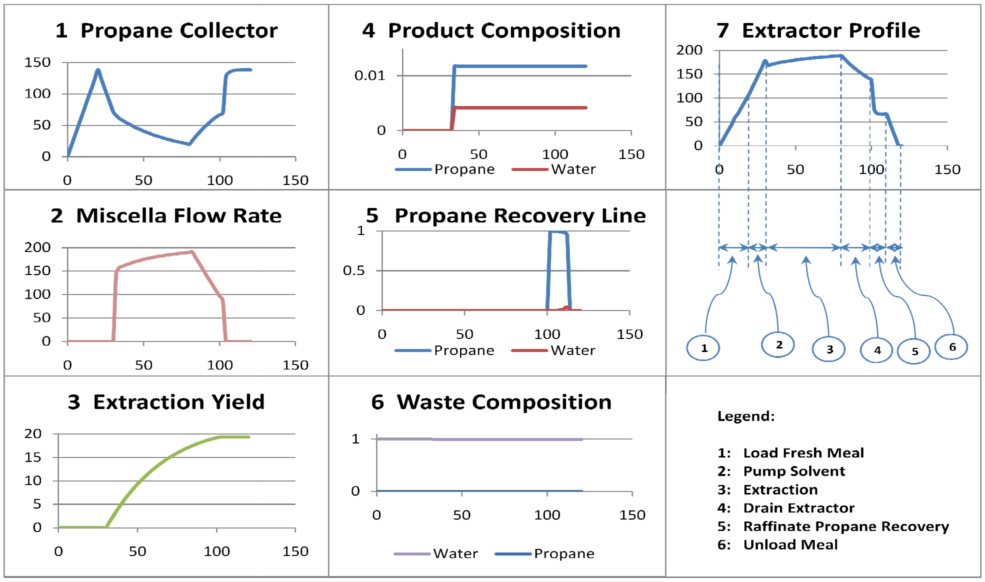

Figure 7.1 Variations in process streams and process variables through each cycle

The figure shows the plots for solvent mass in the propane collector (lb), the miscella flow rate (lb/hr), extraction yield(lb), product composition (wt. fr.), raffine solvent recovery line composition (wt. fr.), waste stream composition and the extractor loading profile respectively. Each of the data is plotted against process time (batch cycle time) and shows how these parameters vary as extraction proceeds to completion. The various steps in the cycle are indicated on plot 7 (Extractor profile) of the graph.

Plot 7 shows the extractor profile for a single batch cycle. During th initial stages (0 ~ 20 mins), the content of the extractor increases as the freash batch of soybean meal is loaded into the extractor; following this, solvent propane is pumped into the extractor, which initially fills the extractor and solvent circulation starts at 30 mins (indicated by 2 on plot 7). Cycle time of 30 to 80 mins (section 3 on plot 7) consists of the extraction (or solvent recirculation) step. Section 4 indicates the solvent drainage step. During the same time interval, the miscella flow rate drops continuously and then stops as the solvent drainage step comes to an end at about 100 mins from the start of the cycle. This is followed by the raffinate solvent recovery stage (section 5) in which, vacuum is applied to the extractor unit to remove any entrained solvent. This results in furthur drop in the extractor mass, with a corresponding increase in the loading of propane collector unit (Plot 1) as the seperated solvent is compressed, cooled and recycled back to the propane collector. Plot 5 shows the purity of the propane obtained during this stage.

The final section of Plot 7 (section 6) represents the unloading characteristics of the system, as the spent meal is removed from the extractor and disposed for further processing. The extraction yield obtained for the process is plotted as a function of time (Plot 3). The initial 30 mins of this plot is at zero as solvent circulation (extraction) begins only 30 mins after the start of cycle, and it increases in the form of a parabola and reaches the maximum extraction yield obtainable for the specified process conditions. Plots 4 and 6 shown on column two of Figure 7.1 show the variations in the product and waste compositions as the process proceeds to completion.

7.1.4 Equipment Sizing and Costing

All the units including major equipment as well as auxiliaries, excluding the vacuum pump have been sized using the ‘Sizing and Cost Estimation’ module available on ChemCAD 5.6. The vacuum pump module not being well defined on ChemCAD, it was decided to size the pump manually. The Chemical Engineering Plant Cost Index (CEPCI) for Dec 2007 was used as the base index to account for inflation [20] (Table7.5).

CEPCI	**Dec '07**
CE INDEX	525.0
Equipment	623.3
Heat Exchangers & Tanks	593.6
Process Machinery	597.9
Pipes, valves & fittings	727.2
Process Instruments	414.4
Pumps & compressors	840.0
Electrical Equipment	436.3
Structural supports & misc	660.8
Construction labor	317.0
Buildings	477.0
Engineering & supervision	356.2

Table 7.5 CEPCI cost index for Dec 2007 [20]

Sizing of the equipment was done for the maximum flow conditions that occur in the system. Since the process deals with edible components, it was decided to use stainless steel for the equipment which process oil, while Carbon steel was employed for the rest of the units. The equipment purchase cost and installation cost for the equipment including the auxiliaries were included in the calculation of the Bare Module Cost. Table 7.6 gives a summary of the cost estimation for the various units involved. The detailed equipment specifications, cost estimation and sizing results are given in Appendix VII.

Unit ID	Notes	FoB ($)	Installation Factor[18]	Installed Cost ($)
16	Working propane feed pump	6603	3.3	21790
13	DVT feed pump	5714.5		18833
19	Miscella Heater	3211	3.17	10178
14	Flash Cooler	2758		8744
20	Propane Condenser	2749		8713
5	Propane Compressor	39629	2.15	85202
7	Oil-Solvent Separator	27867	4.16	115177
3	Water-Solvent Separator	13518		56236
22	Oil Decanter	25709		43705

Table 7.6 Cost estimation details for single, batch processing

The total installation cost for the unit which produces about 500 lb of soybean oil per day comes to be just below $ 370,000 (The install factors are the approximations for setting up an full scale plant; for a pilot plant half of these values can be used. This gives an estimate of about $ 240,000).

Vacuum pump models are not available on ChemCAD. Hence, it cannot be used to determine the size (rating) of the pump required for the system and hence the cost of the unit. The vacuum pump unit used in the lab scale experimental setup was an air driven unit with approximate average capacity of 1.5 scfm for pressure being reduced from 50 psig to 0 psig and 0.11 scfm for pressure reduction below atmospheric pressure. The maximum vacuum achievable by the unit was 27 "Hg for outlet resistance of 15 psig and 23 "Hg for outlet resistance of about 250 psig.

The choice of a vacuum pump should be done based on the maximum vacuum rating achievable and the pump capacity [24] and pump down time, which is a function of these two parameters would be a good option to assess the selection of vacuum pump. The pump down time can be calculated as.

$$\theta = \frac{V}{Q} \ln\left(\frac{P_1 - P_0}{P_2 - P_0}\right)$$

Where,

θ : Pump down time (mins)

V : System volume - including connecting pipes (cu. ft.)

Q : Pump average capacity (scfm)

P_1 : Starting Pressure

P_2 : Pressure to be achieved

P_0 : Minimum pressure achievable

This value is usually increased by 25% to account for any minor leakages in the system. Table 7.7 gives the comparison of the predicted pump performance (pump down time) when using this unit on the lab scale unit and the larger 130 liter DVT unit.

P_0 (“Hg)	Pump Down Time (θ) mins	
	5 L DVT (V = 0.672 ft^3)	130 L DVT (V = 12.823 ft^3)
22.4	4.63	88.32
10.2	16.53	315.40
7.15	37.70	719.40

Table 7.7 Comparison on pump down time for experimental unit and proposed pilot unit

The table gives the pump down time in minutes for three different cases of vacuum pressures. The system volume (V) indicated in the table is the total volume of the extractor, the flash units and the piping connecting these units. As can be seen, the current vacuum unit would require a very high pump down time to reduce the system pressure to about 7 “Hg for the 130 liter extractor process. Hence, it would be advisable to use a unit which has a better capacity rating. A vacuum unit of very high capacity rating would be required to achieve a pressure as low as 7 “Hg with a small pump down time. Calculations for pump down time to achieve 7 “Hg as a function of the pump capacity rating is shown in Table 7.8.

Flow Capacity (scfm)	Pump down Time (mins)
0.11	720
0.5	160
1.0	81
3.0	27
5.0	16
7.0	11.5

Table 7.8 Effect of pump flow capacity on pump down time

From the table it can be concluded that, a vacuum pump with a capacity rating on about 7 ~ 10 scfm would work well for the current system.

7.2 Multiple Alternating Extractor Estimates

The multiple alternating extractors concept was introduced considering that, it would reduce the total cost of the process or will double the production capacity at a small increase in capital costs. The model for this concept was very similar to the single extractor model, with the only difference being the addition of a second extractor unit to the process. The basic parameter values and the operating parameters remain the same.

7.2.1 Scheduling

As in the earlier case, the process is divided into multiple steps. The additional extractor unit has to be fitted into the schedule and the possibility of using more than two units was accessed. Using more than two units would make it necessary to increase the solvent inventory and hence, use of more than two units was not considered. The batch scheduling for each of two extractor units and for the entire process is given in Table 7.9.

Time	DVT - 1	DVT - 2	Time
0 - 10	Load Fresh Meal		0 - 10
10 - 20			10 - 20
20 - 30	Seal and Vacuum		20 - 30
30 - 40	Pump solvent to DVT and solvent circulation		30 - 40
40 - 50			40 - 50
50 - 60			50 - 60
60 - 70		Load Fresh Meal	60 - 70
70 - 80			70 - 80
80 - 90		Seal and Vacuum	80 - 90
90 - 100	Drain Extractor and Dry raffinate	Pump solvent to DVT and solvent circulation	90 - 100
100 - 110			100 - 110
110 - 120			110 - 120
120 - 130	Unload DVT		120 - 130
130 - 140	Load Fresh Meal		130 - 140
140 - 150			140 - 150
150 - 160	Seal and Vacuum	Drain Extractor and Dry raffinate	150 - 160
160 - 170	Pump solvent to DVT and solvent circulation		160 - 170
170 - 180			170 - 180
180 - 190		Unload DVT	180 - 190
190 - 200			190 - 200
200 - 210			200 - 210
210 - 220			210 - 220
220 - 230	Drain Extractor and Dry raffinate		220 - 230
230 - 240			230 - 240
240 - 250			240 - 250
250 - 260	Unload DVT		250 - 260

Table 7.9 Process Scheduling for multiple alternating extractor setup

The second column in the table gives the scheduling for one of the extractors; while column three shows the scheduling for the second extractor. The scheduling is done such that, when one of the extractor is in the extraction loop, the other one would be undergoing the down-time operations (unloading, reloading, etc.). Thus, it can be ensured that there will be a continuous flow of output from the process, though it would still be unsteady.

