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CENTRAL RESEARCH & DEVELOPMENT LABORATORY

FINAL REPORT ON JORDANIAN RAW MATERIALS FOR PILOT PLANT TRIALS

U.N.I.D.O. CONTRACT REFERENCE NO. 91/038G.

CONTENTS

WG/RP18/92/RM.COM.

SECTION 1

INTRODUCTION / OBJECTIVES

Section 1.

Introduction.

The Jordanian raw materials were received on 5th. February 1992, they were described on the airway bills as DEAD SEA MUD.

There were four categories of materials i.e.

Silica Sand.....Internal test ref. no. si/92/ja.

Kaolin 1.....Internal test ref. no. ka1/92/ja.

Kaolin 2.....Internal test ref. no. ka2/92/ja.

Dolomite.....Internal test ref. no. do/92/ja.

There were approximately 140 kilos of each unrefined material.

The bags were cross contaminated, and showed signs of being repacked.

There was no other reference with the consignment, and no chemical analysis from the Jordanian Mining Co. as with the previous small samples.

We noted that the Kaolin 2 appeared to be of an inferior quality to the original small samples, and showed signs of mineralogical contamination.

Objectives.

1) Check the physical and chemical properties of the bulk samples.

2) Determine the grinding times for the hard materials.

3) Formulate a body mix to satisfy the production of cast, jiggered and hand thrown pieces.

4) Determine bisc. and glost firing temperatures and firing cycles.

5) Develop an acceptable fired colour.

6) Set limits for porosity standards.

7) Calculate expansion characteristics, and produce a glaze to match.

8) Provide samples of clay in plastic form, bisc. pieces and finished pieces, glazed with a transparent glaze.

9) Reduce the production and quality problems associated with the introduction of a new body into the plant.

SECTION 2

LABORATORY TEST RESULTS

Section 2.

Laboratory test results.

LAB CARD REF. NO: SAMPLE SI/92/JA.
DESCRIPTION: SILICA SAND.
LOCATION: JORDAN.
PRINTED: 5.3.92.

Raw Analysis. XRF Results:

SiO2	TiO2	Na2O	K2O	CaO	MgO	Fe2O3	Al2O3	Cr2O3
97.4	0.06	0.02	0.04	0.90	0.02	0.09	1.1.	trace.

SPECIAL NOTES.

A) FeO2 is increased by 0.05 % on the original previous small samples.

The silica was washed and sieved to remove any organic material and ball milled as detailed below.

Particle size

%	<	micron
45%		10

The milled material was then combined with crushed dolomite and further milled so that the total of the none plastics were as detailed below.

Particle size.

%	<	micron
56 %		10

Laboratory test results.

LAB CARD REF. NO: SAMPLE DO/92/JA.
DESCRIPTION: DOLOMITE.
LOCATION: JORDAN.
PRINTED: 7.3.92.

The dolomite was crushed in laboratory jaw crusher and samples removed for analysis.

Raw Analysis. XRF Results:

SiO2	TiO2	Al2O3	Fe2O3	CaO	MgO	K2O	Na2O	Loss on Ign.
---------- Standard ----------								43.98 %

Residue of analysed material.

%	<	micron
98.9		20
98		10
95.2		5
84		1

The bulk (crushed) dolomite was added to the milling cycle of the silica and milled together with the following finished results.

Residue of milled silica and dolomite.

%	<	micron
56		10

Laboratory test results.

LAB CARD REF. NO: KA1/92/JA
DESCRIPTION: KAOLIN.
LOCATION: JORDAN
PRINTED: 7.3.92

SPECIAL NOTES:

1) This Kaolin was not identified on the bags as kaolin 1. We have referenced it as this to distinguish it from the other kaolin sample.

2) The residue at 75 micron is more than double that of the previous small sample.

3) The analysis show an increase in FeO2.

4) The blunged and sieved residue results are from samples extracted prior to the body formulation, as it has been agreed that the best procedure is to add the Jordanian kaolins direct into the ball milling cycle to maximise their limited plasticity.

Raw Analysis. XRF Results:

SiO2	TiO2	Al2O3	Fe2O3	CaO	MgO	Na2O	Loss on Ign.
59.3	2.62	26.1	1.49	0.19	0.24	0.08	9.98

Crushed.

The residue was calculated after the sample was put through a cross beater mill having a 1mm screen.

The residue at 75 micron = 7.9 %

Blunged and sieved.

Residue results / particle size analysis.

%	> micron
97	20
88	10
78	5
54	2
30	1
22	0.5

KA1/92/JA. Continued.

XRD ON MATERIAL WITH RESIDUE GROUND IN:

25 quartz. 53 medium ordered kaolinite. 10 mica. 3 interstratified material. 2 alunite. 2 anatase.

Laboratory test results.

