Lawrence Berkeley National Laboratory

Recent Work

Title

EFFECT OF IMPURITIES ON THE RHEOLOGY OF TWO KAOLINS

Permalink

https://escholarship.org/uc/item/3sj6r996

Authors

Langston, R.B. Jenne, E.A. Pask, J.A.

Publication Date

1964-10-13

University of California

Ernest O. Lawrence Radiation Laboratory

TWO-WEEK LOAN COPY

This is a Library Circulating Copy which may be borrowed for two weeks. For a personal retention copy, call Tech. Info. Division, Ext. 5545

EFFECT OF IMPURITIES ON THE RHEOLOGY OF TWO KAOLINS

Berkeley, California

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

To be published in Proceedings of First Meeting of The Clay Mineral Society

UCRL-11671

UNIVERSITY OF CALIFORNIA

Lawrence Radiation Laboratory Berkeley, California

AEC Contract No. W-7405-eng-48

EFFECT OF IMPURITIES ON THE RHEOLOGY OF TWO KAOLINS

R. B. Langston, E. A. Jenne, and J. A. Pask

October 13, 1964

EFFECT OF IMPURITIES ON THE RHEOLOGY OF TWO KAOLINS

by

R. B. LANGSTON, E. A. JENNE, $\frac{1}{}$ AND J. A. PASK

Inorganic Materials Research Division, Lawrence Radiation Laboratory, and Department of Mineral Technology, College of Engineering, University of California, Berkeley, California

ABSTRACT

Two kaolins were given five successive processing steps so as to remove a number of impurities. These processing steps were designed to accomplish the following objectives: (1) removal of nonclay and soluble fractions; (2) removal of organic material; (3) removal of iron oxides; (4) removal of allophane; and (5) removal of three-layer lattice minerals.

After each of these steps, consistency curves were obtained for each of the clays at both 6.5 and 9.0 pH on slurries containing up to 50 weight percent solids. Yield point, plastic viscosity, and apparent viscosity at 250 sec⁻¹ values were calculated. These rheological data were supplemented with chemical analyses, photomicrographs, X-ray and D.T.A. evaluations. The effect that some impurities have on the rheological properties of these kaolins is discussed.

¹ Present address U. S. Geological Survey, Federal Center, Denver 25, Colorado. INTRODUCTION

-2.

The rheological properties of kaolins reported in the literature vary by several orders of magnitude for similar concentration and pH conditions. Since these differences could not reasonably be ascribed to size, shape or counter ion variations of the fundamental kaolin particles, it is assumed that such rheological variations result principally from impurities present. Impurities associated with a kaolin could be both organic and inorganic, water soluble and insoluble, crystalline and amorphous, discrete and coatings.

The objective of this study was to evaluate the effect of naturally occurring organic matter, free iron oxide, amorphous alumino-silicates (allophane) and some three-layer lattice clay minerals on the rheological properties of two kaolins. Inasmuch as the effect of allophane was of particular interest, A.P.I. Kaolins Nos. 2 and 7 were originally chosen to respectively represent low and high allophane contents (Jenne and Langston, 1962). In this work the alumina and silica extracted in 2-1/2 min from the kaolin by a boiling N/2 NaOH solution was called allophane (Hashimoto and Jackson, 1960). Later work has shown that neither of these two kaolins contains more than 1 or 2 percent allophane, but that A.P.I. Kaolin No. 7 itself has a lower degree of crystalline order and a much faster dissolution rate in the boiling caustic than A.P.I. Kaolin No. 2 (Langston and Jenne, 1964).

LITERATURE REVIEW

Organic matter is the only one of the impurities considered in this study whose effect on the rheological properties of kaolin has been reported in the literature. Henry (1955) concluded that much of the increase of plasticity of commercial clays, as a result of weeks' or months' aging under moist conditions, results from microbiological growth. Johnson and Norton (1941) presented data showing much higher apparent viscosities for "Florida Clay" after treatment with H2O2, although the difference is more likely to have been due to the dilute NaOH wash (to provide sodium saturation) and to the additional blunging and screening treatments given the H2O2 treated samples that would have produced differences in shape and size of the particles. They did, however, observe that as increments of NaOH were added to the electrodialyzed specimen, the apparent viscosity increased in the pH range of 6.5 to 9.0 and then decreased at pH 12 to the same value that was obtained between pH 5.9 and pH 6.0. This "hump" in the apparent viscosity vs pH curve did not occur in the H_2O_2 treated electrodialyzed sample. It could be induced by adding a distilled water extract of lignite to the sample; subsequent removal of the lignite by H₂O₂ treatment removed the "hump". According to these authors, "The samples in this region exhibit a pronounced thickening during the period in which the viscosity readings are taken. This phenomenon is a type of 'rheopexy'. The thickening of the slip has been known to continue to increase even after from 10 to 15 hours of operation in the modified MacMichael viscometer ... " They suggested that the sharpness of the peak

