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EVALUATION OF LABORATORY EQUIPMENT FOR COATING COLOR PREPARATION

Evaluation of the Cowles Dissolver for Dispersing Clay Slips

INTRODUCTION

This report will serve as a continuation of Report No. 29, Project 1956 and will deal with the evaluation of a Cowles Dissolver for dispersing clay. Included in this report is a comparative summary of an attempt to study the relative efficiency of a number of methods for dispersing pigments for use in coating color formulations.

The methods that have been evaluated include the following:

1. Ball mill (Abbe 1 gallon jar);
2. Propeller mixer (Model V Lightning laboratory stirrer);
3. Homo-Mixer (Eppenbach SS Size No. 1, 1/4 h.p., 8000 r.p.m.);
4. Kady Mill (Model O, 20 h.p.);
5. Baker Perkins (2-1/2 gallon slow-speed sigma blades, 2 h.p.);
6. Charlotte Colloid Mill (1 h.p.);
- and 7. the Cowles Dissolver.

Other types of mixers which were not included but have been mentioned in the literature are: 1. the two-wheel muller (see Simpson Muller) which has a slow rate of production, is difficult to clean and requires that the consistency be carefully controlled for proper operation; 2. the hammer mill which can handle paste-like consistencies, probably would require much maintenance; it is not used to any great extent for coating color preparation; 3. the multi-roll mill, a high rate of production

dispenser being used by only a few mills, not suitable for hard pigments but can handle high solids and produces high quality coatings; 4. the disk-type mill is said to have a high rate of production and is recommended for use up to 60% solids; and 5. the ribbon-type mixer, which handles fairly high solids coatings, is widely used. The maintenance of the latter is high because of a tendency for leaks at the bearings. It has a slow rate of production and tends to entrain air.

The Cowles Dissolver, Model 5-VT, used in these trials was purchased after a three-month loan period. According to the supplier, Morehouse-Cowles, Inc., the Model 5-VT Dissolver is a mixer characterized by a saw-tooth impeller and a means for tilting the mixing head so as to simplify batch changes. It is belt-driven by a 3 h.p., 3495 r.p.m., 220/440-volt, 3-phase Louis Allis Company induction motor.

There are four different impellers supplied with the unit: a 4-inch double-acting impeller (the teeth are alternately bent up and down), 6-inch single-acting impeller (all the teeth are bent in one direction), 6-inch double-acting impeller, and an 8-inch double-acting impeller. They must be run in a clockwise direction (as seen from the top) with the sloping edge of the vane advancing.

The 8-inch diameter impeller is for use in large batches (25-40 gallons) of liquids (1-500 centipoises) where simple agitation is desired. It can also be used in very heavy dispersion work where the batch is small (under five gallons). In this type of process the impeller is run very close to the bottom of the tank, usually not more than two inches from the bottom. It should not be run faster than 5010 revolutions per minute.

The 6-inch diameter double effect impeller is used at speed ranges of from 1800 to 5600 feet per minute. This impeller is often substituted for the 4-inch impeller when the latter fails to maintain circulation at the desired consistency. Do not run faster than 5650 feet per minute.

The 6-inch diameter single effect impeller can be used nearer the bottom of the tank and permits the use of smaller batches. Do not run faster than 5650 feet per minute.

The 4-inch diameter impeller is usually the one tried first. Speeds of approximately 4000 feet per minute are most popular (see Table I for recommended speeds). Never exceed 4320 r.p.m. Table I suggests impellers and speeds for use in fluids according to the viscosity.

The limiting factors on batch sizes are viscosity and motor load. Higher speeds are used for hard dispersion or disintegration work. In general, the Dissolver is run at impeller rim velocities of 2500 to 5000 feet per minute. The 5-VT Dissolver will generally handle batches from 5 to 40 gallons at viscosities of approximately 50,000 to 100 centipoises, respectively.

Table II, Operating Data, gives the sheave (V-belt pulley) sizes to be used to get different impeller speeds.

TABLE I

IMPELLER - SPEED - VISCOSITY - BATCH SIZE

	<u>1-2500 cps.</u>	<u>2500-5000 cps.</u>	<u>5000-10,000 cps.</u>	<u>10,000-20,000 cps.</u>	<u>20,000-30,000 cps.</u>	<u>Above 30,000 cps.</u>
<u>RPM</u>						
4500- 6000	B-1504	B-1506 B-1504	B-1506 B-1504			
3600- 4500	B-1504 B-1506	B-1504 B-1506	B-1504 B-1506	B-1506	B-1506	
3000- 3600	B-1506 B-1504	B-1504 B-1506	B-1504 B-1506	B-1506 B-1508	B-1506 B-1508	B-1506 B-1508
Below 3000	B-1504 B-1506 B-1508	B-1504 B-1506 B-1508	B-1506 B-1508	B-1506 B-1508	B-1508	B-1508

TABLE II
OPERATING DATA

<u>Motor</u>	<u>Sheave Data</u>		<u>Shaft Data</u>	<u>Impeller Speed - Ft./min.</u>		
	<u>Driven</u>	<u>Ratio</u>		<u>4-in. dia.</u>	<u>6-in. dia.</u>	<u>8-in. dia.</u>
4	12	.333	1200	1260	1890	2510
4	10	.400	1440	1515	2260	3020
6	12	.500	1800	1890	2820	3760
6	10	.600	2160	2270	3400	4520
4	6	.666	2400	2520	3960	5010
10	12	.833	3000	3150	4700	
6	6	1.000	3600	3780	5650	
12	10	1.200	4320	4420		

PROCEDURE

The Cowles Dissolver was fitted with the 4-inch diameter double acting impeller and the speed set at 4320 r.p.m. by using a 12-inch diameter V-belt sheave on the motor and a 10-inch diameter sheave on the driven shaft for a sheave ratio of 1.200.

The tank used was 16 inches in diameter and 16 inches high and had a capacity of 13.8 gallons.

The clay used was "Premax", a premium quality clay from Georgia Kaolin. The brightness is in the range of 87-89 and the particle size is 95% below 2 microns.

Clay slips were prepared, duplicating the formulations of the clay slips used in studying the other makes of laboratory dispersing equipment described in Report 29 of Project 1956. The slips were tested for solids, density, viscosity (at equal solids), and particle size (electron microscope and rate of settling).

EXPERIMENTAL

EFFECT OF TIME OF MIXING ON VISCOSITY

Formulation 1819-64

<u>Material</u>	<u>Parts by weight, g.</u>
"Premax" clay	12,000
Quadrafos	42
Water	5,160

The ingredients were mixed by adding the clay slowly to the Quadrafos solution which was under agitation using the Cowles Dissolver

at the given settings. It took a few minutes to add the clay. The batch was mixed an additional ten minutes and the solids content and viscosity checked.

Solids content: 69.86%

Brookfield Model LV viscosity, Spindle 1

<u>RPM</u>	<u>Cp.</u>
6	372.0
12	237.5
30	137.0
60	94.5

In order to compare the viscosity with the viscosities of the previous type dispersed clay slips of Report 29, Project 1956, the formulation was diluted to the common solids content of 69.6% and the viscosity checked.

Brookfield Model LV Viscosity		
<u>RPM</u>	<u>Spindle 1</u> <u>cp.</u>	<u>Spindle 2</u> <u>cp.</u>
6	318.0	325.0
12	202.5	212.5
30	119.0	125
60	82.5	80

Formulation 1819-65 is the same as Formulation 1819-64 but was mixed an additional 30 minutes and the viscosity and solids content determined.

Solids content: 70.19%

Brookfield Model L V viscosity, Spindle 1

<u>RPM</u>	<u>Cp.</u>
6	320.0
12	202.5
30	116.0
60	78.5

The formulation was diluted to a solids content of 59.6% and the viscosity checked.

<u>RPM</u>	<u>Spindle 1</u> <u>cp.</u>	<u>Spindle 2</u> <u>cp.</u>
6	290.0	310.0
12	185.0	187.5
30	108.0	110.0
60	74.5	77.5

MAXIMUM SOLIDS

A study was made of the capability of the Cowles Dissolver to prepare clay slips of high solids contents.

Formulation 1819-71:

Water, 30 lb., and enough Quadrafos (0.26 lb.) to accommodate about 75 lb. Premax clay at a concentration of 0.35% Quadrafos per clay solids was mixed by the Cowles Dissolver. The clay was added slowly. After about 74 lb. of clay had been added, trouble developed in the belt drive. The belt tended to twist inside-out and ride off the sheaves. The sheaves were slightly out of line. They were shimmed into alignment and the belt was pulled extremely tight. The amperage required for this load was about 3.5 to 4.5 as measured by a clamp-on ammeter. The motor was rated at 8.5 amps.

The formulation was mixed an additional 40 minutes over the 10 to 15 minutes required to add the clay. The viscosity and solids content were determined.

Solids content: 70.82%

Brookfield Model LV viscosity, Spindle 2

<u>RPM</u>	<u>Cp.</u>
6	440.0
12	280.0
30	165.0
60	117.5

Formulation 1819-73:

Additional clay was added to Formulation 1819-71 with mixing. A small amount of clay, 50-100 g., was added at a time. A deposit collected on the side of the bucket in the form of small, undispersed clumps of clay. After 5-15 minutes the deposit was seen to disappear. This was continued until an additional 7-8 pounds of clay had been added. Upon examination of this formulation after about 4-5 hours total adding and mixing time, it was noticed that there were many small undispersed particles throughout the coating.

Water was then added in small quantities and mixing continued until the small, undispersed particles in the coating seemed to disappear. The total mixing time was about eight hours. The solids content and viscosity were determined.

Solids content: 73.5%

Brookfield Model LV viscosity, Spindle 3

<u>RPM</u>	<u>Cp.</u>
6	5900
12	4000
30	1980
60	1300

The formulation was diluted to a solids content of 69.6% and the viscosity checked.

<u>RPM</u>	<u>Spindle 2</u> <u>cp.</u>
6	810.0
12	600.0
30	372.0
60	262.5

After close examination of the coating at 73.5% solids, it appeared that some tiny grit-like particles were still present.

Formulation 1819-75:

Formulation 1819-73 was further mixed and diluted by steps until after several hours the clay slip appeared to have a smooth grit-free texture. The solids content and viscosity were determined.

Solids content: 72.24%

Brookfield Model LV viscosity, Spindle 2

<u>RPM</u>	<u>Cp.</u>
6	3900
12	2520
30	1300
60	796

The formulation was diluted to a solids content of 69.6% and the viscosity checked.

<u>RPM</u>	Spindle 2 <u>Cp.</u>
6	825
12	600
30	370
60	250

The viscosity values of the above prepared clay slips are given in Table III for comparison with the viscosity values of the clay slips prepared with the other types of laboratory dispersers described in Report 29, Project 1956.

TABLE III

CHARACTERISTICS OF CLAY SLIPS DISPERSED UNDER VARIOUS CONDITIONS

Formulation	Dispersing Device	Total Mixing Time, Minutes	Solids, % ³	Viscosity, cp. ⁴		Particle Size		Density
				Spindle 1	Spindle 2	% below 1 micron	% below 0.5 micron	
1819-73	Cowles Dissolver	400-500	73.5	--	262.5	100	87	--
1819-75	Cowles Dissolver	600-700	72.24	--	250	-	-	--
1819-23	Ball Mill	120	69.7	100+	112.5 ²	95	82	--
1879-160	Lightnin' mixer	60	70.2	96	108 ²	95	82	--
1819-27	Baker Perkins	120	70.8	88	92.5 ²	100	88	--
1819-25	Homo-Mixer	45	69.9	81	86 ²	96	83	--
1819-64	Cowles Dissolver	10	69.86	82.5	80	-	-	1.752
1819-65	Cowles Dissolver	40	70.19	74.5	77.5	97	82	1.756
1819-39	Charlotte Colloid Mill	2 ¹	70.0	65.1	70 ²	97	82	--
1879-153-6	Kady Mill (low speed)	41	69.6	61.5	66 ²	95	82	--
1879-156-5	Kady Mill (high speed)	60	69.9	57.5	62.5 ²	-	-	--
Control	Stirred by hand with spatula	-	69.8	--	--	88	68	--
1819-68		-	--	--	--	-	-	1.767
1819-70		-	--	--	--	-	-	1.749

Time in minutes to pass the formulation through the colloid mill. Time to prepare the formulation is not counted.

