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**VISCOSITY MEASUREMENT OF KAOLINITE  
USING THE BROOKFIELD VISCOMETER**

**C J Mitchell**

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British Geological Survey  
Keyworth, Nottingham NG12 5GG, UK

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**British Geological Survey**

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**VISCOSITY MEASUREMENT OF KAOLINITE USING THE  
BROOKFIELD VISCOMETER**

**C J Mitchell**

**1. INTRODUCTION**

The Brookfield viscometer is an instrument for the measurement of low-shear viscosity; it is reputed to be quick and easy to use and to give very repeatable results. Viscosity measurements of kaolinite slurries are essential to their evaluation as possible raw materials for paper making and to their use in ceramic bodies formed by casting processes. The work recorded in this report was carried out primarily to establish a procedure for measurement of viscosity in the BGS laboratories, and secondly to make a preliminary assessment of the effect of kaolin particle size and shape on the high-solids-content suspensions required for viscosity measurement and eventual use. Much of this work was carried out with Mr A. Dawson, Leicester University, during a secondment in the summer of 1991. The work forms part of the ODA/BGS project "Minerals for Development".

**1.1. Theory of viscosity**

Viscosity is a measure of the resistance of a liquid to flow, and the amount of shear required to induce flow is used as a measure of the apparent viscosity. Most clay slurries, which usually contain colloidal size particles, behave as a non-Newtonian fluid, i.e. shear stress is not proportional to shear rate. The fluid behaviour is midway between that of a viscous fluid and an elastic solid (a state known as viscoelastic).

There are several well-defined fluid behaviours in clay slurries which are relevant to viscosity measurement. These include;

- Shear thinning, where apparent viscosity decreases with increasing shear rate.
- Shear thickening (dilatancy), where apparent viscosity increases with increasing shear rate.
- Thixotropy; clay suspensions become viscous on standing, but regain fluidity on stirring (otherwise known as reversible time-dependent flow).

- Rheoplexy; increase in viscosity with time when system is subject to constant shear.

The viscosity of a kaolinite slurry is influenced by a number of factors: viscosity of the fluid medium, solids concentration, particle geometry, particle to particle interaction, particle-size distribution, non-kaolinite mineralogy (especially smectite), soluble salt content, pH and temperature.

## **1.2. The Brookfield viscometer**

The Brookfield viscometer rotates a 'spindle' in the clay slurry and this spindle is connected to a spring which records the torque required to overcome the viscous resistance to the induced movement (see Figure 1). The degree to which the spring is wound is proportional to the viscosity of the fluid and is indicated on a scale of 0 - 100 (see Appendix 1 for the operation of the Brookfield viscometer).

A number of viscosity ranges can be measured by using different combinations of spindle size and speed. For a given viscosity the resistance is greater as spindle size and/or speed increases. The minimum viscosity range is obtained by using the largest spindle at the highest speed and vice versa (see Appendix 2 for appropriate spindle/speed combinations and their respective viscosity ranges).

Even in a fully deflocculated clay slurry the viscosity measurement should be allowed to equilibrate, in order for the slurry to attain optimum dispersion under the measurement conditions. As the measurement of viscosity is by an indirect method it is a measure of apparent viscosity.

## **2. SAMPLE PREPARATION**

Viscosity measurements were carried out on a number of kaolinite samples. Some of these were commercial products (from ECC) and the remainder were processed from the raw state. The raw samples were obtained from Cornwall, England; the East African Rift Valley, Kenya and the Pugu Hills, Tanzania.

For the latter, approximately 2 kg of kaolinite-bearing material was attrition-scrubbed for 2 hours in order to gently break down the clay aggregates and to wash clay from any coarser particles. Some samples required a light grinding of clay lumps prior to scrubbing, using a ceramic pestle and mortar, to increase the disaggregation. The resulting slurry was then wet screened through a stack of sieves down to 63  $\mu\text{m}$ . The material retained on the sieves was dried and examined by binocular microscope. The <63  $\mu\text{m}$  material was dried, sub-samples being removed for analysis, and the remainder was hydrocycloned. The clay was redispersed to form a 10% w/v suspension; this slurry was passed through a pump to enhance dispersion and then

hydrocycloned to produce a cut point of 10  $\mu\text{m}$ . The overflow material, which contained mainly  $<10\ \mu\text{m}$  particles, was predominantly kaolinite and was used for viscosity measurements.

Both prepared and commercial kaolinites were characterized by the following methods. X-ray diffraction analysis, using a Phillips PW 1700 X-ray diffractometer with Cu-K $\alpha$  radiation, to identify the minerals present; thermogravimetric (TG) analysis to determine the amount of kaolinite present; particle-size analysis by X-ray sedigraph; particle shape determination by transmission and scanning electron microscopy.

### 3. VISCOSITY MEASUREMENT

#### 3.1. Method of viscosity measurement

Viscosity measurement of a Newtonian fluid entails its presentation to a viscometer with little preparation, but the viscosity of non-Newtonian fluids depends on a wide range of variables (as briefly outlined earlier) and therefore measurements must be made under strictly controlled conditions. The viscosity measurement procedures carried out were largely based on those described in "ECC Test Methods for the Paper Industry", especially P106 and P107. The amended tests, which are described in the following section, are described in detail in Appendix 1.

In order to gauge a starting point for these tests the first step was to determine the **flowability** of the clay, which is the solids-content at which the clay slurry just begins to flow (similar in concept to the Atterburg liquid limit test). The next test, starting from the flowability solids concentration, is to determine the **deflocculant demand**, which is the minimum amount of deflocculant required to obtain minimum viscosity of the slurry. The final test, starting from a slurry made up to its deflocculant demand, is to determine the **viscosity concentration**, which is the solids concentration of a fully deflocculated clay that has a viscosity of 5 poise at 22°C. All three tests are sequentially related, with flowability concentration required for deflocculant demand, and this in turn being required for viscosity concentration. It is this last test that provides the conditions under which samples of different kaolinites can be compared, the figure of five poise being chosen as this is the viscosity suitable for paper coating, although the viscosity at a specific solids concentration could also be calculated.

The test procedures followed here are specific to the paper industry, although those used in the ceramics industry are similar. In a ceramic viscosity test procedure a slurry is prepared with deflocculant added in small amounts until flowability is reached. The slurry is then left to stand, after which further additions of deflocculant are made, coupled with viscosity measurement, until viscosity no longer falls. At this point water is added until the viscosity is 5 poise, and the solids-content of this final slurry is

known as the **casting concentration**. It is apparent that viscosity concentration and casting concentration are equivalent, and the method outlined in Appendix 1 will provide useful information about kaolinite samples suitability for use in both paper and ceramics.

### 3.2. Results

The following results were all obtained by following the methods outlined in Appendix 1. The results are given in Table 1. Deflocculant demand is plotted as viscosity (in centipoise) against volume of deflocculant (in ml of 25% calgon solution). Viscosity concentration is plotted as solids concentration against a viscosity unit (which is the reciprocal of the square root of viscosity in poise). Figures 2 - 12 give the deflocculant demand and viscosity concentration graphs respectively for each of the samples. Figure 13 contains all the viscosity concentration data plotted together. TEM and SEM photomicrographs of the samples are given in Figures 15 - 30.

#### 3.2.1. SPS

This is a high-grade coating clay produced by ECC and widely used in the European paper and board industries. It is quoted as having 80%  $<2\ \mu\text{m}$  and a viscosity concentration of 69%. The clay was found to contain 79%  $<2\ \mu\text{m}$  with a grade of 82% kaolinite. The flowability was 67.5%, with a deflocculant demand of 0.9 ml 25% calgon soln/100g sample and a viscosity concentration of 66.3%.

#### 3.2.2. Supreme

A very high-grade paper coating clay produced by ECC, that is quoted as having 94%  $<2\ \mu\text{m}$  and a viscosity concentration of 67.5%. The clay was found to contain 92%  $<2\ \mu\text{m}$ , with a grade of 95% kaolinite. The flowability was 66.6%, the deflocculant demand 0.8 ml 25% calgon soln/100g sample, and the viscosity concentration 66.4%.

#### 3.2.3. Dinkie A

A paper coating clay produced by ECC, that was found to contain 70%  $<2\ \mu\text{m}$  with a grade of 88% kaolinite. The flowability was 70.5 %, deflocculant demand 0.72 ml 25% calgon soln/100g sample, and the viscosity concentration 71.1%.

#### 3.2.4. Lee Moor

This Cornish kaolinite was obtained in the raw state and on processing gave a product with characteristics akin to those of a paper coating clay. It contained 55%  $<2\ \mu\text{m}$  and had a grade of 91% kaolinite. The flowability was 64.8%, the deflocculant demand was 1.2 ml 25% calgon soln/100g sample and the viscosity concentration was 69.4%.

### 3.2.5. Standard Porcelain

Standard Porcelain is a ceramic grade china clay produced by ECC, quoted as having 70% <2  $\mu\text{m}$  and a casting concentration of 62%. It was found to contain 65% <2  $\mu\text{m}$  and have a kaolinite grade of 85%. The flowability was 64.6%, with a deflocculant demand of 0.56 ml 25% calgon soln/100g sample and a viscosity concentration of 64.7%.

### 3.2.6. Treviscoe

Treviscoe is another ceramic grade china clay produced by ECC, quoted as containing 42% < 2  $\mu\text{m}$  and having a casting concentration of 62%. It was found to contain 37% <2  $\mu\text{m}$  and have a kaolinite grade of 83%. The flowability was 66.3%, with a deflocculant demand of 3.84 ml 25% calgon soln/100g sample and a viscosity concentration of 62.1%.