7.2.2 Model Parameters

Two cases of the process are investigated. One using two 130 Liter DVT’s with an attempt to double the production rate and the other using two DVT’s half the capacity of the 130 Liter extractor, with an attempt to reduce the costs. The model parameters used for both of these systems was same as that of the single batch extraction model; only the process flow parameters varies for the second system.

7.2.3 Estimated Results

Case 1 was expected to give a 75 % increase in the production rate; while the second was expected to result in a 25 % reduction in the sizes of the down stream units for the same production capacity. The simulations yielded similar findings. For the alternating extractor design, the production capacity was found to almost double; an exact value being a production increase of 83 % for the same amount of process time (68 lbs in 2 cycles). The quality of the oil obtained was same as that obtained for the single extractor model because of the similarities of the down stream processing units and their operating parameters. Figure 7.2 shows the variations in various important parameters of the process as extraction proceeds.

The first three pictures in the figure show the operation parameters for Extractor-1 (DVT 1). A comparison of operation of the two DVT's (Plot 1 and 8) in the figure would show how the operation actually takes place. DVT 1 and DVT 2 operate in an alternating fashion, with their shut down and restart periods overlapping. Plot 5 shows the propane collector profile for such an operation. The small bumps on the plot are because of the alternating nature of the process. Also, plot 6 in the picture gives the extraction yield obtained as the process progresses. There is no stable section on this plot, which indicates that the process downstream units operates continuously and thus a continuous product recovery is achieved.

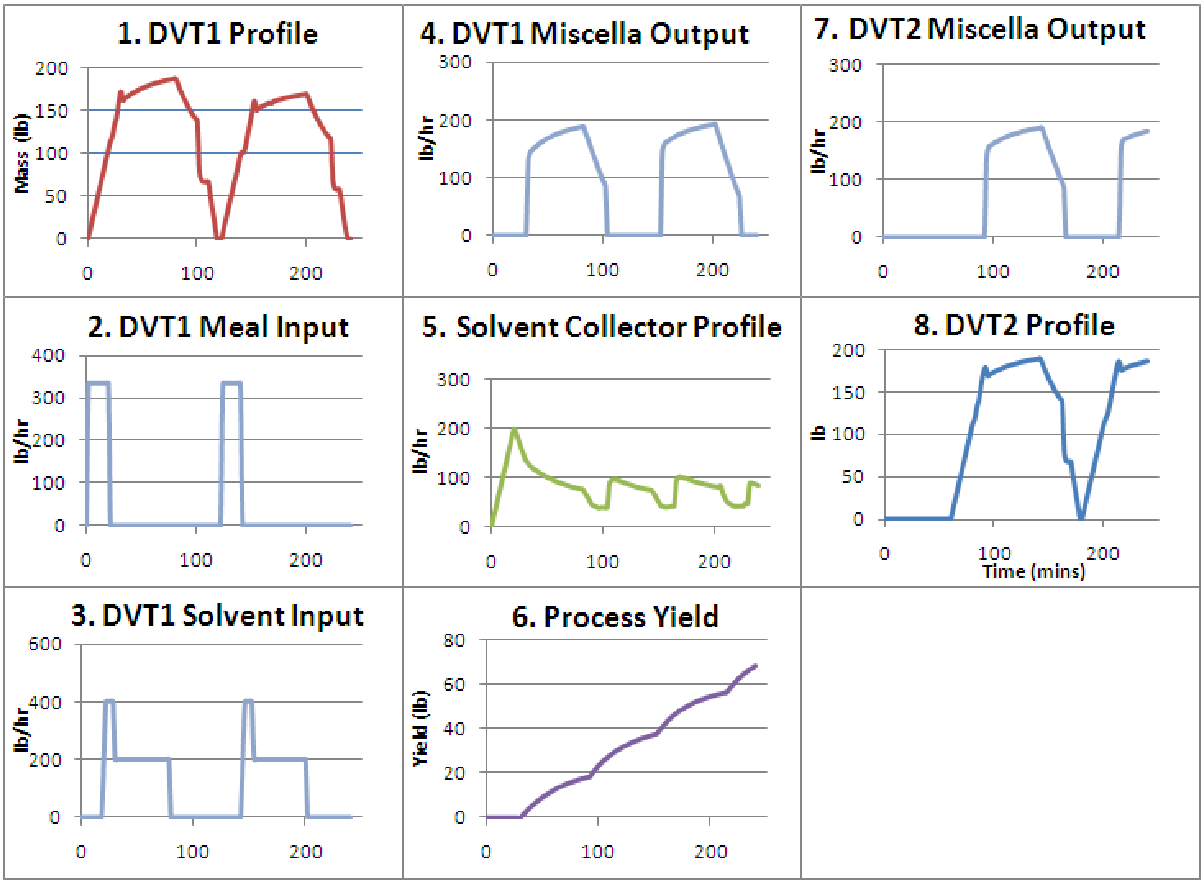

Figure 7.2 Variations in process parameters for multiple alternating extractor setup

In the case of using two DVT's of half the total capacity, a large reduction in equipment sizes was observed. This data of comparison between equipment sizes / rating are presented in Table 7.10.

Unit	Case - I	Case - II
Working propane feed pump	0.0323 hp	0.0194 hp
DVT feed pump	0.0796 hp	0.0395 hp
Miscella Heater	4.06 m^2	2.06 m^2
Flash Cooler	5.78 m^2	2.97 m^2
Propane Condenser	5.52 m^2	3.21 m^2
Propane Compressor	5.66 hp	3.47 hp
Oil-Solvent Separator	1 m (dia.) x 4.8 m	1 m (dia.) x 3.52 m
Water-Solvent Separator	1 m (dia.) x 4.2 m	1 m (dia.) x 3.15 m
Oil Decanter	1 m (dia.) x 3.0 m	1 m (dia.) x 3.0 m

Figure 7.10 Comparison between equipment sizes for the two cases of alternating extractors

CHAPTER 8

CONCLUSIONS

The process for extraction of soy oil using propane as the solvent offers useful advantages over the conventional processes being applied in the industry. Improved product oil quality, reduced energy requirements, significantly suppressed hazardous operating conditions are some of advantages of the current process. Not many literature studies employing propane as a solvent to extract soy bean seeds or gave any design methods and parameters for such a process. Hence, the main objective of the process was to determine the applicability of using propane as a solvent and to generate process models which would be useful in determining the design parameters for a pilot plant scale setup.

Various process alternatives and process models have been generated and simulated using the ChemCAD process simulating software. The model was calibrated using the data obtained from the lab scale unit where the experiments were performed, and simulated to obtain results; which was then compared with experiments conducted at different process operating conditions and these match well. Based on the models and the calibration results, process design parameters were obtained and the operating conditions were set to give the maximum production rate, with maximum quality for minimum product damage and minimum expenses.

8.1 Research Conclusions

Based on the model proposed, the following conclusions have been drawn:

1. The experiments and the simulations indicate that necessary amount of extraction can be obtained at ambient temperatures. Hence, propane is a potentially viable solvent and further detailed estimates for employing a process using solvent propane should be generated before final implementation.
2. The production rate increases at higher extraction temperatures, but remains unaffected by the pressure of the system. But, the pressure is still an important parameter as it has to be made sure that the extractor system operates at a pressure above the vapor pressure of propane, thus keeping the solvent in liquid state.
3. Apart from the extractor temperature, the production rate also depends on various other parameters including the solvent propane flow rate, solvent quality, amount of meal processed and porosity or void area present in the meal. Of these, the solvent flow rate plays a very important role. Increased solvent rates lead to increased extraction rates thus, higher production; but it also led to an increase in the equipment sizes and thus increases the cost of the process.
4. Three process alternatives have been proposed. One using a single extractor in a semi-batch process, the second using multiple extractors in alternating process operation and the final using multiple extractors arranged sequentially in series to give a near steady state or continuous operation.
5. For the multiple alternating extractors process, it would be viable to apply just TWO extractors such that, when one is in its extraction step, the second extractor goes through

the remaining of the process stages. More than two extractors would result in larger inventory for solvent propane and hence the size of the propane collector and also results in increased extraction time.

6. Employing the alternating extractors process alternative, the production rate can be increased by 83 % with just addition of an standby extractor to the existing process. Or, the process downstream units sizes and rating requirements can be reduced by almost 25 ~ 40 % using two DVT's of half the total production capacity.
7. The concept of multiple continuous extractors was introduced considering that it would lead to a continuous production of soy oil at steady state or near steady state conditions. The main advantage of using such a process is that the downstream operations do not remain idle anytime during the process and also operate with lower fluctuations than encountered in a batch or semi-batch process. This alternative seems appealing due to the fact that a near steady state process can be achieved.
8. The Multiple sequential extractors setup has an additional variable as compared to the other processes, which is to decide the number of extractors that could be (has to be) arranged in series to achieve the objective. An ideal case would be to use as large a number as possible. But, for practical purposes and limitations, more than two extractors cannot be operating at the same time, with a third extractor used as a stand by. Increasing the number of extractor units results in a very low down-time, which is practically impossible to achieve or increases the process cost.
9. The downstream units necessary for refining the miscella is a set of flash units and a decanted operated under vacuum conditions to reduce the thermal degradation of the product and increase the crude product quality. Product purity of 98.7 % oil and rest

propane is obtained when the units are operated a pressure of 10 psia (“Hg vacuum). Decreasing the pressure any further would lead to increased costs of the vacuum unit, with no major changes in the product quality.

