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EXAMINATION OF LOCAL KAOLIN FOR USE AS PAPER FILLER

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ABSTRACT

Tests of a sample of raw kaolin from Burma were conducted as subcontract of the UNIDO Project DP/BUR/85/017 "Examination of Local Kaolin for Use as Paper Filler". The kaolin was classified by washing and the clay fraction under 20 μm was tested for the above purpose. In the frame of tests the possibility of using kaolin as filler in the rubber industries and for selected ceramic applications was also evaluated.

As the sample represented a raw material of low quality for paper making costly and complex upgrading methods were applied without a significant effect. There remains one way how to reduce the high abrasivity which consists in testing the kaolin as a component of composite blends which could lead to a significant reduction of necessary imports.

The results of tests of Burmese kaolins "Shwe Daung" and "Wellan" previously carried out by the Research Institute for Ceramics, Refractories and Non-metallic Raw Materials, Pilsen, were considered when recommending the appropriate actions to find and verify other substitutes for improved paper fillers and to find economical applications for the tested sample.

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I. INTRODUCTION

The suitability of the delivered Burmese kaolin sample for the paper making was the principal objective of the tests conducted. The sample represented raw material recently found in Burma which was predetermined as substitute for the imported fillers for Burmese paper industry which is expected to expand due to new industrial projects under consideration.

In principle, there are two applications for kaolinitic minerals in the paper making:
- as fillers,
- as coating agents.

The testing programme was, therefore, set up so as to make possible to investigate gradually all the decisive parameters for the above applications. The kaolin sample was broken down in three basic fractions according to grain size distribution and the main effort was concentrated on the paper making appropriate clay fraction. The parameters followed were the grain size distribution, brightness, abrasivity and the possibility of upgrading by industrial mineral processing methods. The good quality coating kaolins are required to have about 90 per cent of particles in the range under 2 µm and maximum 10 per cent of particles above 10 µm; their abrasivity is usually 10 mg or less and brightness above 80%. The requirements for fillers are less strict; about 55 per cent of particles under 2 µm, abrasivity accounting for 25 - 40 mg and brightness 75 per cent and more.

The testing programme included all the tests specified in the contract to be carried out:
- chemical and mineralogical analyses in raw state
- washing of raw kaolin to separate:

a/ sandy fraction
b/ silty fraction
c/ clay fraction

- chemical and mineralogical analyses of fractions a, b, c
- grain size distribution of fractions a, b, c
- technological properties of fractions b, c
- magnetic separation of fraction c with subsequent chemical and mineralogical analyses of non-magnetic, semi-magnetic and magnetic fractions together with their particle distributions
- the sample was also subjected to chemical bleaching.

Based on the results of the above tests the quality of the sample from the point of view of paper making was evaluated, results concluded and relevant actions recommended. As the sample under test did not represent raw material of sufficient quality information gathered during previous tests of other Burmese kaolins conducted by the Research Institute for Ceramics, Refractories and Non-metallic Raw Materials in Pilsen was considered in recommending appropriate steps.

The sample of kaolin was also evaluated from the point of view of possible utilization in the production of rubber and selected ceramics.

II. CONCLUSIONS AND RECOMMENDATIONS

1. The kaolin from Burma is a raw material applicable as filler in the rubber industries and as component of selected ceramics.

2. The tested kaolin from Burma contains a relatively high portion of clay fraction under 20 μm (66.1%) which is not suitable for paper coating because of a high abrasivity (70.7 - 96.6 mg) and a low brightness (67.7 abs. %).

3. The clay fraction has not adequate parameters to be used as paper filler without expensive and complex upgrading. The fraction has appropriate grain size distribution but its brightness is low and abrasivity high considering the papermill requirements which are 25 - 40 mg abrasivity and 75% brightness.

4. To suit to the above requirements the kaolin must be subjected to upgrading. The brightness can be increased up to 78% absolute by boiling in hydrochloric acid which is a costly method demanding for energy, stainless equipment and disposal of waste.