### 3.2.7. Eburru

This kaolinite was collected from the East African Rift Valley of Kenya and formed by hydrothermal alteration of obsidian. It was processed from the raw state and found to contain 63% <2  $\mu\text{m}$ , with a kaolinite grade of 97%. The flowability was 64.6%, with a deflocculant demand of 1.52 ml 25% calgon soln/100g sample and a viscosity concentration of 61.7%.

### 3.2.8. Muranga 5, 6 and 7

This kaolinite is a composite of three samples (combined to provide enough material for viscosity measurement) collected from Kenya, and consisted of a mixture of halloysite and kaolinite. The sample was processed from the raw state to give a product containing 51% <2  $\mu\text{m}$  and 97% kaolinite. The flowability was found to be 64.9%, with a deflocculant demand of 1.28 ml 25% calgon soln/100g sample and a viscosity concentration of 57.0%.

### 3.2.9. Muranga 12 and 14

This is a composite of two sample (combined to provide enough material for viscosity measurement) from Kenya, containing halloysite and kaolinite. It was processed from the raw state to give a product containing 49% <2  $\mu\text{m}$  and 94% kaolinite. The flowability was found to be 48.6%, with a deflocculant demand of 1.92 ml 25% calgon soln/100g sample and a viscosity concentration of 52.9%.

### 3.2.10. Pugu Hard

Pugu Hard was separated from a kaolinite-bearing sandstone from the Pugu Hills of Tanzania and is composed of 'Pugu D', a *b*-axis disordered kaolinite, which, unusually, has well-formed hexagonal crystals. The processed product contained 79% <2  $\mu\text{m}$ , and had a kaolinite grade of 98%. The flowability was 45.7%, the deflocculant demand 1.1 ml 25% calgon soln/100g sample and the viscosity concentration 62.0%.

### 3.2.11. Pugu Soft

Pugu Soft has the same derivation as Pugu Hard, but it is composed of 'Pugu K', a structurally well-ordered kaolinite. This material was processed from the raw state, the product containing 62% <2  $\mu\text{m}$  and 85% kaolinite. The flowability was found to be 47.9%, the deflocculant demand 1.04 ml 25% calgon soln/100g sample and the viscosity concentration 71.2%.

### 3.2.12. Goonvean kaolinite R9-55

This is a kaolinite from the Goonvean and Rostowrack China Clay Company. It contains 59% kaolinite and has 47% <2  $\mu\text{m}$ . The flowability was found to be 54%, but the deflocculant demand and the viscosity concentration could not be determined. This was due to the high proportion of smectite (4.5% Ca smectite) present.

## 3.3. Discussion of results

Ideally paper manufacturers require a kaolinite that will produce a fluid, high-solids-content slurry that can be pumped, screened and applied to paper via high-speed blades without any production problems. The slurry should contain between 60-70% solids (by weight), with a viscosity in the region of 5 poise.

Before discussing the results in detail it is necessary to briefly outline the effect of particle geometry on viscosity. Kaolins with a narrow size distribution will form a slurry with an 'open' structure, due to the inefficient packing of closely sized particles. For a given solids-content the viscosity will be higher than for a kaolin with a broad size distribution, as more water is required to fill the gaps between particles. A kaolin with an 'open' structure is known to give a good coating, with good optical properties (high brightness) and printability. Ideally the interstices should be 0.3-0.7  $\mu\text{m}$  in diameter, which is the average diameter of the  $\text{TiO}_2$  pigment particles used to whiten paper.

Kaolins with a broad particle size range have better packing, resulting in lower viscosities for a given solids-content, as small particles fill the interstices which water would occupy. The higher degree of packing means that for a given viscosity the



solids-content is higher, which leads to denser coating films and agglomerations in filled sheets, giving rise to inferior printing properties. Therefore a balance must be struck between kaolinites with close or broad particle size ranges to obtain a slurry with acceptable viscosity, and optical and printing properties.

To gauge the 'closeness' of the particle-size distributions of the samples their 'inclusive graphic standard deviations' were calculated (see Table 1) which were then plotted against viscosity concentration. Closely sized material has a low standard deviation and vice versa. The samples combined (excluding the Muranga samples) gave a negative correlation coefficient, of about -0.5. This indicates the opposite to that expected, material with a close size distribution has higher solids-contents than that with a broad size distribution. The correlation coefficient of all the samples is only -0.2.

In an attempt to gauge the influence of other properties on the viscosity of the samples investigated, correlation coefficients were calculated for viscosity against the following properties; flowability, deflocculant demand, graphic mean particle size, clay grade, and kaolinite grade. None of the values exceeded 0.5, and although the better of the correlations were obtained from the last three (see Table 2) they are not high enough to draw any meaningful conclusions.

Particle shape influences viscosity via the effective volume (or 'swept' volume) a particle occupies during movement of the slurry. The swept volume of non-spherical particles, such as plates (kaolinite) and rods (halloysite), is controlled by the largest diameter, and volume increases disproportionately with increase in size. The two Muranga samples have low viscosity concentrations due to their high viscosities, which is probably due to the rod-like shape of their halloysitic components. This increase in viscosity is thought to be more a result of the rods tangling during movement than to the volume they occupy.

The samples investigated show similar responses to the various viscosity tests performed. The deflocculant demand plots all characteristically show a decrease in viscosity with addition of deflocculant until the viscosity either levels out or begins to rise again. SPS kaolinite deflocculant demand is a good example of the later response. The initial viscosity can be between 100 - 7000 centipoise, decreasing sharply to the deflocculant demand viscosity, which ranges from 30 - 2000 centipoise, depending on the sample.

The viscosity concentration plots show an increase in viscosity with increasing solids concentration. The viscosity concentration is taken at 5 poise, which corresponds to 0.447 viscosity units and most samples plot around this value. This makes extrapolation of the viscosity at 5 poise reasonably reliable. However the Pugu samples do not plot around 5 poise and this seems to make the extrapolation less reliable, even

though their extrapolated viscosity concentrations are close to those of the other samples. This can be seen to better effect in Figure 13 where the Pugu samples plot at the low viscosity and low solids concentration end of the graph. This is probably due to the presence of soluble salts in the Pugu samples. If the plots in Figure 13 were to be extended to encompass lower and higher solids concentration the data would plot as a curve. The low solids concentration viscosities would approach that of the suspending medium and the high solids concentration that of a solid. The viscosity concentration is taken at five poise, which occurs on the bend in the curve.

Figure 13 shows a broad similarity between the kaolinities in their relationship of viscosity to solids-content. No relationship with any physical properties are apparent which fit this scenario, except that broadly those with high solids-contents (65%+) at 5 poise are paper grade, those with solids-contents from 55-65% are ceramic grade and those below 55% have viscosities too high for these applications.

The problems in trying to determine the rheological properties of the Goonvean kaolinite demonstrate the influence of smectite upon viscosity. Figure 14 shows the deflocculant demand graph: viscosity is seen to rise with addition of deflocculant and only falls when an excessive amount is added. The increase in viscosity is possibly due to the exchange of the Ca ions in the smectite for the Na ions in the deflocculant.

#### **4. CONCLUSIONS**

The use of the Brookfield viscometer in measuring the viscosity of kaolinite products has been found to be quick and easy. However, the sample preparation is critical and in order to produce meaningful results, such as the viscosity concentration, slurries that meet strictly defined properties are required. The viscosity concentration is the solids concentration of a slurry with a viscosity of 5 poise at 22°C, with the required flowability (solids concentration at which a slurry just flows) and deflocculant demand (amount of deflocculant required to produce the minimum viscosity). The study of the influence of particle geometry on viscosity was made difficult by the range of kaolinitic material investigated, but with each future sample studied the database of information will grow and make this study more accurate and reliable. The present investigation has shown that viscosity measurement can be used in the evaluation of kaolinite samples and in comparison with commercial paper and ceramic grades to indicate their industrial suitability.

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**Table 1. Mineralogy and physical properties of kaolinities**

Sample	Kaolinite content (Wt %)	Remaining mineralogy	Particle size (µm) <10 (Wt %)	<2 (Wt %)	Graphic * mean Std dev (µm) (phi)	Flowability (Wt %)	Deflocculant demand **	Viscosity concentration (Wt%)
SPS	82	Qtz, musc.	97	79	1.0 0.52	67.5	0.9	66.3
Supreme	95	Qtz, musc.	98	92	0.7 0.27	66.6	0.8	66.4
Dinkie A	88	Qtz, musc, K-fld.	97	70	1.4 0.51	70.5	0.7	71.1
Lee Moor	91	Qtz, musc, biot, horn, tour.	94	55	2.8 0.40	64.8	1.2	69.4
Standard Porcelain	85	Musc, qtz.	97	65	1.8 0.47	64.6	0.6	64.7
Treviscoe	83	Musc.	89	37	4.1 0.51	66.3	3.8	62.1
Eburru	97	Qtz, Fe ox.	96	63	2.3 0.61	64.6	1.5	61.7
Muranga 5, 6 and 7	97	Qtz, musc, haem, K-fld.	92	51	3.0 0.57	64.9	1.3	57
Muranga 12 and 14	94	Qtz, musc, haem, K-fld.	95	49	3.0 0.39	48.6	1.9	52.9
Pugu Hard	98	Qtz, sol. salts.	90	79	1.5 0.58	45.7	1.1	62
Pugu Soft	85	Qtz, sol. salts.	97	62	2.0 0.43	47.9	1.0	71.2
Goonvean R9-55	59	K-fld, Na-Sm, qtz, alb.	96	47	2.9 0.74	54	-	-

**Key**

Qtz = Quartz, Musc = Muscovite mica, K-fld = Potassium feldspar, Alb = Albite feldspar, Biot = Biotite mica, Horn = Hornblende, Tour = Tourmaline, Fe ox = Iron oxides, Haem = Haematite, Sol. salts = Soluble salts, Na-Sm = Sodium smectite.