10. The solvent-water separator flash unit and the upstream heat exchanger can be removed from the process as the presence of this flash unit does not make a significant impart on the solvent purity (99.6 %) obtained compared to the purity (99.3 %) when no solvent-water separator is used.

8.1 Future Work

The following additions can be incorporated in the present model to further improve it:

1. The current model does not consider the presence of impurities in the oil or the solvent (after under going extraction). Compounds like tocopherols, sterols, sugars can be incorporated to further improve the prediction horizon.
2. Multiple continuous (sequential) extractor process can be investigated and modeled to generate predictions which can be compared with some experimental unit results.
3. Though the current model gives acceptable solutions, an alternate method to model the solid – oil mixture might improve the predictions.
4. One of the unresolved issues with the model is the raffinate desolventizing operation model. This model predicts an almost instantaneous flashing of the solvent from the raffinate which leads to a very high vapor flow rate in the process line; Future works should focus on eliminating this concern.

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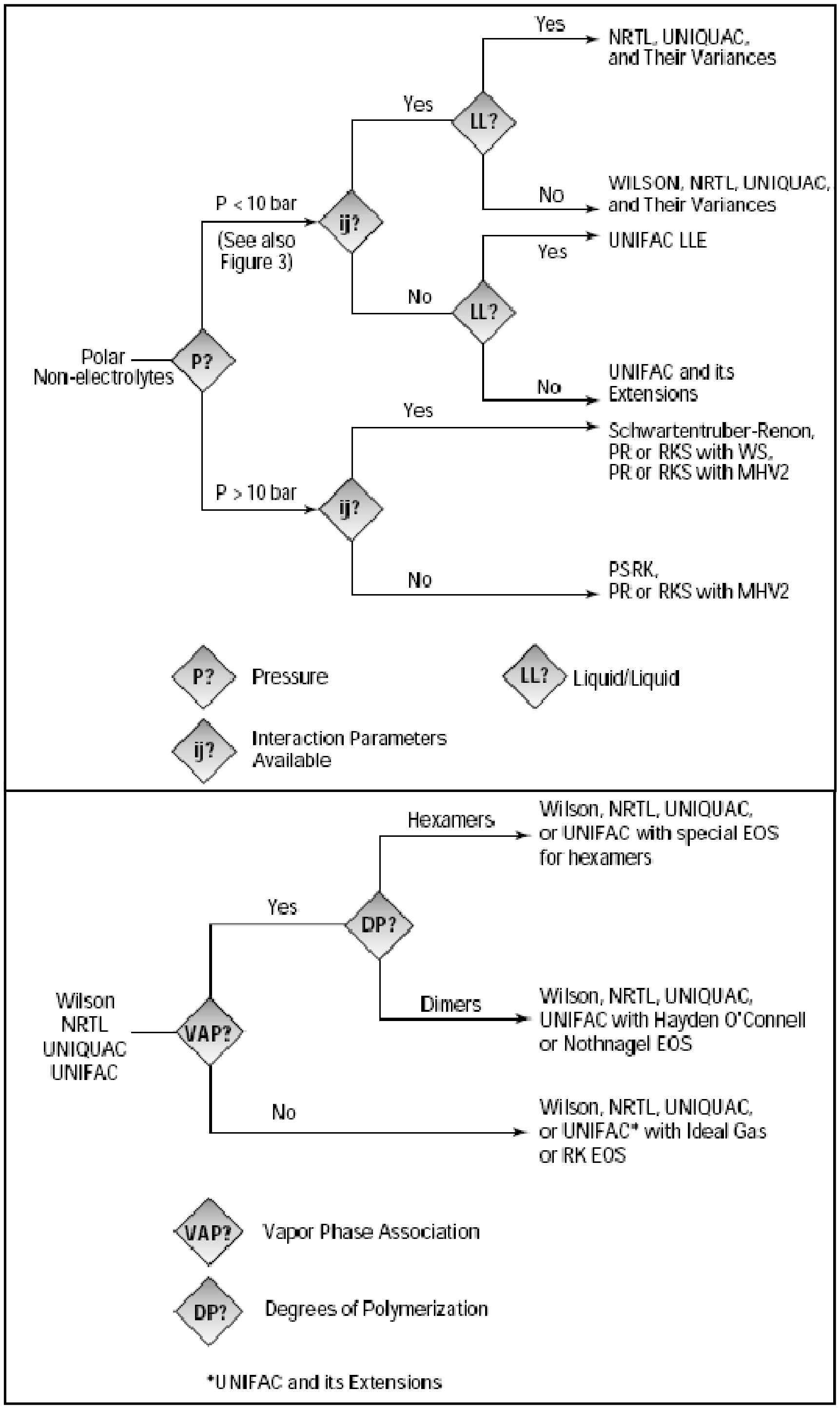
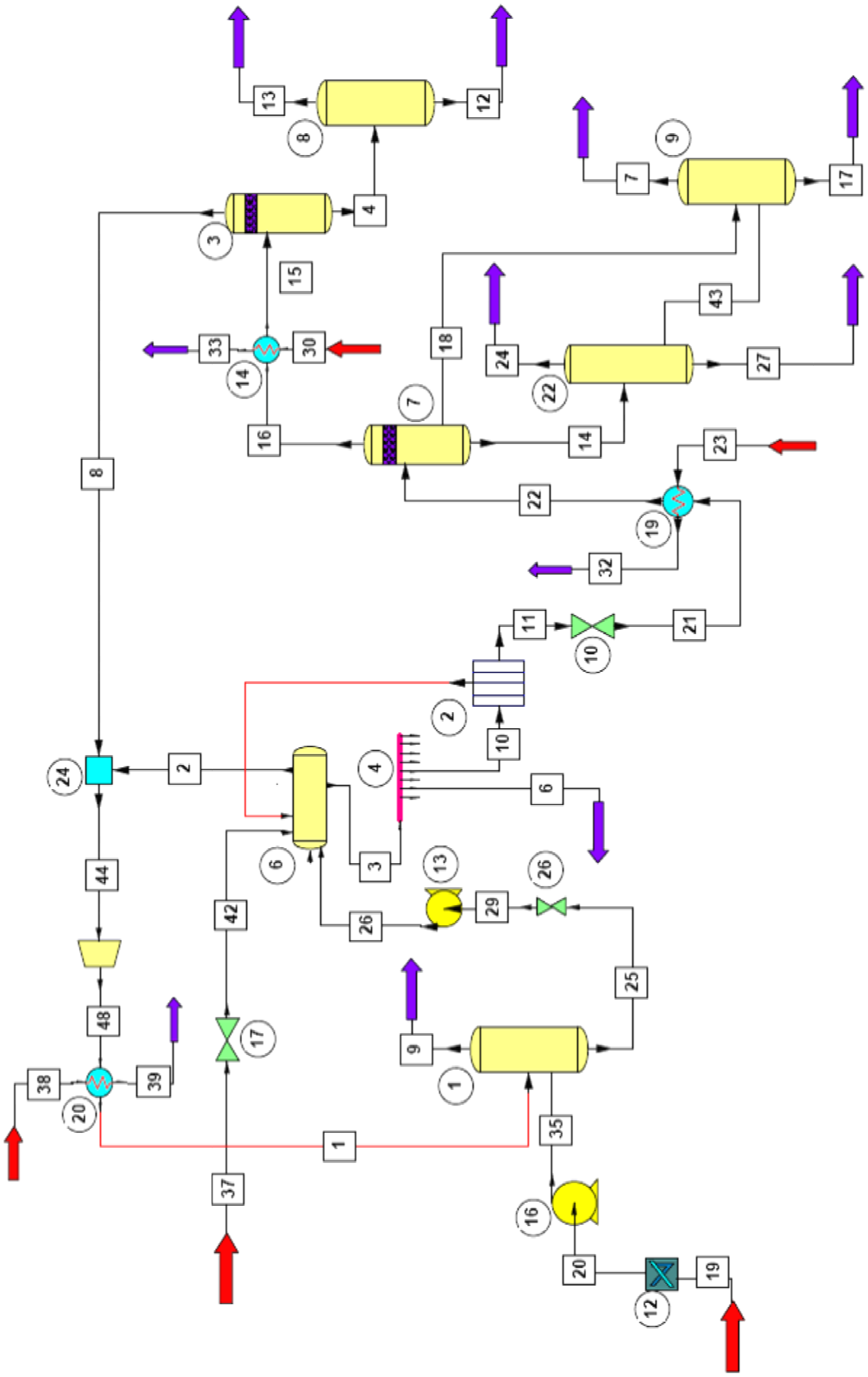
APPENDIX – I
Thermodynamic Models [16]