LAB CARD REF. NO: KA2/92/JA.
DESCRIPTION: KAOLIN.
LOCATION: JORDAN.

SPECIAL NOTES:

1) This kaolin was not identified on the bags as kaolin 2. We have referenced it as this to distinguish it from the other kaolin sample.

2) The blunged and sieved results are from samples extracted prior to the body formulation, as it has been agreed that the best procedure is to add the Jordanian kaolins direct into the ball milling cycle to maximise their limited plasticity.

Raw Analysis. XRF Results.

SiO2	TiO2	Al2O3	Fe2O3	CaO	MgO	K2O	Na2O	Loss on Ign.
65.2	2.11	21.7	1.89	0.02	0.35	0.77	0.07	7.98

Crushed.

The residue was calculated after the sample was put through a cross beater mill having a 1mm screen.

The residue at 75 micron = 8.7 %

Blunged and sieved

Residue results / particle size analysis.

%	>	micron
89		20
75		10
68		5
47		2
32		1
17		0.5

XRD ON MATERIAL WITH RESIDUE GROUND IN: %.

31 quartz. 51 medium ordered kaolinite. 10 mica. 3 interstratified mineral. 2 anatase. 2 alunite.

SECTION 3

PILOT PLANT TRIALS

Pilot plant trials.

A) Body preparation.

Work previously carried out on the small samples analysed in 1991, together with the small body trials previously carried out have suggested a body recipe incorporating a maximum of 23% of an English ball clay. The ball clay previously used was the SKD blend of devonian ball clays.

The samples submitted, after sorting, removing contamination, then blunging / sieving were of an effective weight to give a maximum of 500 kgs of plastic body when proportionally combined into the recommended body recipe.

SPECIAL NOTES:

1) We feel that one of the kaolins submitted was not as the original sample (KA1/92/JA) This is evidenced by the general appearance of the clay which looked to be dirty. Also the analysis (page 5 Lab. test results) show a marked increase in Fe02, and a residue of more than double the original clay samples. This may be due to the clay being mixed with a degree of overburden, or taken from a part of the mine site where inferior deposits are located.

This is the most probable reason for the finished fired results being a 'less white ' appearance than the original fired body trials completed in 1991.

Procedure:

The body recipe judged to be the most suitable for this trial was a follows.

Kaolin 1.....11 %)		
Kaolin 2.....11 %)		
SKD Ball clay.....23 %)	Clays at 45 %	
Silica sand.....40 %)		
Dolomite.....15 %)	Non plastics at 55 %	

Body preparation continued.

The materials were milled in a standard batch ball mill porcelain lined, using high alumina grinding media.

The quartz - as silica sand - was washed then added to the ball mill and ground for two hours and a residue test showed a particle size of 38% < 10 micron.

The milling was continued for one further hour and a residue test showed a particle size of 45% < 10 micron.

The crushed dolomite was then added to the ball mill and grinding was continued for a further one hour. The particle size at this point was 51% < 10 micron. The mill was further ground for 30 mins and a reading taken of 55% < 10 micron.

This therefore gave a particle size of the combined none plastics of 55 % < 10 micron.

The kaolins 1 & 2 were added to the ball mill and ground for one further hour.

0.1 % $BaCO_3$ was added at this stage (0.1% of the body)

No cobalt whitener was added.

The SKD Ball clays were blunged in a standard high speed blunger and a slip free of defloculant at 26 ozs per pint was obtained in the blunger.

The ball clay and milled materials were blended together and screened through a 125 micron screen into a separate pumping reserve storage ark and filter pressed at 10 bar for two and a half hours.

The filter press was opened and filter cakes of 19.5 % moisture were produced.

The filter press was re - charged to completely use the milled slip and a second batch of filter cakes were obtained at 18.9 % moisture content.

Body preparation continued.

The chemical analysis of the combined body recipe is:

SiO2	TiO2	Al2O3	Fe2O3	CaO	MgO	K2O	Na2O3	L.O.I.
66.20	0.74	12.50	0.63	5.65	2.56	0.68	0.08	10.98

Particle size analysis:

% less than	20	10	5	2	1	0.5	micron
	91	74	53	34	25	18	

Modulus of rupture. (Green strength)

	KG / CM. 2.	LBS / IN. 2.
Mean.....	31	
Mean.....		439

Water absorption - of bisc. fired sample.

At buller ring 8 (1000 C.) 21.5%

Seven filter cakes were reserved for the preparation of casting slip.

The balance of the filter cakes were passed through a single deck de-airing pugmill (extruder) at a vacuum of 25 Lbs / in 2.

SPECIAL NOTES:

1) It was observed that the extruded clay was not as plastic as had been evidenced in the earlier trials. We must assume that this is again due to the differential in the received kaolin as mentioned earlier.