5. The abrasivity is difficult to reduce, the only way being the testing of "composite mixtures" with other local materials or imported ones.

6. The content of rubber poisons, 0.021% Fe, 0.0015% Cu, 0.00015% Mn, corresponds to the required parameters of rubber fillers. However, the application must be verified on semi-industrial scale.

7. The sample of clay fraction was also fired at 1 410°C temperature. The fired specimens were of grey-white colour and well vitrified. The kaolin can be considered as a component of bodies to produce

fine stoneware. Vitrified floor tiles, decorative fencing bricks and other different types of earthenware ceramics could also be produced.

8. It is recommended to look for other kaolins or non-metallic minerals which would suit better to the requirement of paper mills with simple upgrading. There exist in Burma deposits of kaolins from which samples were tested in the Research Institute for Ceramics, Refractories and Non-metallic Raw Materials, Pilsen, to verify their applicability in the production of fine ceramics. The kaolins "Shwe Daung" and "Wellan" should be tested for the application as fillers since their chemical and mineralogical compositions indicate their suitability.

9. The kaolin tested is recommended to be verified for the applications in the production of stoneware, sanitary ware and earthenware and vitrified structural ceramics and for the application in the rubber industry. Semi-industrial scale is necessary for the rubber industry applications and laboratory scale for ceramics.

10. In order to reduce imports of paper filling materials highly specialized tests to verify composite blends of the kaolin with other materials including those imported are recommended. Thus the required parameters of abrasivity and brightness could be reached.

11. A two-week mission to Burma of two experts, one non-metallic mineral expert and one industrial economist, can be expected to accelerate and economize the subsequent research of Burmese paper fillers. The experts should collect and study the available information, take samples for the tests as outlined above and negotiate and formulate the need in Burma for relevant technical assistance.

III. TECHNOLOGICAL TESTS

1. Methods Applied

The sample of delivered raw material was at first evaluated in a macroscopic way from the point of view of mineralogy and petrography. Further it was analysed chemically by the methods of classical wet analysis /by gravimetric, titrimetric and photometric methods/, combined with the method of absorption spectrophotometry /AAS 403, Perkin - Elmer, USA/. Mineralogical analyses were executed by the X-ray method and by the thermal methods. The X-ray goniometer /DRON 4/ was used. (The shift of an arm was 1°/min., screen 10, 0.52).

Thermal analyses, i.e. differential and gravimetric thermal analyses, were carried out by means of derivatographic method /Q-1500 D, MOM, Hungary/ using 300 mg of weighed samples and the heating rate 10° C/min. Coarse fraction a was separated by sieve, silty fraction b and clay fraction c were separated by sedimentation. Particle size distribution of silty fraction and clay fraction was determined by sedimentometric method /Sedigraph 5000, Micromeritics, USA/. Abrasivity of the fraction c was ascertained by the device equivalent to the apparatus Einlehner AT 1000. The device OPTON ELREPHO RFA 2 , West Germany, was used for measurement of brightness. For greater detail we refer to the Czechoslovak state standards.

2. Macrospecification of Raw Material

The delivered sample of 50 kg mass was marked as "Kaolin Burma". It represented a sediment with aulerotic-pelitic structure - lutite of the line quartz - mica - clay. The size of raw material fragments ranged from 20 cm to 0.5 cm and less.

The colour of the sample which had a very low natural moisture was grey-white as far as white. The faces of fracture which were either regular or conchoidal were stained with russette-brown very fine schliers of iron oxides and iron hydroxides. The material was fragile, non-reinforced. The natural moisture - 1.1%; pH of wash-out - 4.98.

3. Washing

The sample was blunged in distilled water and liquefied by addition of 0.9 wt per cent (related to dry matter) of sodium silicate (water glass). Three fractions were separated. By means of wet screening the fraction above 63 µm was separated, further referred to as fraction a. The minus mesh was divided by three-fold free sedimentation into two fractions:
fraction b containing grains 63 µm - 20 µm
fraction c containing clay particles under 20 µm.