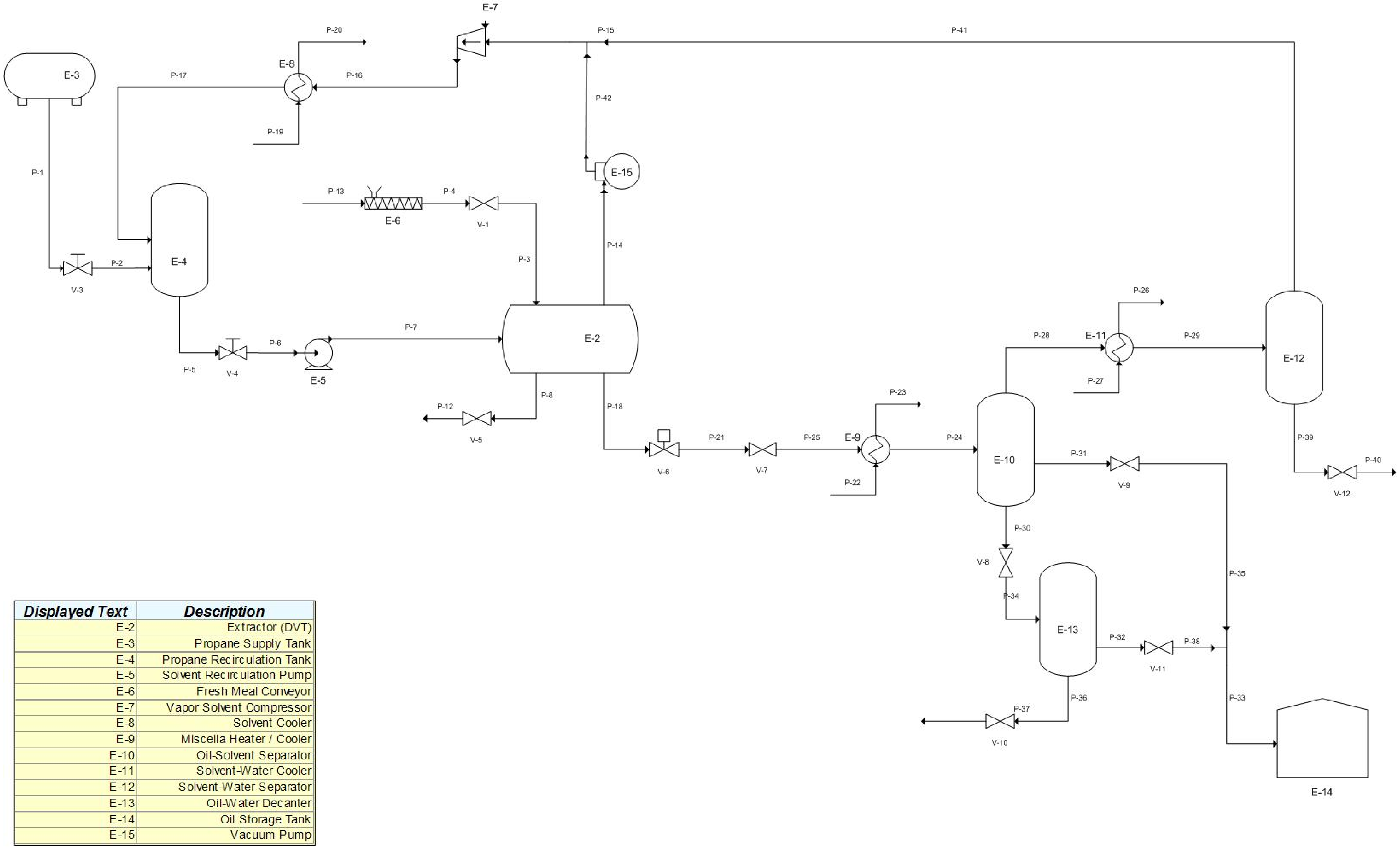
The choice of the thermodynamic model for a process depends on a variety of factors such as the type of system under consideration (liquid system, vapor liquid system, etc.), type of chemical substances to be analyzed, the availability of interaction parameters etc. The figure shown below give a heuristic for selection of the type of thermodynamic model to be used based on the conditions discussed above.

The diamonds in the figure represent questions that modeler needs to ask himself about the process. E.g. is the pressure range of interest below 10 bars? If the answer to this question is true then, are the interaction parameters for the species involved are available? If yes and the process is an liquid liquid system then NRTL or the UNIQUAC and their variations would give good predictions of the thermodynamics of the system. Similarly, the other branches can be used depending on the various parameters that affect the process.

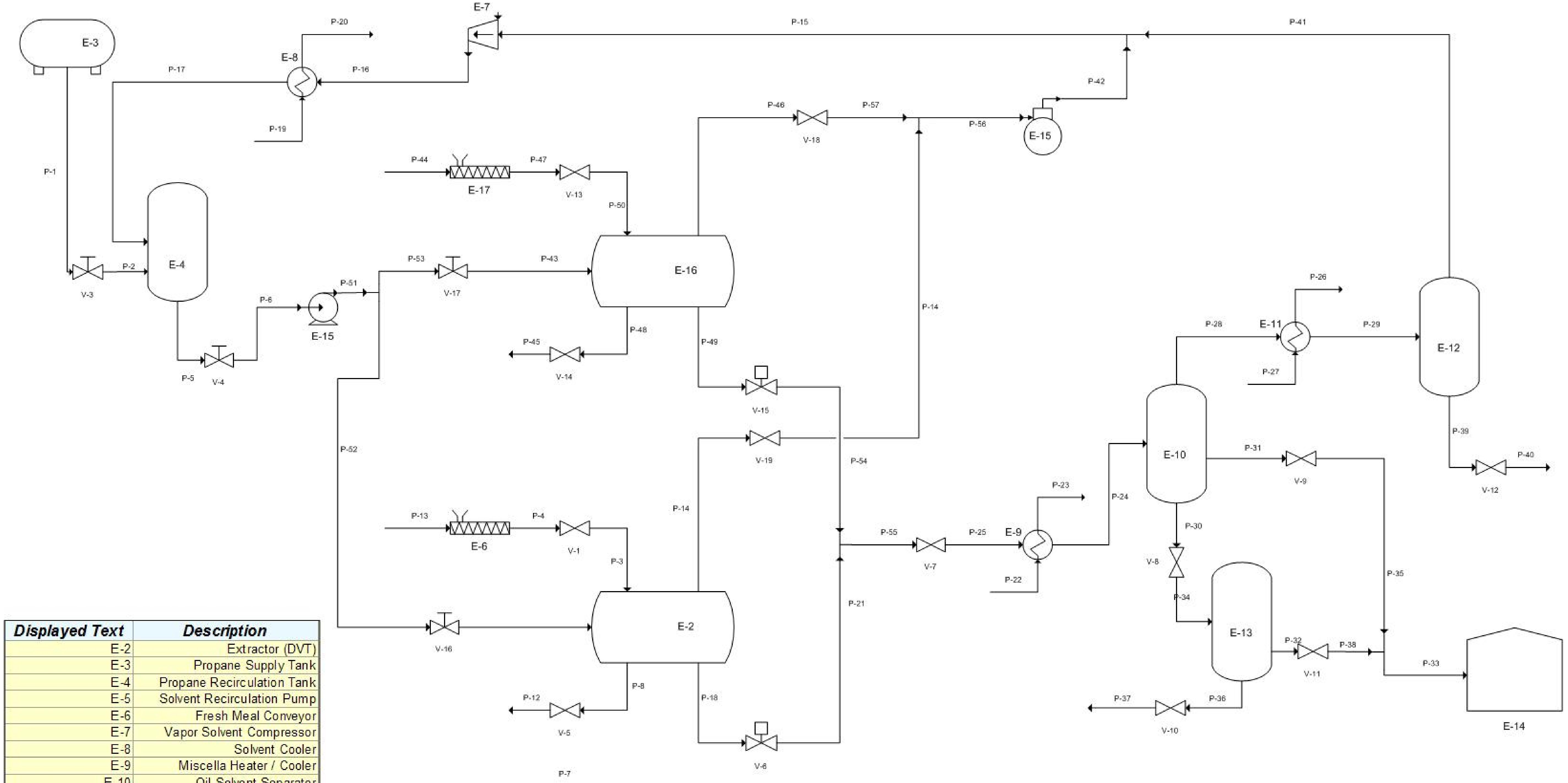

Polar Non-electrolytes
P?
P < 10 bar
(See also Figure 3)
ij?
Yes
LL?
Yes
NRTL, UNIQUAC, and Their Variances
No
WILSON, NRTL, UNIQUAC, and Their Variances
No
LL?
Yes
UNIFAC LLE
No
UNIFAC and its Extensions
P > 10 bar
ij?
Yes
Schwartentruber-Renon, PR or RKS with WS, PR or RKS with MHV2
No
PSRK, PR or RKS with MHV2
P? Pressure
LL? Liquid/Liquid
ij? Interaction Parameters Available
Wilson NRTL UNIQUAC UNIFAC
VAP?
Yes
DP?
Hexamers
Wilson, NRTL, UNIQUAC, or UNIFAC with special EOS for hexamers
Dimers
Wilson, NRTL, UNIQUAC, UNIFAC with Hayden O'Connell or Nothnagel EOS
No
Wilson, NRTL, UNIQUAC, or UNIFAC* with Ideal Gas or RK EOS
VAP? Vapor Phase Association
DP? Degrees of Polymerization
*UNIFAC and its Extensions

APPENDIX – II
Process Flow Diagrams





Displayed Text	***Description***
E-2	Extractor (DVT)
E-3	Propane Supply Tank
E-4	Propane Recirculation Tank
E-5	Solvent Recirculation Pump
E-6	Fresh Meal Conveyor
E-7	Vapor Solvent Compressor
E-8	Solvent Cooler
E-9	Miscella Heater / Cooler
E-10	Oil-Solvent Separator
E-11	Solvent-Water Cooler
E-12	Solvent-Water Separator
E-13	Oil-Water Decanter
E-14	Oil Storage Tank
E-15	Vacuum Pump