2) The modulus of rupture results indicate a good green strength, and we would suggest an increase in kaolin at the expense of 2% silica in any further trials.

Body preparation continued.

B) Casting slip preparation.

The casting slip was prepared from filter cakes at a moisture content of 19.5 %.

A charge of 50 lbs of filter cake was used.

The standard method of slip preparation was used, i.e.

The water / clay ratio to be in the order of 75% water to 25% clay.

The defloculants used were sodium silicate and soda ash in the proportion 2 : 1.

A mixture of defloculant equal by weight to 1% of the calculated dry weight of the clay was prepared in 0.25 pints of warm water.

The basis for the calculation of the casting slip is as follows.

Filter cake charge.....50.00 lbs (800 ozs)
At 19.5% m.c there is water in the cake of.. 9.75 lbs (156 ozs)
The dry clay content is therefore.....40.25 lbs (644 ozs)
The total defloc. weight is therefore..... 0.40251bs(6.44ozs)

Based on the clay water ratio stated:

Total dry clay will be.....644 ozs (75%)
Total water will be.....215 ozs (25%)

	Dry clay.	Water.
	644 ozs.	156 ozs (in filter cake)
		59 ozs (2.8 pints) to be added
Total	644 ozs (75%)	215 ozs (25%)

For the purposes of this trial the water content used in the preparation of the defloculant was disregarded.

Casting slip preparation continued.

Method.

50% of the total water allowed was introduced into a geared down blunger. The speed being reduced due to the small amount of body being prepared.

50% of the defloc. mixture was introduced.

50% of the total filter cake was introduced in small hand broken down pieces.

The slip was mixed for 15 minutes.

Whilst mixing continued:

The balance of the total water allowance was introduced

15% of the balance of the defloc. mixture was introduced.

The balance of the filter cake was introduced in small hand broken down pieces.

The body was mixed for ten minutes.

Sample 'A' - approximately two pints - was drawn from the blunger and sieved through an 80' s mesh sieve and the slip characteristics tested on a Galenkamp torsion viscometer, see Graph - Casting Slip. The tested slip was returned back into the main mixture.

A further 10 % of the defloculant mix was added and mixing continued for seven minutes.

Sample 'B' was drawn off and testing procedure repeated.

A further 5% of the defloculant mixture was added and mixing continued for five minutes.

Sample 'C' was drawn off and tested.

The characteristics of sample 'C' were within the range of earthenware casting slip parameters, and the whole slip was then sieved through an 80's mesh sieve into a slow stirring arc to mature.

Production Pilot Plant Trials.

Plastic clay making.

Machine making.

See roller making, observations.

The de - aired pugged clay was used on a Service Engineers model 'B' heated head four stage roller machine.

The following items were manufactured.

Dinner Plates.

Small oatmeal bowls

Tea saucers.

The following items are yet to be produced.

Side plates.

Tea cups and coffee mugs.

The tea cups and coffee mugs will be produced from the original pugged clay, but re - processed through the integral in line de - airing pug within the fully automatic Service Engineers cup / mug roller making production unit.

Semi - automatic making machine with batting out facility.

See semi - automatic making observations.

Dinner plates / side plates have been produced on this unit.

Hand Jigger with batting out machine.

See hand jigger making observations.

Tea cups and dinner plates have been produced on this machine.

Hand throwing.

We have produced hand thrown samples but due to artistic individuality of this process, and the need for the hand thrower to become familiar with the clay body we w ll not proceed with this further as the limited amount of clay body could be wasted. We therefore propose to enclose sufficient of the body for the usage of the Queen Alia hand throwers, to enable them to familiarise themselves with the clay.

Roller making observations.

Due to the fact the we are able to produce only around .75 tonne of the finished clay body in filter cake form because of the limited material supplied, we were obliged to pug the clay through a 3 in. extrusion pugmill. It was not possible for the larger 6 in. extruder to be used, as it takes over 1 tonne to fill the chamber.

We were therefore only able ' introduce into the production process 3 in. extrusions instead of 6 in. extrusions which are always used for larger shaped items such as side plates and dinner plates.

The small oatmeal bowls and tea saucers made quite well on the roller machine. The dinner plates were made with difficulty due to our having to physicaly squeeze the slices taken from the 3in. extrusion, into an approximately 6 in. cross section.

See loss percentages.

Semi - automatic making observations.

We produced dinner plates and side plates with reasonable ease. We found that the clay was a little ' short' for the larger plates, and some difficulty was observed in getting the batting out spreader to spread a ten inch blank. We had to exercise closer control over the water spray gun on the spreader, giving a wider angle of spray than normally used.

See loss percentages.

Hand jigger making observations.

We made dinner plates and tea cups on this machine. The tea cups were produced with ease, but a little ' creasing of the clay when running up the inside of the mould was observed.