The balance of washing

fraction a - sandy	>63 µm	2.2 wt%
fraction b - silty	63 µm - 20 µm	31.7 wt%
fraction c - clay	<20 µm	66.1 wt%
		100.0 wt%

4. Chemisms and Mineralogy

The chemical analyses of raw material and particular fractions are given in Table 1. The low content of Al_2O_3 and high contents of SiO_2 and alkalis indicate that marginal raw material is in question.

The fraction a - sandy was not put into mineralogical

analyses. To evaluate it chemical composition and grain size distribution is sufficient for the classification as unuseful waste. If the fraction were to be used for certain special purposes, special tests would have to be done the economic value of which would be questionable considering only 2.2% representation in the raw material. So the following applies to the raw material and fractions b and c.

The X-ray characteristics of mineral components found indicate the line kaolinite - mica - quartz. (Table 2) The domination of quartz is not surprising taking into consideration the chemical composition. Other principal minerals are kaolinite and mica. The clay fraction contains 30% of kaolinite which is the maximum while the minimum 20% is contained in silty fraction. The sample is also characteristic for high contents of mica which corresponds to the share of K_2O in particular fractions (Table 1). Feldspars were not found in the sample. The poor technological properties of the raw material are due to high contents of mica and, above all, of quartz.

In Table 3 there are presented thermogravimetric data that prove that the raw material has a very low content of clay portion which is represented by kaolinite as witnessed by goniometric difractometry.

5. Grain Size Distribution (fraction b,c)

The grain size distribution was fixed by means of Sedigraph 5000, Micromeritics, USA. The observed suspensions were prepared from the dry material and 0.2% concentrate of sodium hexametaphosphate. The acidity of suspension was fixed by adding NH_4O_4 at pH 9. The distribution curves for fractions b and c are presented as Figure 1 and 2 respectively. Table 4 shows the grain size distribution of particular fractions expressed in per cent.

6. Technological Properties and Upgrading

The decisive parameters to evaluate the applicability of kaolin as paper filler are:

- brightness
- abrasivity

The device OPTON ELREPHO RFA+2 was used to fix the brightness in the usual way of measuring in the sphere of defined blue spectrum and expressed in absolute per cent. A low abrasivity is desired because of wear of paper machinery, namely paper screens and printing matrixes. The parameter was fixed by a device equivalent to current apparatus Einlehner AT 1000 and expressed in mg of mass reduction of screen worn at standard conditions by the tested material.

The above tests were applied only for the clay fraction c:

- brightness 67.7 abs. %
- abrasivity 70.7 - 96.6 mg

Bleaching by High Gradient Magnetic Separation

The clay fraction was tested to enhance the brightness by high intensity magnetic separation of colouring oxides of titanium and iron and other admixtures. High gradient magnetic separator SALA, HGMS 10-15-20 was applied under following conditions:

- magnetic field intensity	1.9 T
- matrix	fine
- vessel (diameter)	1.5 inch
- flow velocity	7 mm sec^{-1}
- loading of matrix	1 g cm^{-3}
- concentration of suspension	170 g of dry matter per 1 litre of suspension

- dispersion	0.6% sodium hexametaphosphate
- delamination	mixer 5 min. 10 000 rpm

Three products were the result of magnetic separation P - principal, non-magnetic product; O - non-magnetic by-product; M - magnetic product.

Balance, contents of colouring oxides and brightness of non-magnetic usable product

Product	Share wt%	Colouring oxides wt% Fe_2O_3	TiO_2	Brightness abs. %
P	71.0	1.17	0.85	70.6
O	23.2	1.56	1.14	
M	5.8	1.95	1.44	
Total	100.0			

Even though the yield of non-magnetic product was satisfactory the magnetic separation was not sufficient to enhance the brightness so as to reach the required minimum for fillers 75 abs. per cent.