in the apparent viscosity vs pH curve would tend to indicate the degree of purity of the sample relative to organic material. Mitchell and Henry (1943) reported the observation that the viscosity was greatly increased in a "soft" Georgia kaolin by H_2O_2 treatment. Mitchell and Poulas (1959) reported that in five out of seven Georgia kaolins the viscosity was decreased by H_2O_2 treatment and in the other two, there was not much change; however, only the samples that were treated for organic matter removal were blunged, and evaluation of the effect of the H_2O_2 treatment itself was not obtained.

No references were found showing the effect of iron oxides on the rheological properties of kaolins. However, Hauser and Reed (1937) found that 2.47 percent positively charged iron oxide (dialyzed) mixed with electrodialyzed bentonite resulted in a gel with a thixotropic setting time in excess of 130 hr and that additional ferric oxide lowered the thixotropic setting time to a few minutes. They cite a private communication to the effect that positively charged aluminum oxide (alumina gel) produced a similar effect.

EXPERIMENTAL

Materials

Two samples of raw kaolins from the A.P.I. series of reference clays were obtained from Wards Natural Science Establishment and used in this study. The preliminary A.P.I. reports on project 49 describe Kaolinite-2 as coming from the Birch Pit near Macon, Georgia, and Kaolinite-7 as

-4-

coming from the Dixie Rubber Pit near Bath, South Carolina. Typical chemical analyses of materials from these areas are presented in Table 1. According to Kerr, Main, and Hamilton (1950), the impurities found in Kaolinite-2 include: quartz, halloysite, sericite, leucoxene, and carbonaceous material; while the impurities found in Kaolinite-7 include: quartz, vermiculite, feldspar, mica (sericite), titanium bearing minerals (ilmenite or rutile, sphene altering into leucoxene), and some zircon.

Processing of the Materials

Pretreatment. - Any carbonates, subfates, or manganese dioxide present in the raw clays were removed with a 1-N sodium acetate (5.0 pH, buffered with 27 ml glacial acetic acid per liter) treatment. The procedure of blending the sample in the sodium acetate solution, followed by wet sieving (to separate the +2 mm fraction), allowed the nonclay and soluble fractions to be discarded. The clay fraction was recovered by centrifugation. Each sample was processed through three extraction cycles with the slurry being heated at about 80°C for 15 min before centrifugation (Jackson, 1956, paragraph 2-6). The centrifuged solids were then washed twice with 1-N NaCl to remove the acetate ions and then washed with dis-. tilled water, using sufficient methanol to maintain the clay in a deflocculated state, until the decanted liquor gave a negative chloride test with AgNO3. In both cases the supernatant was passed through No. 03 filter candles to insure that no clay was lost. The recovered washed solids were then dried at 60°C, crushed to -100 mesh, and stored. The resulting samples were numbered Ka-2 I and Ka-7 I.

<u>Organic Matter Extraction</u>. — In each case the organic matter present in the samples was removed by subjecting the solids from treatment I to a H_2O_2 treatment (Jackson, 1956, paragraph 2-15). These extractions were carried out using a total of 40 ml of 30 percent H_2O_2 per 10 g of clay solids in 400-ml beakers in a hot water bath. After the peroxide treatment the samples were washed, dried, and crushed as previously described in the preceding paragraph. The resulting samples were numbered Ka-2 II and Ka-7 II.