We would not recommend that articles were made 'direct' in the mould. i.e. it is necessary to 'finger run' up the clay within the mould.

The dinner plates were again a little difficult to spread on the batting out machine, but judicious use of a small trickle of wat assisted this.

See loss percentages.

Clay making loss percentages.

The following percentages were lost in the clay making process. Some of this is due to the machine operator not being familiar with the clay, and this is a factor that must be taken into consideration.

Acceptable clay losses on the particular machine process are noted in brackets.

Roller flat machine making.

Item.	% clay loss.	
Oatmeal bowls	5.2.	(4.00)
Tea saucers	4.6.	(4.00)
Dinner Plates	14.7.	(10.00)

Semi - automatic making.

Item.	% clay loss.	
Dinner plates	9.6.	(6.00)
Side plates	7.4.	(6.00)

Hand jigger making.

Item.	% clay loss	
Dinner plates	8.9.	(3.00)
Tea cups	4.1.	(3.00)

Casting making.

The articles produced were from slip sample 'C' as detailed on page 13.

In order minimise wastage from the limited amount of casting slip available, and to develop a 'technique' in using the slip, the first articles produced were small cream jugs.

The procedure was as follows.

a) The slip was agitated in a small portable mixer at a speed of 20 r.p.m. This allowed any residual air to escape from the mixture. The mixture was allowed to 'mature' for twenty four hours to ensure full dispersion of the defloculants.

b) After 24 hours the slip was again tested, and the fluidity had dropped from 325 overswing to 288. a small amount of the defloculant mixture was added and the overswing was brought back to 320 which is within normal paramaters. The thixotropy was 24.

c) Slip was poured into 10 identical plaster moulds in the normal way. After three minutes mould one was emptied and allowed to drain.

This procedure was carried out progressively on all the moulds, at two minute intervals, reclaiming the slip each time.

The moulds were allowed to dry 'leatherhard' and each mould was checked for the gradual build up of the cast thickness.

The results were as follows.

Sample 1) After 3 mins.....1.07mm.

From the above results, and the normal casting thickness for

standard tableware dinnerware being around 6mm dry / unfired, we are satisfied that the clays produce a good slip with acceptable casting times to suit a production line cast system. Further development work could refine the slip characteristics even more.

We then proceeded to cast a variety of coffee pots / cream jugs and sugar bowls. All the items produced released from the mould easily, and were of good 'green' strength.

No production losses were recorded from any of the cast trials, due to the good working characteristics of the slip.

Biscuit firing / Thermal expansions.

The items produced were fired in a standard gas fired semi muffled tunnel kiln. The flatware pieces were stacked, but not bedded in sand. The castware pieces were free standing on the kiln shelves.

The kiln firing curve was NOT fast fire, but rise of temperature was at between 140 & 180 C. per hour.

Only five articles cracked on the biscuit fire. This is 2.3% of the total passed through the first fire of the kiln.

At this stage we re - checked the linear thermal expansion of the prefired biscuit, and at this point we became aware of a great anomaly in the results from this firing, and the results from the first trial exercise.

The thermal expansion of the first trials at 1065 C. was....0.38%

The thermal expansion of the bulk pilot trials at 1065 C. was.....0.45%

This result determines the fact that no commercial glaze can be easily manufactured to 'fit' the ceramic body.

We proceeded to re - run all laboratory tests from the first stage of analysis, and after exhaustive trials and double checking have arrived at the conclusions presented in the section that follows.

SECTION 4

CONCLUSION

The bulk trials have been only partially successful.

The materials performed satisfactorily in mixing, blending, filter pressing, extruding, making by casting, hand, semi automatic jiggering, and by roller machine making.

Biscuit firing was carried out at 1065 C. in a conventional gas fired open flame tunnel kiln, with oxidising atmosphere.

Extensive trials have been carried out to formulate a glaze to 'fit' the biscuit (first fired) body. The body has exceptionally high thermal expansion characteristics. These have not been successful. A commercial glaze to fit the current biscuit body would not be economically viable.

We have endeavoured to reduce the thermal expansion by pre firing the body to partial vitrification at 1200 C. but only small improvements in expansion were made, and a poor fired colour was obtained at this temperature.

In order to evaluate why the expansion values of the body produced in these bulk pilot plant trials varies so much from the original expansions obtained from the very first small analysis / laboratory testing carried out in an earlier contract, we have re traced every analysis and laboratory procedure of the materials provided to us.

We have no doubt that the basic reason is the variation in DOLOMITE supplied to us.

The Dolomite supplied for the original trials was in very fine micronised powder form. It consisted of 99% less than 20 microns, and 85% less than 2 microns.