From Table I, Report No. 29, Project 1956.

These are the solids content values of the formulations as mixed. All of the formulations were reduced to the solids content of 69.6% before determining viscosity.

Brookfield Model LV viscosity at 60 r.p.m., solids adjusted to 69.6% solids.

RESULTS AND OBSERVATIONS

The 20 h.p. Kady Mill produced clay dispersions having the lowest viscosities. The Charlotte Mill gave the next lowest viscosity followed by the 70% dispersions made in the Cowles Dissolver, Homo-Mixer, Baker Perkins Mixer, Lightnin' Mixer, and the Abbe Ball Mill respectively. The high solids (72.2 and 73.5%) dispersions made with the Cowles Dissolver were highest in viscosity on dilution to a common solids content. This may be due to insufficient electrolyte for deflocculating the clay in a fine state of subdivision or to some other effect such as entrainment of air.

The Baker Perkins sigma blade mixer produced dispersions with the highest per cent of fine particles. However, the mixing time was long, the consistency was critical, and the remaining large particles were bigger than the large particles in some dispersions produced with impact types of mills. At least four hours of kneading appears to be desirable for good breakdown of the clay.

Particle size reduction in general is a function of the amount of work expended. For a true comparison this factor should have been kept as a common base; however, some compensation for this aspect was included by varying the time over a wide span with some of the dispersers. Also the large horsepower units were evaluated with correspondingly (or roughly so) larger size batches.

From the overall point of view the Cowles Dissolver showed up very well as a laboratory unit. It was capable of producing dispersions with small size particles. Its batch size range was broad and encompassed the requirements of a pilot size coating machine.

The Kady Mill used has an elevated rotor and could not be used successfully if the amount of material used did not cover the rotor. The Kady Mill Model O (not the small laboratory mill), on the other hand, has a real advantage in that it can be used for preparing the final coating color including the adhesive without entraining air, it is claimed. Indeed, it has been suggested for use for deaerating on-machine coating color.

The Charlotte Mill did not work well with high solids content clay dispersions, unless the gap between the rotor and stator was increased to about 0.03 inch. It performed well on more fluid mixes; such as might be used with an air doctor.

The Homo-Mixer, being an impact type of mixer, would probably show up better if the time of mixing had been increased and the gap between the stator and rotor regulated.

Future work should probably include the "relative sediment volume." According to Robinson (1) the relative sediment volume is completely independent of particle size, although affected by the ratios of particle dimensions and proportions present. He also notes that a high relative sediment volume of very small particles is an indication of a high degree of particle agglomeration just as it is with large particles.

REFERENCES CITED

1. Robinson, J. V., Tappi, 42, No. 6, pages 432-438.
2. Parts List and Instructions for Installing and Operating Model 5-VT Cowles Dissolver, Morehouse-Cowles, Inc., 1150 San Fernando Road, Los Angeles 65, California.
3. Preparation of Paper Coating Colors, Tappi Monograph Series, No. VI.
4. Asdell, Bernard K., The Rheological Properties of Clay-water Suspensions, Paper Mill News, June 26, 1948.

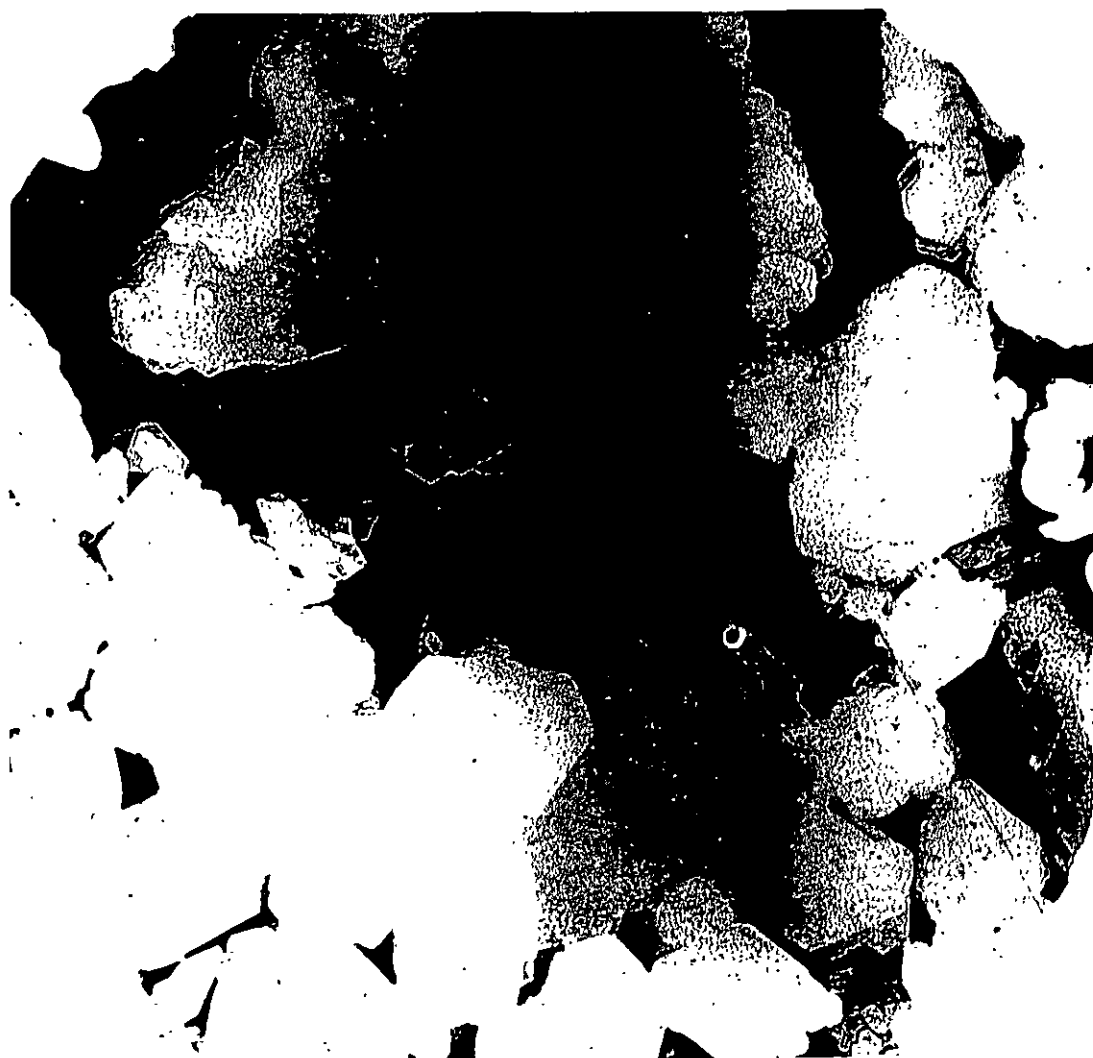


Figure 1

1679-160
Premax Clay, 70.2% Solids
Mixed with Model V Lightnin Mixer for 30 Minutes
22,000X



Figure 2

1919-25
Premax Clay, 69.9% Solids
Mixed with a $\frac{1}{4}$ h.p. Homo-Mixer for 20 Minutes
(45 minutes total time)
22,000X

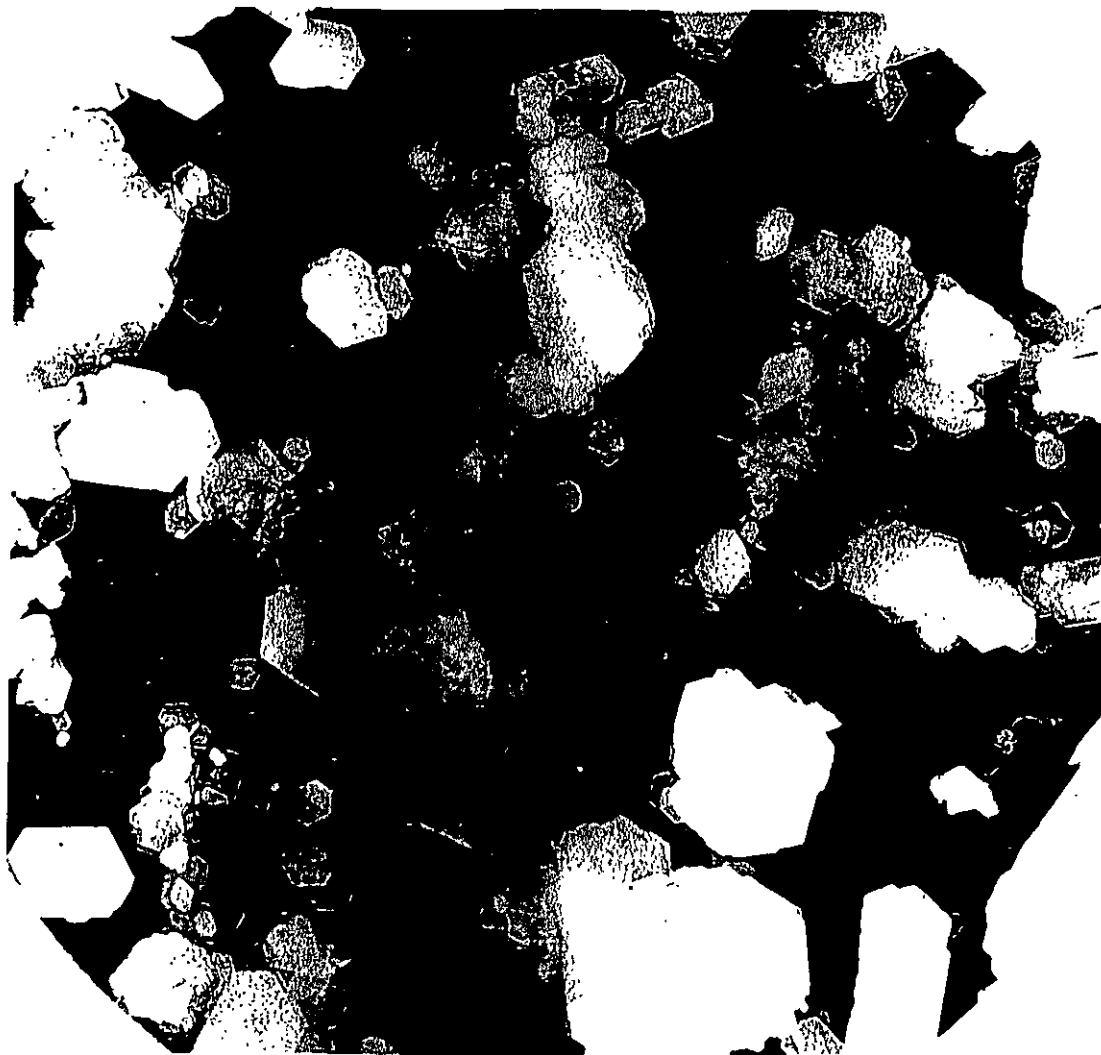


Figure 3

1819-39
Premax Clay, 70% Solids
3 Passes Through Charlotte Mill set at 0.036" Gap
22,000X