\* Formulae for calculating graphic mean and inclusive graphic standard deviation are given in Table 2.

\*\* Deflocculant demand values represent ml of 25% calgon solution required per 100g of sample to attain minimum viscosity.

**Table 2. Viscosity concentration correlation coefficients**

<b>Property</b>	<b>All samples (- Muranga)</b>	<b>All samples (+ Muranga)</b>
<b>Kaolinite content</b>	-0.32	-0.49
<b>Percentage &lt;2 <math>\mu\text{m}</math></b>	0.11	0.4
<b>Graphic mean</b>	-0.25	-0.47
<b>Inclusive graphic standard deviation</b>		
- $\mu\text{m}$	-0.53	-0.21
- phi (log)	-0.49	-0.21
<b>Flowability</b>	0.06	0.29
<b>Deflocculant demand</b>	-0.47	-0.41

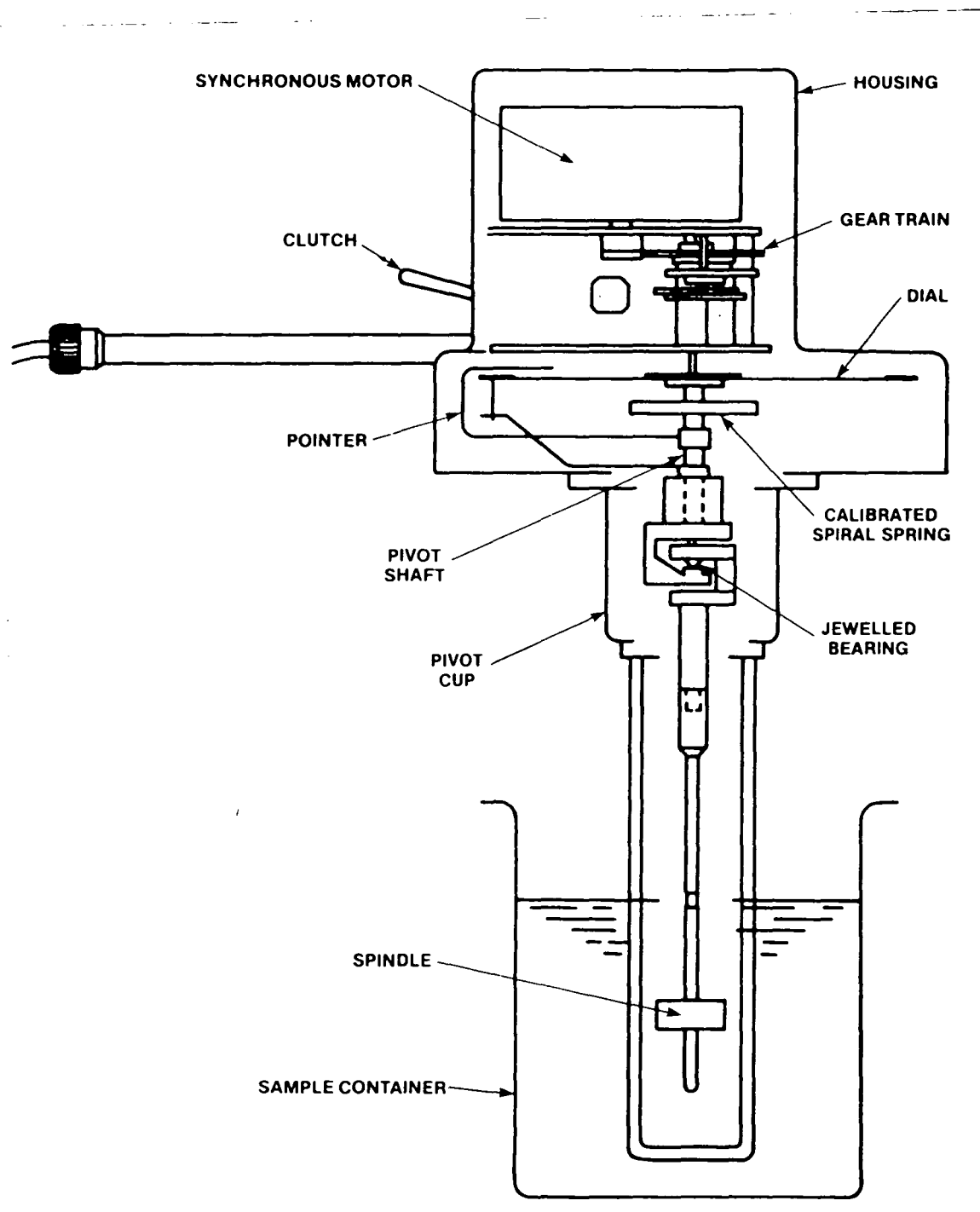
**Graphic mean formula**  $(d_{84} + d_{50} + d_{16}) / 3$

**Inclusive graphic standard deviation**

-  $\mu\text{m}$  formula  $(d_{84} - d_{16} / 4) + (d_{95} - d_5 / 6.6)$

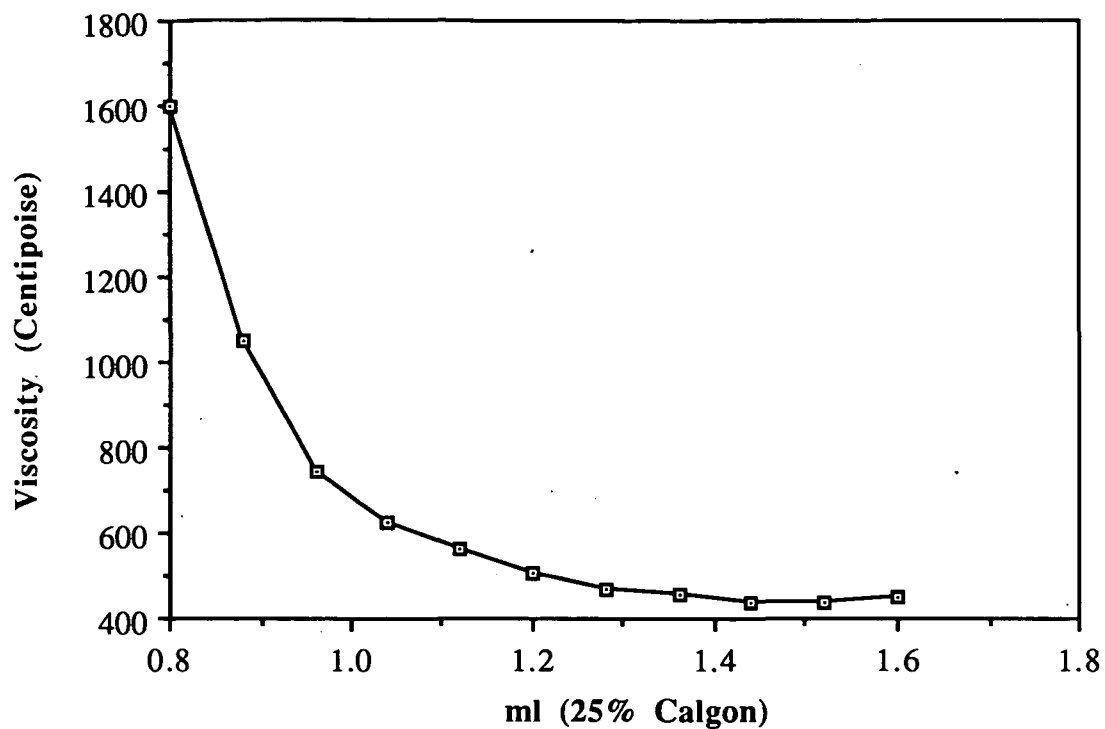
- phi formula  $(\phi_{84} - \phi_{16} / 4) + (\phi_{95} - \phi_5 / 6.6)$

(d = diameter at corresponding cumulative percentage.  $\phi$  = Log of d.)