Chemical Bleaching

Two processes to enhance the brightness were tested :
a/action of hydrochloric acid ,
b/action of $Na_2S_2O_4 + H_3PO_4$.

a/ The process is based on the reaction of boiling hydrochloric acid with the colouring pigments which are converted into chlorides which are soluble and separable by recurring washing out with water.

b/ Bleaching by $Na_2S_2O_4$ + H_3PO_4 is based on the reduction of trivalent iron compounds which react into $Fe_3/PO_4/_2$ which is colourless and insoluble so that its presence in the material is without objections. The residual H_3PO_4 is then neutralized by calcium hydrate.

The results of the reactions

bleaching process	brightness /abs. %/	brightness increment
before bleaching	67.7	0
boiling HCl	77.4	9.7
$Na_2S_2O_4$ + H_3PO_4	72.8	5.1

IV. FINAL NOTE

The kaolin from Burma consists of 3 mineral components - kaolinite, mica and quartz which dominates. The comparatively high share of SiO_2 (66.75% in raw material and 63.77% in clay fraction) corresponds to the mineralogical composition.

The material contains 66.1% of clay fraction and the rest is made up by sand and silt which are without significance for the paper making industry. Consequently, the technological tests concentrated on the clay fraction.

The tests carried out issued in the conclusion that kaolin from Burma is not suitable as coating material for paper making. The usable clay fraction is coarsely grained, its brightness is low and abrasivity high. As for the paper filling the clay fraction is of suitable grain size distribution but the whiteness and abrasivity do not correspond to the quality requirements of paper mills. The high abrasivity which is obviously on the high content of free quartz occurring in whole the grain spectrum is very difficult and questionable to reduce.

The possibilities of improving the brightness were verified both by high-gradient magnetic separation and chemical bleaching. It is worth mentioning that bleaching by $Na_2S_2O_4 + H_3PO_4$ is more suitable than boiling in HCl when industrial scale is considered.

The magnetic separation was efficient and the increment of brightness was 2.9 abs. %. So was the chemical bleaching when 9.7% increment was the result of HCl action and 5.1% of $Na_2S_2O_4 + H_3PO_4$ action. All the three results are significant, however, the initial whiteness is as low as 67.7%

so that the chemical bleaching by hydrochloric acid is the only process efficient enough to reach the minimum paper filling required value 75% absolute whiteness which is sufficient to produce standard printing and writing papers.

The tests conducted enabled to judge other applications of the raw materials. Kaolin specimens were made up and fired at temperature 1 410°C. This preliminary orientation test shows possibility of using the material in certain ceramic manufactures. In addition, the content of rubber poisons was analysed with the result that the material can be considered as filler in the rubber industry.

Based on the above results of tests relevant actions were recommended both to utilize the tested kaolin industrially in other sectors and to look for more appropriate paper filling and coating minerals which are probable to occur in Burma. A couple years ago the Research Institute for Ceramics, Refractories and Non-metallic Raw Materials in Pilsen carried out tests of kaolin samples (Shwe Daung and Wellan) which had promising chemical and mineralogical compositions to be verified for the paper making. No other conclusions about their value for paper making can be drawn because the kaolins were tested for ceramics and fully consumed during the tests.

ANNEXES

Table 1 Kaolin from Burma
Chemical Analyses of Raw Material
and of Fractions a, b, c, Respectively

Component	Raw material	Fraction a >60 µm	Fraction b 63-20 µm	Fraction c <63 µm
L.O.I.	3.87	2.18	3.14	4.70
SiO_2,	66.75	84.85	74.71	63.47
TiO_2	0.96	0.46	0.86	0.99
Al_2O_3	20.61	8.23	14.60	21.89
Fe_2O_3	1.10	0.87	0.98	1.47
MgO	1.06	0.48	0.88	1.25
CaO	0.03	0.06	0.08	0.22
Na_2O	0.07	0.06	0.06	0.07
K_2O	5.01	2.14	3.88	5.44
Total	99.46	99.33	99.19	99.50