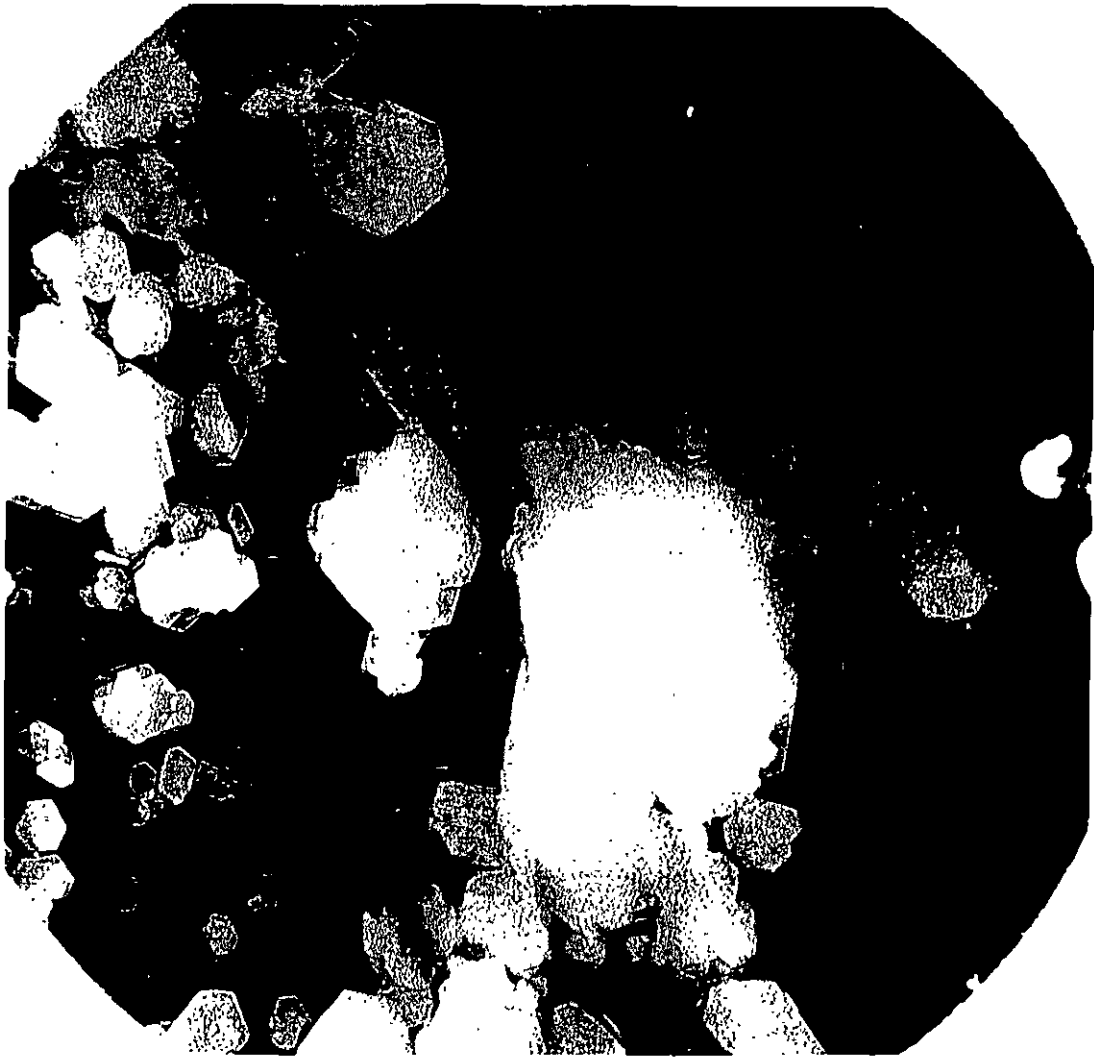


Figure 4

1219-21
Premax Clay, 70.1% Solids
Mixed at 72.5% Solids for 1½ Hours
In Baker Perkins Sigma Blade Mixer
22.00°C

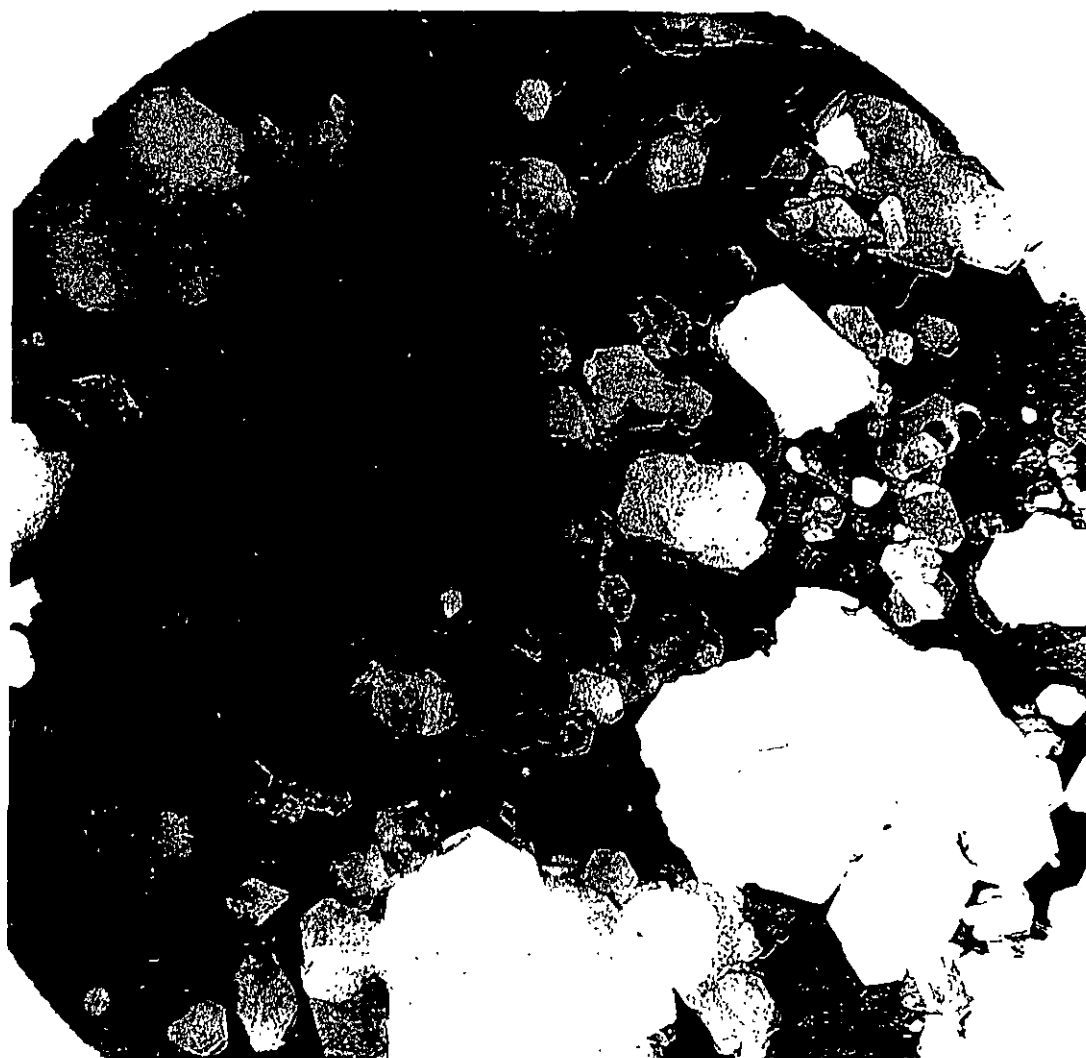


Figure 5

1919-27
Promax Clay, 76.6% Solids
Mixed in Baker Perkins Sigma Blade Mixer for $1\frac{1}{2}$ Hours
22,000X