**Figure 1 Brookfield low-shear viscometer.**

### Deflocculant demand



### Viscosity concentration

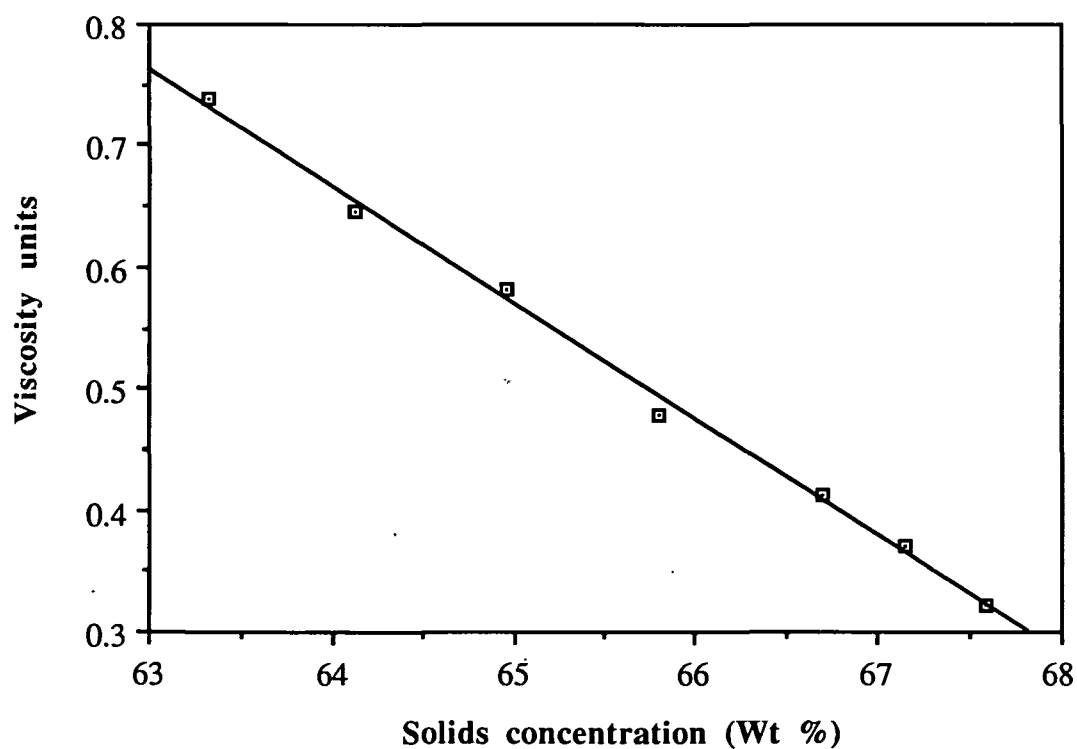
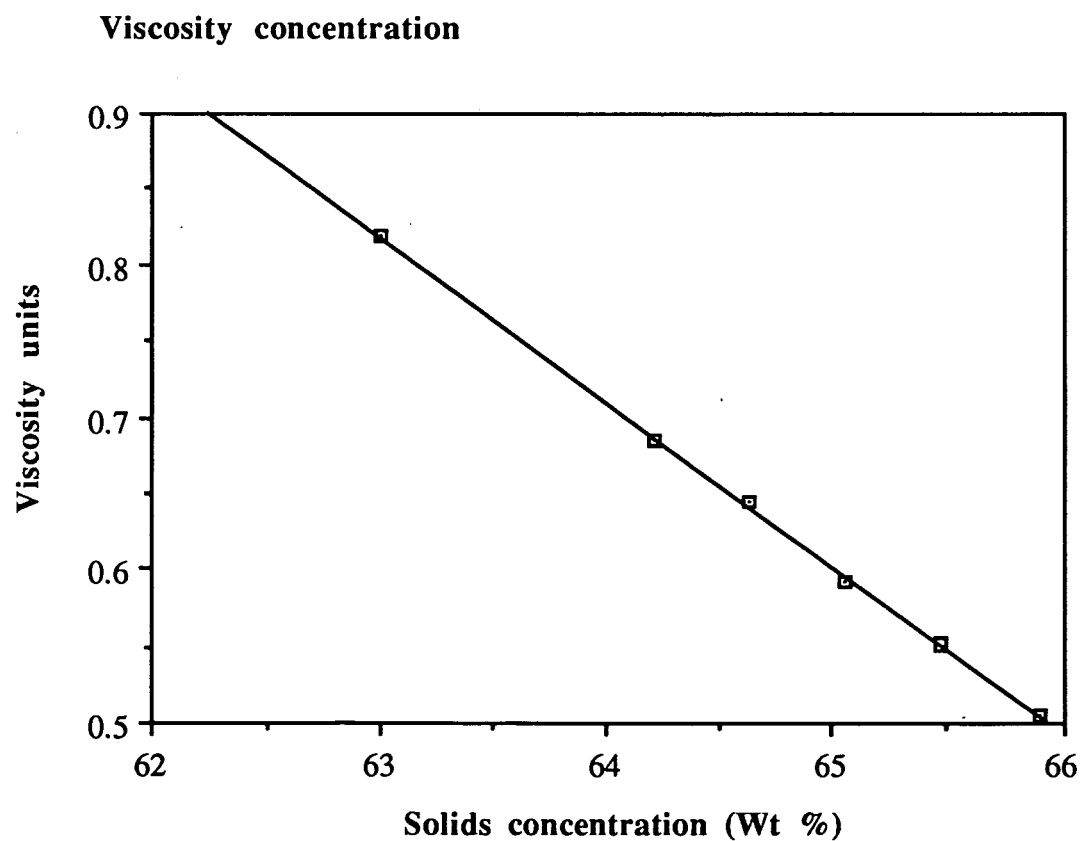
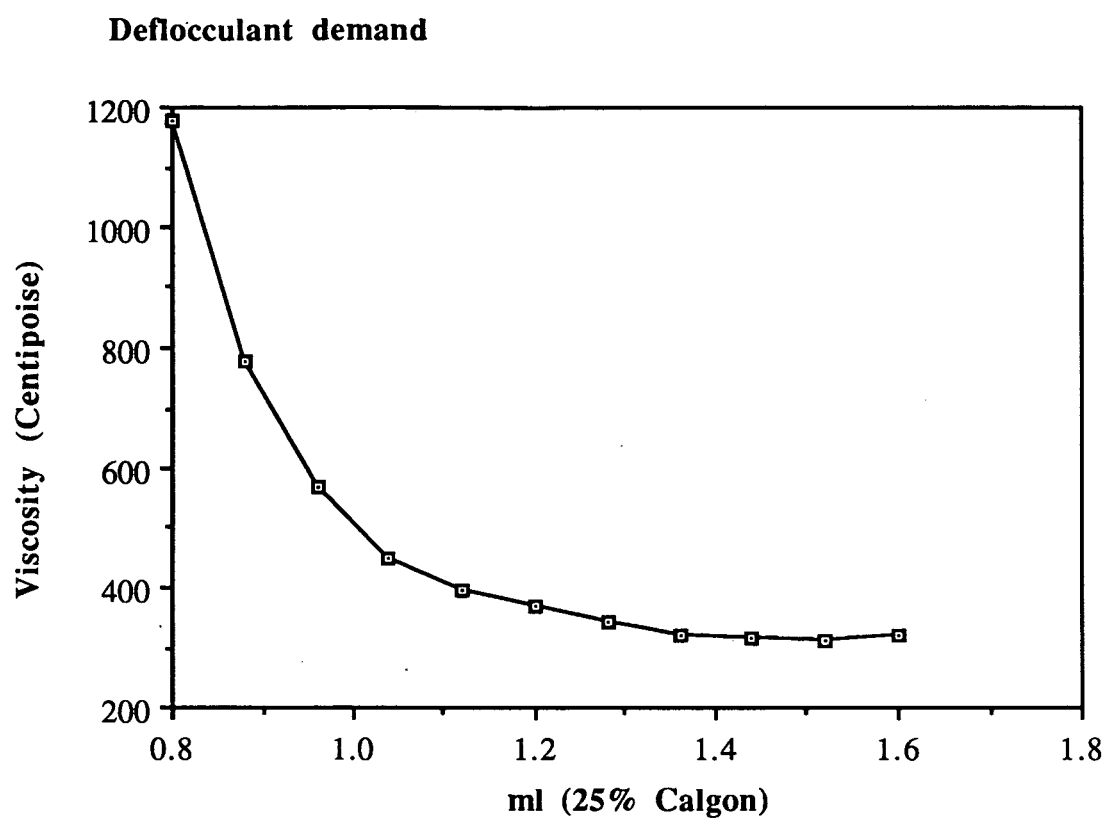
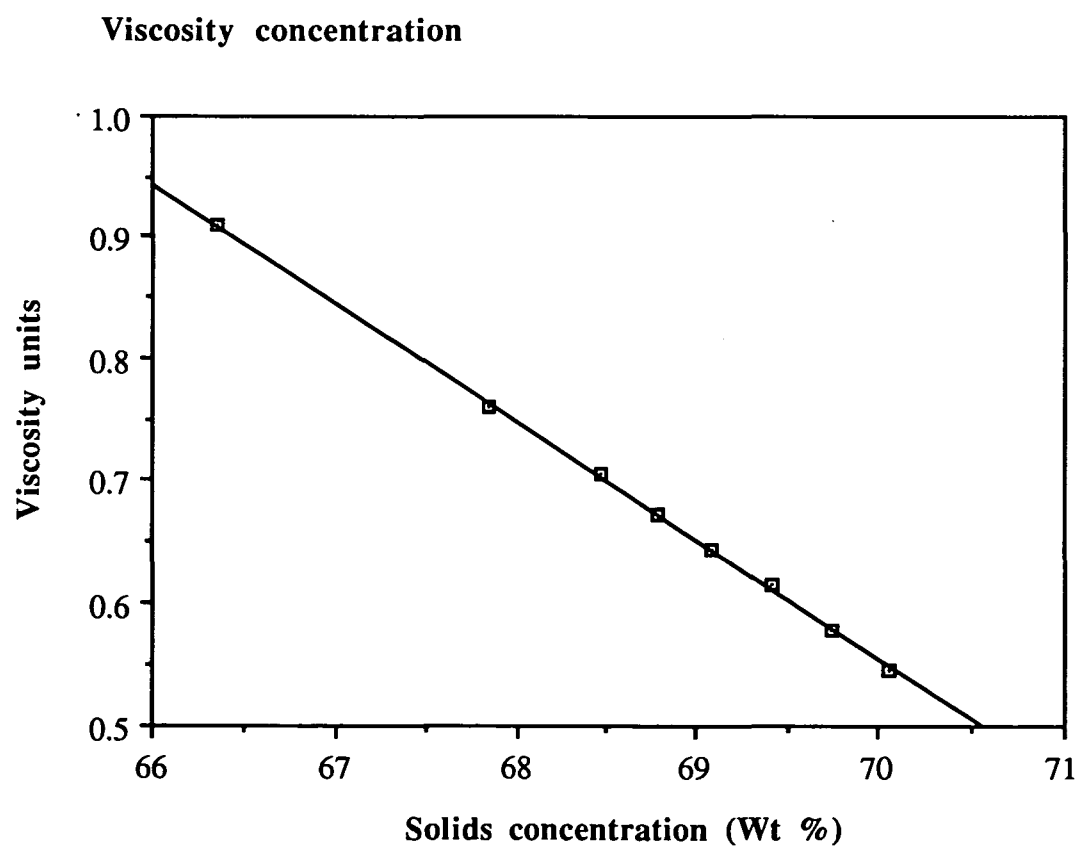
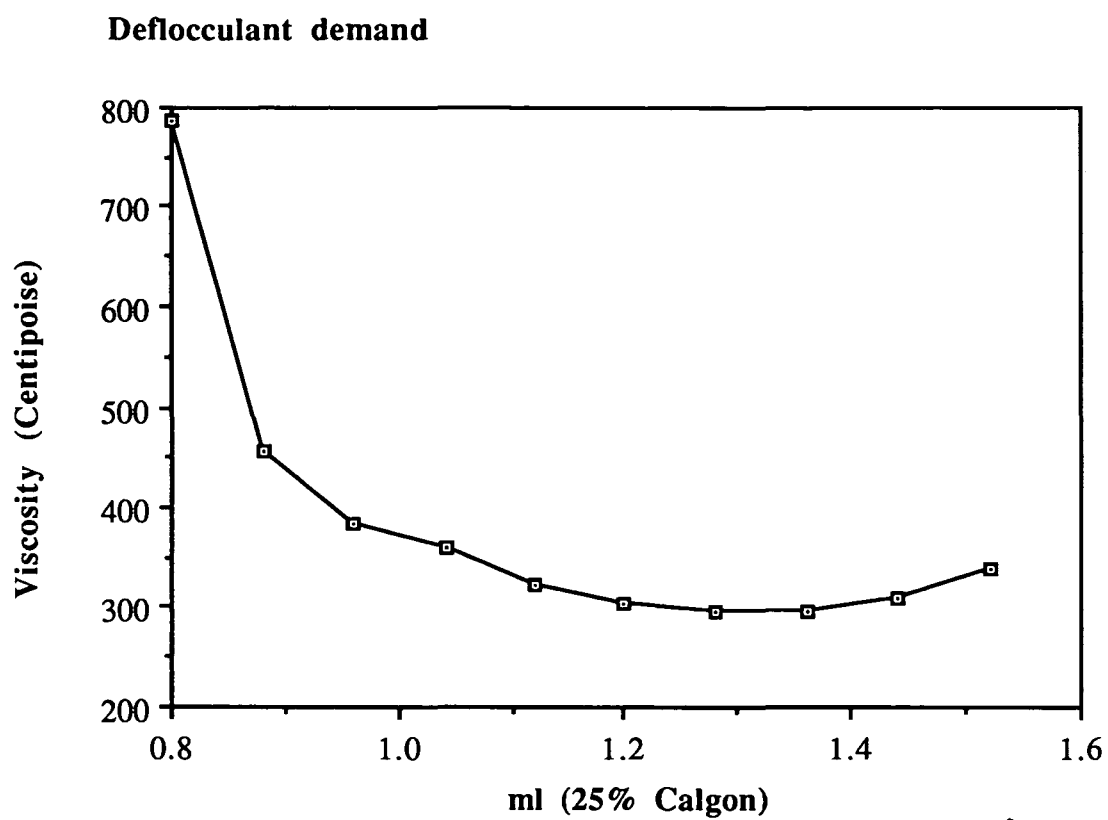


