

EFFECT OF HEATING WITH AN ADDITIVE ON
PARTICLE SIZE AND ATTERBERG LIMITS
OF MONTMORILLIC CLAYS

By

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CHAPTER I

INTRODUCTION

Actual field trials have proven that extremely clayey soils are unsuccessful for stabilization work. In the field, trials have shown that clay soils are difficult if not impossible to mix with the stabilizing agent whether portland cement or asphaltic cutback. These soils are difficult to pulverize when dry and when water is added for mixing, the soil becomes too sticky for homogeneous final products; so it has become necessary in the design and construction of soil cement and soil asphalt to have a limiting criteria on the type of soil to be used. Various agencies use different criteria but all list a maximum plastic index, a maximum liquid limit, and a maximum percentage of grain size smaller than 0.005 mm, among other limiting factors.

Large areas of the United States are covered with extremely clayey soil. These same areas are usually devoid of mineral aggregate, making the highways built in these areas expensive since aggregate of some type must be transported long distances to the proposed highway site. But if the undesirable properties of the soil could be eliminated by heating the soil with an additive, the soil on location might be utilized. This

would lower the cost per mile of the highways, allowing construction of more miles of highways for the same cost.

Thus, this investigation was instigated to see if the Atterberg limits and particle size of clayey soil could be altered by heating the soil with an additive. It is the purpose of this paper to report the results and conclusions obtained from the investigation. Two different clays were used, one was bentonite which was almost 100 percent montmorillonite. The other was Dennis clay containing about 50 percent montmorillonite.

CHAPTER II

REVIEW OF THE LITERATURE

There are principally three factors that contribute to changing the undesirable properties of high-plastic clays. One method would be to lower the Zeta Potential on individual grains either by decreasing the charge or by decreasing the thickness of the double layer. If the crystal lattice water is removed, the lattices can realine and allow more complete crystals to be formed. The addition of a cementing agent to bind the grains together would be another method of effecting the desired results.

Atterberg (5)¹, in his original investigation on soil plasticity, was interested in finding to what extent the different minerals, from which soils are composed, affect plasticity. Consequently, he ground various minerals to give particles the size of clay and measured their plasticities. His studies showed that only those minerals that have a platy or sheetlike structure exhibit plasticity when ground. Quartz and feldspar, whose crystals are made up of linked tetrahedra, are non-plastic. On the other hand, kaolinite, talc, mica, and others whose crystal are built up in sheets are plastic. These differ-

¹The number in parentheses indicates the reference in the Bibliography.

ences are attributed to a greater surface area and increased contact in the case of plate-shaped particles. Although it is realized that few soils contain sufficient amounts of those primary minerals to effect plasticity very markedly; nevertheless, the fact that the secondary clay minerals have sheet-like structures similar to the aforementioned plastic minerals, helps to explain the plasticity of clays.

One of the characteristics of a colloidal suspension is that the particles carry an electric charge. This charge may be either positive or negative. Most clay particles possess a negative charge. They migrate to the positive pole if placed between electrodes of unlike signs. The greater the speed of migration, the higher the negative potential. According to the Helmholtz electric double layer Theory, this charge, the shape of the particle, and the dielectric constant of the medium express the Zeta Potential.

$$Z = \frac{4 \pi e d}{D}$$

Z = Zeta Potential

e = electric charge

d = thickness of double layer

D = Dielectric Constant

The higher the electric charge the more water a soil particle can hold by alining the Dipolar water to the charges; hence, the higher the liquid and plastic limits. Soils with low Zeta Potential are less plastic. Bayer (1) shows that the Zeta Potential may be lowered by the addition of an electrolite which contains an ion that is not

contained in the clay complex.

Na-clay +KCl gives \rightarrow K-clay+NaCl - which lowers the potential through exchange
 K-clay+KCl - which lowers the potential through repression

Jenny's (2) investigations have shown that if clay has highly hydrated cations, such as sodium, the Zeta Potential is high because of the thick double layer and that the use of a high volume exchange ion such as aluminum will cause the Zeta Potential to be decreased by the absorption of a more tightly bound cation. The magnitude of the Zeta Potential, being a function of the distance the ions in the outer layer are from the inner layer, is decreased because the smaller, less hydrated ions are closer to the inner layer. The quantity of change depends on both the exchange ion and the absorbed ion originally on the clay.

The concepts of Shaber and Russell (3) offer an explanation of the center of the attractive forces in soil colloids for water molecules. Even though the amount of adsorption of moisture is a function of the total surface, it is also dependent to a great extent on the nature of the clay mineral and the character of the forces associated with crystal lattices. They suggest also that there are two kinds of adsorbed water. First, there is the broken bond water which is adsorbed through the orientation of water molecules by the free bonds of the mineral constituents on the exposed edges of a broken crystal

lattice. This type of adsorption is considered relatively strong. Second, they define planer water as that adsorbed by the unsaturated valences or free electric fields on the surface of the sheets of the crystal lattice.

If the clay is raised to high temperature, the loss of the adsorbed water allows the broken crystal lattice to realine and combine to form larger and more chemically inert particles having lower Zeta Potential. For a given crystal lattice, the number of water molecules which can be packed into one cavity is dependent upon the number and size of the exchangeable ion in the cavity.

Experience in the chemistry of ferric hydroxide has shown that this colloid becomes almost completely irreversible upon dehydration. There is sufficient evidence to suggest that this irreversibility of colloidal iron hydroxide is the important factor in the production of stable aggregates in certain soils. Studies by Lutz (4) on the relation of free iron to aggregation have indicated a very close relationship between the amount of free iron and the quantity of water stable aggregate. He suggests that iron may serve a dual purpose in aggregation. That part which is in solution may act as a flocculating agent and the other part which is more gelatinous in nature may exert a cementation action. He also suggested that colloidal alumina might play a role similar to iron but the importance of metallic colloids in effecting the properties of soil has not been evaluated to date.

In reviewing the literature, it appears the best method of changing the particle size and Atterberg limits would be to effect an ionic exchange within the clay and then apply heat to dehydrate the clay. If the additive would, in addition to affecting the necessary ionic exchange, serve as a cementing agent the final end product should have a particle size increase due to cementation. Bentonite was chosen for the original material for it should be the most difficult of the clays to change. The additives chosen were calcium chloride, ferric chloride, and aluminum nitrate because all should effect the ionic changes wanted.

CHAPTER III

MATERIALS AND LABORATORY PROCEDURE

For the purpose of this investigation, it seemed logical to examine the effect of heat with an additive on montmorillic clay. It has a very high liquid limit and plastic index. Any small percentage of this clay in a soil has a very undesirable effect on the use of the soil for stabilization.

Atterberg limits and particle size tests were run on the material in the pure state without additives and results were compiled. This was to show the effect of the heat alone on the material. An ionic exchange was affected in the material and heat applied to the altered material. The tests were completed on the altered material to investigate the effect of heat coupled with the use of an additive. Three different additives were used to affect the ionic exchange with the bentonite.

After completing the tests on the bentonite, the most effective additive, aluminum nitrate, was used in testing a locally occurring clay, the Dennis clay loam.

Material

The bentonite used in the major portion of this investigation was a commercial bentonite obtained from the Barjod Drilling Mud Company located at Perry, Oklahoma.

The bentonite is used commercially in the drilling of oil wells. The bentonite is almost 100% montmorillonite, a highly plastic clay. The final clay tested was the Dennis clay located at 1-1/2 miles Northwest of Skedee, Oklahoma in Pawnee County. The sample was taken in the "B" horizon approximately 36 inches below ground level. The clay was approximately 13% sand, 42% silt, and 45% clay. Of the clay fraction approximately 50% was montmorillonite (6).

For the exchange with the sodium ion on the clay, it was decided that any element with a positive valence that is below sodium on chemistry elements table, would effect a certain amount of desired results. The amount of additive necessary is decreased by using a material with a high positive valence. The change in Atterberg limits is proportional to the strength of the new bond with the clay. It was decided that, among others, calcium, iron and alumina would be satisfactory for the tests. The chlorides of these elements furnish ions with ease, so the additives chosen were calcium chloride, ferric chloride and aluminum chloride. The aluminum chloride proved too dangerous to handle without experience so aluminum nitrate was substituted. All additives were of technical grade obtained from the Chemistry Department of Oklahoma State University.

Ionic Exchange With Clay

Before effecting the ionic exchange, it was necessary

to calculate the required amount of additive that was to be added. The exchange capacity of the sodium clay, approximately 120 milli-equivalent, was obtained from a chemistry table. Equivalent weight was computed by dividing the molecular weight by the equivalent replaceable hydrogen. This answer divided by one thousand gave the milli-equivalent exchange capacity of the additive. The product of the two milli-equivalent gave the necessary parts of additive per 100 parts of clay. The necessary amount of additive was computed for each 1000 grams of clay.