Iron Oxide Removal. —The solids from treatment II were subjected to three successive iron oxide extractions (Jackson, 1956, paragraph 2-56). In each of these extractions 10 g of the specimen were placed in a solution of 40 ml of 0.3 <u>M</u> sodium citrate to which 5 ml of $1-\underline{M}$ sodium bicarbonate was added. The temperature was then raised to 80° C and 1 g of $Na_2S_2O_4$ added. The mixture was then stirred constantly for 1 min and the treatment was continued, with occasional stirring, for 15 min. The liquor was separated using centrifugation after each extraction, after deflocculation with 10 ml of saturated NaCl solution. Finally the solids were washed, dried, and crushed as described previously and the resulting samples were numbered Ka-2 III and Ka-7 III.

<u>Allophane Removal.</u> — Amorphous silica and alumina were extracted using the method of Langston and Jenne (1964). This process consisted of placing 3 g of Ka-2 III or 0.3 g of Ka-7 III in 200 ml of boiling N/2 NaOH for 12 min, with stirring. After the extraction, the deflocculated solids were washed, dried, and crushed as previously described. The recovered solids were numbered Ka-2 IV and Ka-7 IV. <u>Particle Size Classification and Removal of Three-Layer Lattice</u> <u>Minerals.</u>—Identical particle size separations on the Ka-2 IV and Ka-7 IV samples were made to obtain clean, narrower size range particles. The separations were carried out in distilled water which contained 2 g of sodium carbonate per 18 liters with a clay concentration of 2 g per liter. The coarsest fraction was removed by centrifugation. The limit of the finest fraction was set so that X-ray diffraction studies of orientated and glycolated specimens indicated only a trace of three-layer expandable minerals to be present. The remaining fractions were washed, dried, and crushed as previously described. Since only comparable sized specimens were desired, accurate particle size determinations were not made. The finished specimens were numbered Ka-2 V and Ka-7 V.

EVALUATION AND RESULTS

X-Ray Diffraction. - The solids from the various extraction processes were studied using the X-ray diffraction techniques that have been previously described by Langston, Trask, and Pask (1958). In their method, oriented specimens are prepared on glass microscope slides. The slides have a density of 20 mg of sample per square inch and were successively given treatments of (a) drying at 110°C, (b) glycolation for 1 hr at 65°C, and (c) firing for 1/2 hr at 600°C with an X-ray diffraction pattern being obtained after each of the treatments. Table 2 shows the effect the different treatments had on some selected characteristic spacings of the various processed solids ... Powder patterns were also obtained on -325 mesh material that was dried at 110°C. The diffraction patterns were obtained with a NORELCO diffractometer that was operated using nickel filtered copper radiation at a scan rate of 1° 2 θ per The data in Table 2 indicate that kaolin is the major constituent minute.

in both of the samples. The stronger reflections for Ka-2 suggest a higher degree of crystallinity. A three-layer lattice minerals, 12.6 A° when dried at 110°C that expands to 17 A° when treated with ethylene glycol and contracts to 9.4 A° when heated to 600°C is present in Ka-2. It was removed during the particle size separation step (treatment V). Ka-7 also has an expandable three-layer lattice mineral present. It has a 10 A° spacing when dried at 110°C that expands to 18.4 A° when treated with ethylene glycol and contracts to 10 A° when fired at 600°C; the latter line, however, was very weak on the Ka-7 I specimen but appeared stronger on the Ka-7 II specimen. This expandable material was no longer present after the iron oxide removal step (treatment III). Some hydrous micaceous-like (10 A°) material appears in the Ka-7 series after the iron oxide removal processing (treatment III) which is subsequently removed during the particle size separation step (treatment V). Samples of the Ka-7 IV solids were not available for analysis.

Differential Thermal Analysis. —Samples were heated at a rate of 12° C per min in equipment so as to measure the differences in temperature between the sample and a standard inert material (calcined Al₂O₃). A general review of the D.T.A. curves reveals the following: (1) both the exothermic and endothermic effects observed in Ka-2 specimens were stronger than those observed in Ka-7 specimens, and (2) as the material progressed through the various impurity removal steps, the kaolinite reaction peaks generally became sharper and more intense with the other smaller thermal effects diminished. This effect was more pronounced for treatment III of the Ka-7 specimens.

<u>Chemical Analysis</u>. —Conventional analytical methods were used to evaluate silica, alumina, iron oxide, and volatile matter content of the various processed solids. There were no significant changes in the contents of either series of specimens (Ka-2 or Ka-7) due to any of the processing steps. Lack of change in the iron contents was confirmed using X-ray fluorescence.