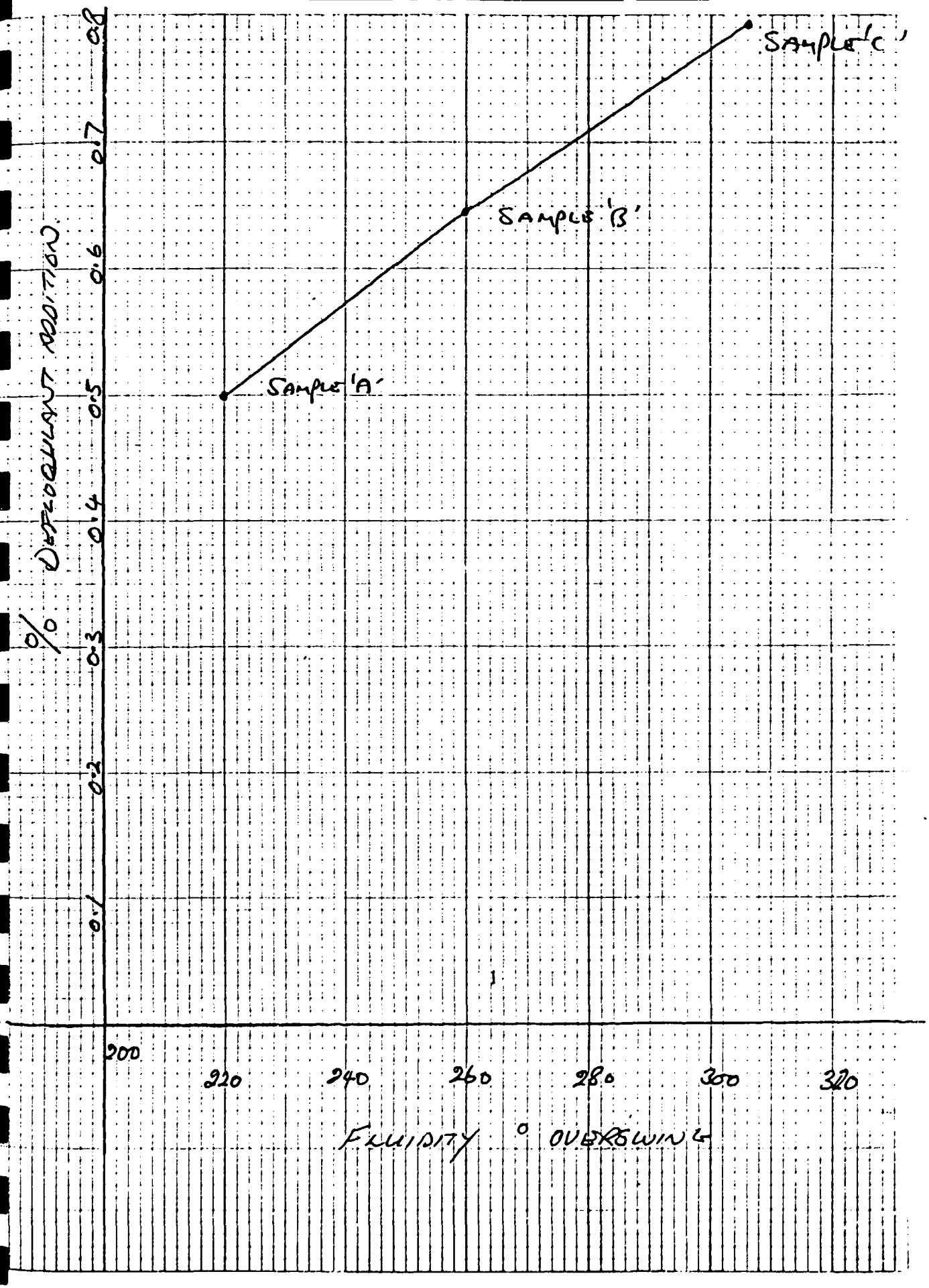
The Dolomite provided for the bulk pilot plant trials was basically too coarse, and has not fully fluxed into the fired body.

We are confident that if a micronised (fine powdered) Dolomite is used a very good conventional earthenware pottery body can be produced from the materials provided, using a conventional glaze on the finished product.