The additive was dissolved in water and mixed with clay. Water was added to the mixture until it became a thin slurry. It was continuously mixed in the Hobart C-100 mixer for five minutes until it was homogeneous. After mixing, the material was transferred to a large basin and extra water added to wash the sodium chloride from the mixture. Particles were allowed to settle and the water was siphoned off. The washing process was repeated three times. The mixture was dried in a constant temperature oven at 105 C.

Material was passed through the No. 100 sieve. Sample amounts of the material were heated to the required temperature in a Hoskins Electric Furnace, Type FD 2020. Control of the temperature was accomplished by inserting a Chromel-Alumel thermocouple in the material. The thermocouple measured the temperature with the aid of a Wheelco Instrument, Model No. 192. When the material reached the desired temperature, it was removed from the furnace and test-

ed according to the AASHO specification for particle size analysis and Atterberg limits.

Particle Size Analysis

The particle size of the soil was determined according to AASHO Designation: T88-54.

An appropriate sample of the material to be sized was weighed and placed in a 250-ml beaker, covered with 125-ml of a stock solution of the dispersing agent, sodium hexametaphosphate, and allowed to soak for a minimum of 12 hours. After soaking, the contents of the beaker were washed into a dispersion cup, distilled water added until the cup was more than half full, and the contents dispersed for a period of one minute in the mechanical stirring apparatus.

After dispersion, the mixture was transferred to the glass graduate and distilled water was added until the mixture attained a volume of 1000-ml. After shaking the mixture, a hydrometer was inserted and a reading taken at the end of two minutes. The hydrometer was read at the top of the meniscus formed by the suspension around its stem. Hydrometer with scale A was used and read to the nearest 0.5 gm. per liter. Subsequent readings were taken at intervals of 5, 15, 30, 60, 250, and 1440 minutes after the beginning of sedimentation. Readings of the thermometer placed in the soil suspension were made immediately following each hydrometer reading and recorded.

After each reading, the hydrometer was removed from the

soil suspension and placed with a spinning motion in a graduate of clean water. About 25 or 30 seconds before the time for a reading, it was taken from the graduate of clear water, slowly immersed in the soil suspension and allowed to come to rest before the reading was made.

Hydrometer readings made at temperatures other than 67 F. were corrected by adding appropriate temperature correction factors. Temperature correction curves of this type were determined experimentally for each hydrometer used.

The percentage of the dispersed soil in suspension, represented by different corrected hydrometer reading, depended upon both the amount and the specific gravity of the soil dispersed. The percentage of dispersed soil remaining in suspension was calculated as follows:

$$P = \frac{Ra}{W} \times 100$$

where P = percentage of originally dispersed soil remaining in suspension.

R = corrected hydrometer reading.

W = weight in grams of soil originally dispersed minus the hygroscopic moisture.

a = constant depending on the density of the suspension. For hydrometer A, the value of a, for a specific gravity, G, and a water density at 67 F. (19.4C) of 0.9984 was obtained from the following formula:

$$a = \frac{2.6500 - 0.9984}{2.6500} \times \frac{G}{G - 0.9984}$$

It was sufficiently accurate for ordinary tests to select the constant for the specific gravity nearest to that of the particular soil tested.

The maximum diameter, d , of the particles in suspension, corresponding to the percentage indicated by a given hydrometer reading, were calculated by the use of Stokes Law.

According to Stokes Law:

$$d = \sqrt{\frac{30 nL}{980 (G - G_1)T}}$$

where: d = maximum grain diameter in millimeters.

n = coefficient of viscosity of the suspending medium (water) in poises. Varies with the change in temperature of the suspending medium.

L = distance in cm. through which soil particles settle in a given period of time.

T = time in minutes, period of sedimentation

G = specific gravity of soil particles

G_1 = Specific gravity of the suspending medium (approximately 1.0 for water)

Liquid Limit T89-54

The liquid limit of a soil is the water content at which the soil passes from a plastic to a liquid state as determined in accordance with the following procedure.

The soil sample was placed in an evaporating dish and thoroughly mixed with about 20 cc of water by alternately and repeatedly stirring, kneading and chopping with

a spatula. Further additions of water were made in small increments with thorough mixing between each increment.

When sufficient water had been thoroughly mixed with the soil to form a uniform mass of stiff consistency, a small quantity was placed in the cup of the liquid limit device. This soil was spread and the bubbles worked out with the spatula. The excess soil was removed. Immediately thereafter, the soil was cut with the grooving tool along the center line of the cup.

The cup containing the sample was lifted and dropped at the rate of two revolutions per second until the sample came into contact at the bottom of the groove along a distance of about one half of an inch. The number of blows required to close the groove was recorded.

A soil sample was taken across the groove. The sample was weighed for a wet weight and placed in an oven and dried at 110 C. After the material was dried a day, weight was obtained.

This procedure was repeated until two samples were taken at less than 25 blows and two samples at more than 25 blows. A "flow curve" was plotted on a semilogarithmic graph paper. Moisture content, expressed as a percentage of dry soil, was plotted on the arithmetic abscissa. Number of blows was plotted on the logarithmic ordinate. The flow line was drawn as a straight line through the points as nearly as possible. The moisture content corresponding to the intersection of the 25 blow ordinate is taken

as the liquid limit of the soil.

Plastic Limit T90-54

Immediately after completing the liquid limit test, the plastic limit test was run. A small portion of the plastic soil, enough to form a thread about six inches long and one-eighth of an inch in diameter, was used. The soil was formed into a uniform mass and rolled between the fingers and a piece of absorbent paper until it reached a thread about one-eighth of an inch in diameter. The material was reformed into a mass and the process repeated until the one-eighth of an inch thread crumbled under the pressure of rolling.

The crumbled soil was gathered together and placed in a container and weighed. After drying, the loss in weight was recorded as the weight of water. The plastic limit was expressed as the moisture content in percentage of dry weight soil.

CHAPTER IV

RESULTS

The results of the liquid limit and plastic limit tests on the clays have been listed in Table I.

Tables II, III, IV, V, and VI contain the particle size distributions of the clays investigated.

TABLE I

THE EFFECT OF TEMPERATURE ON LIQUID LIMIT
AND PLASTIC LIMIT OF MONTMORILLIC CLAYS

Temp.	Pure Bentonite		Calcium Bentonite		Ferric Bentonite		Aluminum Bentonite		Aluminum Dennis	
	LL	PL	LL	PL	LL	PL	LL	PL	LL	PL
100C	545	42	170	43	170	45	102	42	36	23
250C	549	42	171	42	172	44	100	40	36	23
350C	548	41	169	43	121	43	71	42	29	24
450C	545	42	124	41	61	44	NP	NP	NP	NP
550C	109	42	80	42	NP	NP	NP	NP	NP	NP
650C	56	41	NP	NP	NP	NP	NP	NP	NP	NP
750C	NP	NP	NP	NP	NP	NP	NP	NP	NP	NP

TABLE II

PARTICLE SIZE ANALYSIS OF PURE BENTONITE AFTER HEATING
(Percent by Weight Smaller)

Heating Temp.	Diameter of Particles (mm x 10 ⁻²)							
	6.0	4.3	2.6	1.5	1.1	0.73	0.37	0.15
100C	100	100	100	100	100	100	92	86
250C	100	100	100	100	100	100	92	86
350C	100	100	100	100	100	100	92	86
450C	100	100	100	100	100	100	92	86
550C	100	99	98	95	91	88	76	66
650C	81	76	67	53	47	42	33	26
750C	77	70	50	34	26	13	0	0

TABLE III

PARTICLE SIZE ANALYSIS OF CALCIUM BENTONITE AFTER HEATING
(Percent by Weight Smaller)

Heating Temp.	Diameter of Particles (mm x 10 ⁻²)							
	6.0	4.3	2.6	1.5	1.1	0.73	0.37	0.15
100C	100	100	100	100	100	95	92	88
250C	100	100	100	100	100	95	93	88
350C	100	100	100	100	96	92	90	80
450C	100	100	100	93	91	86	79	67
550C	96	93	88	81	73	70	54	23
650C	74	63	45	30	23	21	16	10
750C	68	53	32	20	16	13	8	0

TABLE IV

PARTICLE SIZE ANALYSIS OF FERRIC BENTONITE AFTER HEATING

(Percent by Weight Smaller)

Heating Temp.	Diameter of Particles (mm x 10 ⁻²)							
	6.0	4.3	2.6	1.5	1.1	0.73	0.37	0.15
100C	100	100	100	100	100	100	96	88
250C	100	100	100	100	100	100	97	87
350C	100	100	100	98	95	93	88	77
450C	96	94	92	90	84	80	69	58
550C	73	65	56	42	31	22	16	7
650C	65	56	45	32	23	17	7	2
750C	55	44	33	19	12	10	3	0