<u>Rheological Measurements</u>. —Viscosity and yield stress measurements on prepared clay-water slurries were made using a modified Precision Interchemical coaxial cylinder rotational viscometer. This instrument is equipped with a constant temperature bath which was maintained at $25 \pm 0.2^{\circ}$ C, a stepwise controller of the rotational speed (0 to 400 rpm), and with a selection of cups, bobs, and torsion springs that produced a working range of shear rates from zero up to 1700 sec⁻¹. It was calibrated against standard viscosity materials obtained 'from the National Bureau of Standards.

Water suspensions of each of the samples were prepared over the concentration ranges of 5 to 50 weight percent solids as indicated in Table 3. After standing overnight and just before the consistency curves were run, the pH of each slurry was adjusted to either 6.5 or 9.0 pH with a hydrochloric acid or sodium hydroxide solution. After completion of each consistency curve, pH evaluations showed that in no case had the pH varied more than 0.3 pH units. The pH determinations were made with a Beckman Model H-2 pH meter.

Consistency down-curves for each of the suspensions were obtained using a suitable bob, cup, and torsion spring combination. These curves were obtained by determining a number of points, at 50 rpm intervals.

-9-

Starting with the viscometer at a steady state at 400 rpm, the rotational speed was decreased and the reading for that speed recorded when the viscometer reached equilibrium which in most cases required 2 or 3 min. Between the points, in each case, the viscometer rotational velocity was returned to equilibrium at 400 rpm before the next point on the consistency down-curve was determined. From these consistency curves, plots of shear stress vs shear rate, graphical values were obtained so as to allow calculations to be made of the plastic viscosity, the apparent viscosity at 250 sec⁻¹, and yield stress. The results are summarized in Tables 3, 4, and 5, respectively. The data for plastic viscosity vs clay concentration, for suspensions at pH 9.0, are presented graphically on semi-log paper in Fig. 1. The observed relationships found in Fig. 1 are also reproduced on arithmetic paper in Fig. 2. The plastic viscosity data of the 6.5 pH suspensions as well as the yield point and apparent viscosity data for both the pH series show relationships similar to Figs. 1 and 2.

<u>Microstructure</u>. —Examination of 6.5 pH and 9.0 pH suspensions containing 0.1 percent clay (by weight) with transmitted light revealed considerable dispersion of the particles. A number of aggregates, however, were observed. Figures 3-a and 3-b present micrographs of the pH 9 suspensions of Ka-2 III and Ka-7 III, respectively. The specimens were prepared by placing a few drops of suspension between a cover glass and microscope slide which were held apart by a few 80μ grains. In both of these cases, as well as the cases with the other specimens, the suspended particles varied greatly in size with the larger particles having a diameter of some 50 to 100 times that of the smaller particles. Furthermore, the Ka-2

-10-

specimens had larger particles than the Ka-7 specimens. The suspended particles remained in constant motion (Brownian type), usually vibrating around a stationary point in the suspension. The larger particles vibrated only slightly. The mottled appearance of the background of the micrographs is due to the vibration of the smaller particles which are not well defined due to the length of time required to properly expose the film (3 to 5 sec).

Figures 4-a and 4-b are electron micrographs of the clay solids of Ka-2 I and Ka-7 I, respectively. The specimens were prepared from the pH 9.0, 0.1 percent clay suspensions using a technique consisting of spraying, freezing, and drying by sublimation. They further illustrate the greater crystallinity and larger particle size of Kaolin 2.

DISCUSSION

The rheological behavior, in this case specifically coefficient of viscosity, of a clay suspension is sensitive to the structure of the suspension. In a general way, this sensitivity can be expressed in terms of a modified Einstein's equation as presented by Langston and Pask (1958) in Eq. (1),

$$\eta = \eta_0 [1 + k(\gamma_h + \gamma_c) \theta_{\text{solid}}]$$
(1)

in which $\gamma_{\rm h}$, an effective hydrodynamic volume factor; $\gamma_{\rm c}$, a particle interference factor; and k, a shape factor, are functions of $\theta_{\rm solids}$, the weight fraction of the solid, and particle size distribution in some complex way. The $\gamma_{\rm h}$ factor is an overall term that converts the $\theta_{\rm solid}$ factor into the corresponding volume fraction of the effective dispersed phase; it increases with any hydration, gelation, adsorption of water, or entrapment of water in any structure that may form due to interaction between the solid phase particles. The γ_c factor is a term that takes into account any interactions between and within the portions of the more rigid phase of the system and thus becomes more important at higher concentrations, and with an increase in the number of particles for a given weight or volume of solid. The data (Table 3) showing the relationship between viscosity and concentration can be interpreted in a general way on the basis of these principles.