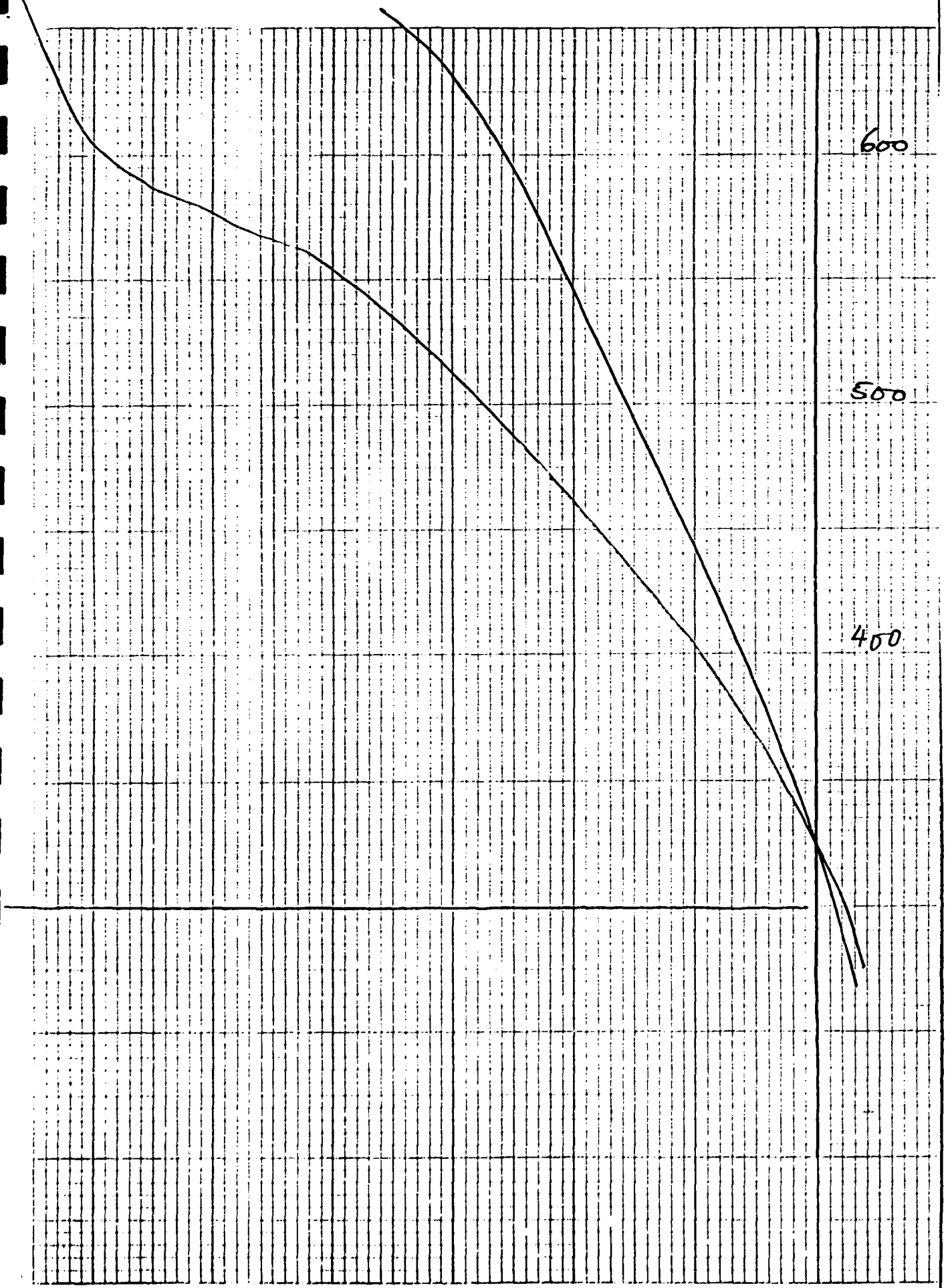
APPENDIX A.

Technical specification graphs and body development charts.

CASTING SLIP DEFLOCULATION GRAPH.



BODY THERMAL EXPANSION GRAPH 1065 C.