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E-2	Extractor (DVT)
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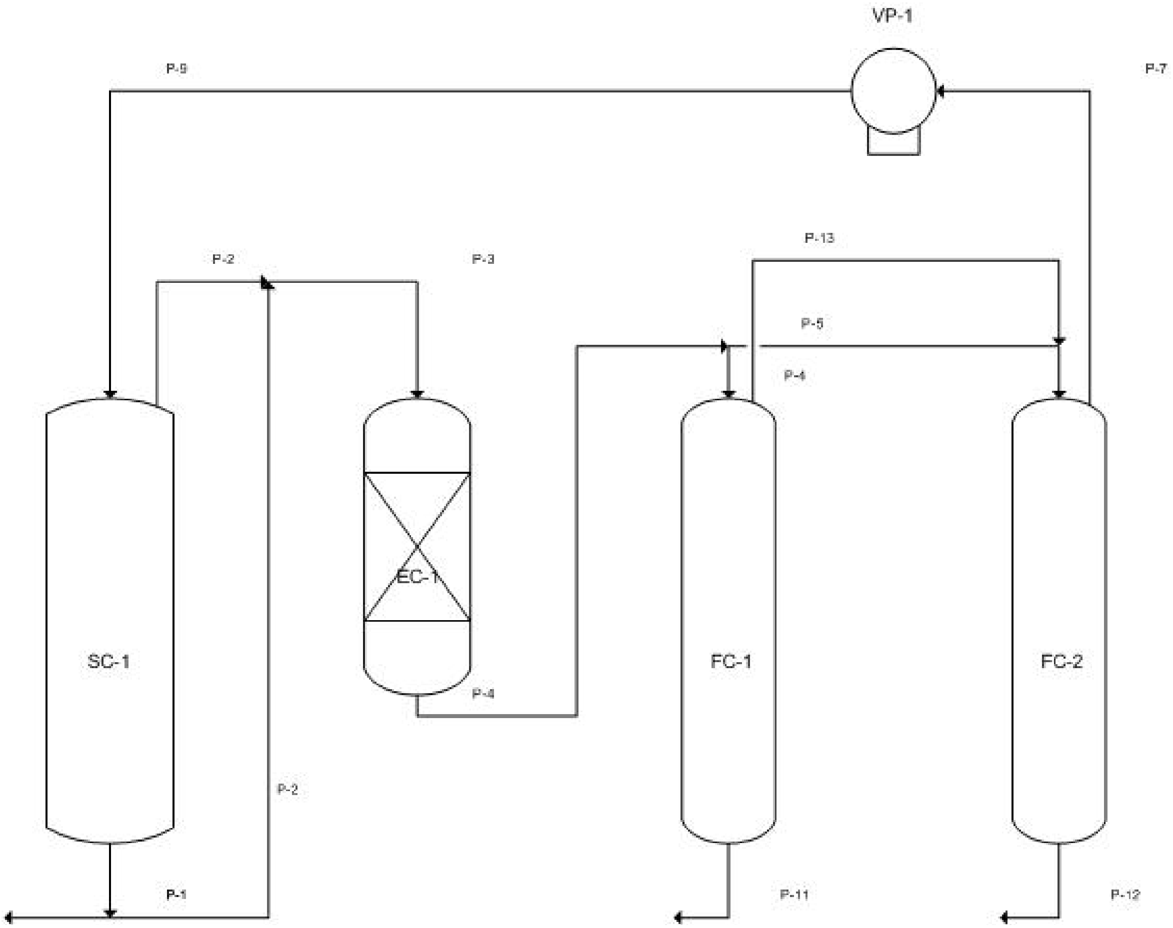
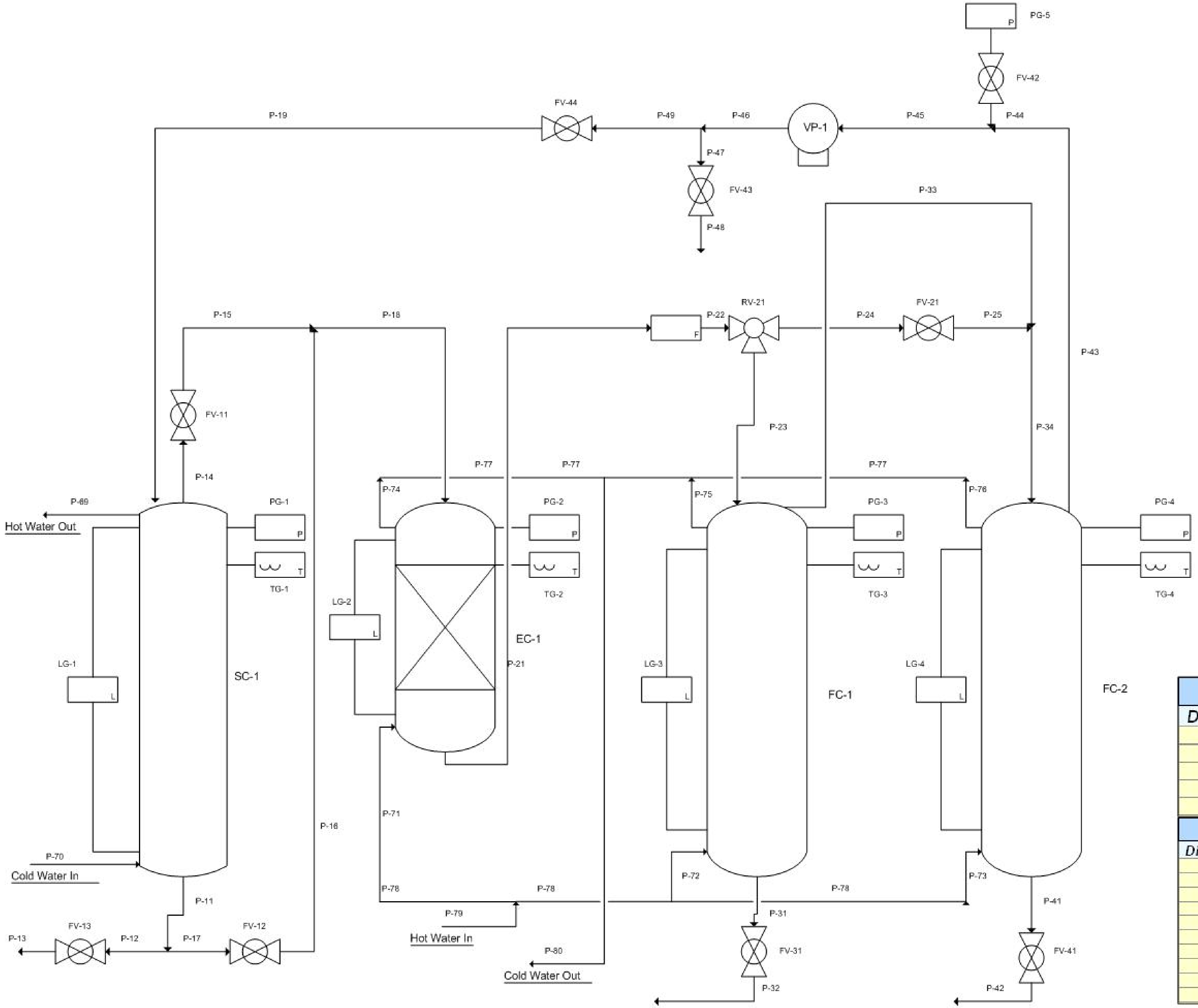
E-9	Miscella Heater / Cooler
E-10	Oil-Solvent Separator
E-11	Solvent-Water Cooler
E-12	Solvent-Water Separator
E-13	Oil-Water Decanter
E-14	Oil Storage Tank
E-15	Vacuum Pump

APPENDIX – III a
Experimental Setup





Equipment List

Displayed Text	Description	Manufacturer	Material	Model
EC-1				
FC-1				
FC-2				
SC-1				
VP-1				

Valve List

Displayed Text	Description	Line Size	Valve Class	Manufacturer	Model
FV-11					
FV-12					
FV-13					
FV-21					
FV-31					
FV-41					
FV-42					
FV-43					
FV-44					
RV-21					

APPENDIX – III b
Operating Procedure

Operating Procedure

1. EVACUATE SYSTEM

Objective: Remove air (oxygen) from system.

a. ENTIRE SYSTEM

- i. Open valves FV-11, RV-21, FV-21, FV-42 (vacuum gauge), FV-43 (vent), all others closed.
- ii. Turn on pump VP-1, until entire system us under vacuum (about 6.5 ~ 7 in. H2O).
- iii. Turn off VP-1 and wait for few minutes to ensure that no gross leaks are present.

b. EXCLUDING PROPANE TANK

- i. Open valves RV-21, FV-21, FV-42 (vacuum gauge), FV-43(vent), all others closed.
- ii. Same as ‘a(i)’
- iii. Same as ‘a(ii)’
- iv. Proceed to next step

2. PROPANE FILL

Objective: To input fresh propane to system (to SC-1).

Continue to this step only after confirming that the system is totally evacuated.

- i. Detach PG-5 from the system and attach filling line to the system.

ii. Open valves FV-11, RV-21, FV-21, FV-42, FV-44, all others closed.
iii. Turn on VP-1.
iv. Monitor LG-1 (tank should not be more than ¾ full)
v. Turn off VP-1
vi. Close FV-42 and detach filling line from the system
vii. Reinstall PG-5
viii. Proceed with step ‘1(b)’
ix. Proceed to next step

3. LOADING EXTRACTION MEAL

Objective: To input fresh meal for extraction into the system (to EC-1)

i. Detach line P-18 from the system
ii. Dismantle top of extractor EC-1 and remove the filter
iii. Detach TG-2 (causes obstruction while loading meal)
iv. Load EC-1 with the fresh meal (in a pouch/bag)
v. Reinstall filter, TG-2 and reattach the extractor top
vi. Reattach line P-18
vii. Check whether all the joints are screwed in tight.
viii. Proceed to next step

4. EXTRACTION PROCESS

Objective: To extract oil from the fresh extraction meal.

i. Open valves FV-12, RV-44, all others closed
ii. Adjust RV-21 for desired flow of miscella
iii. Turn on VP-1
iv. Make sure to maintain a visible liquid level in SC-1
v. After running the process for desired time, turn off VP-1
vi. Close valve FV-12
vii. Open valve FV-11 and start VP-1 (to drian EC-1 of miscella and return solvent back to SC-1)
viii. Once liquid is drained, close FV-11

ix. Proceed to next step

5. MEAL RECOVERY

Objective: To remove extracted meal from the system (from EC-1).

i. Open valves RV-21, FV-21, FV-42 (for PG-5), FV-44, all others closed
ii. Monitor PG-5 for steady vacuum
iii. Turn off VP-1 and close FV-44
iv. Open valve FV-41 to break vacuum on the system and drain liquid from FC-2
v. Open valve FV-31 and drain liquid from FC-1
vi. Dismantle line P-18 and top of EC-1, to remove the extracted meal.
vii. Reseal EC-1 and reattach P-18

APPENDIX – IV
ChemCAD – Excel Interface

The initially formulated models of the process could be use to represent the process, but it was very cumbersome to use as the user had to change the process dynamic run time at the end of each cycle. Hence, to sort this issue, equipment modules which can govern the flow through the system depending on the cycle times are necessary. Valves are the most commonly used for such purposes in a process. But, the valve module available on ChemCAD gives a constant throughput during the entire span of the process, which is undesirable. So, it is necessary to have user defined equipment modules, which can switch flows in accordance with the required cycle time.