Since the consistency measurements were made on clays that were, after all treatments, washed with a NaCl solution to remove excess treating chemicals, then washed to remove the excess chloride ions, and finally adjusted to either pH 6.5 or 9.0, it can be assumed that they were essentially Na-clays. The relatively minor rheological differences between the two pH series support this assumption, although the plastic viscosities and yield points, as expected, are lower at pH 9.0 for otherwise equivalent suspensions. The observed changes in plastic viscosity for either clay for a given clay concentration must then be due to structural changes in the suspension that have resulted from the various treatments to which the clays were subjected.

The specific effects of all of the various treatments on the clays themselves have not been evaluated. The principal indications of changes have been provided by the X-ray diffraction analyses. Besides the kaolinite lines, some lines for expandable three-layer lattice minerals are present. These minerals are different in the two specimens; those in Kaolin-7 were more susceptible to the chemical treatments. The Ka-7 III specimen indicated that the "iron oxide removal" treatment did attack and probably removed the expandable fraction. D.T.A. runs support this conclusion in that the kaolinite thermal peaks of Ka-7 III are larger and sharper than those of the Ka-7 I and Ka-7 II specimens. The lack of a significant change in the total iron content suggests that the remaining phases have essentially the same fraction of iron content as the removed phase.

-13-

Of further interest is the appearance of diffuse diffraction in the 10 to 12 A° region on the Ka-2 V specimen heated to 600°C; it may indicate the presence of disordered three-layer material which would account for the higher total silica content in this specimen. These indications of phase compositional differences are not readily deducted from a chemical analysis.

The removal of impurity three-layer minerals in the discarded fine fraction in the preparation of the Ka-2 V and Ka-7 V specimens suggests that they were removed as individual fine particles or as aggregates. The aggregates were of lower density caused by association of water with the solids either as a hydrosphere about individual particles, as interlayer water or as interstitial water in agglomerates. It is thus evident that Stokes' law calculations could be misleading for systems whose microstructure is not known under the existing test conditions. The Ka-2 V and Ka-7 V fractions showed the lowest plastic viscosities and yield points of their respective series for corresponding weight suspensions but were not equivalent; the former had lower values presumably because of larger (and, consequently, a smaller number) particles with smaller shape factors,

as seen in Fig. 4.

The change in plastic viscosity with increasing clay concentration follows a different pattern for the two kaolins which can be attributed largely to the nature and amount of three-layer lattice minerals present. The Ka-7 suspensions generally show a higher viscosity at the lower clay concentrations and a lower viscosity at the higher concentrations (compared to the Ka-2 suspensions). Also, the greater sensitivity of Ka-7 to chemical treatment is reflected in the viscosity data. The reduction in plastic viscosity after the carbonaceous removal treatment is expected on the basis that the presence of such material would generally be expected to aid in the formation of agglomerates or of a gel-like structure. The compositional changes caused by subsequent chemical treatments, as discussed previously, are also shown by the viscosity results. The Ka-2 III and Ka-2 IV suspensions exhibit only minor changes in plastic viscosity from that of Ka-2 II. The Ka-7 III and Ka-7 IV suspensions, however, show significant changes. The "iron oxide removal" treatment apparently attacks the three-layer lattice mineral with a resulting decrease of aggregate structures as suggested by the correspondingly large decrease in viscosity. The "allophane removal" treatment apparently removed amorphous cementing material between kaolinite crystallites resulting in a larger number of smaller particles with larger shape factors and consequently higher viscosities. The lower viscosities of Ka-7 I to Ka-7 IV suspensions (compared to the Ka-2 specimens) at the higher concentrations can be attributed to particles and structures that can be more easily distorted and sheared as might be expected from the presence of an appreciable fraction of threelayer lattice mineral and of thinner, smaller and more numerous clay particles.