Table 2 Kaolin from Burma
X-ray Powder-pattern lines of the Raw Material and Fractions

Silty fraction		Clay fraction		Raw material		Minerals+
d /nm/	I	d /nm/	I	d /nm/	I	
1.005	6.5	1.005	21	1.005	15	M
0.720	3	0.720	8.5	0.720	7	K
0.501	4	0.501	11	0.501	8	M
0.448	8	0.451	12	0.451	10	M,K
0.427	35	0.427	28	0.427	35	Q
0.388	2	0.390	3	0.390	3.5	M
0.374	2	0.374	3	0.375	3	M
0.366	2	0.368	5	0.367	3.5	M
0.358	4	0.359	7.5	0.358	6	K
0.351	4	0.351	5	0.352	4	M
0.335	100	0.335	100	0.335	100	Q
0.321	2.5	0.321	5	0.321	4.5	M
0.307	2	0.307	3	0.307	3.5	M
0.300	3	0.299	5	0.300	4	M
0.389	2.4	0.287	4.5	0.287	3.5	M
0.280	2	0.280	3	0.280	3	M
0.256	9	0.256	12	0.257	13	M
0.246	13	0.246	13	0.246	15	Q
0.239	3	0.239	4.5	0.239	4	M
0.228	10	0.229	9	0.228	11	Q
0.224	7	0.223	5	0.224	6.5	Q
0.315	2	0.215	3	0.214	3	M
0.213	10	0.213	9	0.213	10	Q
0.200	5	0.199	9	0.200	6	M
0.198	7	0.198	8	0.199	7.5	Q
0.182	18.5	0.182	17	0.182	19	Q

+/ K - Kaolinite
M - Mica
Q - Quartz

Table 3 Kaolin from Burma

Thermogravimetry

a/ Raw Material

temperature interval °C	reduction of mass /wt%/
23 - 130	0.5
130 - 450	0.5
450 - 570	1.66
570 - 755	1.5
755 - 1000	0.33
	4.49

b/ Fraction b (silty)

temperature interval °C	reduction of mass /wt%/
21 - 405	0.82
405 - 520	1.15
520 - 730	1.47
730 - 1000	0.16
	3.60

c/ Fraction c (clay)

temperature interval °C	reduction of mass /wt%/
22 - 120	0.66
120 - 470	0.66
470 - 580	1.82
580 - 760	1.99
765 - 1000	0.17
	5,30

Table 4 Kaolin from Burma
Grain Size Distribution

Interval /µm/	Share /wt%/	
	clay fraction	silty fraction
< 1	26.5	2.5
1 - 2	14.5	4.5
2 - 3	11.0	3.0
3 - 5	15.0	3.5
5 - 10	18.0	14.5
10 - 20	9.0	47.5
20 - 30	6.0	18.0
30 - 50	-	6.5
> 50	-	-
TOTAL	100.0	100.0