TABLE V

PARTICLE SIZE ANALYSIS OF ALUMINUM BENTONITE AFTER HEATING
(Percent by Weight Smaller)

Heating Temp.	Diameter of Particles (mm x 10 ⁻²)							
	6.0	4.3	2.6	1.5	1.1	0.73	0.37	0.15
100C	100	100	100	100	100	100	92	86
250C	100	100	100	100	97	95	88	78
350C	96	95	94	92	90	88	83	67
450C	93	89	84	73	70	67	52	40
550C	80	70	53	36	30	23	16	10
650C	70	63	46	30	23	16	10	3
750C	50	38	26	19	14	8	1	0

TABLE VI

PARTICLE SIZE ANALYSIS OF
ALUMINUM-DENNIS CLAY AFTER HEATING

(Percent by Weight Smaller)

Heating Temp.	Diameter of Particles (mm x 10 ⁻²)							
	6.0	4.3	2.6	1.5	1.1	0.73	0.37	0.15
100C	88	75	65	42	39	37	34	33
250C	88	75	65	42	39	37	34	33
350C	75	62	48	35	31	26	22	20
450C	67	54	38	27	22	16	13	10
550C	62	48	32	22	17	12	8	4
650C	55	40	23	13	11	7	3	0
750C	50	32	18	11	7	3	0	0

CHAPTER V

DISCUSSION OF RESULTS

Effect of Heating on Particle Size of Pure Bentonite

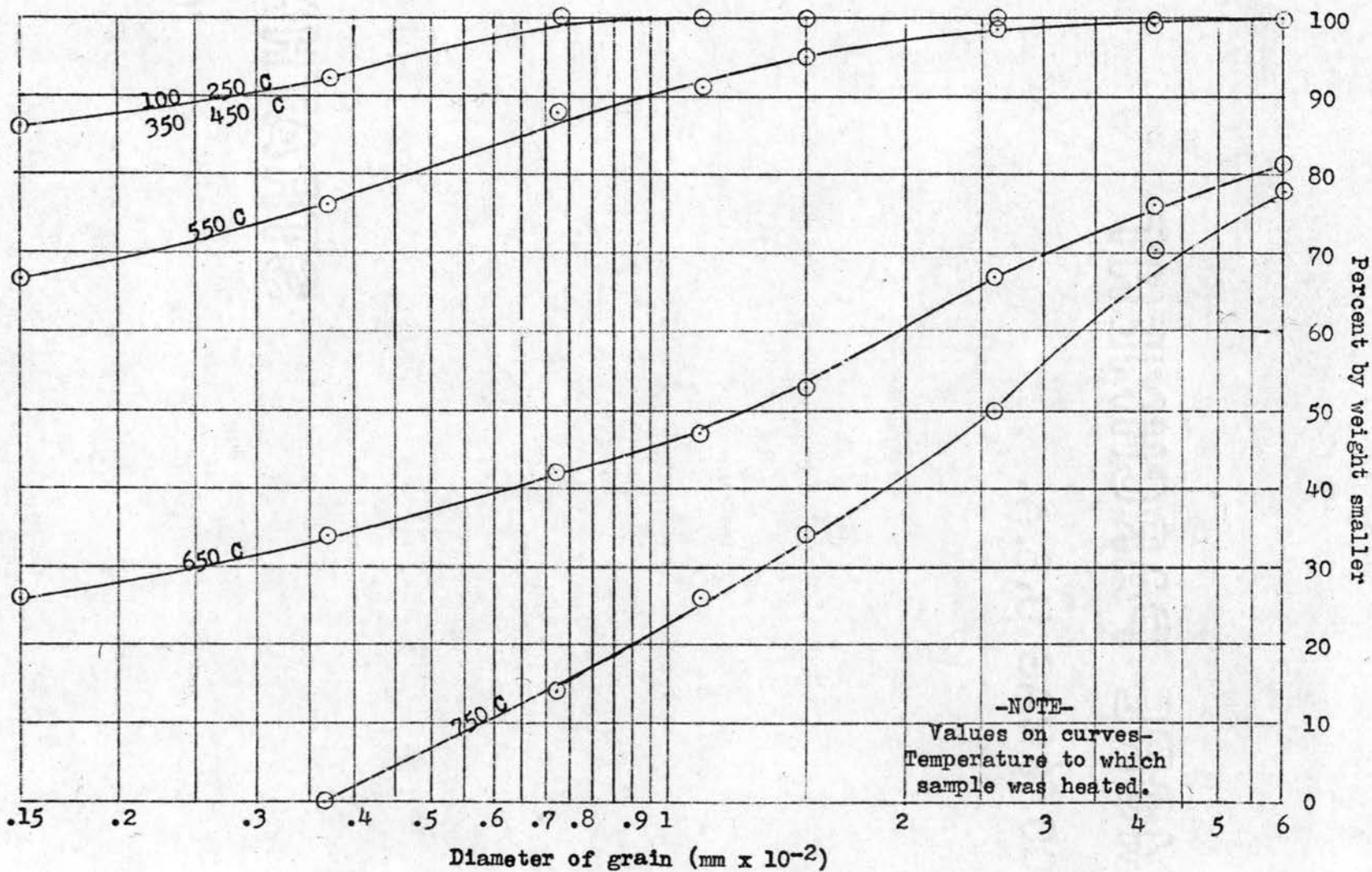
As evidenced by the results in Figure 1, the heat alone had very little effect on particle size until a temperature of 550 C was attained in the Bentonite. At 550 C, a small percentage had changed to a size larger than silt and approximately one quarter of the material was in the silt range.* Continued heating caused a change in size with an increase in both sand and silt size. At the 750 C temperature, the material had completely changed from clay with approximately 30 percent in the sand range. This change in size may be due to the realining of the crystals. Previous tests (1) have shown that there is a shattering of the crystal lattice in Bentonite above 550 C.

Effect of Heating on Particle Size of Bentonite

After Calcium Ionic Exchange

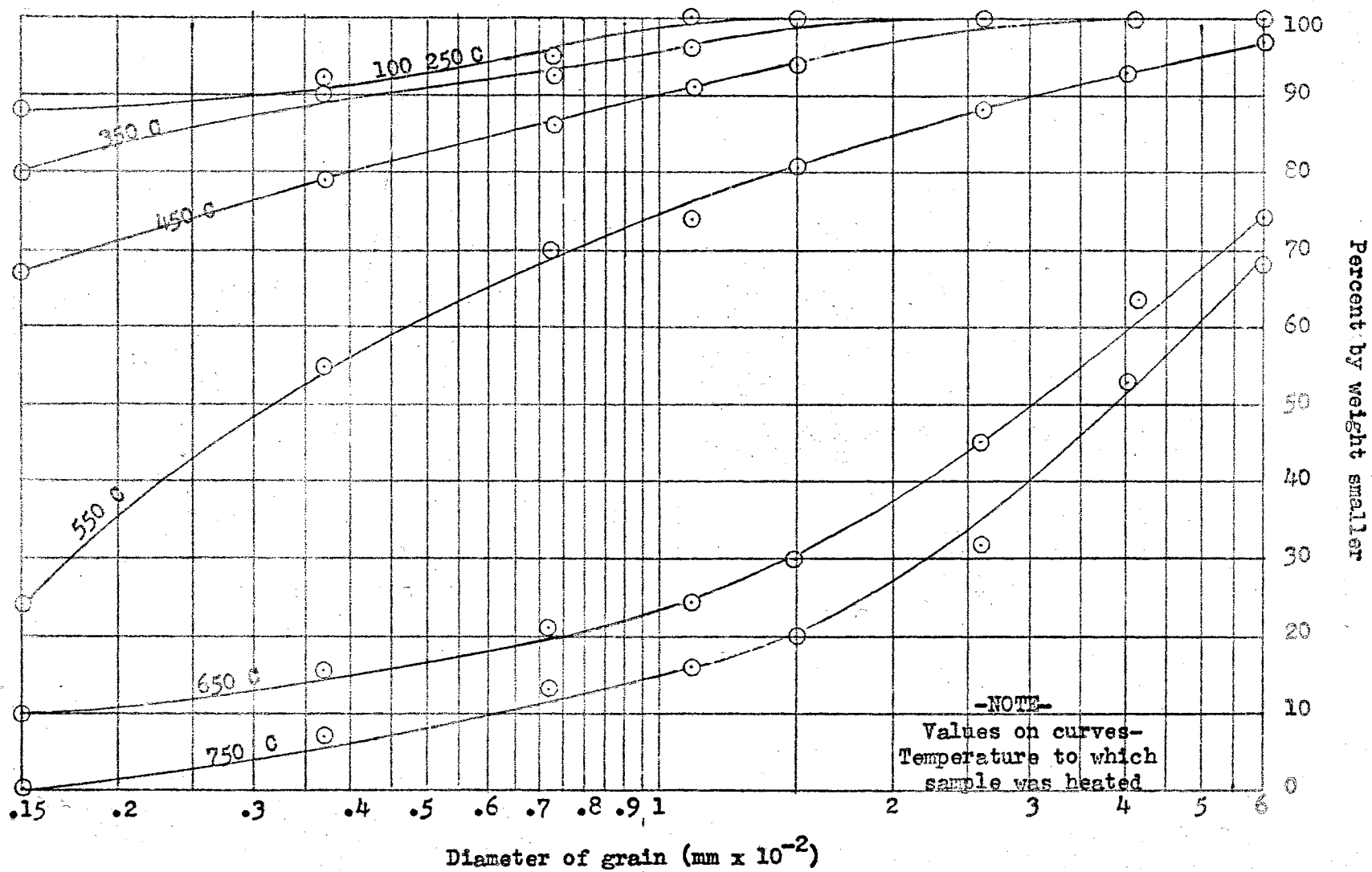
As evidenced by the result in Figure 2, there was no noticeable effect on the calcium bentonite by the 250 C heating. The 350 C heating increased the particle size.