-14-

CONCLUSIONS.

-15-

The rheological behavior of kaolinite suspensions free of three-layer lattice minerals is similar; differences are primarily due to differences in the size distribution and shape factor of the kaolinite particles. The presence of impurities of the gel and expanding layer lattice mineral types causes large changes in rheological behavior by especially affecting the effective hydrodynamic volume factor. Some of these phases are more sensitive to any chemical treatments to which they may be subjected which, in turn, results in changes in rheological behavior.

A more quantitative analysis would require a detailed study of the structure existing in the suspension for a given set of conditions for which adequate techniques have not yet been developed. A further complicating factor is that, in most cases, the structure in the suspension is dynamic and undergoes continual changes with the nature of the shear pattern.

ACKNOWLEDGMENTS

The authors express their sincere thanks for the laboratory assistance of Ronald Anderson, Donald Warner, and Larry Hartsough as well as the financial assistance provided by the National Science Foundation (Grant G-9435), the California Division of Mines, and the Inorganic Materials Research Division of the Lawrence Radiation Laboratory.

REFERENCES

Hashimoto, I. and Jackson, M. L. (1960) Rapid dissolution of allophane and kaolinite-halloysite after dehydration: in <u>Clays and Clay</u> <u>Minerals</u>, 7th Conf., Pergamon Press, New York, pp. 102-113.

Hauser, E. A. and Reed, C. E. (1937) Studies in thixotropy. II. The thixotropic behavior and structure of bentonite: <u>J. Phys. Chem.</u>, v. 41, pp. 911-934.

Henry, E. C. (1955) Clay technology in ceramics: <u>Clays and Clay</u>

<u>Technology</u>, Calif. Division of Mines Bull. 169, pp. 257-266. Jackson, M. L. (1956) <u>Soil Chemical Analysis—Advanced Course</u>: Published by the author, University of Wisconsin, Madison, 991 pp. Jenne, E. A. and Langston, R. B. (1962) Alkali extraction of silicon and aluminum oxide impurities from kaolins: Presented at the 11th Conf.

on Clays and Clay Minerals, Ottawa, Canada, August 13-17.

Johnson, A. L. and Norton, F. H. (1941) Fundamental study of clay: Preparation of a purified kaolinite suspension: <u>J. Amer. Ceram.</u> <u>Soc.</u>, v. 24, pp. 61-69.

Kerr, P. F., Main, M. S. and Hamilton, P. K. (1950) Occurrence and microscopic examination of reference clay minerals: Preliminary Report No. 5, Amer. Pet. Inst. Project 49.

Langston, R. B. and Jenne, E. A. (1964) NaOH dissolution of some oxide impurities from kaolins: in <u>Clays and Clay Minerals</u>, Minutes of 12th Conf. on Clays and Clay Minerals, pp. 633-647 (in press).
Langston, R. B. and Pask, J. A. (1958) Analysis of consistencies of kaolin-water systems below the plastic range: in <u>Clays and Clay Minerals</u>, Natl. Acad. Sci. -Natl. Res. Council. pub. 566, pp. 4-28.

Langston, R. B., Trask, P. D. and Pask, J. A. (1958) Effect of mineral composition on strength of central-California sediments: <u>Calif.</u> J. Mines and Geology, v. 54, No. 2, pp. 215-235.

Mitchell, L. and Henry, E. C. (1943) Nature of Georgia kaolin: I. Evidence of chemical and colloidal analysis: <u>J. Amer. Ceram. Soc</u>., v. 26,

pp. 105-113.

Mitchell, L. and Poulas, N. E. (1959) The relationship of structure of Georgia kaolin to its viscosity: Georgia Institute of Technology Engineering Experiment Station Bull. 23, 41 pp.

÷		Birch Pit Macon, Georgia <u>l</u> /	Dixie Rubber Bath, South Car	Pit olina <u>l</u> /	Theoretical Kaolin
		Ĭ.			,
_Si02		45.20	45.40		46.55
A1203		37.82	35.74	· ·	39.49
Fe ₂ 03	(total Fe ex- pressed as Fe ⁺⁺⁺)	0.34	1.91		
MgO		0.47	0.01	 _ 4	· · · · · · · · · · · · · · · · · · ·
CaO		0.52	0.30		40 °C =7
K20	n an an Arran an Arra an Arra. An Arra an Arr Arra an Arra an	0.49	0.57	· · · · · ·	
Na ₂ 0		0.36	0.40		
TiOz		1.26	1.48		
H ₂ 0 -		1.55	• 0.83		••••
H20 +		13.27	13.11	1	13,96
				•	

Table 1. - Chemical Analysis of Kaolins (weight percent)

. •

-19

 $\frac{1}{P}$ F. Kerr et al., Preliminary Report No. 7, American Petroleum Institute Project 49 (1951).