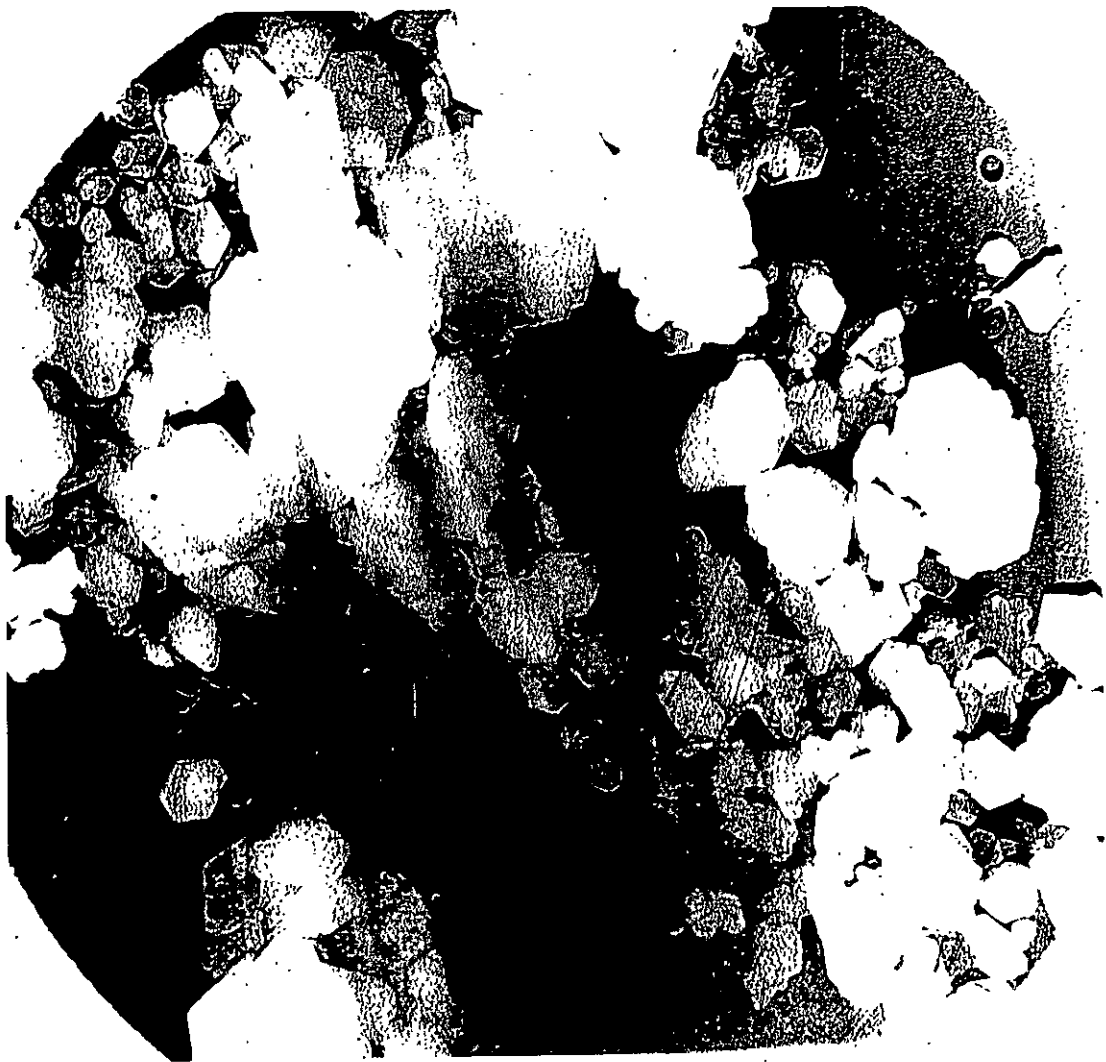


Figure 6

1919-23

Premax Clay, 69.7% Solids

Ground for 2 Hours in a 1-gallon Abbe Ball Mill

20,000X

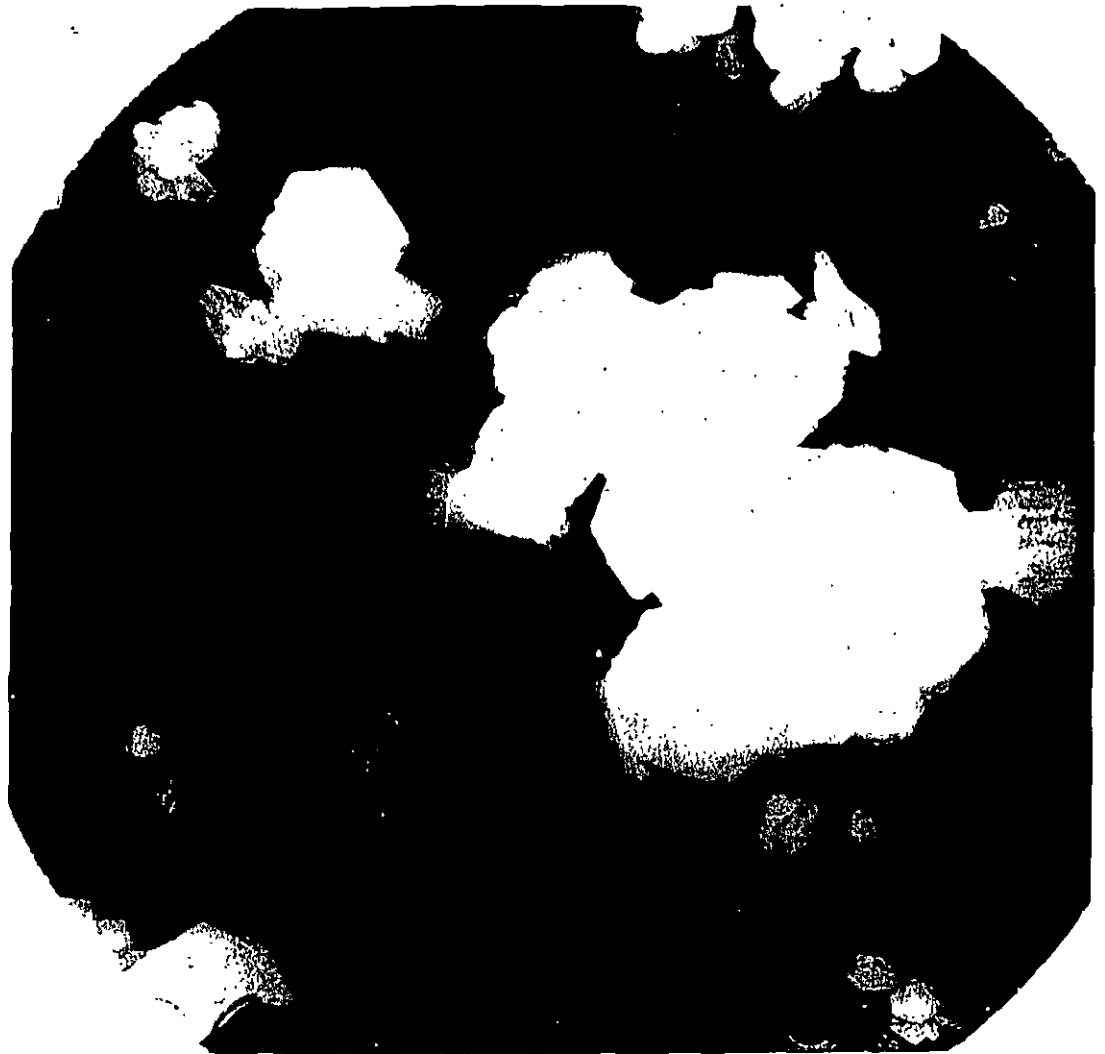


Figure 7

1679-153-1
Premax Clay, 69.6% Solids
Mixed for 5 Minutes in Kady Mill, 3000 r.p.m.
22,000X

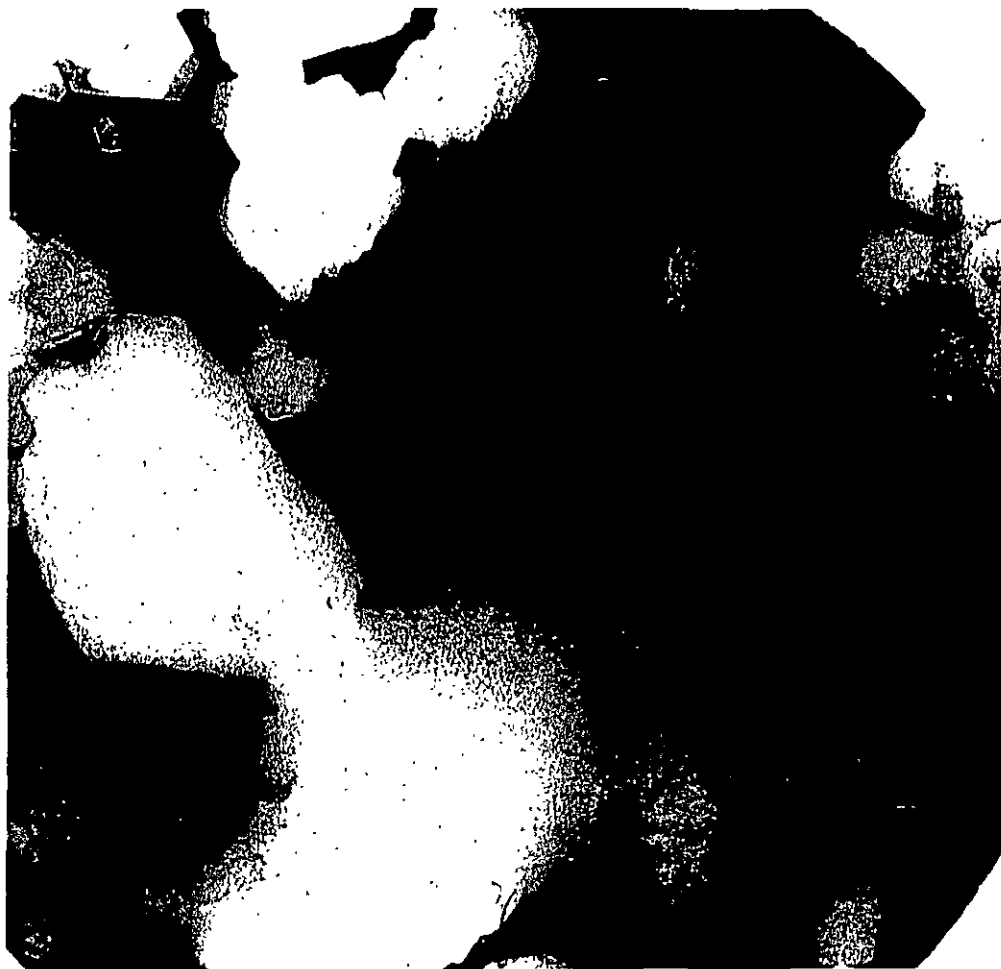


Figure 8

1679-153-2
Premax Clay, 69.6% Solids
10 Minutes in Kady Mill, Model C
22.000X

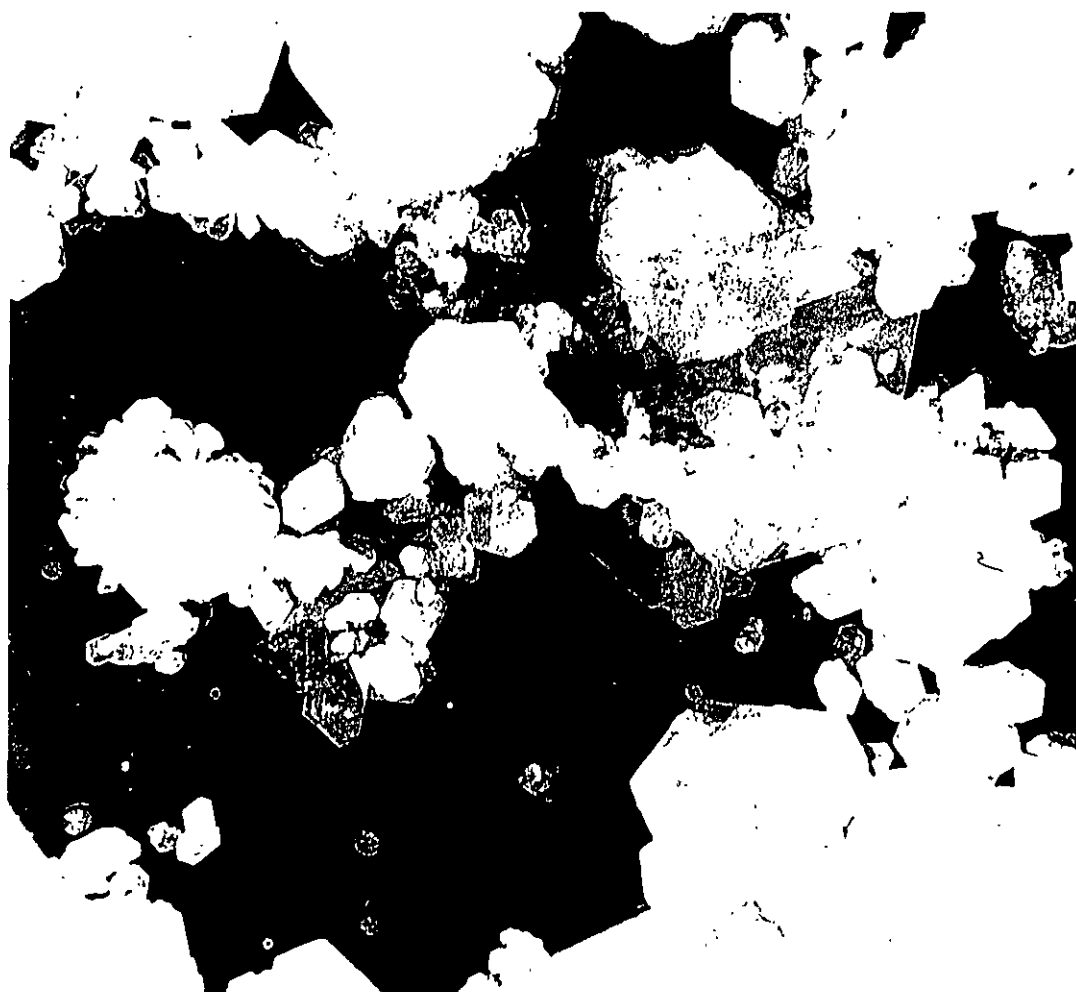


Figure 9

1679-153-3

Premax Clay, 69.6% Solids

Ground in Kady Mill for 15 Minutes at 3000 r.p.m.