The utility of ChemCAD-Excel interface has been used to design such valve modules. The actual equipment module is programmed as Excel Macros build on the Visual Basic Applications programming language. ChemCAD connects to the excel user defined modules through a set of COM interfaces, which are built into two types of libraries available on ChemCAD viz. cc5.tlb and ccx2xls.tlb. Various ChemCAD functions and data from the model are made available to excel through these interfaces. The user can transfer data such as unit operation parameters, stream flow information, thermodynamic parameters, etc. to and from ChemCAD and excel. The user can also perform operations like flash, retrieve the entire flowsheet to excel in form of unit IDs, perform engineering unit conversions, etc.

Four unit operation modules have been programmed in VBA to be used in this process. Each of these functions as valves and is placed at different locations on the flowsheet. As the modules are programmed to function in a similar manner, the codes are also very similar. The four user modules created for this model are: ext34.xls ext310.xls, ext312.xls and ext317.xls. The module ext312 and ext317 operate on the solvent feed and soy bean feed respectively, while ext310 and ext34 are placed downstream on the solvent storage and meal storage respectively and are used to control the flow of material into the extractor module where are actual process of extraction is simulated.

The module ext312 functions as the heart of the process by manipulating the other units as and when required. Thus, most of the utilities of the ChemCAD-VBA interface are accessed through this module. Since this module manipulates other user defined units in the process, it is very important for this module to keep track of the process time and cycle time for each step. This is done using the following code that is shown below:

```
tym = CC5.getdynamictimeinminute
```

Where, tym keeps track of the process run time

Other user variables that are used in this module are:

tm	keeps track of the cycle time
run	keeps track of number of runs processed
i	counter variable for number of components involved
stp	Dynamic time step used for solving the process
CC5	ChemCAD model as an object
chk1	solvent flow rate
chk2	meal input rate

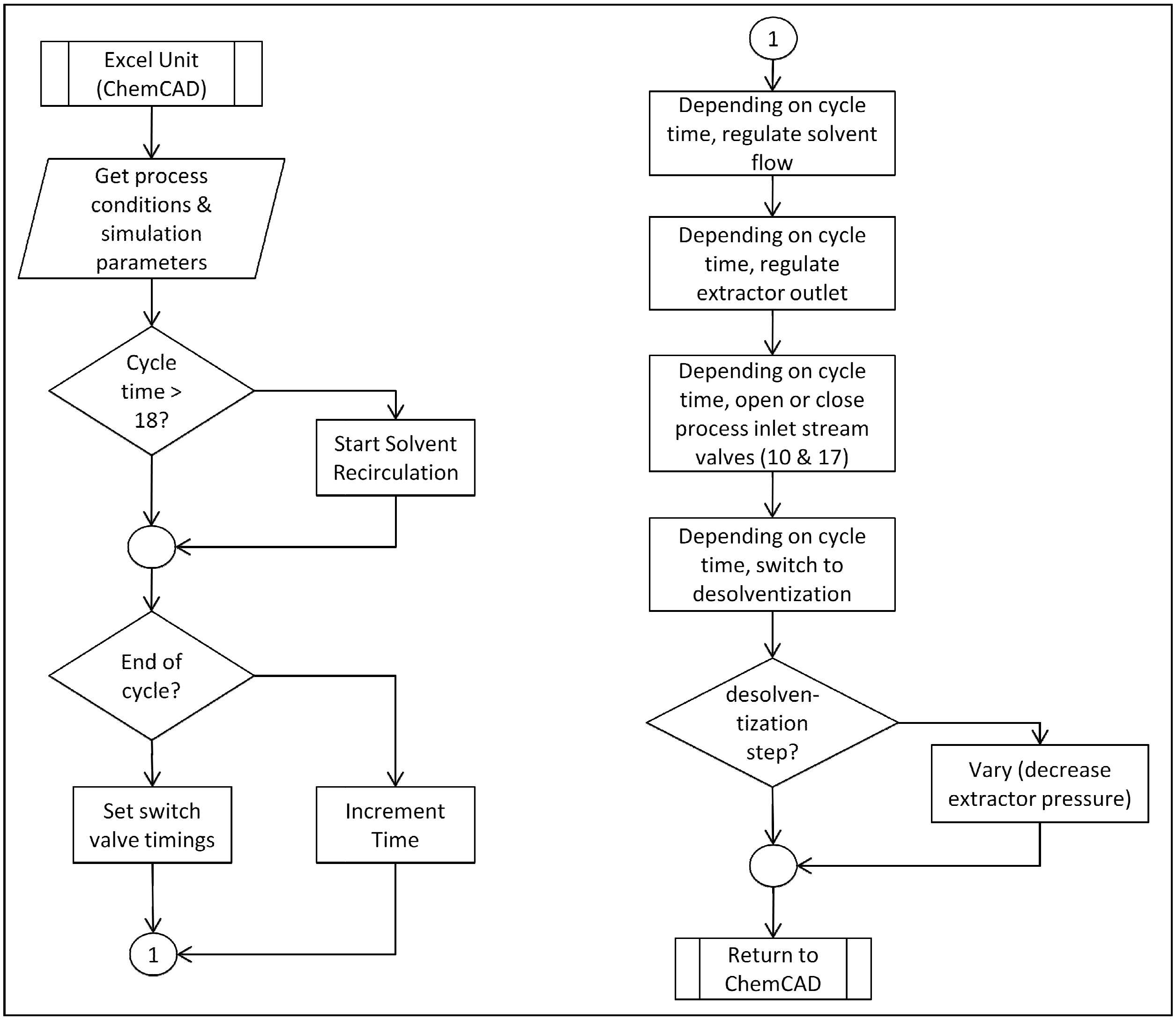
Apart from these user defined variables, other predefined variables are also made available by the COM interfaces that are made available in the ChemCAD libraries. These variables usually include, ‘user unit operation as an object’, ‘process streams involved in the user unit operation as objects’, ‘components array’, ‘flash mode’ etc. The predefined objects are extremely useful for accessing the ChemCAD based functions that are required to perform calculations in the unit. The important coding syntax involved for the user defined unit module ext312.xls is given below:

```
 Set CC5 = CreateObject("CHEMCAD.VBServer")        ‘access ChemCAD model
tym = CC5.getdynamictimeinminute                   ‘get process run time
stp = CC5.getdynamictimestepinminute               ‘get process step
```

```
check = uopInfo.GetUnitOpPar(chk1, 29, 1)          ‘get unit operation parameter
check = uopInfo.PutUnitOpPar(9, 21, 0)             ‘put specific value for unit
                                                    operation parameter
```

Apart from the interfaces, VBA file handling techniques have also been used to store specific data while executing the process. The program logic is explained along with each step of coding available on VBA macro ‘Sub ExcelUop’, which can be found in the file ‘ext312.xls’.

The other important unit module in this process is ‘ext34.xls’ which operates on the soy meal output from ‘unit 6’, the soy meal storage and extraction unit. This module, has a single inlet stream coming from ‘unit 6’, but unlike the other user defined modules, it contains two outlets, necessary for switching from extraction step to desolventizing step. This switch is performed with the help of the cycle time that is calculated by unit ‘ext312.xls’ and stored into a text file (tfile.txt) thus making these variables available to use in other unit operations.