Orientated, dried 110°C	Orientated, glycolated		Orientated, fired	600° C
12.6 A° 7.2 A°	17 A° 7.2 A°	:	9.4 A°	

Table 2. -- Summary of X-Ray Diffraction Results⁺

Peak	12.6 A° 7.2 A°	17 A° 7.2 A°	9.4 A°
Source of peak	3-layer Kaolin	3-layer Kaolin	3-layer
Ka-2 I	500 790	560 740	200
Ka-2 II	380 770	650 790	150
Ka-2 III	d 830	440 830	d 80
Ka-2 IV	560 770	575 780	120
Ka-2 V	0 1010	0 1050	d 7 to 9°2 θ
· · · · ·	· · ·		,

Treatment	Orientated, dried 11	<u>0° C</u>	Orientate	ed, glyc	olated	Orientated,	fired 600°C
Peak	10.0 A° 7.2 A°		18.4 A°	10 A°	7.2 A°	10 A	lo lo
Source of peak	3-layer Kaolin		3-layer		Kaolin	3-1a	yer
	•				1		
Ka-7 I	13 490		26	· 0 ·	560		8
Ka-7 II	24 650	•	26	0	7,70	2	0
Ka-7 III	80 800		0	d 12	790	s 4	0
Ka-7 IV			NO S	AMP	LE		-
Ka-7 V	0 760		12	0	780		d

s = sharp

Treatment

- d = diffused
- [†]Intensities in counts per second

			. 9	.0 pH							5.5 pH			
Specimen		We	ight perce	nt concent	tration	2	`.'	1.		Weight per	cent conce	ntration		
	5	·10 ·	20	. 30	-35	40	50	5	10	20	30	35	40	50
											·	- ⁻		
Ka-2 I	0.0184	0,0324	0,0515	:	0,830		5.48	0, 0181	0.0326	0,0763		0.414	••	4, 93
Ka-2 II	0.0134	0.0203	0,0366	0, 331		0,714		0,0137	0.0214	0.0410	0,319	**	0,230	
Ka-2 III	0.0139	0. 0229	0.0517	0.368		0. 792	 ,	0,0162	0.0238	0.0656	0,407	 '	0,506	
Ka-2 1V	0. 01 30	. 0, 0213	0,0560	0.312		0.530	, 	0,0126	0.0210	0.0581	0, 287		0.469	
Ka-2 V	0.00997	0.0112	0.0149	0.0240	1	0.0402	· •• !	0.0106	0.0128	0.0165	0.0277	• ·	0.0410	
			•	•		. •						· ·		
Ka-7 I ·	0.0352	0,0675	0, 203		0. 525			0, 0173	0.0648	0, 333		0,958		
Ka-7 II	0.0218	0.0298	0, 186	0.375* ;	••	0, 479		0.0210	0.0304	0, 191	0.442*		1.58	
Ka-7 111	0.0142	0, 0130	0.0445	0, 145		0, 195		0.0156	0.0148	0.0448	0,0891		0.114	
Ka-7 IV	0,0161 /	0.0283	0, 0805	0.144		0, 338		0.0206	0.0294	0. 132	0.332	'	1,30	
Ka-7 V	0.0100	0.0144	0.0207	0.0566		,		0,0129	0.0162	0. 0245	0.0867			
L						· · ·	·		÷		· .'			

Table 3, -- Plastic Viscosity (poise) of Kaolin Suspensions

21

[†]The viscosity of water at 25°C was 0.00893 poise.

*327.