20,000X

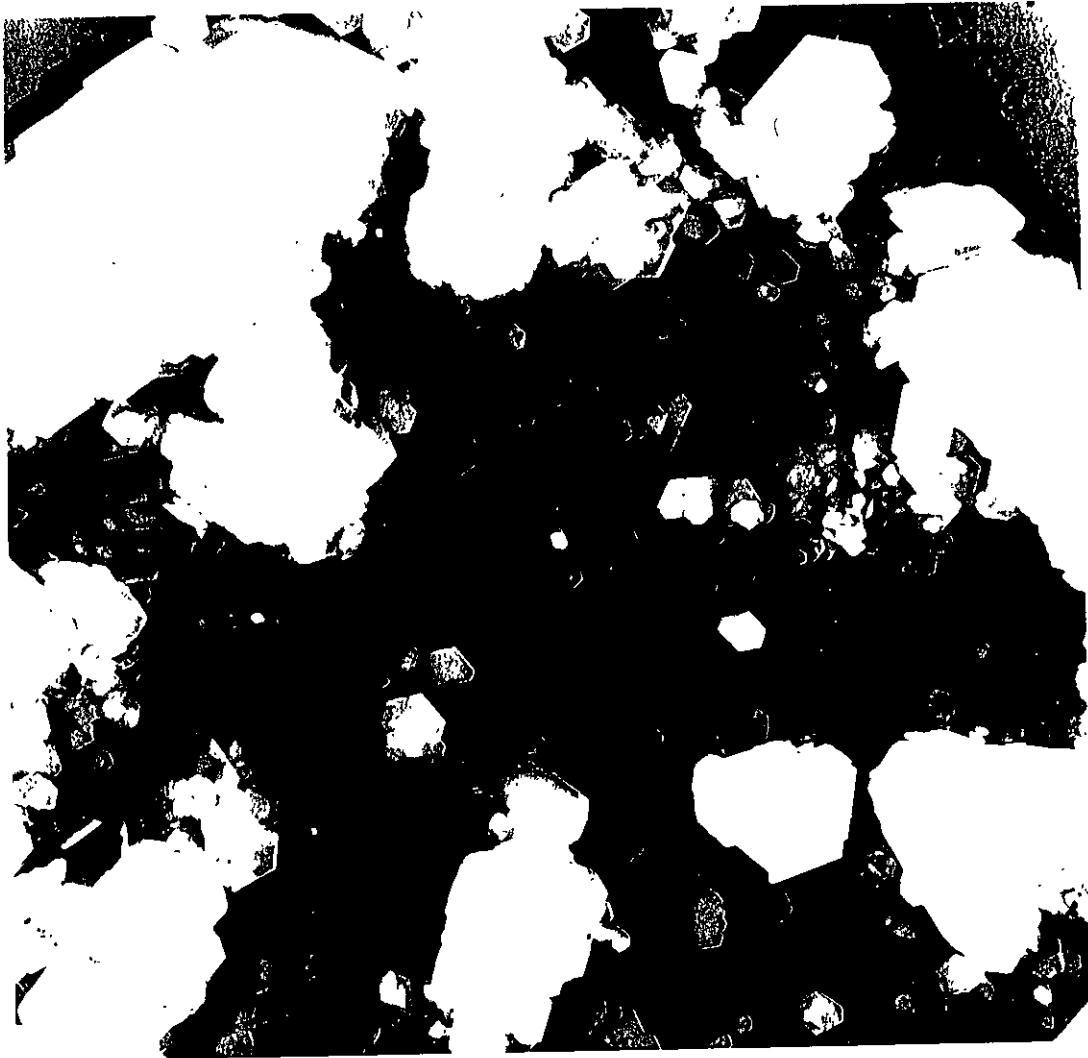


Figure 10
1679-153-4
Premax Clay
20 Minutes in Kady Mill
22,000X

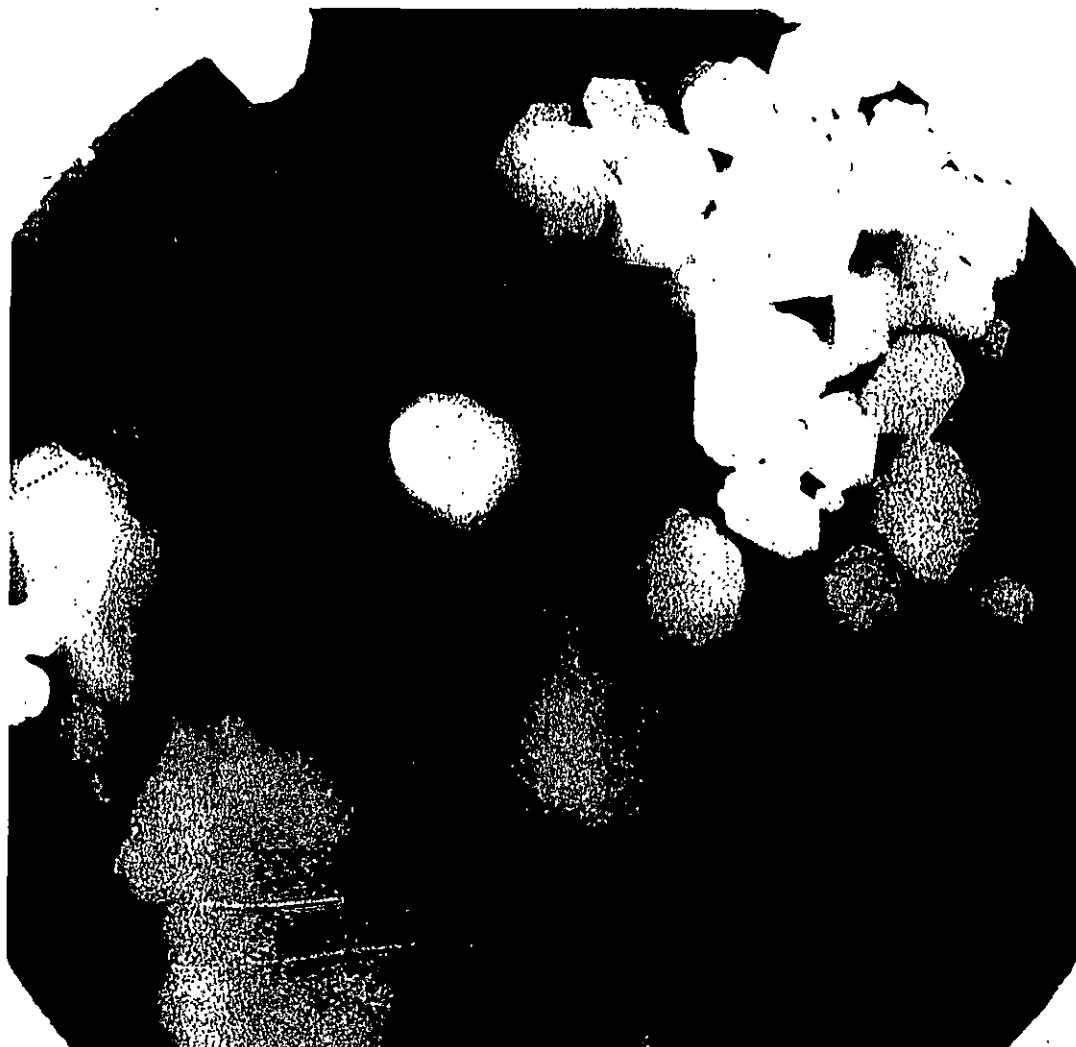


Figure 11

1679-153-5
Premax Clay, 69.6% Solids
Mixed in Kady Mill Model C, 25 minutes at 3000 r.p.m. (slow speed)
22.000X

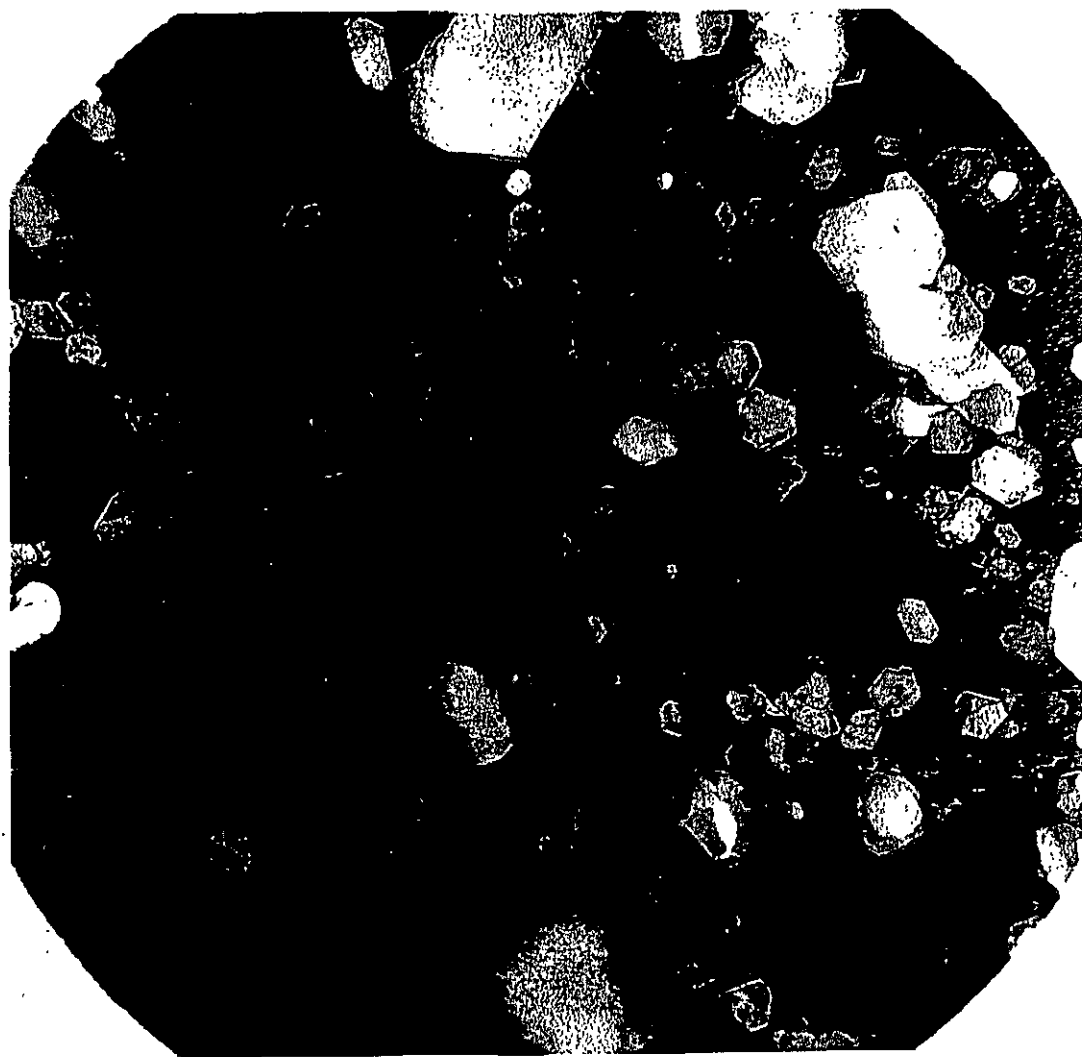


Figure 12

1673-153-6
Premax Clay, 69.6% Solids
Mixed for 30 Minutes in Kady Mill
22,000X

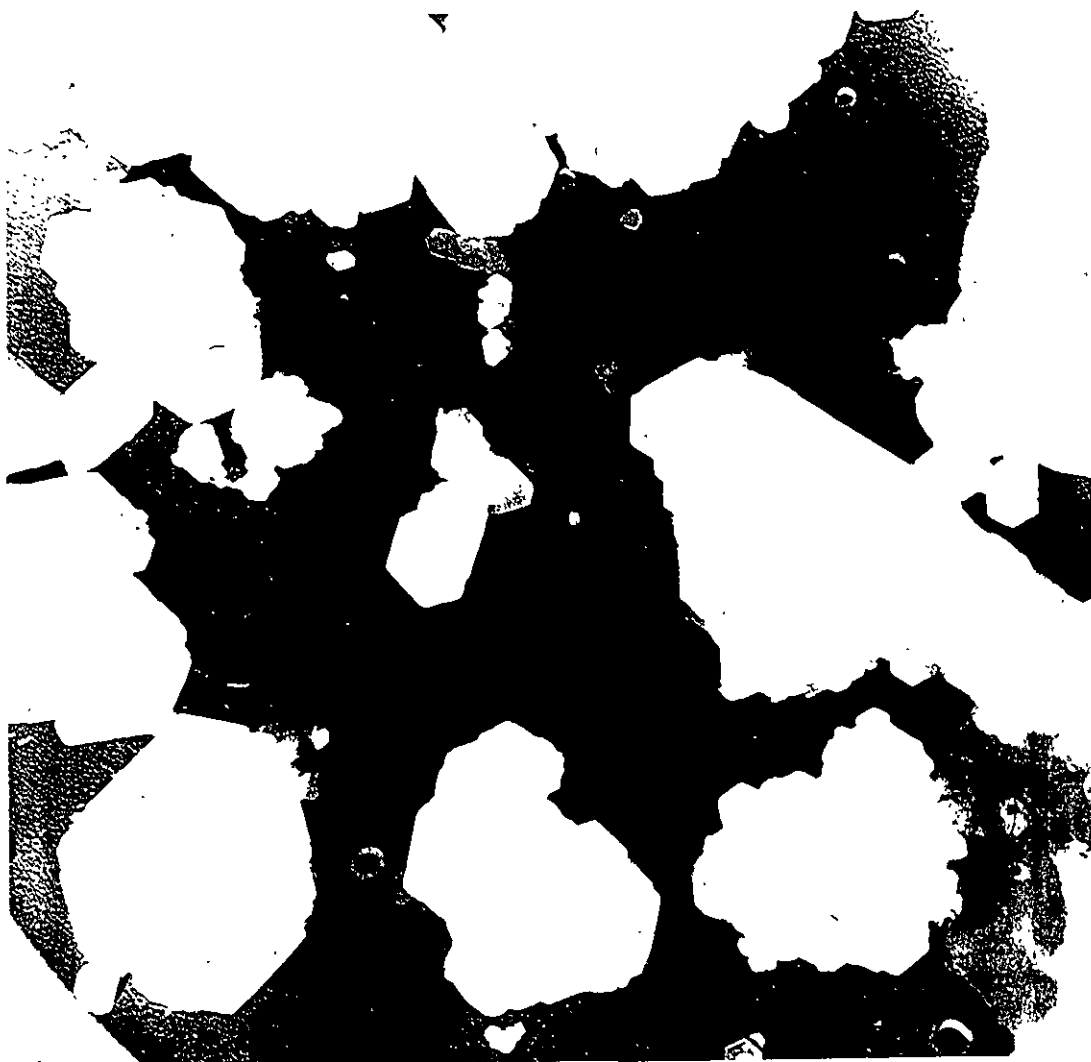


Figure 13

1679-156-1

Premax Clay, 77.1% Solids
5 Minutes in Kady Mill at 6200 r.p.m.
22.000X



Figure 14

1679-156-2
Premax Clay, 70.2% Solids
11 Minutes in Kady Mill at 6200 r.p.m.
22,000X