Flowchart for the accompanying program

User Defined Codes:

```
    Dim tm As Integer
    Dim run As Integer
    Dim i As Integer
    Dim tym As Integer
    Dim stp As Integer
    Dim CC5 As Object
    Dim chk As Single
    Dim chk1 As Single
    Dim chk2 As Single
    Dim chk3 As Single
    Dim chk4 As Single
    Dim op As Integer

    ' PART 3, user calculations (author your codes here)
    run = 1                                               'Counter to determine number of runs
    Set CC5 = CreateObject("CHEMCAD.VBServer")        'Connect to ChemCAD
    tym = CC5.getdynamictimeinminute                      'Get current process run time
    stp = CC5.getdynamictimestepinminute                  'Get current step increase in time
    check = uopInfo.GetUnitOpPar(chk, 62, 1)          'get parameter 62 (mass hold uo) of unit 1 at
                                                      'start of each cycle

'Perform the valve type operations to regulate process flow
    If tym = 0 Then
        'These set of operations are performed at the start of the process
        check = uopInfo.GetUnitOpPar(chk1, 29, 1)     'takes in the values of process variables
        check = uopInfo.GetUnitOpPar(chk2, 29, 6)     ‘set by the user at the start of the run
        check = uopInfo.PutUnitOpPar(9, 21, 0)
```

```
    check = uopInfo.PutUnitOpPar(6, 21, 0)
    check = uopInfo.GetUnitOpPar(chk4, 6, 6)
    'If time (tym) is ZERO set the run number to ZERO
    run = 0
    op = 0
    Open ActiveWorkbook.Path & "\tfile1.txt" For Output As #2
      Print #2, chk1, chk2, chk3, chk4              'stores data into an external text file
    Close #2
  Else
    'These set of operations are performed anytime after the start ie tym > 0
    check = uopInfo.GetUnitOpPar(chk1, 6, 16)     'takes in the values of process variables
    check = uopInfo.PutUnitOpPar(1, 6, chk1)      'resulting from the previous step
    check = uopInfo.GetUnitOpPar(chk1, 6, 6)      'increment in time
   'Depending on the simulation time, regulates the solvent inlet flow (to the solvent
   'accumulator). This is the initial step carried out during start up of the process
    If tm < 18 Then
      check = uopInfo.PutUnitOpPar(6, 6, chk1)
    Else
    'If time > 18 close input to solvent accumulator and start solvent recirculation
      check = uopInfo.GetUnitOpPar(chk1, 6, 13)
      check = uopInfo.PutUnitOpPar(6, 6, chk1)
    End If
    Open ActiveWorkbook.Path & "\tfile1.txt" For Input As #1
      Input #1, chk1, chk2, chk3, chk4         'get previously stored process data from text file
    Close #1
    Open ActiveWorkbook.Path & "\tfile.txt" For Input As #4
      Input #4, tm, run, op                    'get previously stored cycle time and run
    Close #4
  End If
```

```
'Thsi set of code determines the cycle time and run number
If tm >= 120 Or tym = 0 Then
    tm = 0
    run = run + 1
    op = op + 1
    'This set of code is used to switch between the extraction cycle and raffinate removal
    check = uopInfo.PutUnitOpPar(6, 6, chk4)
    check = uopInfo.PutUnitOpPar(4, 2, 120 * (op - 1) + 2)
    check = uopInfo.PutUnitOpPar(4, 3, 108 + 120 * (op - 1))
    check = uopInfo.PutUnitOpPar(4, 5, 112 + 120 * (op - 1))
    check = uopInfo.PutUnitOpPar(4, 6, 120 * op)
Else
    tm = tm + stp
End If

If tm >= 98 And chk < 10 Then
'used to start feed to solvent storage, if mass of solvent in the storage falls below 10 lbs
    run = 1000
End If

Open ActiveWorkbook.Path & "\tfile.txt" For Output As #3
    Print #3, tm, run, op                    'store updated values of cycle time and run
Close #3
'Sets the pressure, temperature and flash mode for outlet stream (For the excel unit operation)
outletTempR(1) = inletTempR(1)
outletPresPsia(1) = inletPresPsia(1)
outletFlashMode(1) = 1
```

```
'These set of codes are used to regulate the process steps occurring after the solvent
draining
'from extractor. Performs pressure reduction in extractor and switches between steps.
If tm >= 96 And tm <= 110 Then
    check = uopInfo.PutUnitOpPar(6, 29, 0)
    check = uopInfo.PutUnitOpPar(6, 30, 0)
ElseIf tm > 110 Then
    check = uopInfo.PutUnitOpPar(6, 29, chk2)
    check = uopInfo.PutUnitOpPar(6, 30, chk3)
End If

If tm > 78 or tm < 20 Then
    check = uopInfo.PutUnitOpPar(1, 29, 0)              'Stops solvent input to extractor
ElseIf tm < 30 Then
    check = uopInfo.PutUnitOpPar(1, 29, 2 * chk1)     'Extractor fill up step
Else
    check = uopInfo.PutUnitOpPar(1, 29, chk1)          'Normal extraction cycle
End If

'Extractor unit regulation for pre extraction and extraction stages
If tm < 18 Then
    check = uopInfo.PutUnitOpPar(6, 29, 0)
    check = uopInfo.PutUnitOpPar(6, 30, 0)
ElseIf tm > 28 Then
    check = uopInfo.PutUnitOpPar(6, 29, chk2)
    check = uopInfo.PutUnitOpPar(6, 30, chk3)
End If

'Computes component flow rate for outlet stream
'Controls flow through user added excel unit
If (tm < 20 And run = 1) Then
```

```
    outletCompRatesLbmol_Hr(1, 1) = inletCompRatesLbmol_Hr(1, 1)
    outletCompRatesLbmol_Hr(1, 2) = inletCompRatesLbmol_Hr(1, 2)
    outletCompRatesLbmol_Hr(1, 3) = inletCompRatesLbmol_Hr(1, 3)
    outletCompRatesLbmol_Hr(1, 4) = inletCompRatesLbmol_Hr(1, 4)
    outletCompRatesLbmol_Hr(1, 5) = inletCompRatesLbmol_Hr(1, 5)
    outletCompRatesLbmol_Hr(1, 6) = inletCompRatesLbmol_Hr(1, 6)
    outletCompRatesLbmol_Hr(1, 7) = inletCompRatesLbmol_Hr(1, 7)
    outletCompRatesLbmol_Hr(1, 8) = inletCompRatesLbmol_Hr(1, 8)
    outletCompRatesLbmol_Hr(1, 9) = inletCompRatesLbmol_Hr(1, 9)
ElseIf (tm < 10 And run = 1001 And chk < 66.67) Then
    outletCompRatesLbmol_Hr(1, 1) = inletCompRatesLbmol_Hr(1, 1)
    outletCompRatesLbmol_Hr(1, 2) = inletCompRatesLbmol_Hr(1, 2)
    outletCompRatesLbmol_Hr(1, 3) = inletCompRatesLbmol_Hr(1, 3)
    outletCompRatesLbmol_Hr(1, 4) = inletCompRatesLbmol_Hr(1, 4)
    outletCompRatesLbmol_Hr(1, 5) = inletCompRatesLbmol_Hr(1, 5)
    outletCompRatesLbmol_Hr(1, 6) = inletCompRatesLbmol_Hr(1, 6)
    outletCompRatesLbmol_Hr(1, 7) = inletCompRatesLbmol_Hr(1, 7)
    outletCompRatesLbmol_Hr(1, 8) = inletCompRatesLbmol_Hr(1, 8)
    outletCompRatesLbmol_Hr(1, 9) = inletCompRatesLbmol_Hr(1, 9)
Else
    outletCompRatesLbmol_Hr(1, 1) = 0
    outletCompRatesLbmol_Hr(1, 2) = 0
    outletCompRatesLbmol_Hr(1, 3) = 0
    outletCompRatesLbmol_Hr(1, 4) = 0
    outletCompRatesLbmol_Hr(1, 5) = 0
    outletCompRatesLbmol_Hr(1, 6) = 0
    outletCompRatesLbmol_Hr(1, 7) = 0
    outletCompRatesLbmol_Hr(1, 8) = 0
    outletCompRatesLbmol_Hr(1, 9) = 0
End If
```

```
'Valve 10
If tm > 78 Then
   check = uopInfo.PutUnitOpPar(26, 6, 1)
Else
   check = uopInfo.PutUnitOpPar(26, 6, 0)
End If

'Valve 17
If tm > 18 Then
   check = uopInfo.PutUnitOpPar(17, 6, 1)
Else
   check = uopInfo.PutUnitOpPar(17, 6, 0)
End If

If tm >= 96 And tm <= 102 Then
   check = uopInfo.PutUnitOpPar(6, 6, 100 * Exp(0.1155 * (102 - tm)))
ElseIf tm > 102 And tm <= 110 Then
   check = uopInfo.PutUnitOpPar(6, 6, 5 * Exp(0.3745 * (110 - tm)))
End If
```

APPENDIX – V
Experimental Data[25]

Date:	4/4/2008
Sample:	Kerry Soy
ID:	Ksoy-4-4-1

Fresh Meal Data:

Bag #	10
Bag Weight	0.0405
Filled Weight	2.913
Sample used	2.8725

Experimental Observations:

Time (mins)		Solvent Collector	Extractor	Flash 1	Flash 2	Comments
0	T (F)	45	64	77	53	No water to solvent collector, vacuum to 6.2", closed 10/20 valve, turned on hot water
	P (psi)	125	0	0	0	Opened liq. Valve - filled immediately
3		68	75	74	58	
		126	131	42	48	
10		93	70	67	57	Primary receiver has light yellow liquid at 8 mins
		171	176	46	53	
20		106	68	54	54	
		202	207	47	54	
30		116	67	38	51	Solvent collector cold, hot water turned on
		230	235	48	56	
40		124	66	37	50	Hot water to solvent receiver turned on
		250	255	49	57	
46		98	66	37	50	hot on / cold off extractor
		156	161	49	56	
51		92	67	39	51	Solvent receiver too full and too cold - need to replace hot water pump
		168	173	51	59	
60		110	65	40	45	
		215	220	51	57	
66		120	64	42	47	Closed liquid valve from solvent receiver (end extraction), hot off/ cold on for solvent receiver
		210	156	53	62	
76		91	57	49	50	Extractor empty
		200	81	55	64	
84		93	58	55	50	opened 10/20 valve
		200	53	44	46	
116		55	74	72	53	Vacuum to 6.4"
		125	0	0	0	

APPENDIX – VI
Assumptions

1. Density of the oil components involved remain fairly constant and equal to 57.81 lb / ft^3 [3] throughout the process
2. Soy bean oil is a mixture of 0.1 % tetradecylic Acid, 10.3 % hexadecanoic Acid, 54.1 % linoleic Acid, 22.5 % oleic Acid, 4.7 % stearic Acid and 8.3 % linolenic Acid [23]
3. Soybean feed composed of 70 % solid matter, 10 % water, 20 % soy oil [3]
4. Complete mixing in the extractor
5. Constant operating conditions for all equipments during the extraction stage
6. Density of solvent propane remains constant (31 lb / ft^3[17]) during extraction
7. No significant pressure drop across heat exchanger
8. No material or energy losses throughout the process
9. No or negligible pressure drops in pipes
10. Extraction Ratio - Solvent to Meal Ratio = 2 (vol. / vol.)