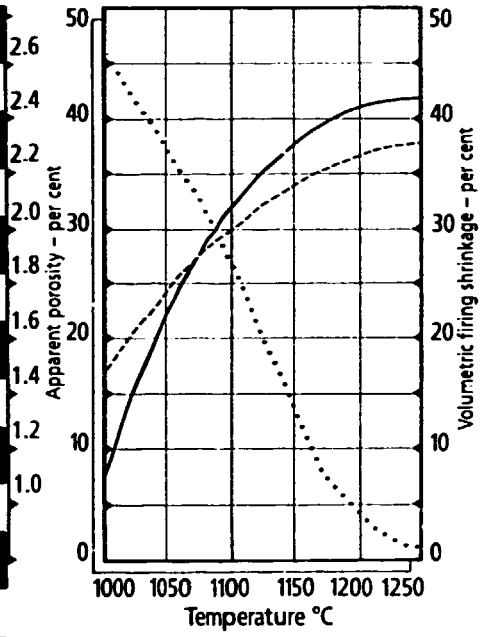


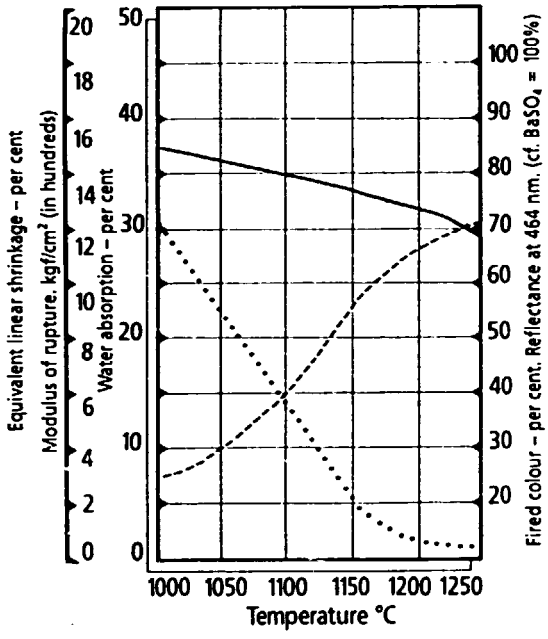
SKD CLAY DATA SHEET

FIRED PROPERTIES

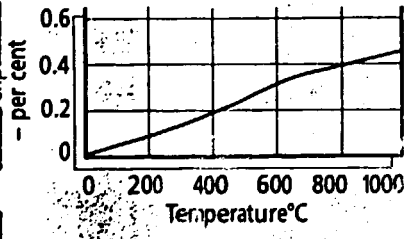
Devon Clays

- BULK DENSITY
- VOLUMETRIC FIRING SHRINKAGE
- APPARENT POROSITY
- FIRED COLOUR
- MODULUS OF RUPTURE
- WATER ABSORPTION





Reversible Thermal Expansion
(Prefired at 1200°C)



NOTES

The technical data given here are indicative only, and your attention is particularly drawn to the fact that all sales are undertaken strictly in accordance with our 'Conditions of Sale' which can be referred to on the reverse of our notepaper and invoice forms.

Ball Clay SKD

Devon Clays

A composite blend specially formulated for porous earthenware.

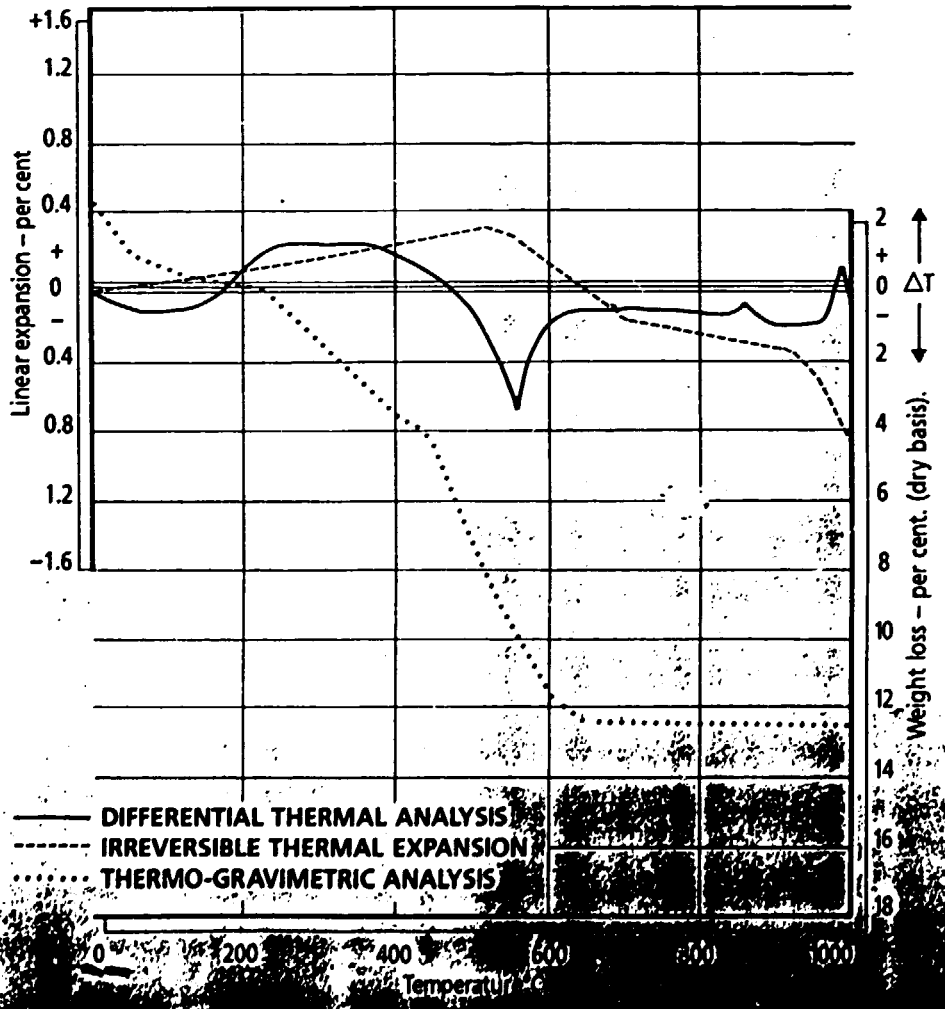
MINERALOGY

Dominant Minerals
Partially disordered kaolinite and hydrous mica.