Figure 2 Deflocculant demand and viscosity concentration of SPS kaolinite



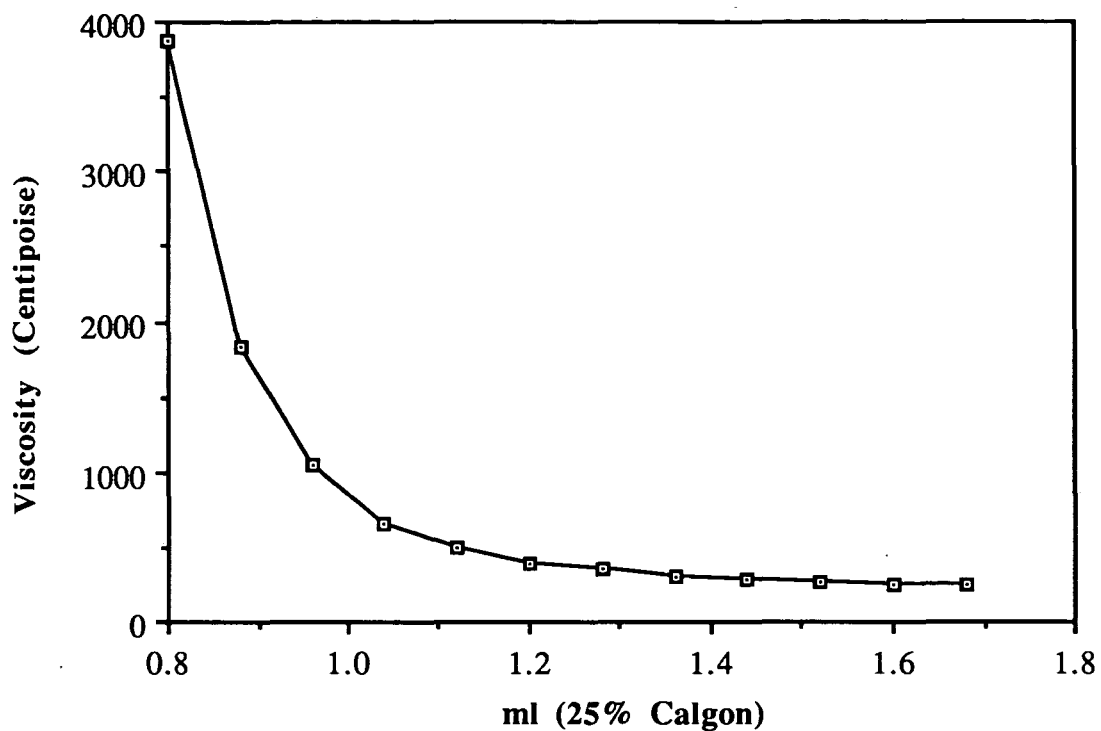


**Figure 3 Deflocculant demand and viscosity concentration of Supreme kaolinite**



**Figure 4 Deflocculant demand and viscosity concentration of Dinkie A kaolinite**

### Deflocculant demand



### Viscosity concentration

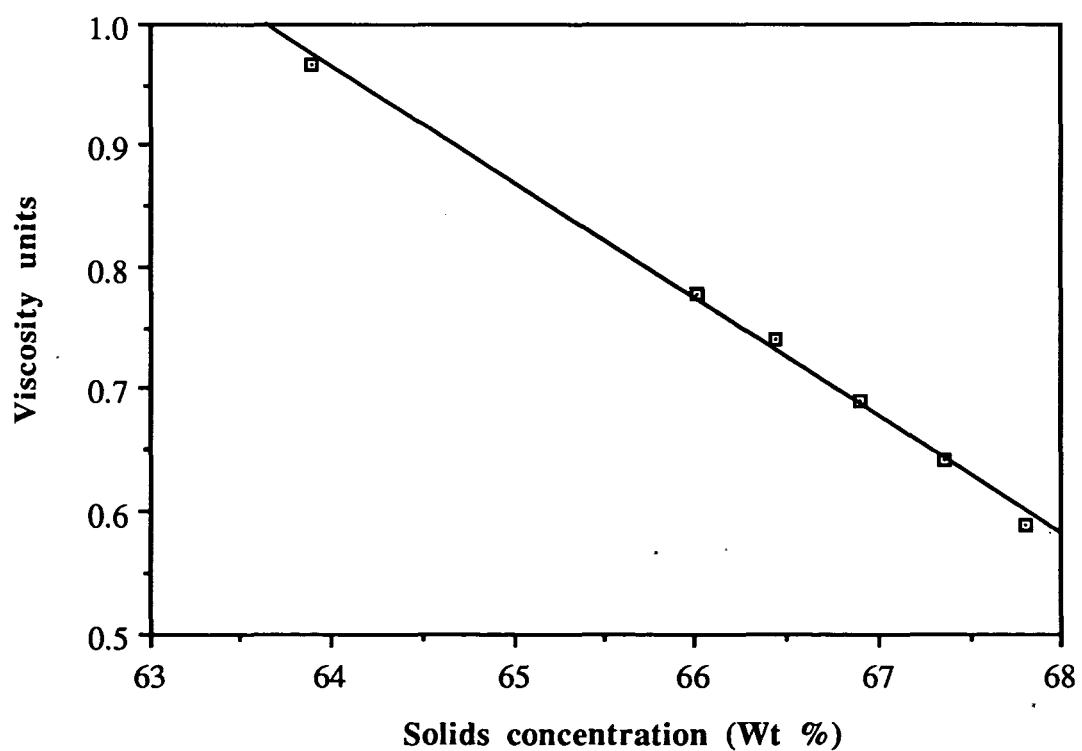
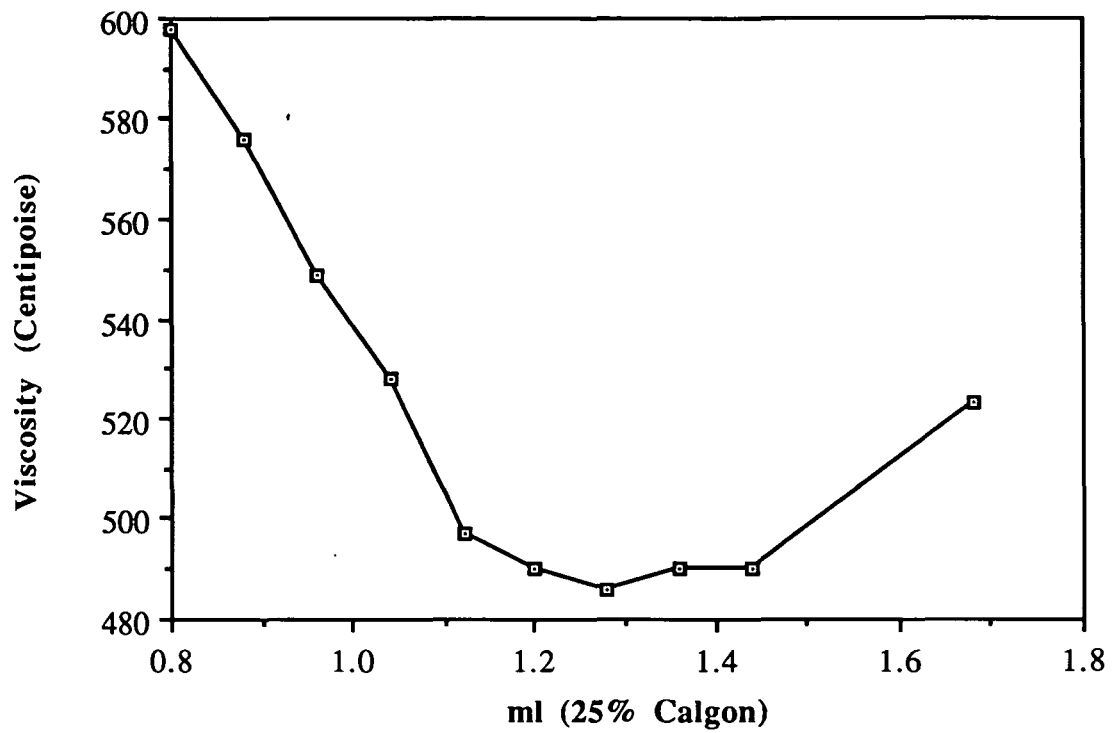