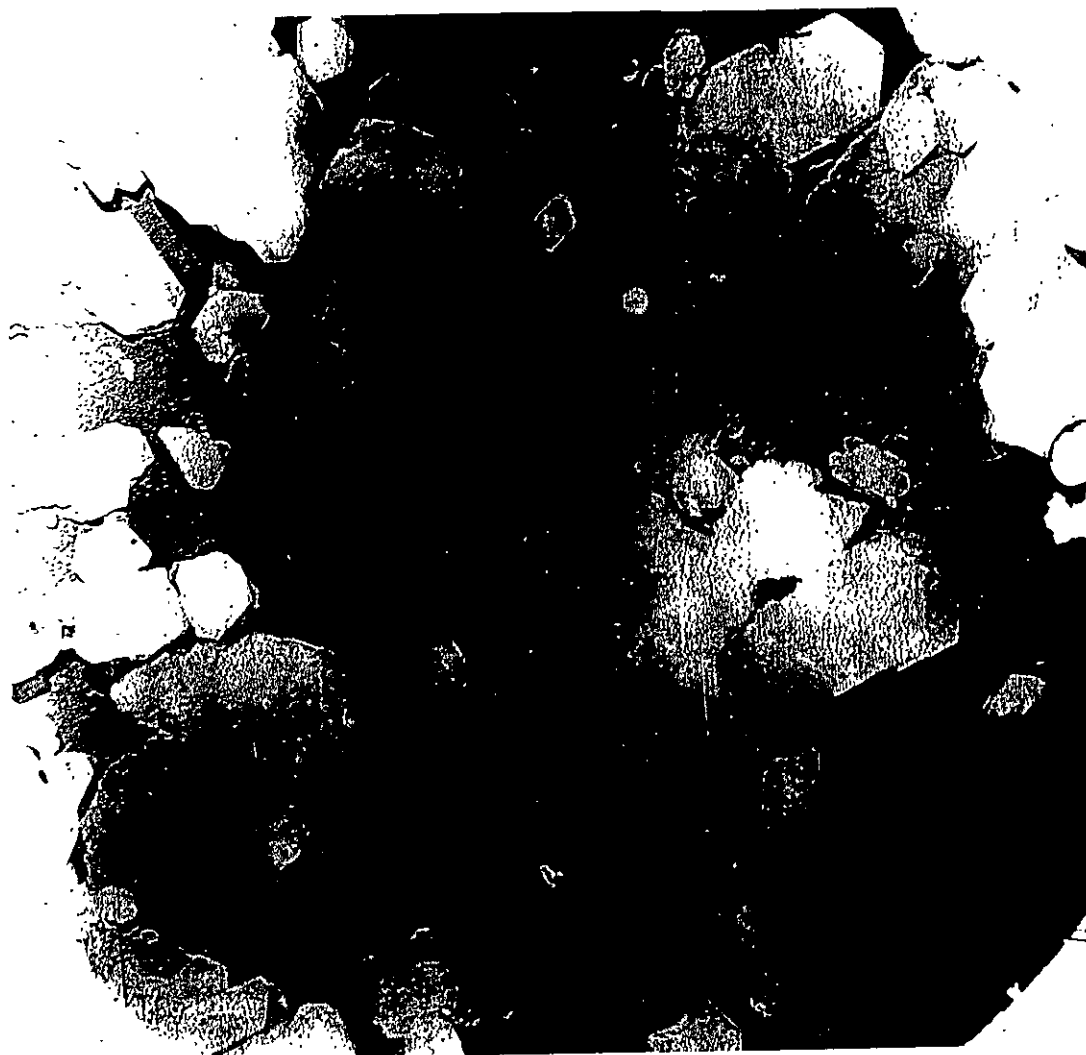


Figure 15

1679-156-3
Premax Clay 69.2% Solids
16 Minutes in Kady Mill, 6200 r.p.m.
22.000X



Figure 16

1679-156-4
Premax Clay, 70% Solids
21 Minutes in Kady Mill at 6200 r.p.m.
22,000X

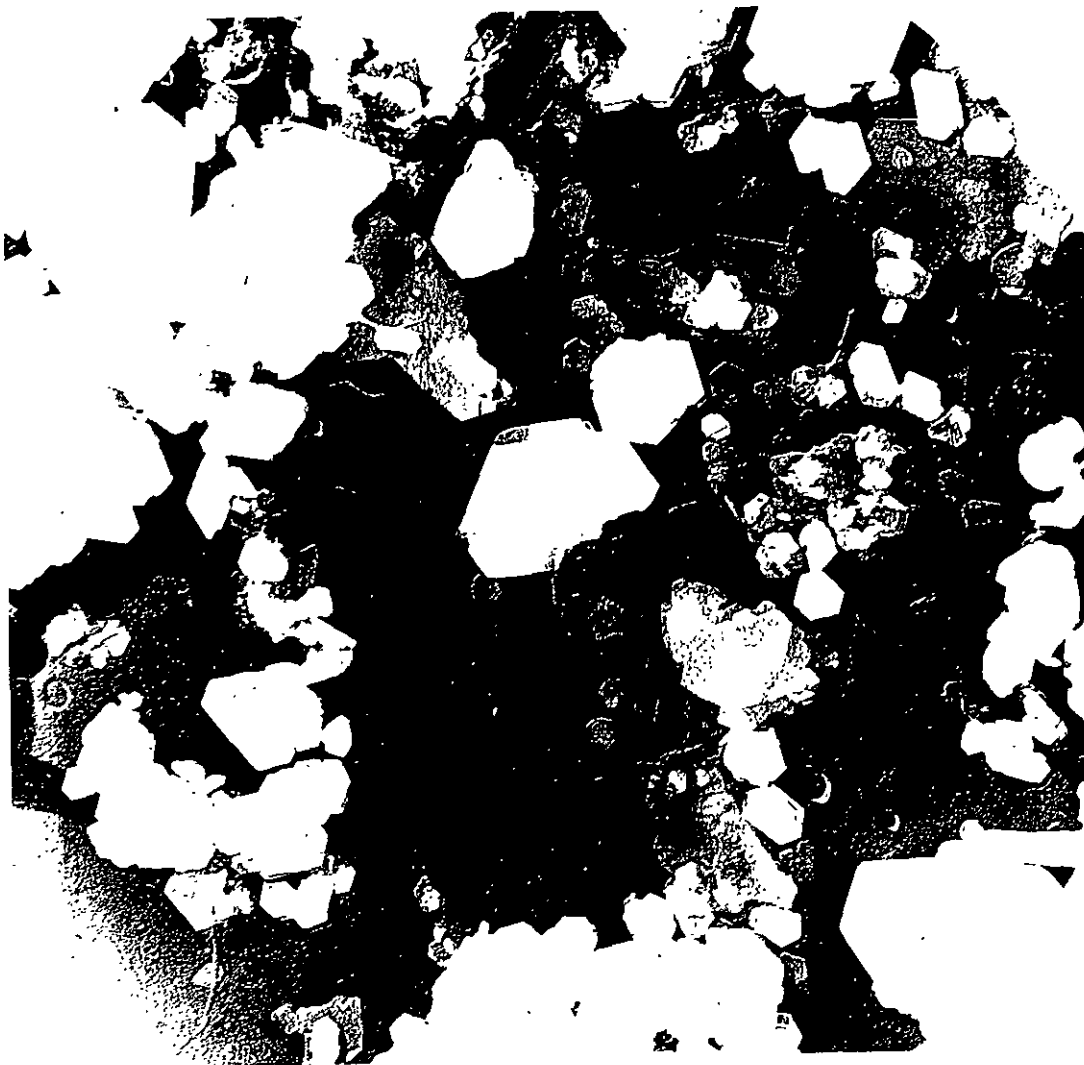


Figure 17

1679-156-5
Premax Clay, 70% Solids
26 Minutes in Kady Mill at 6200 r.p.m.
22,000X

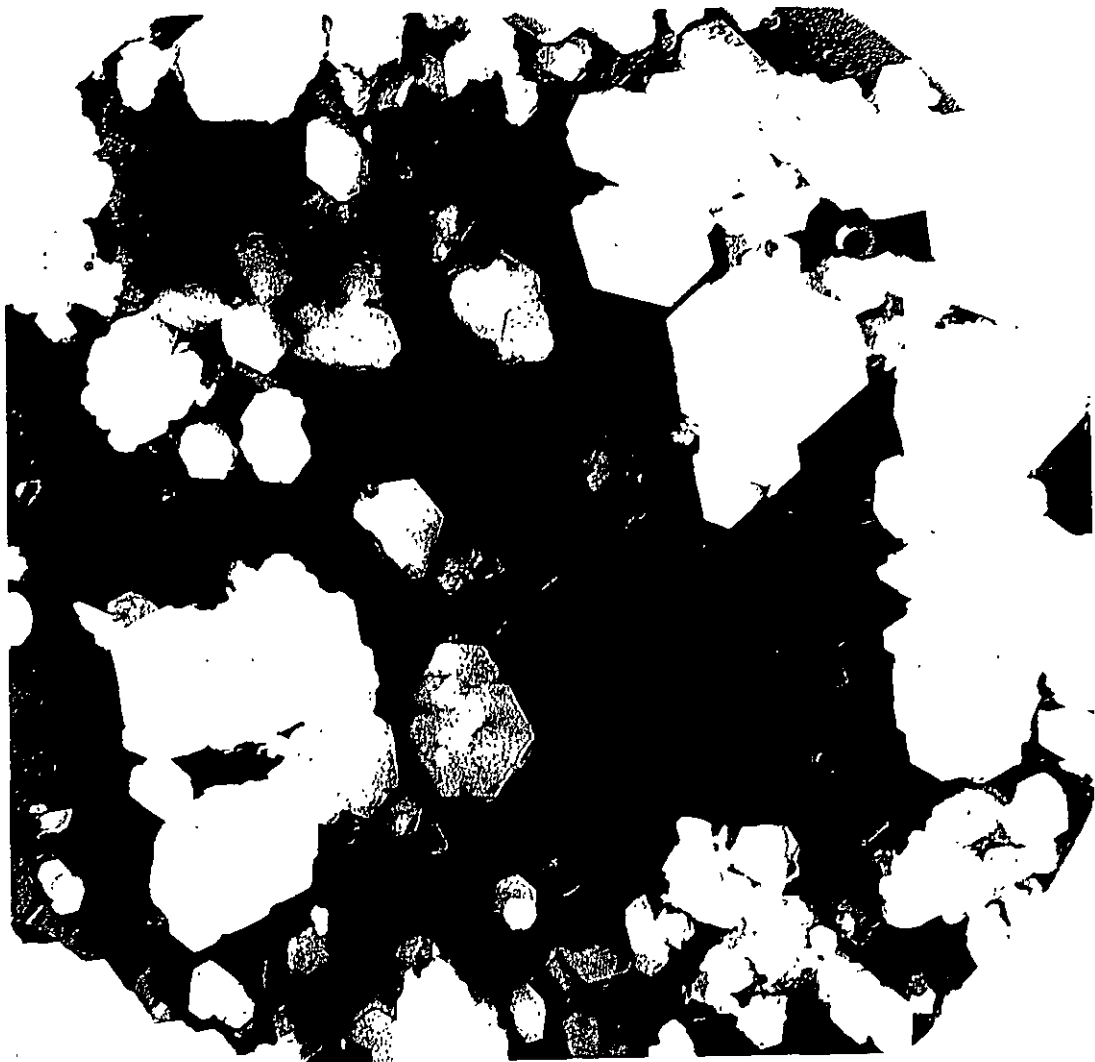


Figure 18

1679-156-6
Premax Clay, 70.0% Solids
Mixed in Kady Mill 31 Minutes at 6200 r.p.m.
22,000X