Minor Constituents
Quartz, anatase and carbonaceous matter.

Calculated Mineralogical Composition (Rational Analysis)

Kaolinite	67
Potash Mica	14
Soda Mica	2
Quartz	11
Carbonaceous matter	3



TECHNICAL DATA

Devon Clays

Ball Clay

SKD

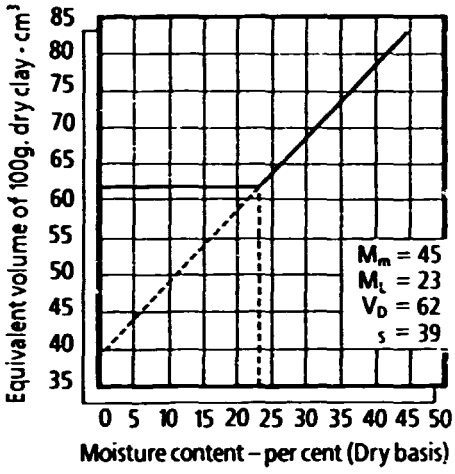
SKD CLAY DATA CHEET

CHEMICAL & PHYSICAL PROPERTIES

	Ultimate Analysis (per cent)	Calculated Fired Analysis (per cent)
SiO_2	50.0	57.2
TiO_2	1.0	1.1
Al_2O_3	32.9	37.6
Fe_2O_3	1.2	1.4
CaO	0.2	0.2
MgO	0.3	0.3
K_2O	1.6	1.8
Na_2O	0.2	0.2
Loss on ignition	12.6	
Carbon	1.6	

Shrinkage on Drying
Volumetric shrinkage (wet basis) of specimen prepared at 31 per cent moisture.
26 per cent.

Volumetric Drying Shrinkage



pH
pH value 5.0

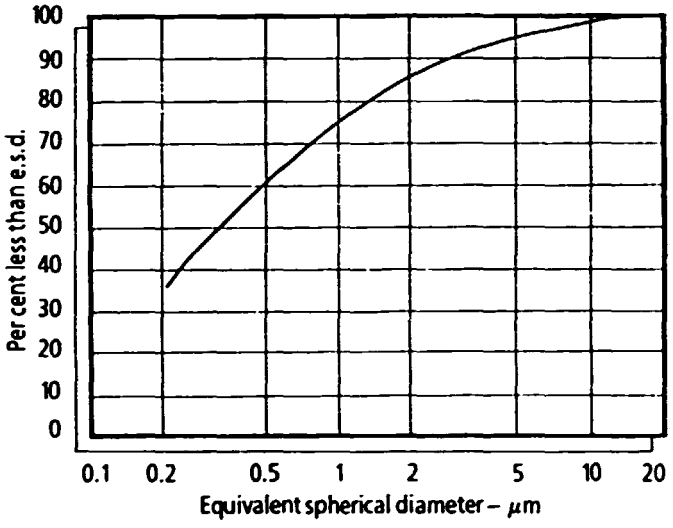
Residue
On 120's mesh (125 μm)
average less than 0.5 per cent

Modulus of Rupture
56-63 kgf/cm²
800-900 lbf/in²

Refractoriness
Rate of Heating 50°C per 5 min.
Cone 33 - 1730°C

Surface Area
(by nitrogen adsorption)
25 m²/g.

Particle Size Distribution

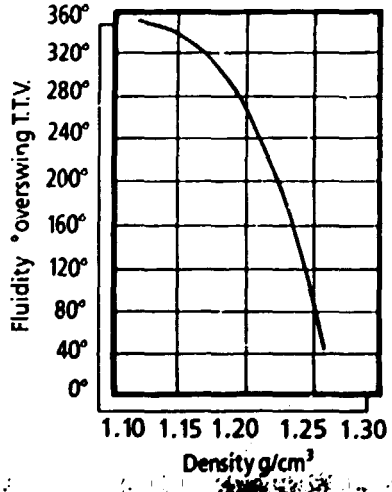


FLUID PROPERTIES

Viscometer
Technico torsion viscometer fitted with 30 s.w.g. torsion wire and 11/16" bob.

Deflocculant
Sodium Silicate C 100
Sodium Carbonate anhydrous
Ratio 3:1

Fluidity/Slip Density



Fluidity/Density/Deflocculant Addition