Figure 5 Deflocculant demand and viscosity concentration of Lee Moor kaolinite

### Deflocculant demand



### Viscosity concentration

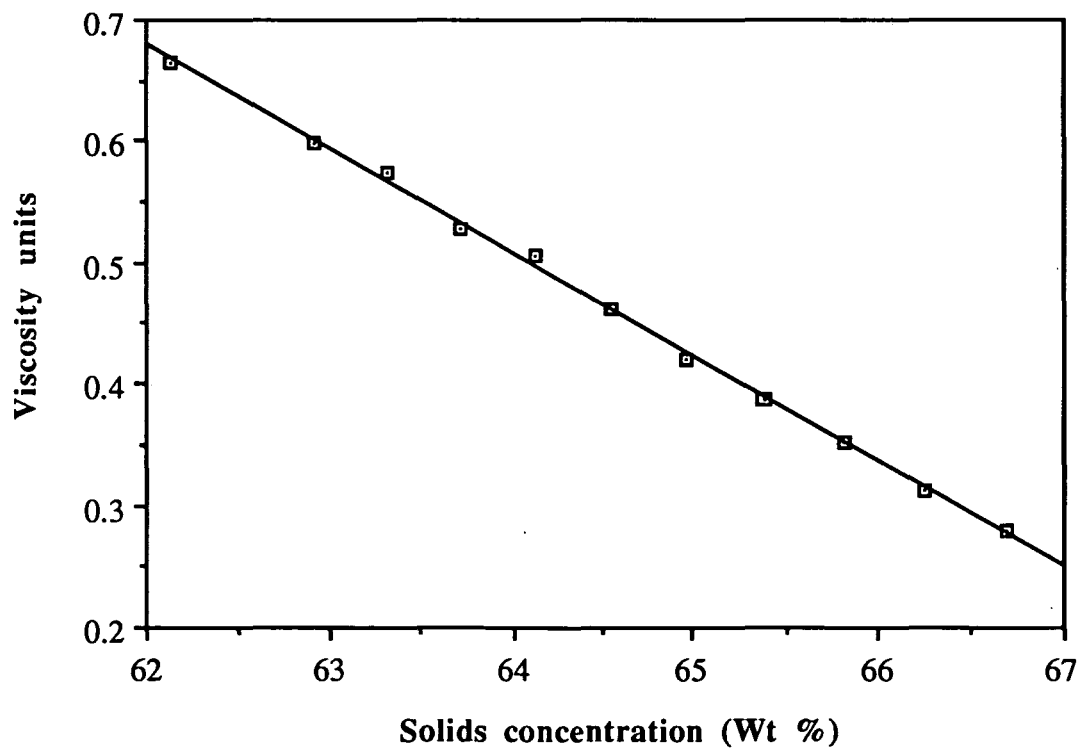