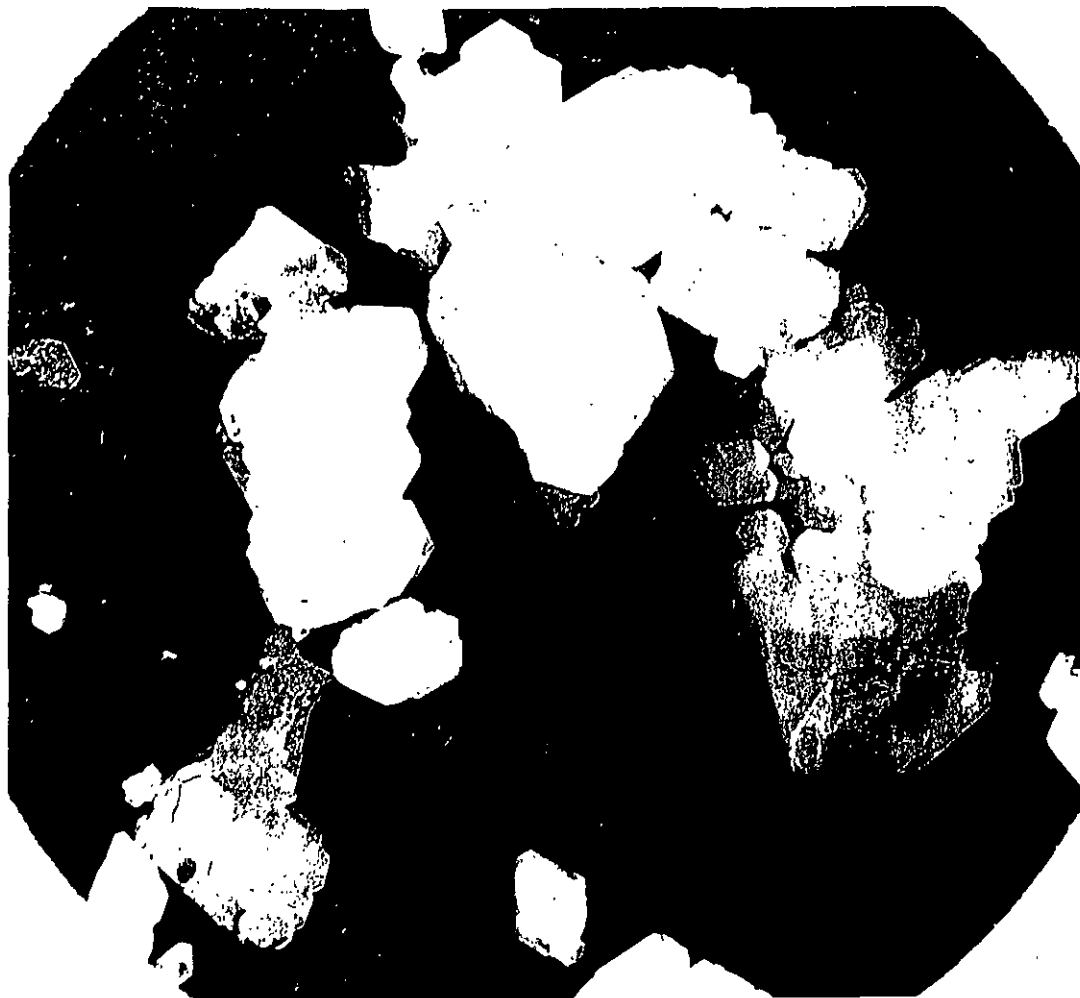


Figure 19

1679-137
Premax Clay, 71.2% Solids
Ground in Kady Mill for 7¹/₂ Minutes (Dilutant)
22,000X

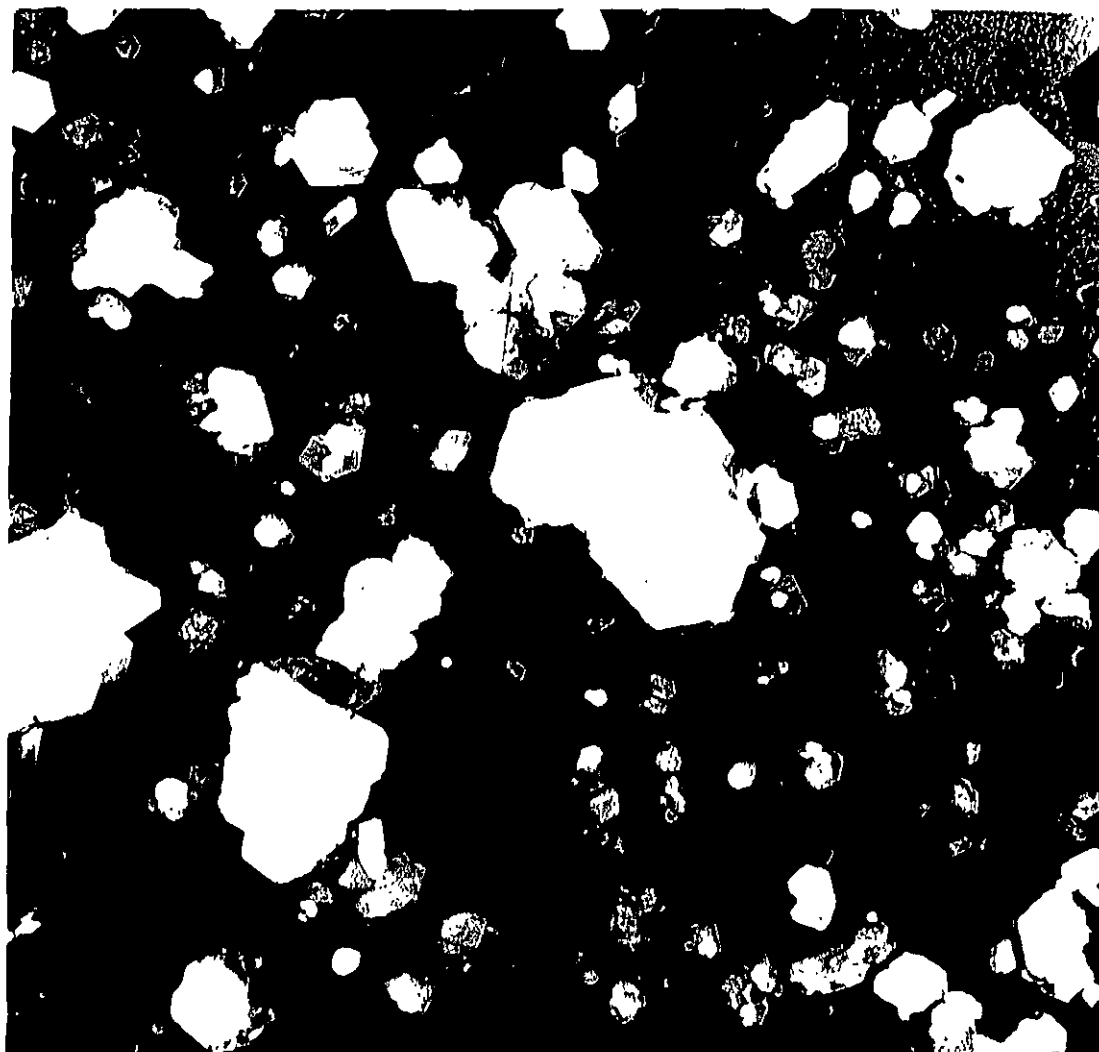


Figure 20

1819-64

Premax Clay, 79.9% Solids

Mixed with a Cowles Dissolver for 10 Minutes

22,000X

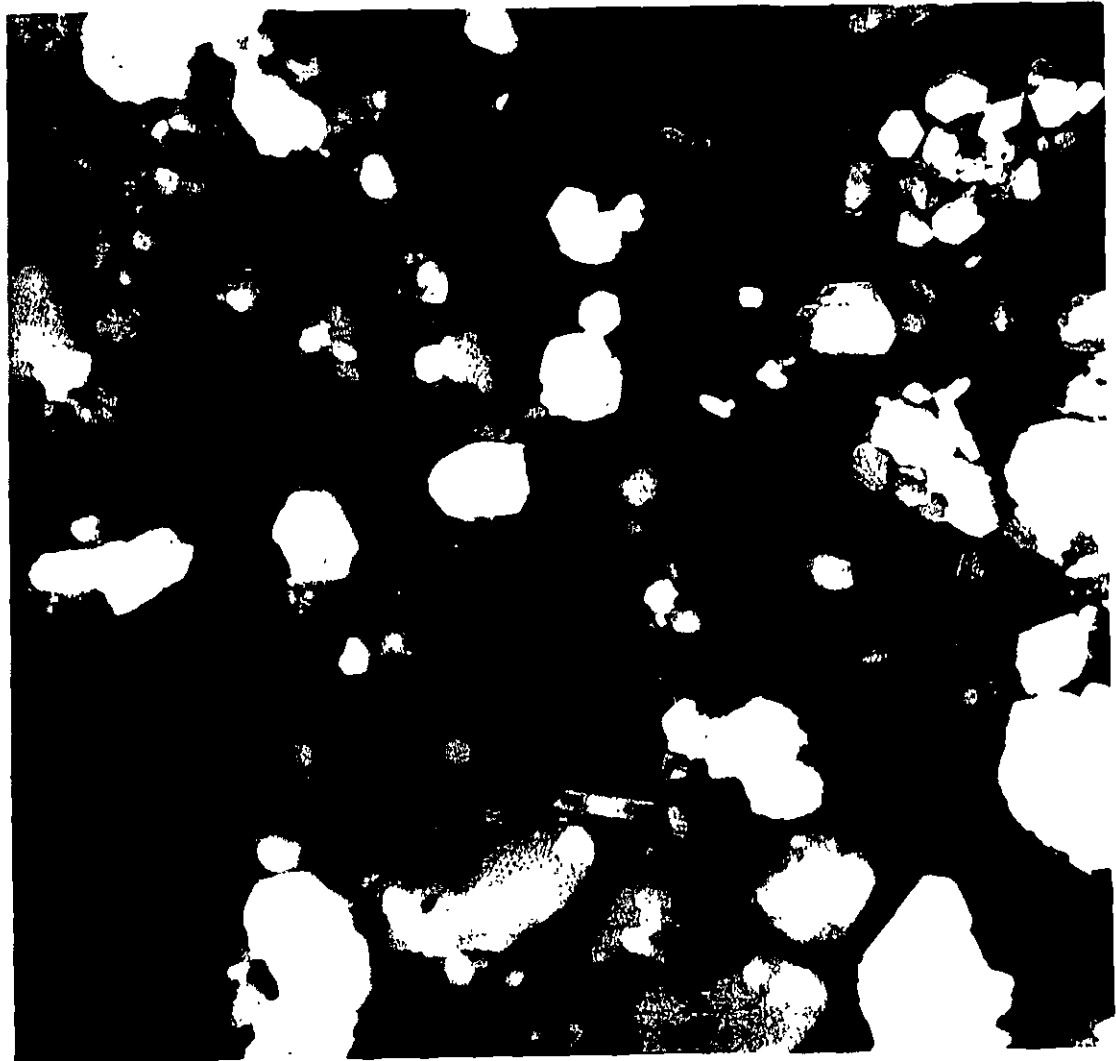


Figure 21

1919-65
Premax Clay, 70.19% Solids
Dispersed with a Cowles Dissolver for 40 Minutes
20.000X