FIGURE 1

0.063–0.020 mm

PARTICLE SIZE DISTRIBUTION

SAMPLE IDENTIFICATION SILTY FRACTION 1147.86

DATE 4.11.1986

Density 2.6 g/cc LIQUID _____ Density _____ g/cc Viscosity _____ cp

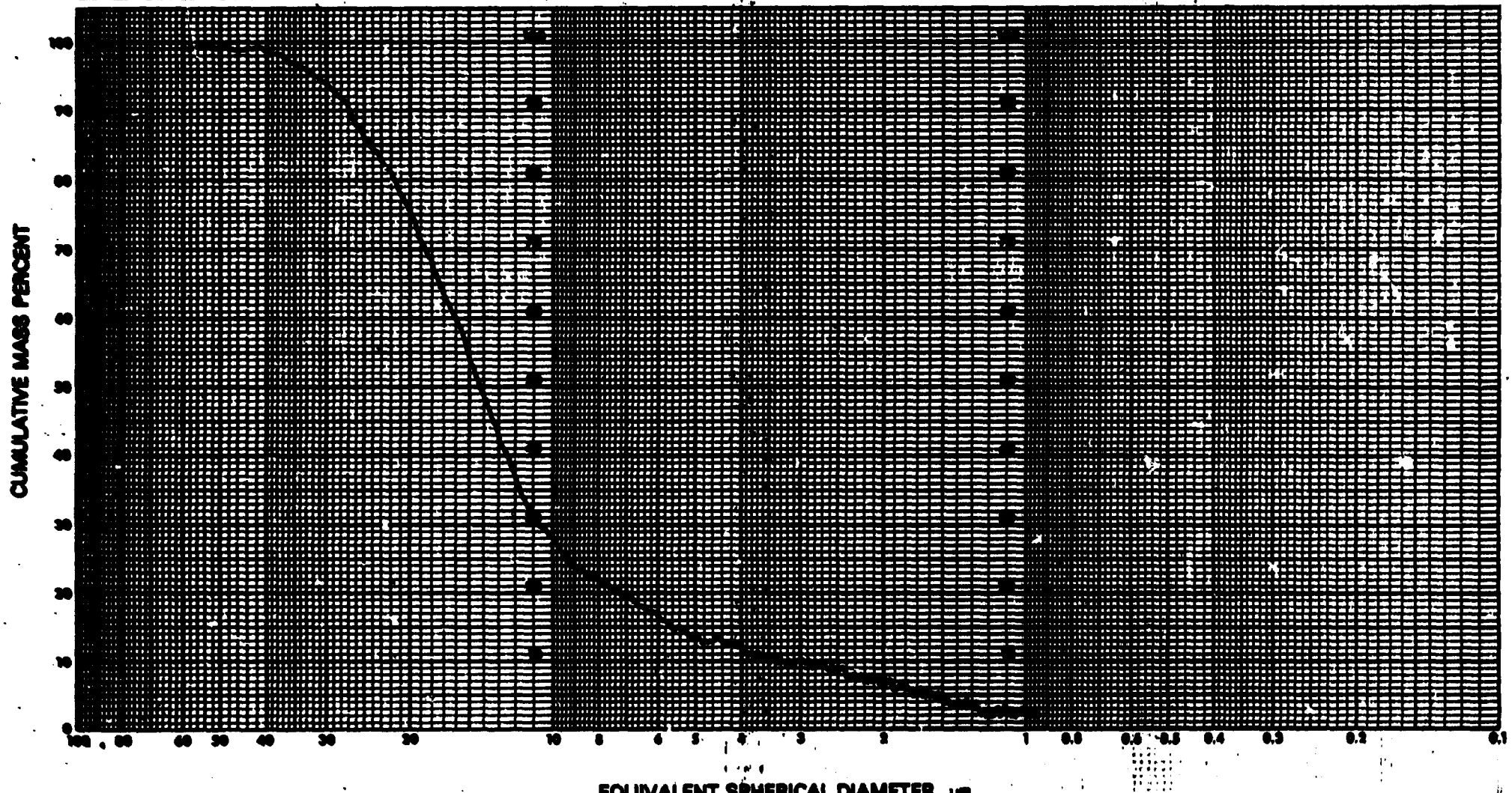
BY _____

Preparation KAOLIN FROM BURMA

TEMPERATURE 33 °C

SODIUM HEXAMETAPHOSPHATE, 0.2% SOLUTION

RATE 7.81 START DIA. 60 µm



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3016 FIGURE 2