*U.S. Bureau of soil classification lists clay size as smaller than 0.005mm, silt size as between 0.05-0.005mm, and sand as larger than 0.05mm.



PURE BENTONITE
CHANGE IN PARTICLE SIZE DUE TO TEMPERATURE

Figure 1



CALCIUM-BENTONITE
 CHANGE IN PARTICLE SIZE DUE TO TEMPERATURE
 Figure 2

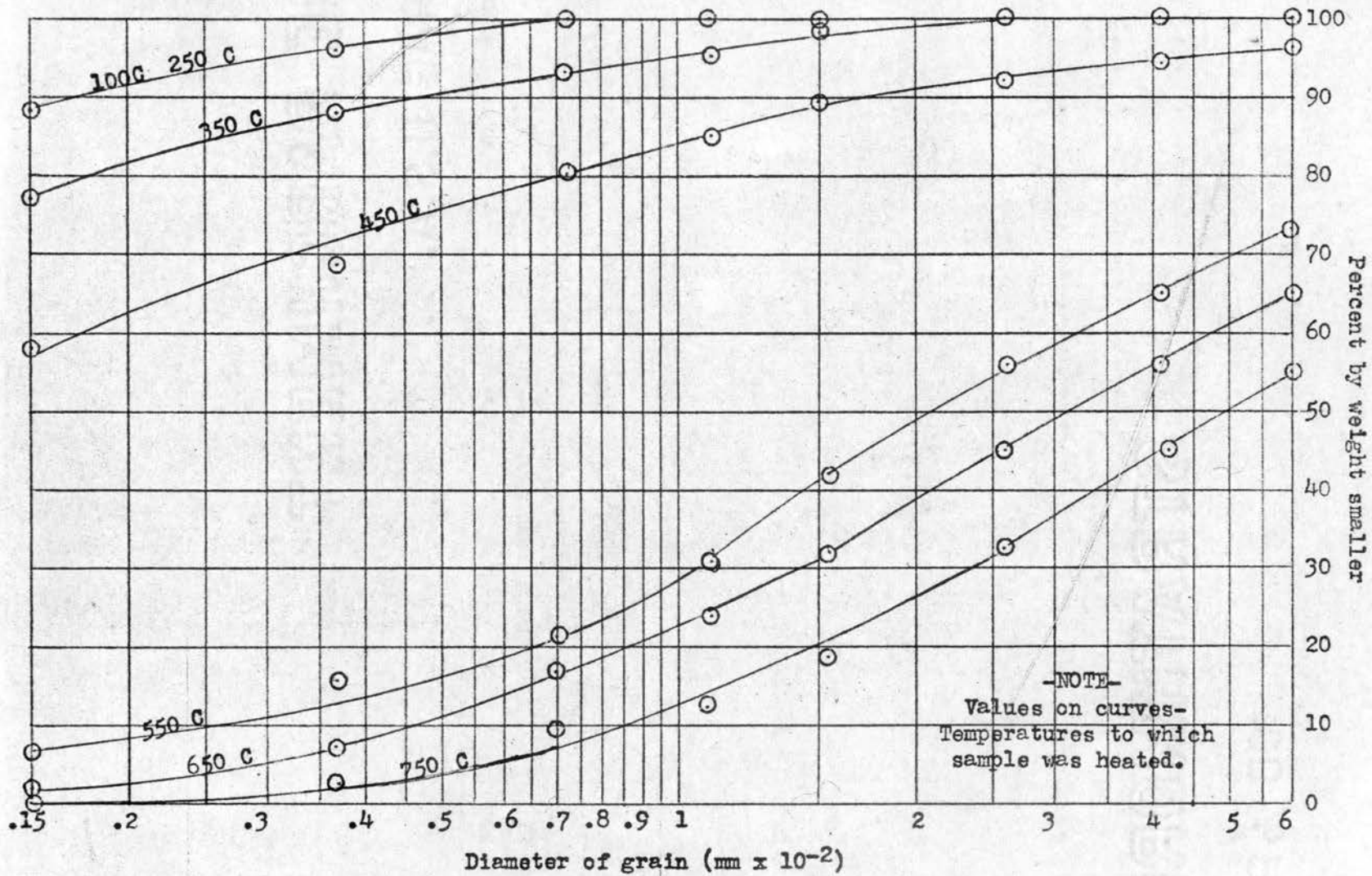
The rate of change between 250 C and 350 C continued through the 450 C and 550 C heating. There was a large change in particle size between 650 C and 550 C. The shape of the curve had changed from concave upward to concave downward, showing a large change in particle size. The 750 C curve shows a continued increase in particle size. At this temperature, there was only 8 percent of clay size particles remaining in the material.

Effect of Heating on Bentonite After Ferric Ionic Exchange

The results in Figure 3 show there was an increase in particle size between the 250 C and the 350 C heating. The same rate of change seemed to continue up through the 450 C and 550 C heatings. As evidenced by the Figure, after heating to 650 C, the material continued to change in particle size. At the 750 C temperature, the material was almost devoid of particles in the clay size range.

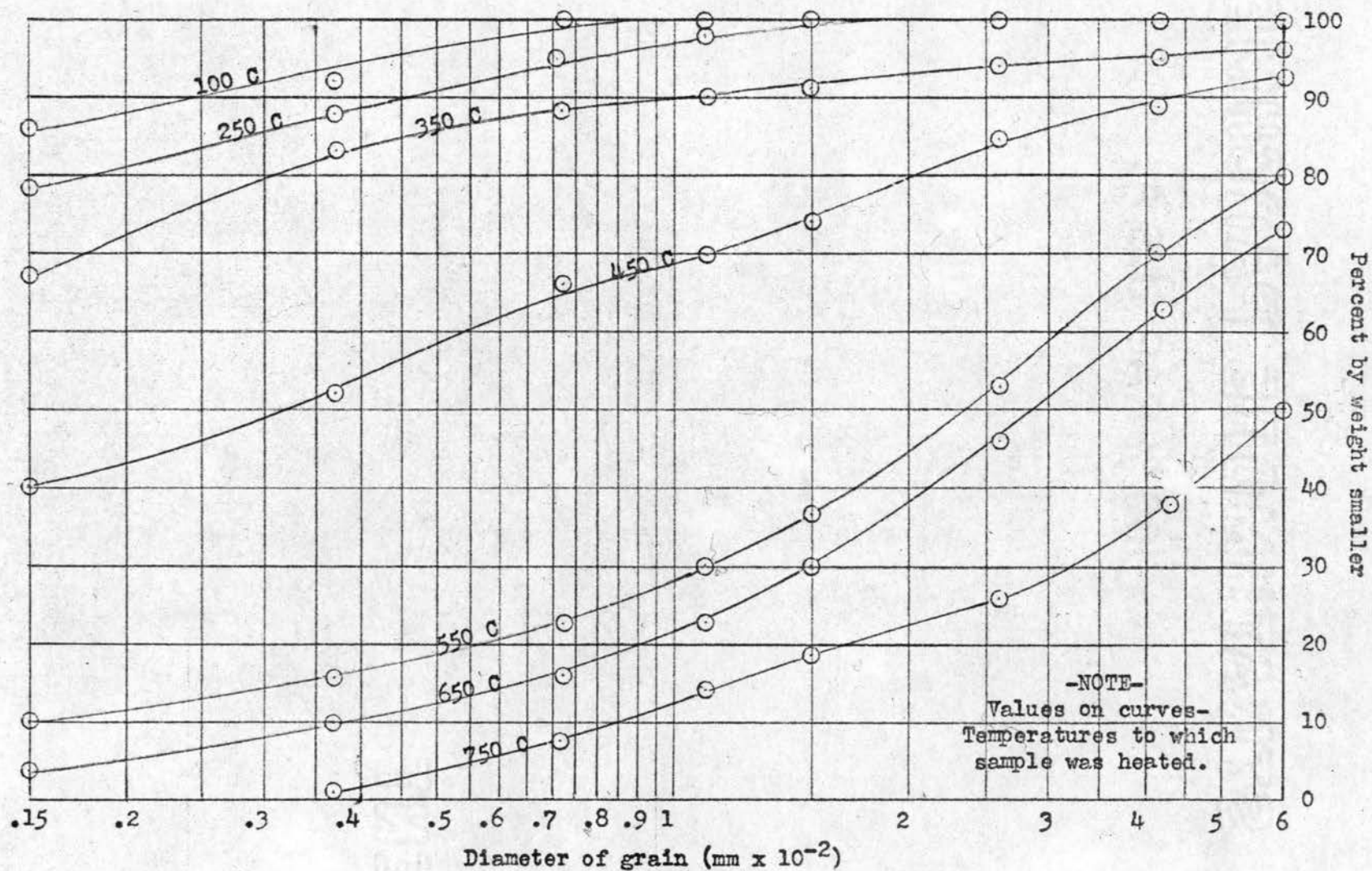
Effect of Heating on Particle Size of Bentonite After Ionic Exchange With Aluminum