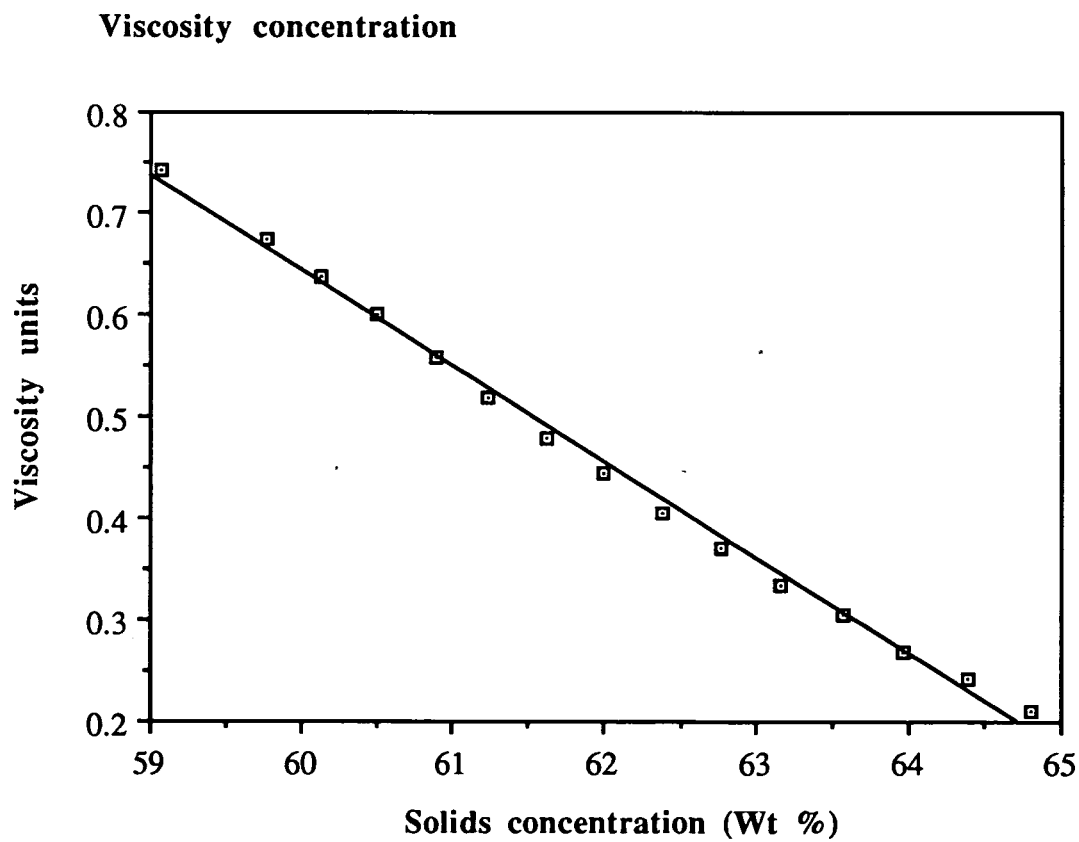
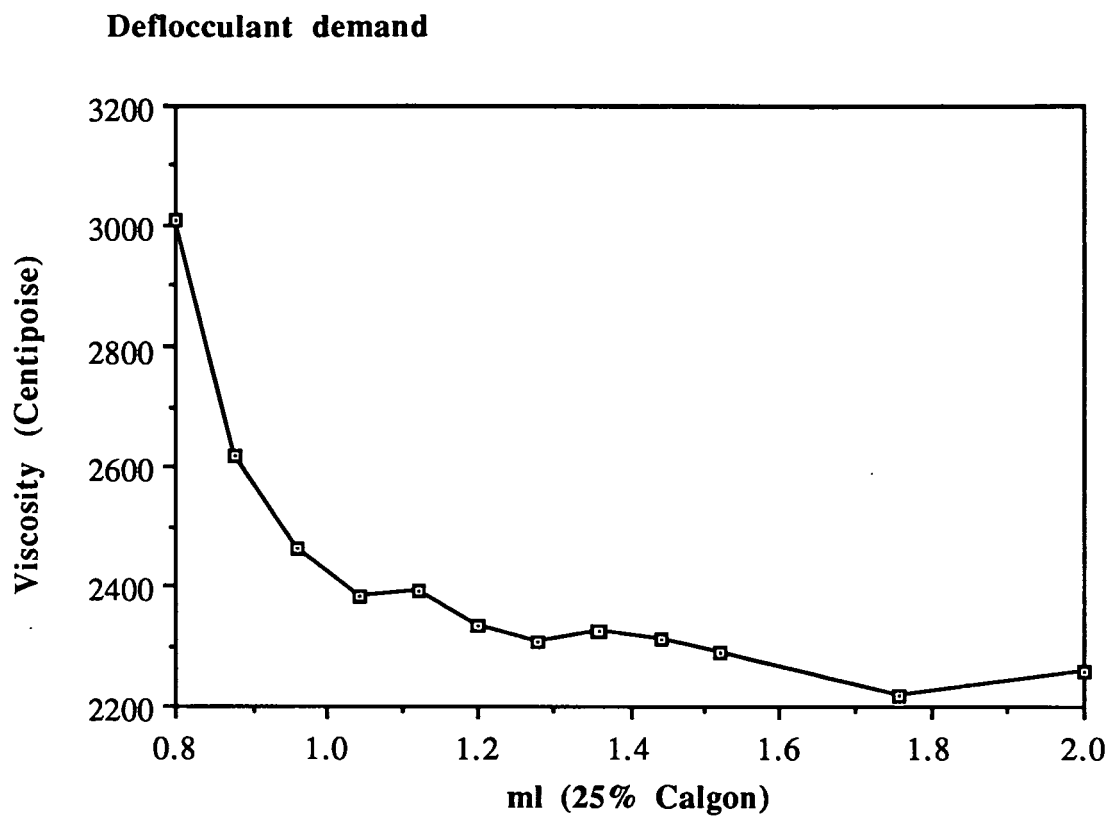
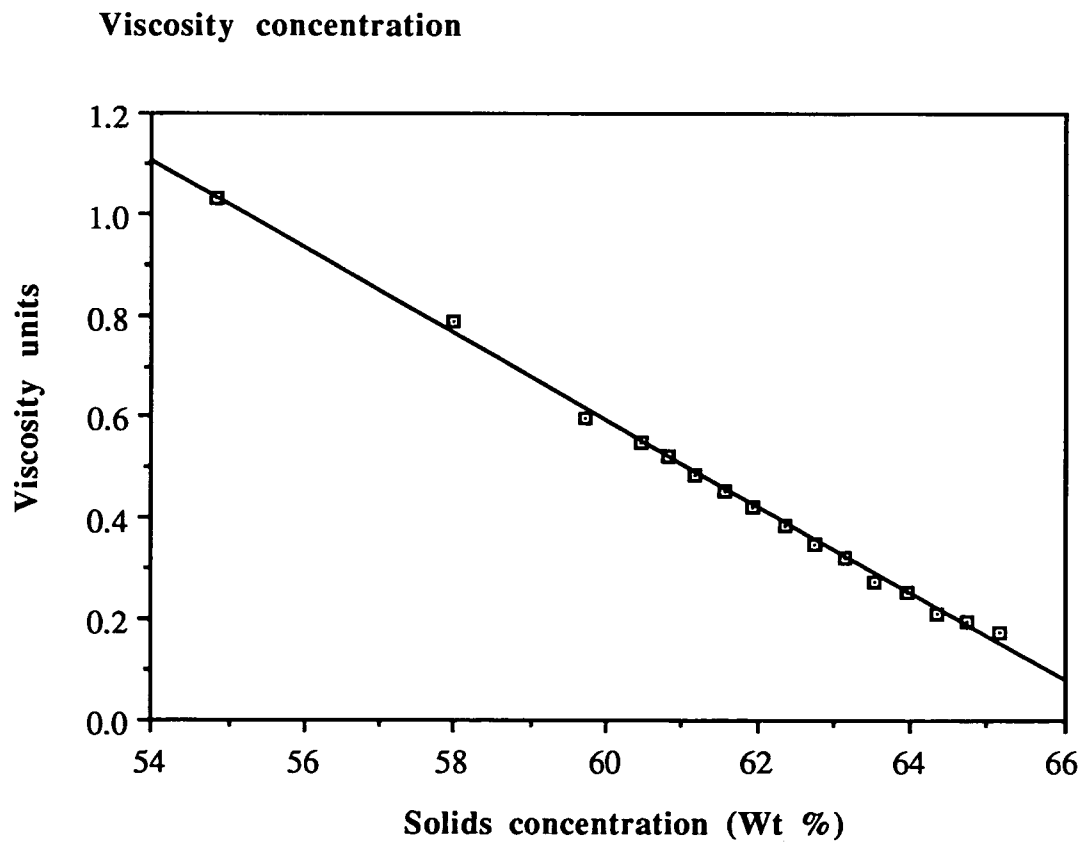
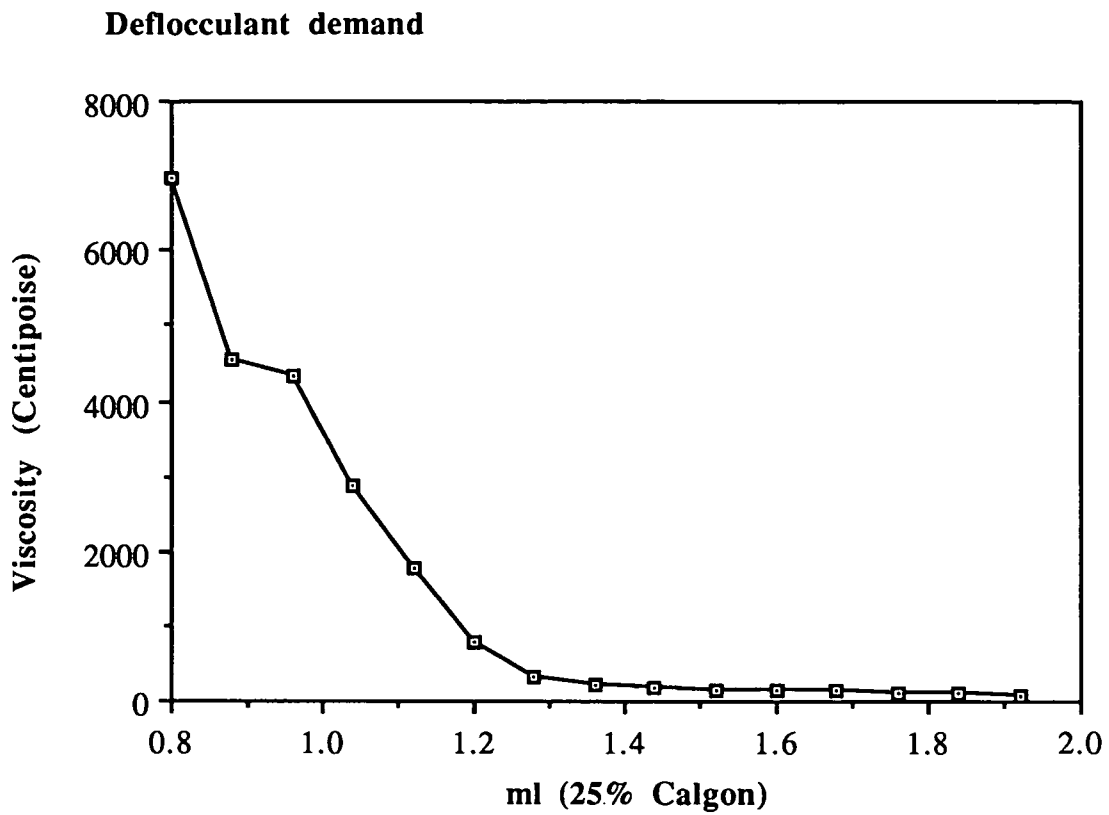