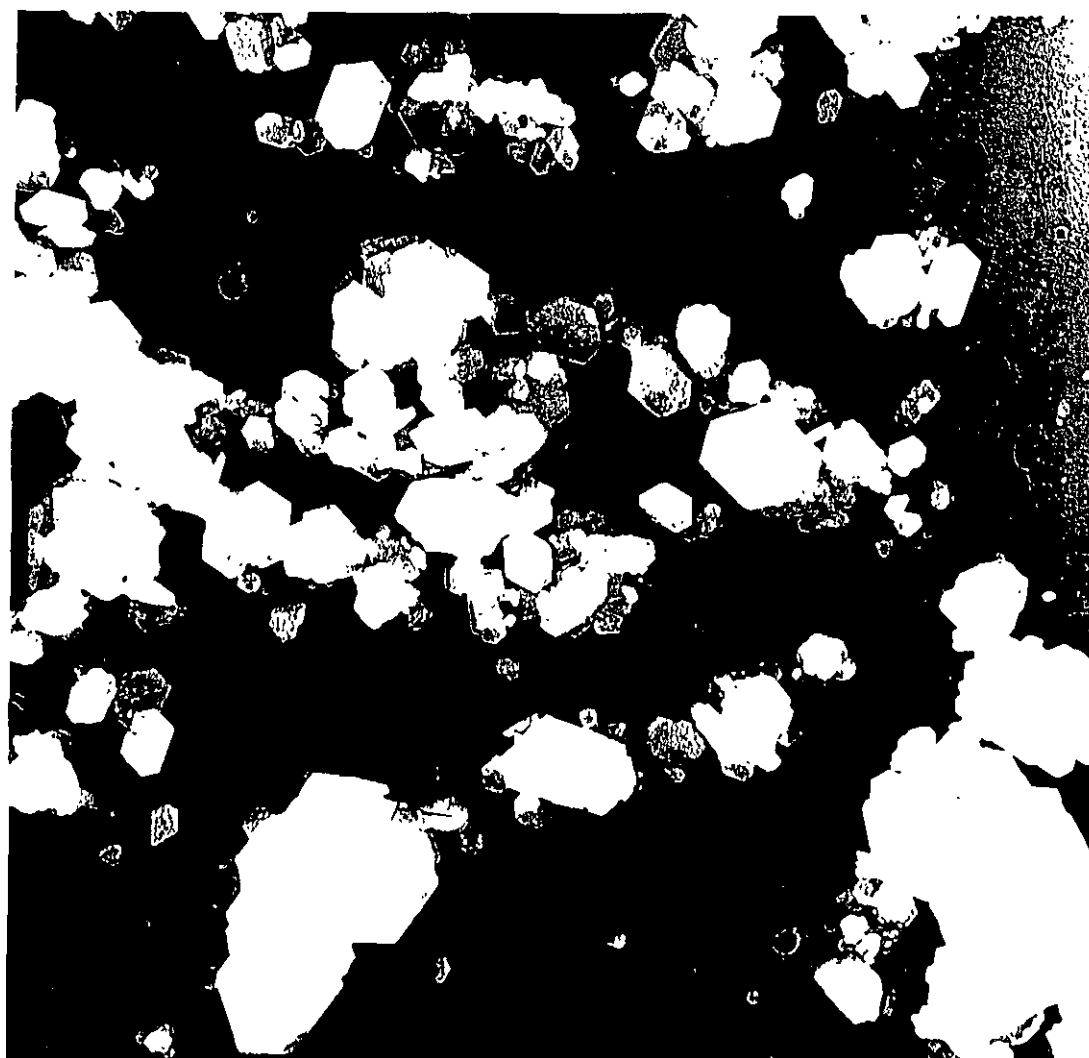


Figure 22

1819-71
Fremax Clay, 70.8% Solids
Mixed for 40 Minutes with Cowles Dissolver
22,000X

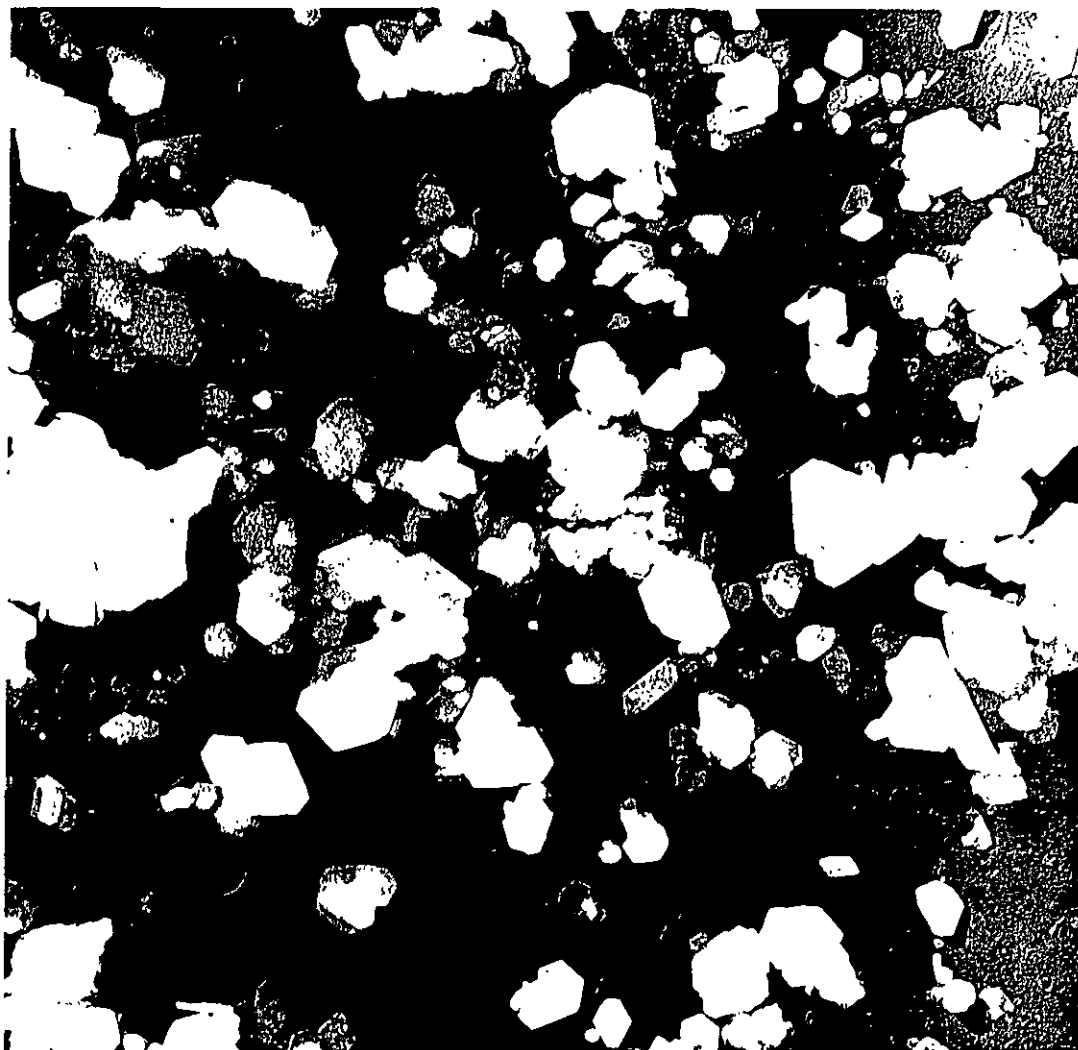


Figure 23

1919-75
Premax Clay, 72.2% Solids
Dispersed with Cowles Dissolver over 10 Hours
22.000%

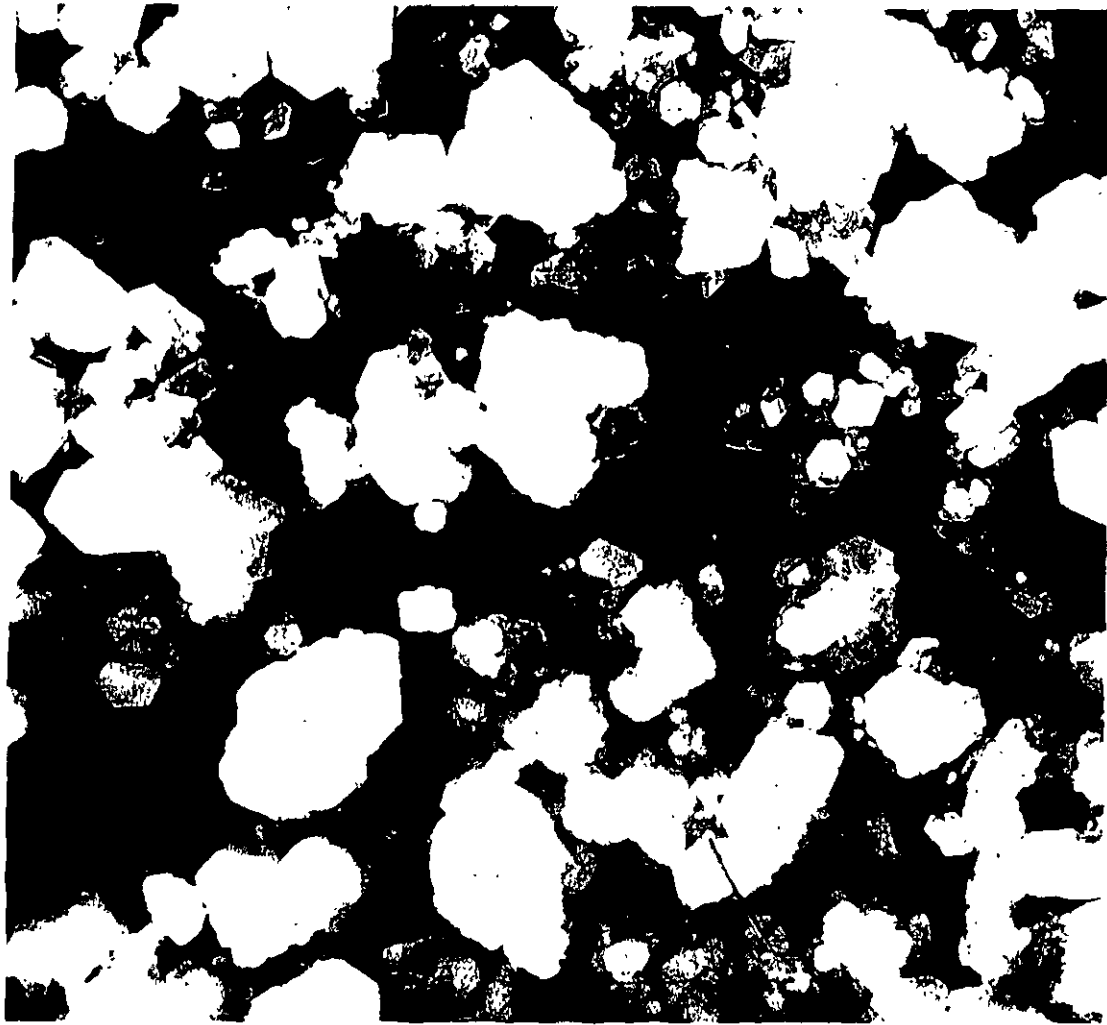


Figure 24

1919-73
Premax Clay, 73.5% Solids
Dispersed with a Cowles Dissolver for 8-9 Hours
22,000X