The results in Figure 4 show a change in particle size of aluminum exchanged bentonite after heating to 250 C, which was the lowest temperature that a change was noted. The size of the particles continued to increase with an increase of temperature. Between the 350 C and 550 C, the particle size of the material exhibits a major



FERRIC BENTONITE
CHANGE IN PARTICLE SIZE DUE TO TEMPERATURE

Figure 3



-NOTE-
Values on curves-
Temperatures to which
sample was heated.

ALUMINUM BENTONITE
CHANGE IN PARTICLE SIZE DUE TO TEMPERATURE

Figure 4

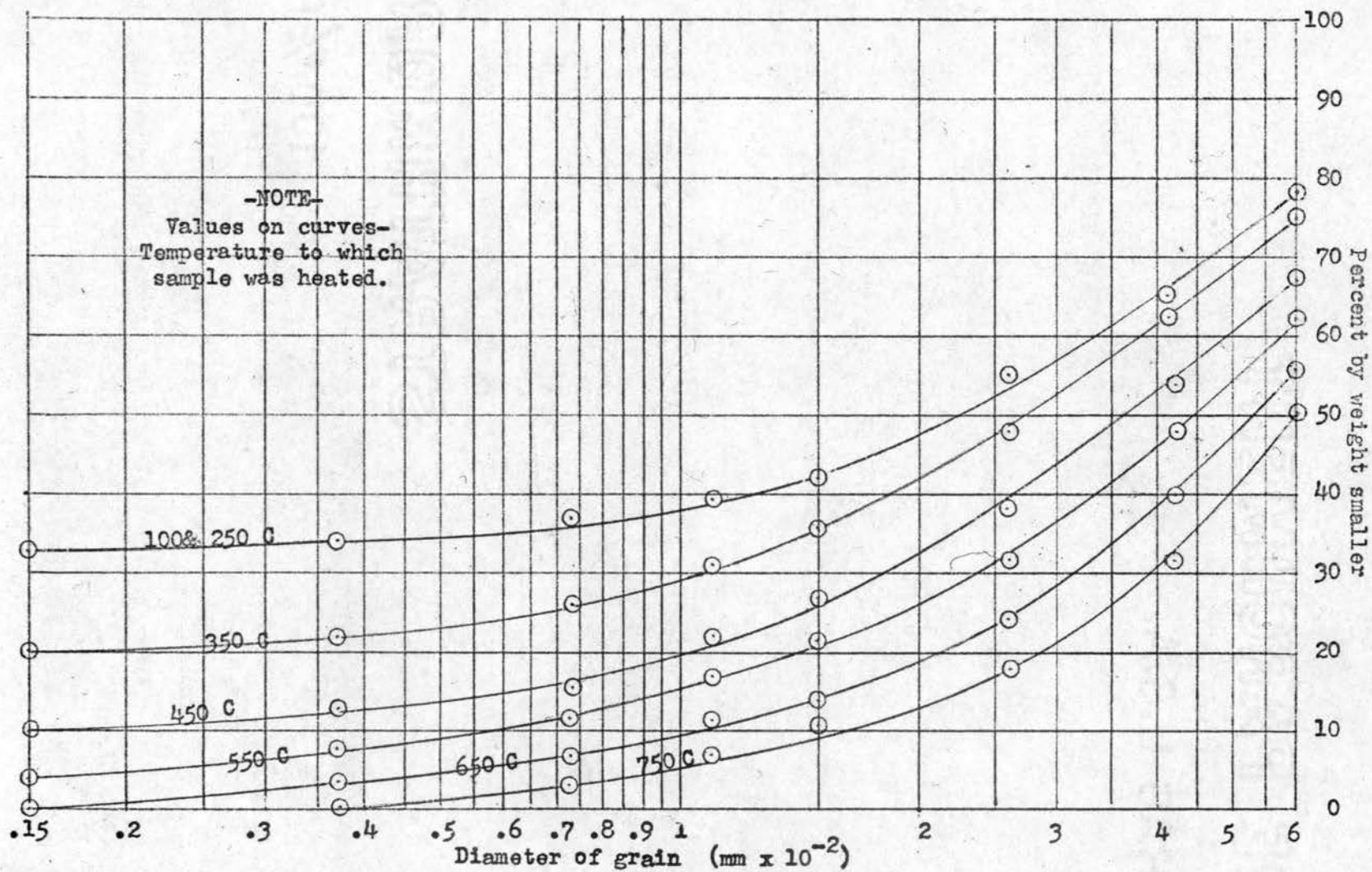
portion of the changing. The 450 C temperature seems to be near the temperature of the major change. After heating to 750 C only 5 percent of the particles were of clay size.

Effect of Heating on Particle Size of Dennis Clay After Ionic Exchange With Aluminum

As evidenced by the results shown in Figure 5, the 250 C heating had no effect on the particle size of the aluminum exchanged Dennis clay. The 350 C heating caused an increase in particle size. The change continued up through the 750 C heating. The curves all follow the same pattern with each increase in temperature causing an increase in particle size. The results on the Dennis clay compare favorably with the results of the aluminum exchanged bentonite. The change in particle size began at the same temperature, 350 C, in both clays. In both clays, approximately 2 percent of the particles were of clay size after heating to 750 C.

Discussion of The Effect on Particle Size

As expected, all additives had the desired effect on particle size although the additive without heating had no noticeable effect on particle size. Of the three additives used, the aluminum proved to be the most effective, followed closely by the iron. The percent passing curve of the 550 C of the pure bentonite corresponded to the 450 C of the calcium, to the 350 C of the iron, and to the 250 C



ALUMINUM-DENNIS CLAY
CHANGE IN PARTICLE SIZE DUE TO TEMPERATURE

Figure 5

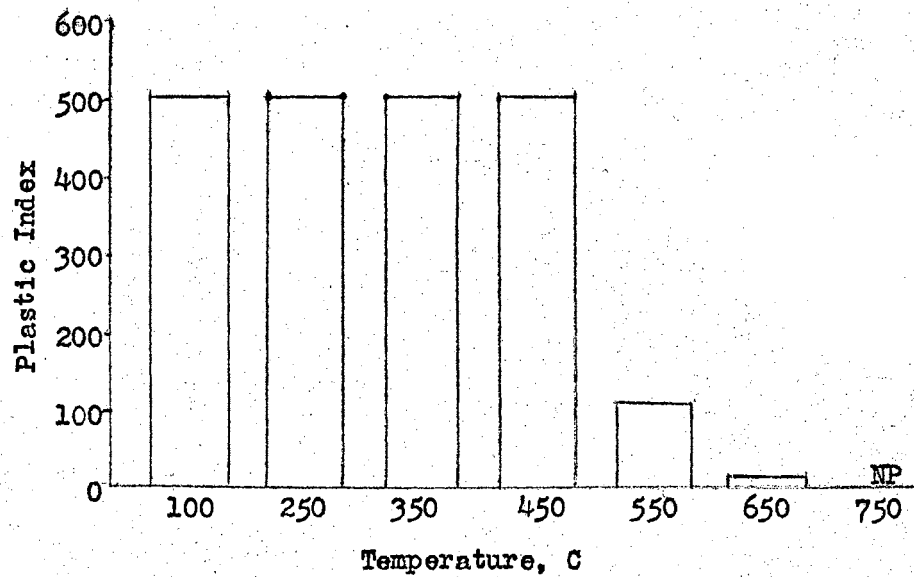
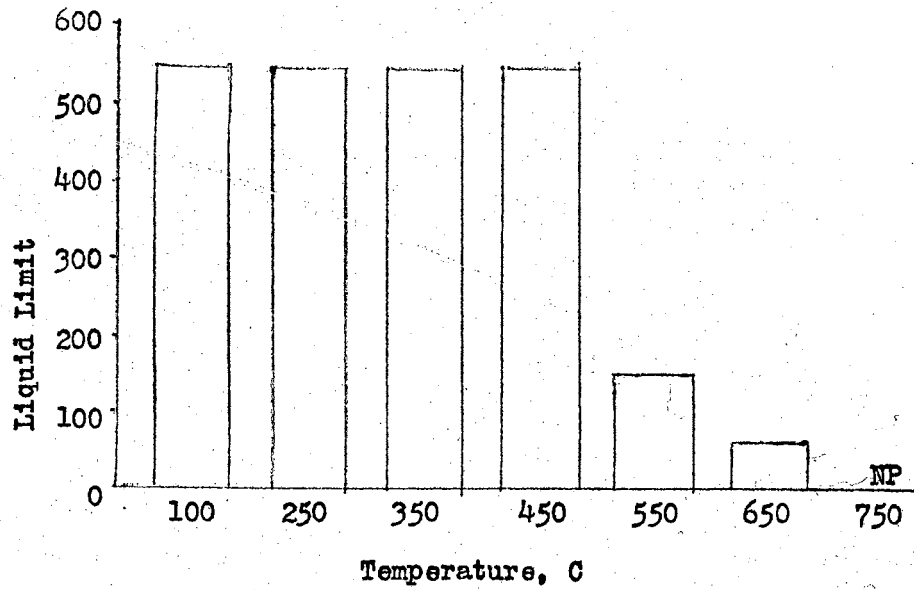
of the aluminum. The changes of grain size could probably be accounted for by the realination of the crystals after the loss of water. The positive exchange ions had probably neutralized the negative charge on the clay.