PARTICLE SIZE DISTRIBUTION

< 0.020mm

SAMPLE IDENTIFICATION CLAY FRACTION 1148-86

DATE 4.11.1986

Density 2.6 g/cc LIQUID _____ Density _____ g/cc Viscosity _____ cp

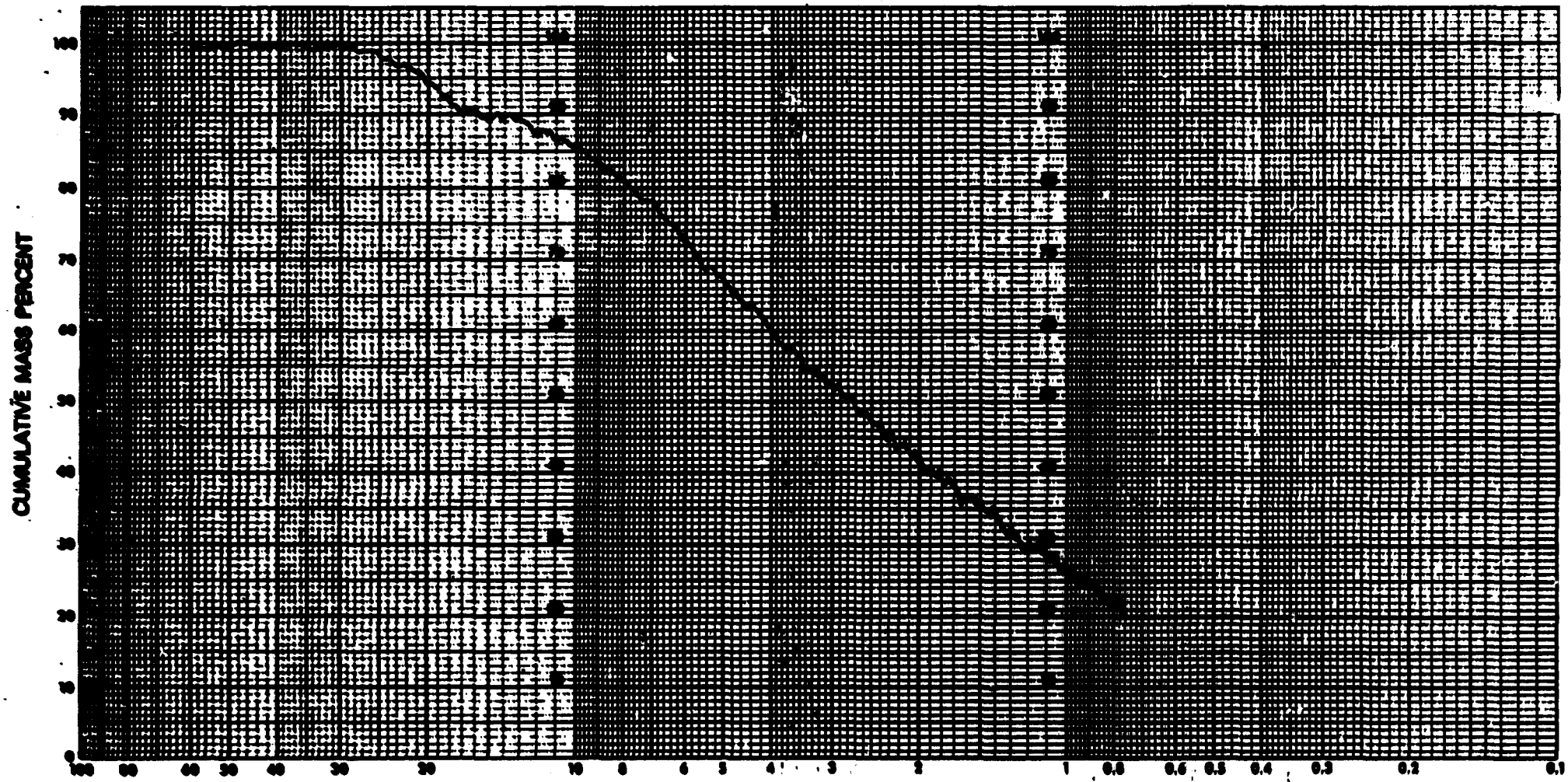
BY _____

Preparation KAOLIN FROM BURMA

TEMPERATURE 33 °C

SODIUM HEXAMETAPHOSPHATE, 0.2% SOLUTION

RATE 781 START DIA. 60 µm



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