J 2 /6

۰.		• :		Table	4. —Yield	Point (dy	nes/cm ²) of	Kaolin S	Suspension	16		•			
			•	9.0 pH	· · ·			.		6.	5 pH				
Spec imen			Weight perc	ent conce	ntration				" Weight percent concentration						
	∽ ` 5	10	20	- 30	35	40	50	5 1	/ 10	20	30	35	40	50	
	*	·					lite p				•	· · · ·			
Ka-2 I	0	່	1.2		657	·	2, 340	.0	0	7.9		1,075		3,59	
Ka-2 11	0	0	32.2	171	·	442		0	: 0 '	37.8	164		196		
Ka-2 III :	ο'.	0.8	24, 3	135	·	289		0	1,1	27. 5	228	••	. 374		
Ka-2 IV	o .	0	21.6	235		309		0	0	15.0	135		366		
Ka+2 V	o	0	1.3	3.0		5,9	••	0	0	3,5	8.6		12.1	·	
		•		· · ·							÷1				
Ka-7 I	18.9	103	471		3,310			3.7	64.3	539		2,180		 	
Ka-7 II	7.2	73	670	813*		2,190		29.4	64.6	841	994*		2,560	`	

• •

••

910

340

65.4

43

907

4.0

21.1

1.6

•

3, 5

84, 3

7,5

4. A

368

1,170

254

972

• •

--

2,750

*32%

Ka-7 III

Ka-7 IV

Ka-7 V

0

,6.9

0,8

1,6

1.3

17

96

270

10. 9

398

[411

50

· · ·				9.0 pll		· ·			. 6.	,5 рН	•	· · ·	• .
Specimen		We	ight perce	ent concen	tration			We	ight perce	ent conceni	ration	:	
	5	10	20	30	35	40 50	5	10	20	30	35 .	40	50
	•		· .			÷		•	•				
Ka-2 I	0.0184	0.0324	0.0564		3.47	14,8	0.0181	0.0326	0,109	• ••	4.70		19.0
Ka-2 11	0.0134	0, 0203	0, 166	1, 02	••	2, 51 ,	0.0139	0.0214	0.176	0.975	•• *	1.01	77
Ka-2 III	0.0139	0.0261	0.148	0,906		2, 17	0.0162	0,0282	0.176	1.32		2. 26	
Ka-2 IV	0.0130	0.0213	0.142	1,25	**	1. 75	0. 0126	· 0. 0210	0, 118	0.825	.	1,92	
Ka-2 V	0. 00997	0,0112	0.0202	0.0365		0.0618	0.0106	0.0128	0, 0304	0,0622		0.0898	
	· ·		· ·		·			· · · ·					
Ka-7 I	0.110	0, 480	2.07	1	13.8		0,0382	0, 322	2.49		9, 58		
Ka-7 11	0.0510	0,322	2,86	3, 58*	·, 🛥 🗇	9.13	0.138	0, 288	3.56	4.38*	· ••	11.7	
Ka-7 III	0.0142	0,0189	0.428	1.73	**	3.84	0.0317	0, 0288	0.306	1,56		4.00	• ••
Ka-7 IV	0. 0433	0.0961	1.16	1,89		5.63	0.105	0.366	3,76	4.97		12. 2	• ••
Ka-7 V	0.0134	0.0192	0.0645	0, 225			0,0194	0,0462	0.197	1,10			
							L		•. 			· · ·	

Table 5. — Apparent Viscosity (poise) at 250 sec⁻¹ of Kaolin Suspensions

*32%

FIGURE CAPTIONS

-24-

Figure 1. Semilogarithmic plots showing the effect of the five processing steps on plastic viscosity as a function of clay concentration for 9.0 pH slurries of the two kaolins.

> Key: O Treatment I; + Treatment II; \triangle Treatment III; Treatment IV; O Treatment V

Figure 2. Replot of "best lines" of Fig. 1 on arithmetical coordinates showing the effect of the five processing steps on plastic viscosity as a function of clay concentration for 9.0 pH slurries of the two kaolins.

Figure 3. Transmitted light microphotographs of 9.0 pH, 0.1 weight percent clay suspensions. Ka-2 III (on the left) and Ka-7 III (on the right). The dark blotch areas are agglomerates while the mottled background appearance was produced by vibration of the smaller particles.

Figure 4.

Electron micrographs showing differences in particle size distribution between Ka-2 I (on the left) and Ka-7 I (on the

right).



-25-

MUB-4188

Fig. 1





This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

- A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
- B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

<u>, z</u>. . 6 e e