Effect of Heating on the Atterberg Limits of Bentonite

The Atterberg limit tests were very time consuming to run on the pure bentonite because of the difficulty in getting a homogeneous mix with the water. The extremely high liquid limit (see Figure 6) illustrates the amount of water needed to mix with the bentonite. There was no noticeable change in the liquid limit until after the bentonite was heated to 550 C. The change in liquid limit occurred at the same temperature as a large change in particle size. The liquid limit continued to decrease with an increase in temperature until the material completely lost its plasticity at 750 C. There was no noticeable change in the plastic limit throughout the test, so the plastic index followed the same pattern as the liquid limit.

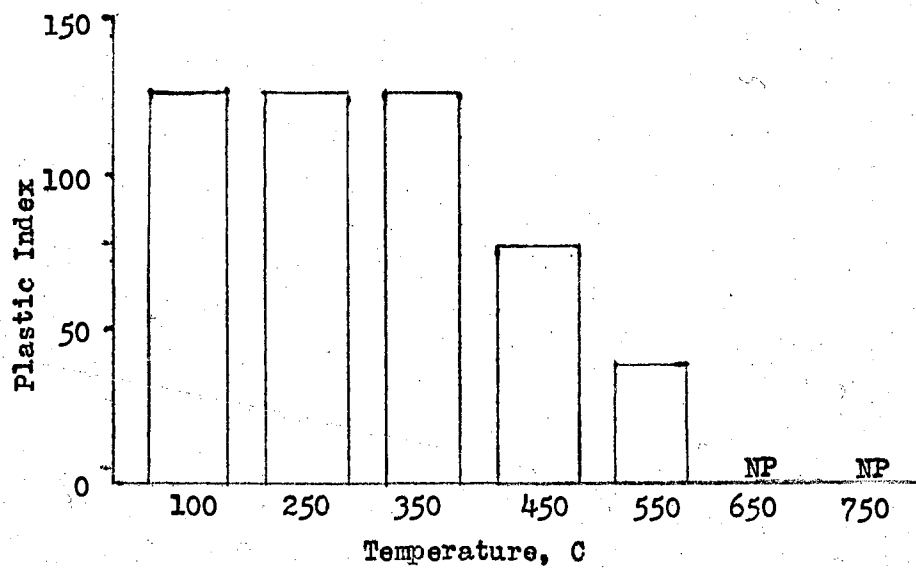
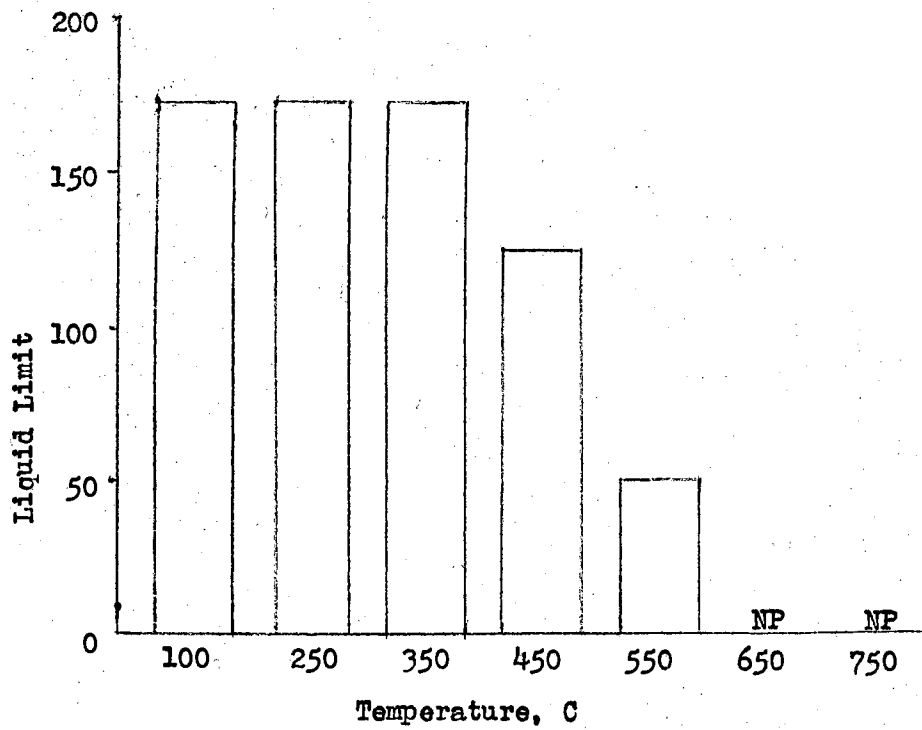
Effect of Heating on Atterberg Limit of Calcium Bentonite

The ionic exchange of calcium with bentonite lowered the liquid limit to approximately one-third of the liquid limit of the original material. This change was due to the exchange of the original sodium ion with a more tightly bound calcium ion. The liquid limit (see