APPENDIX – VII
Cost Estimation & Sizing

PUMPS	
Name	**Solvent Feed Pump**
Equip. No.	16
Pressure increase psi	20.0000
Efficiency	0.6000
Calculated power hp	0.0323
Calculated Pout psia	150.0000
Head ft	92.3409
Vol. flow rate ft3/hr	13.3061
Mass flow rate lb/h	415.0000
Cost estimation flag	1
Motor type	2
Motor RPM	2
Install factor	1.6500
Basic pump cost $	5961
Basic motor cost $	642
Total purchase cost $	6603
Total installed cost	10895 $

Name	**Solvent Recirculation Pump**
Equip. No.	13
Pressure increase psi	50.0000
Efficiency	0.6000
Calculated power hp	0.0796
Calculated Pout psia	200.0000
Head ft	234.9544
Vol. flow rate ft3/hr	13.1183
Mass flow rate lb/h	402.0000
Cost estimation flag	1
Motor type	2
Motor RPM	2
Install factor	1.6500
Basic pump cost $	5226
Basic motor cost $	481
Total purchase cost $	5707
Total installed cost	9416 $

HEAT EXCHANGERS

Name	**Miscella Cooler**	**Name**	**Solvent Flash cooler**
Equip. No.	19	Equip. No.	14
1st Stream T Out F	80.0000	1st Stream T Out F	40.0000
U Btu/hr-ft2-F	80.0000	2nd Stream T Out F	60.0000
Calc Ht Duty MMBtu/h	0.0318	U Btu/hr-ft2-F	70.0000
LMTD (End points) F	97.6823	Calc Ht Duty MMBtu/h	0.0040
LMTD Corr Factor	1.0000	LMTD (End points) F	10.0000
Utility Option:	1	LMTD Corr Factor	1.0000
Calc U Btu/hr-ft2-F	80.0000	Utility Option:	1
Calc Area ft2	4.0633	Calc U Btu/hr-ft2-F	70.0000
1st Stream Pout psia	10.0000	Calc Area ft2	5.7856
2nd Stream Pout psia	25.0000	1st Stream Pout psia	10.0000
Cost estimation	1	2nd Stream Pout psia	15.0000
Delta T2 (2nd Stream)	-50.0000 (F)	Cost estimation	1
Shell and tube	1	Install factor	1.6000
Install factor	1.6000	Material factor	1.0000
Material factor	1.1869	Pressure factor	0.8645
Pressure factor	0.8469	Type factor	0.3842
Type factor	0.3721	Basic cost $	4861
Basic cost $	5025	Total purchase cost $	2758
Total purchase cost $	3211	Total installed cost	4413 $
Total installed cost	5137 $	Design pressure psia	100.0000
Design pressure psia	100.0000		

Name	**Solvent Condenser**
Equip. No.	20
1st Stream T Out F	80.0000
U Btu/hr-ft2-F	90.0000
Calc Ht Duty MMBtu/h	0.0411
LMTD (End points) F	82.7327
LMTD Corr Factor	1.0000
Utility Option:	1
Calc U Btu/hr-ft2-F	90.0000
Calc Area ft2	5.5241
1st Stream Pout psia	150.0000
2nd Stream Pout psia	25.0000
Cost estimation	1
Delta T2 (2nd Stream)	60.0000 F
Install factor	1.6000
Material factor	1.0000
Pressure factor	0.8622
Type factor	0.3826
Basic cost $	4878
Total purchase cost $	2749
Total installed cost	4398 $
Design pressure psia	180.0000

COMPRESSORS

Name	**Solvent Compressor**
Equip. No.	5
Pressure out psia	150.0000
Type of Compressor	1
Efficiency	0.8000
Actual power hp	6.0004
Cp/Cv	1.1437
Theoretical power hp	4.8003
Ideal Cp/Cv	1.1354
Calc Pout psia	150.0000
Motor type	2
Motor RPM	2
Install factor	1.2000
Basic compressor $	39319
Basic motor cost $	1324
Basic driver cost $	473
Total purchase cost $	41117
Total installed cost	49340 $
Cost estimation flag	1
Calc. mass flowrate	176 lb/h

FLASH UNITS

Name **Oil-Solvent Separator**

Unit # 7

Loadings and Properties

	Vapor	Light	Heavy
Flowrate	178.4893 lb/h	9.5058 lb/h	2.5140 lb/h
Flowrate	2325.2727 ft3/hr	0.1711 ft3/hr	0.0404 ft3/hr
Density	0.0768 lb/ft3	55.5729 lb/ft3	62.2101 lb/ft3

K constant	0.2574 ft/sec
Min disengaging height	2.0000 ft
Min inlet nozzle to HLL	3.0000 ft
Mist eliminator	0.3000 ft
Design pressure	30.0000 psia
Allowable stress psia	15015.0000
Shell joint efficiency	1.0000
Head joint efficiency	1.0000
Head type	Ellipsoidal
Corrosion allowance	0.0104 ft
Vessel density	489.0240 lb/ft3
Weight percent allowance	20.0000
Surge time	1.0000 min.
Holdup time	5.0000 min.
Surge height	2.0000 ft
Light outlet to baffle	0.7500 ft
Inside diameter ID	2.5000 ft
V_max	6.9217 ft/sec
Length	11.5813 ft
Length / Diameter ratio	4.6325
Shell thickness	0.0208 ft
Head thickness	0.0208 ft
Inlet to mist eliminator	2.0313 ft
Liq to inlet	5.0000 ft
Baffle to liq	0.5000 ft
Heavy liq to light outlet	1.0000 ft
Heavy liq height	1.0000 ft
Shell weight	934.4168 lb
Head weight	194.3632 lb
Total weight (empty)	1128.7800 lb
Total vessel volume	60.9402 ft3
Total weight (full)	4919.8767 lb
Total weight (full) w/allow.	5145.6328 lb

Material = Stainless steel 316
Vertical vessel
Fm = 2.1
Cb = $ 11272.7
Ca = $ 2736
Base cost index = 347.5
Current cost index = 623.3
Cost (purchase) = $ 47368.5
Cost (installed) = $ 94737

Name **Oil Decanter**

Unit # 22

Loadings and Properties

	Vapor	Liquid
Flowrate	0.0038 lb/h	2.5140 lb/h
Flowrate	0.0644 ft3/hr	0.0403 ft3/hr
Density	0.0585 lb/ft3	62.3730 lb/ft3

K constant	0.0237 ft/sec
Min disengaging height	0.5000 ft
Min liq to inlet height	1.0000 ft
Mist eliminator	0.0000 ft
Design pressure	35.0000 psia
Allowable stress	15015.0000 psia
Shell joint efficiency	1.0000
Head joint efficiency	1.0000
Head type	Ellipsoidal
Corrosion allowance	0.0104 ft
Vessel density	489.0240 lb/ft3
Weight percent allowance	20.0000
Inside diameter ID	0.5000 ft
V_max	0.7748 ft/sec
Surge time	5.0000 min.
Retention time	1.0000 min.
High liquid level HLL	0.0205 ft
Normal liquid level NLL	0.0034 ft
Length	1.5205 ft
Length / Diameter ratio	3.0411
Shell thickness	0.0208 ft
Head thickness	0.0208 ft
Shell weight	25.3473 lb
Head weight	16.1637 lb
Total weight (empty)	41.5110 lb
Total vessel volume	0.3313 ft3
Total weight (full)	62.1739 lb
Total weight (full) w/allow.	70.4761 lb

Material = Stainless steel 304
Vertical vessel
Fm = 1.7
Cb = $ 5760.88
Ca = $ 198.119
Base cost index = 347.5
Current cost index = 623.3
Cost (purchase) = $ 17921.7
Cost (installed) = $ 35843.3

Name	**Solvent-Water Separator**	
Unit # 3		
Loadings and Properties		
	Vapor	Liquid
Flowrate	176.6558 lb/h	1.8335 lb/h
Flowrate	2135.3274 ft3/hr	0.0294 ft3/hr
Density	0.0827 lb/ft3	62.3195 lb/ft3

K constant	0.2574 ft/sec
Min disengaging height	2.0000 ft
Min liq to inlet height	0.6000 ft
Mist eliminator	0.3000 ft
Design pressure	30.0000 psia
Allowable stress	15015.0000 psia
Shell joint efficiency	1.0000
Head joint efficiency	1.0000
Head type	Ellipsoidal
Corrosion allowance	0.0104 ft
Vessel density	489.0240 lb/ft3
Weight percent allowance	20.0000
Inside diameter ID	1.0000 ft
V_max	7.0606 ft/sec
Surge time	5.0000 min.
Retention time	1.0000 min.
High liquid level HLL	0.0037 ft
Normal liquid level NLL	0.0006 ft
Length	2.9037 ft
Length / Diameter ratio	2.9037
Shell thickness	0.0208 ft
Head thickness	0.0208 ft
Shell weight	94.8749 lb
Head weight	42.2586 lb
Total weight (empty)	137.1335 lb
Total vessel volume	2.5424 ft3
Total weight (full)	295.5744 lb
Total weight (full) w/allow.	323.0011 lb

Material = Carbon steel
Vertical vessel
Fm = 1
Cb = $ 6544.96
Ca = $ 522.582
Base cost index = 347.5
Current cost index = 623.3
Cost (purchase) = $ 12676.8
Cost (installed) = $ 25353.7

VITA

Patrachari Anirudh Ramanujan

Candidate for the Degree of

Master of Science

Thesis: PROCESS SIMULATION, MODELING & DESIGN FOR SOYBEAN OIL EXTRACTION USING LIQUID PROPANE

Major Field: Chemical Engineering

Biographical:

Education: Completed the requirements for the Master of Science in Chemical Engineering at Oklahoma State University, Stillwater, Oklahoma in December 2008.

Bachelor of Engineering in Chemical Engineering, Mumbai University, Mumbai, India, May 2006.

Experience: Employed as a Teaching Assistant by Oklahoma State University for ENGR 1412 – Computer Programming for Engineers (Fall ’07 to Present) and ENSC – 3233 - Fluid Mechanics (Summer ’08).

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Professional Memberships: Member of the Honor Society of Phi Kappa Phi – Oklahoma State University Chapter

Member of AIChE – Oklahoma state University Chapter

Name: Patrachari Anirudh Ramanujan Date of Degree: December, 2008

Institution: Oklahoma State University Location: Stillwater, Oklahoma

Title of Study: PROCESS SIMULATION, MODELING & DESIGN FOR SOYBEAN OIL EXTRACTION USING LIQUID PROPANE

Pages in Study: 141 Candidate for the Degree of Master of Science

Major Field: Chemical Engineering

Scope and Method of Study: This study investigates the use of liquid propane for soybean oil extraction and the use of commercial software for process modeling and simulation

Findings and Conclusions:

Soybean oil extraction using liquid propane is feasible and requires lower operating temperatures and pressures compared to processes using other solvents such as hexane or carbon dioxide. Process models for a 5 liter extraction unit have been developed and validated using experimental data. These models were then used to design, size, and cost a 130 liter extraction process. Modification and alternatives to improve process performance have also been suggested.

ADVISER'S APPROVAL: Dr. ARLAND H. JOHANNES