CHANGE IN ATTERBERG LIMITS OF PURE BENTONITE
DUE TO TEMPERATURE

Figure 6



CHANGE IN ATTERBERG LIMITS OF CALCIUM BENTONITE
DUE TO TEMPERATURE

Figure 7

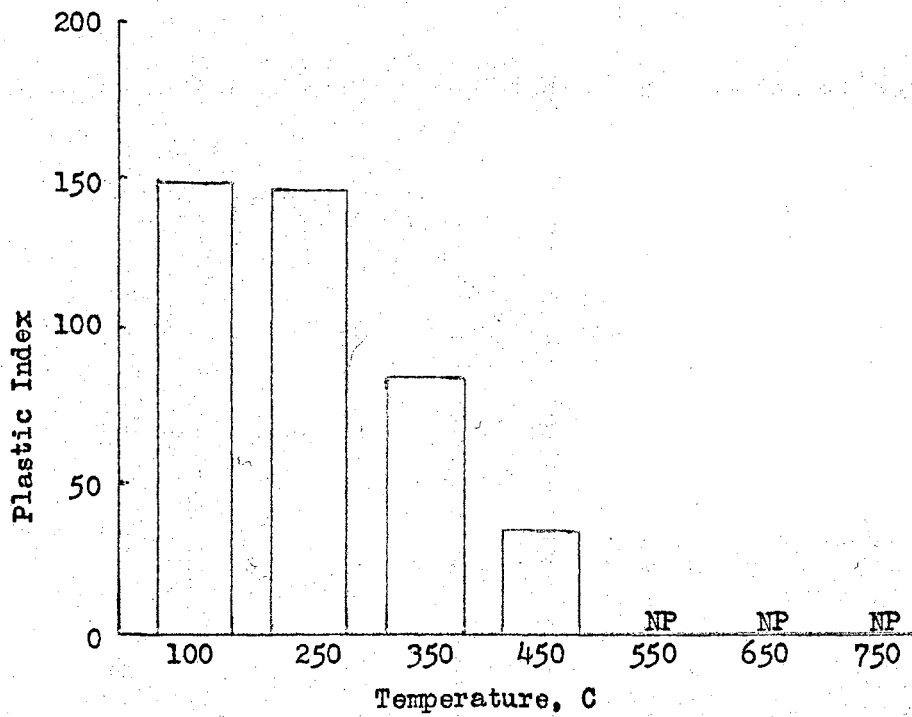
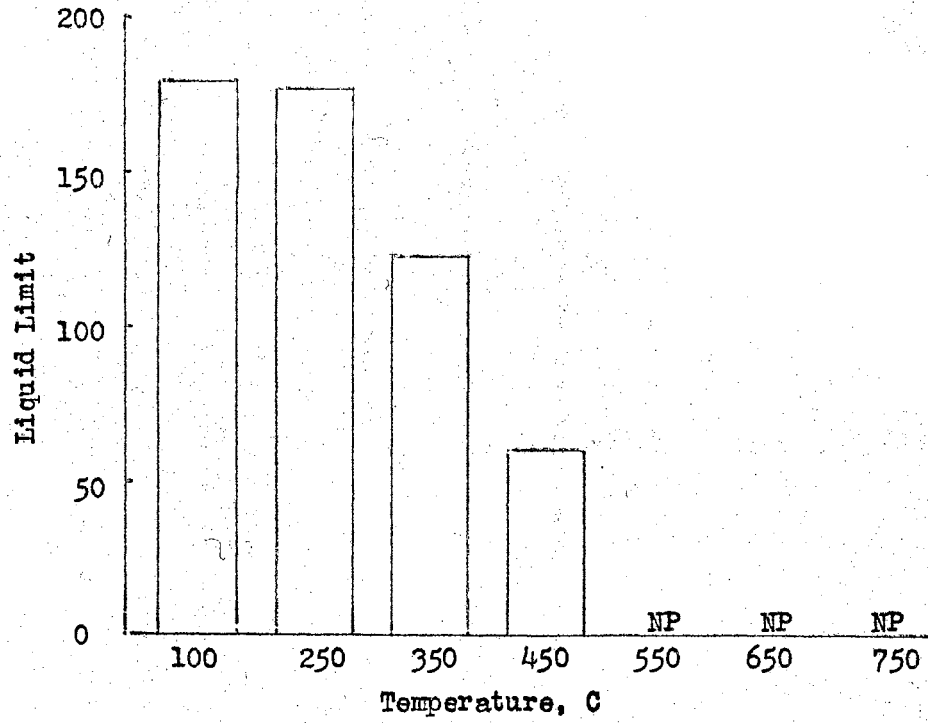
Figure 7) was unchanged until the material was heated to a temperature of 450 C. The liquid limit continued to decrease until the temperature reached 650 C at which temperature the material had turned nonplastic. The plastic limit of the material was unaffected by heating after the ionic exchange. Since the plastic limit was unchanged, the plastic index followed a pattern similar to the liquid limit.

Effect of Heating on Atterberg Limit of Ferric - Bentonite

The ionic exchange of ferric with bentonite lowered the liquid limit of the bentonite to approximately one-third of the liquid limit of the original material. This change was due to the exchange of the original sodium ion with the more tightly bound ferric ion. The liquid limit (see Figure 8) began to change when the material had reached a temperature of 350 C. The liquid limit continued to change until the material was heated to 550 C at which temperature the material exhibited no plasticity. The plastic limit was unchanged throughout the test so the plastic index followed a pattern similar to the liquid limit.

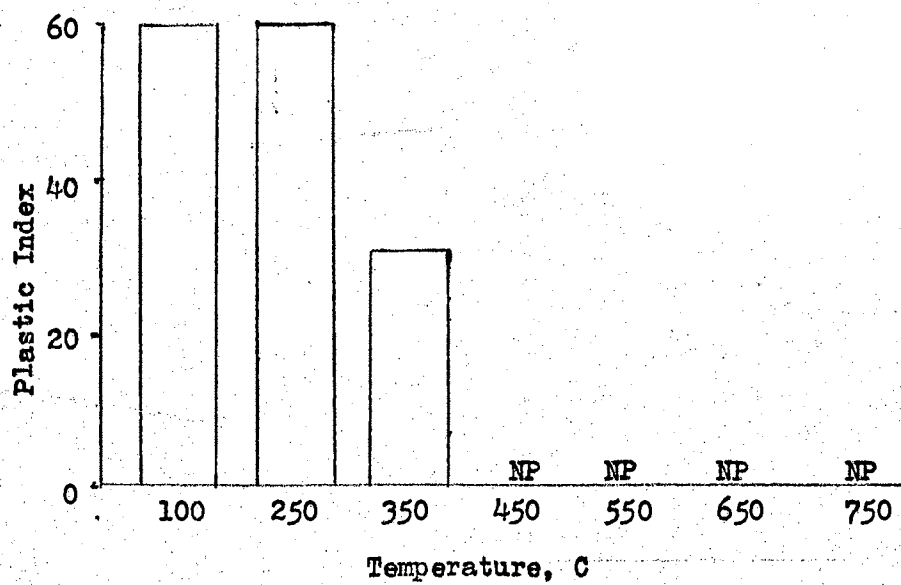
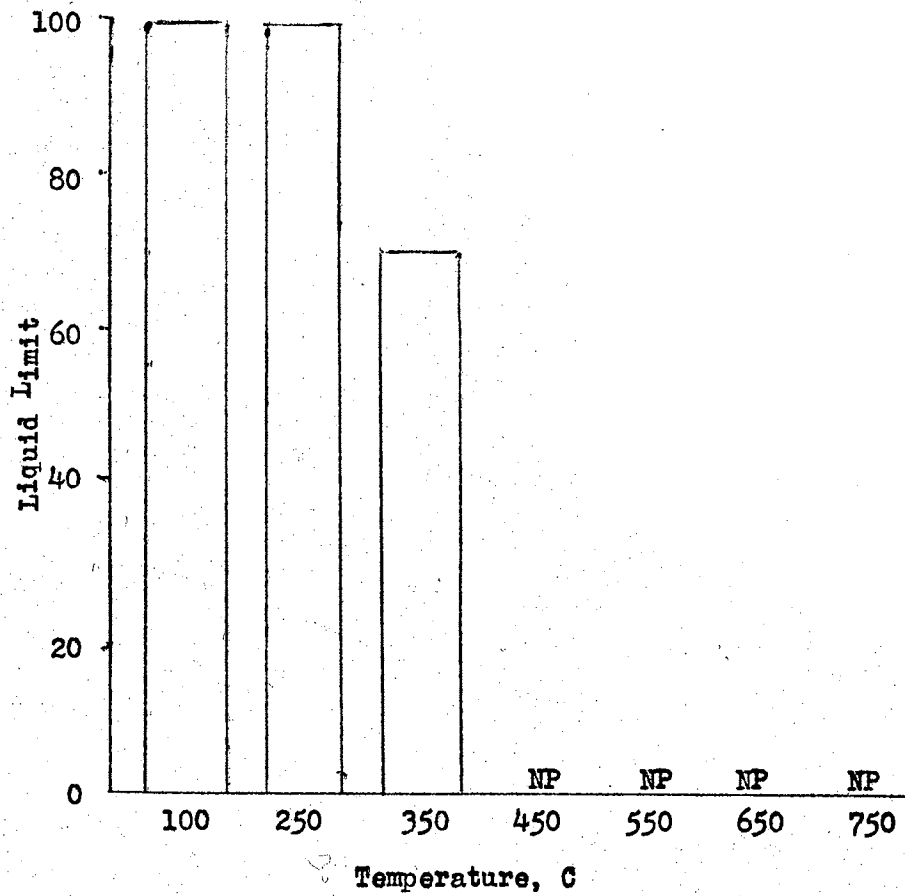
Effect of Heating on Atterberg Limits of Aluminum Bentonite

The ionic exchange of aluminum with bentonite lowered the liquid limit of the bentonite to approximately one-fifth



CHANGE IN ATTERBERG LIMITS OF FERRIC BENTONITE
DUE TO TEMPERATURE

Figure 8



CHANGE IN ATTERBERG LIMITS OF ALUMINUM-BENTONITE
DUE TO TEMPERATURE

Figure 9

of the liquid limit in the original material. This change was due to the exchange of the original sodium ion with a more tightly bound aluminum ion. The liquid limit (see Figure 9) remained at 100 until the material reached a temperature of 350 C at which time a noticeable change occurred. When the material reached a temperature of 450 C, the material exhibited no plasticity. The plastic limit remained the same throughout the test, so the change in plastic index was similar to the change in the liquid limit.

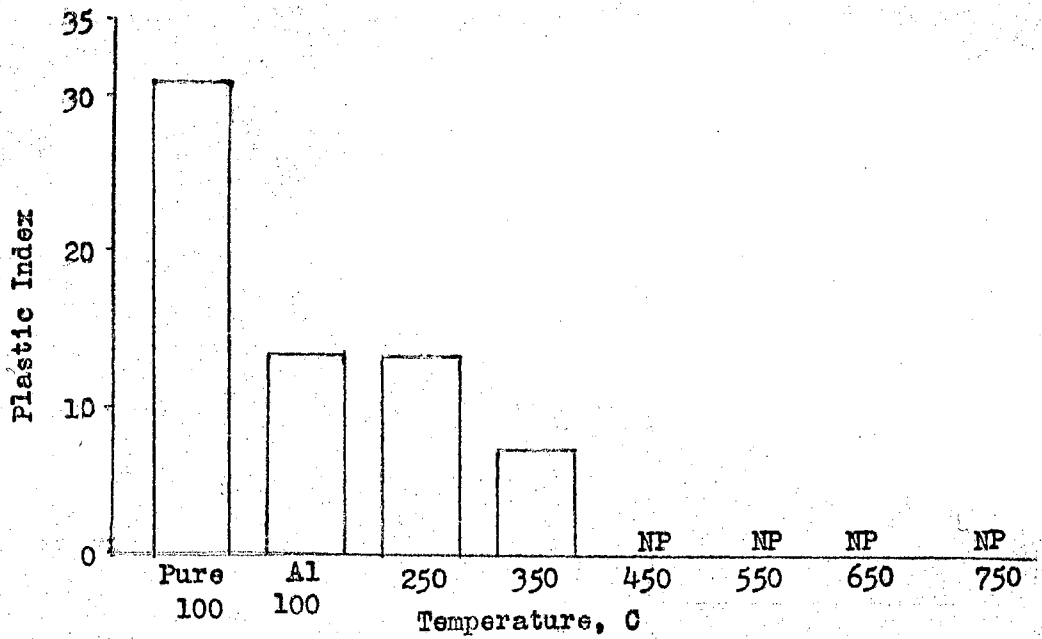
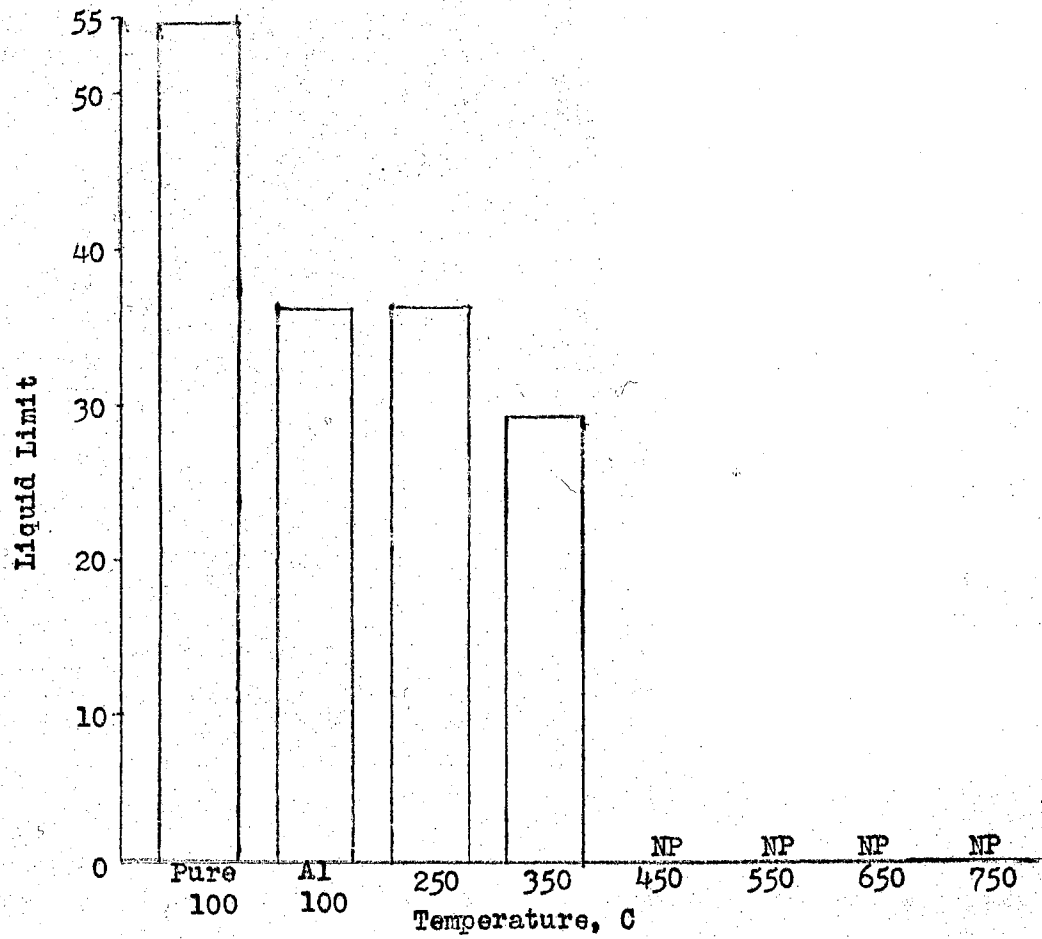
Dennis Clay

The liquid limit (see Figure 10) on the Dennis clay was 55 before the ionic exchange with aluminum. The exchange with the more tightly bound ion lowered the liquid limit to 36. The next change was noted after heating the material to a temperature of 350 C. The clay exhibited no plasticity at the temperatures above this.

The plastic limit of the Dennis clay was 23 in the original material and remained the same throughout the tests.

The Dennis clay, in its original state, was not suitable for a stabilization material. After the exchange and heating to 350 C, the material would pass the requirements for gradation and Atterberg limits for stabilization work.

The particle size on the Dennis clay began changing when the material was heated to 350 C. The change was approximately regular throughout the heating.



CHANGE IN ATTERBERG LIMITS OF ALUMINUM-DENNIS CLAY
DUE TO TEMPERATURE

Figure 10

CHAPTER VI

CONCLUSIONS

The evaluation of the effect of heating with an additive on particle size and Atterberg limits on montmorillic clay has been made to see if it is possible to sufficiently change some of the undesirable properties of clay to utilize it for stabilization work. The results of this investigation show a definite change in particle size and liquid limit of the clays tested will take place when heated with an additive. The use of an additive without heating might be deemed possible if a material was unfit for use by plasticity requirements alone.

Conclusions: Effect of Additives

1. The ionic exchange lowered the liquid limit of the clays a considerable amount.
2. The ionic exchange had no apparent effect on particle size.
3. The ionic exchange had no apparent effect on plastic limit.

Conclusions: Effect of Heat Alone

1. The heat began to change the particle size of the bentonite at 550 C.
2. The bentonite, after heating to 750°, had

changed from clay particle size to silt particle size.

3. The liquid limit changed at approximately the same temperature as the particle size. This is reasonable because the amount of water that can be held by a soil is related to particle size.

Conclusions: Heating with an Additive

1. The temperature necessary to change the properties investigated was considerably lowered by the use of additives.

2. Of the three additives used, the aluminum nitrate showed the best results.

3. Neither the ionic exchange nor the heating had any appreciable effect on the plastic limit.

The result of this investigation show that particle size of the clays was increased by heating both alone and with additives. The use of the additive with the heat, lowered the intensity of heat necessary to effect the desired change in particle size. The ionic exchange between the clay and the additive decreased the liquid limit and plastic index of the clays; and the application of heat caused a further decrease in liquid limit and plastic index. The plastic limits of the clays were unchanged by neither the heat nor the ionic exchange.

A SELECTED BIBLIOGRAPHY

1. Baver, L. D., "Soil Physics", John Wiley & Sons, p. 10.
2. Jenny, Hans, Studies on the Mechanism of Ionic Exchange in Colloidal Aluminum Silicates, (1932), Vol. 36, pp. 2217-2258.
3. Shaber, G. M. and Russell, M. B., The Thermal Method as a Quantitative Measure of Clay Mineral Content, Soil Science, (1942), Vol. 53, pp. 353-364.
4. Lutz, J. F., The Physicochemical Properties of Soils Affecting Soil Erosion, Missouri Agr. Exp. Station Research Bulletin, p. 212.
5. Baver, L. D., "Soil Physics", John Wiley & Sons, p. 14.
6. Wilkinson, Guy and Gray, Fenton, A Clay Mineralogical Study of Certain Reddish Prairie Soils of Oklahoma, Soil Science Soc. Am. Proc., (1954), Vol. 18, p. 264.
7. AASHO Designation: T88-54, Standard Specifications for Highway Materials and Methods of Sampling and Testing, American Association of State Highway Officials Specifications, Part II, 7th Edition, p. 257.
8. AASHO Designation: T89-54, Standard Specifications for Highway Materials and Methods of Sampling and Testing, American Association of State Highway Officials Specifications, Part II, 7th Edition, p. 270.
9. AASHO Designation: T90-54, Standard Specifications for Highway Materials and Methods of Sampling and Testing, American Association of State Highway Officials Specifications, Part II, 7th Edition, p. 274.

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