Figure 6 Deflocculant demand and concentration of Standard Porcelain kaolinite

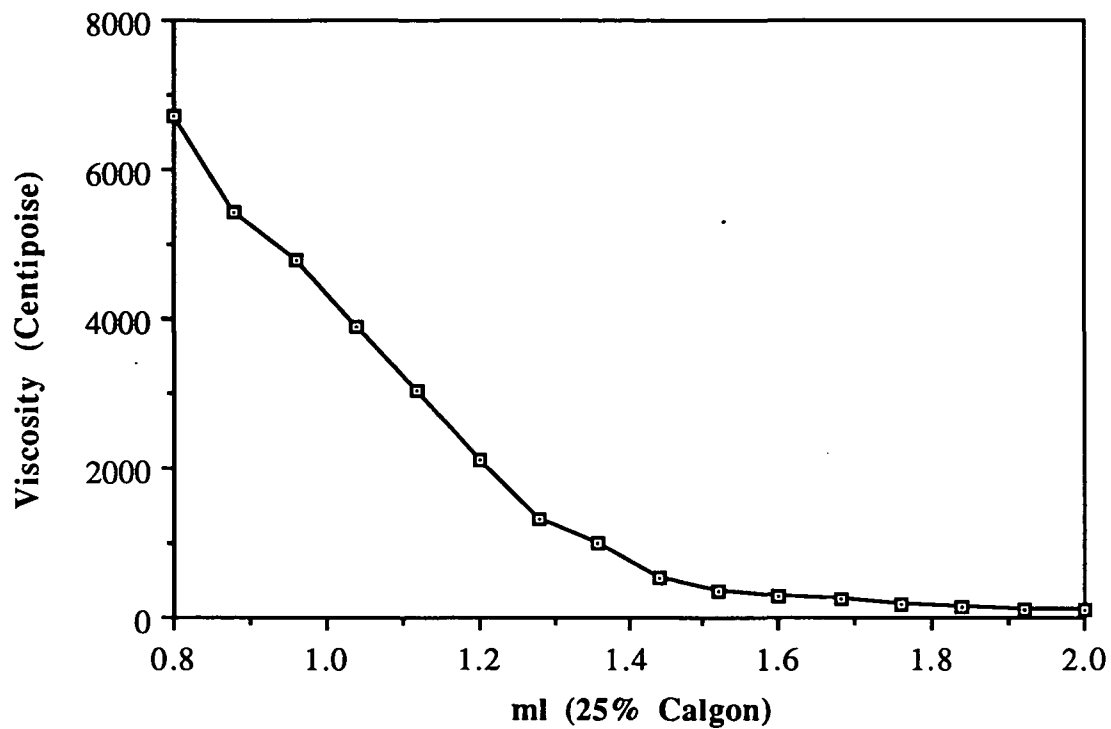


**Figure 7 Deflocculant demand and viscosity concentration of Treviscoe kaolinite**

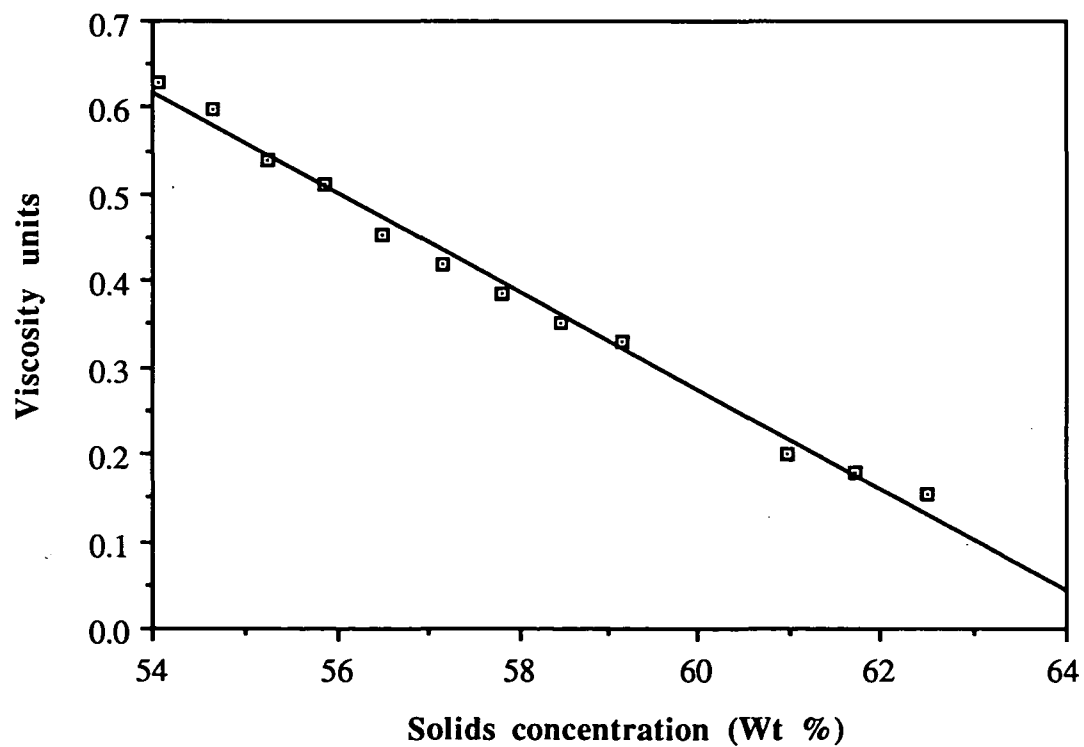


**Figure 8 Deflocculant demand and viscosity concentration of Eburru kaolinite**

### Deflocculant demand

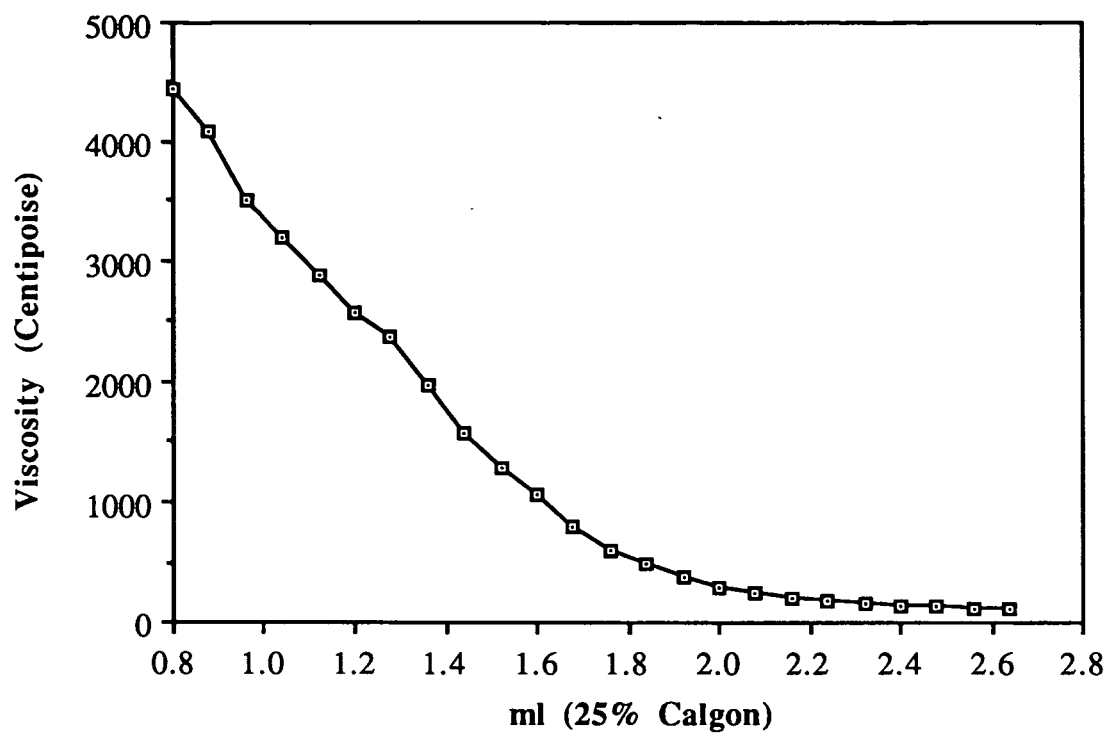


### Viscosity concentration

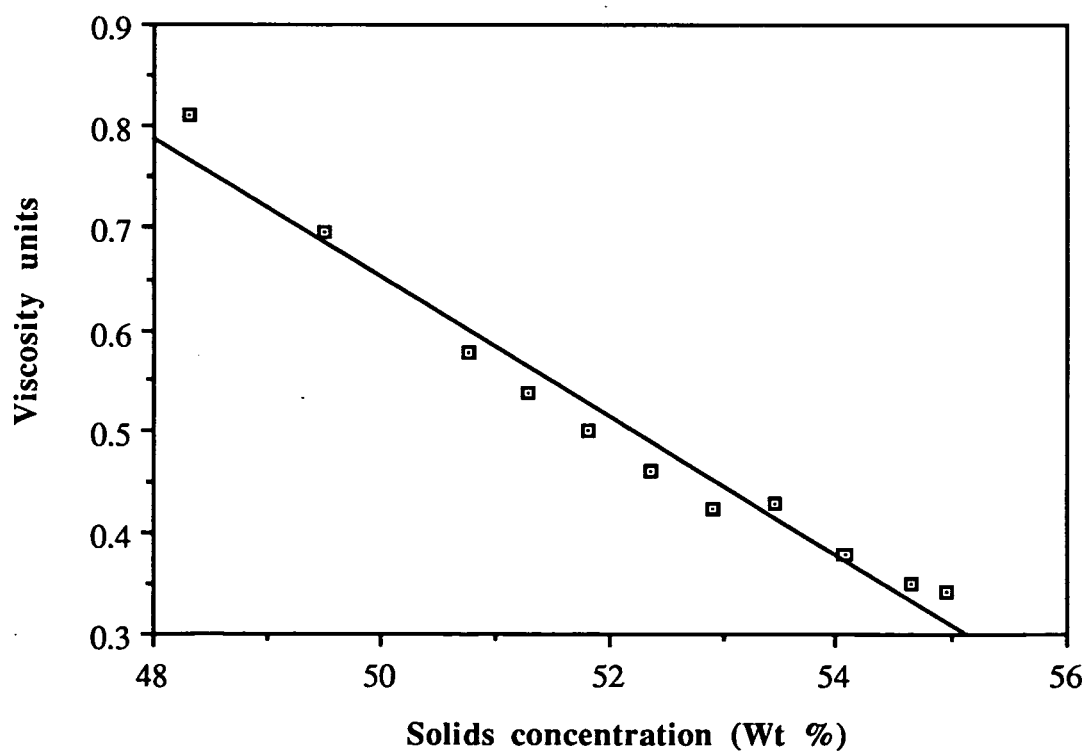


**Figure 9 Deflocculant demand and viscosity concentration of Muranga 5, 6 and 7 kaolinite**

### Deflocculant demand



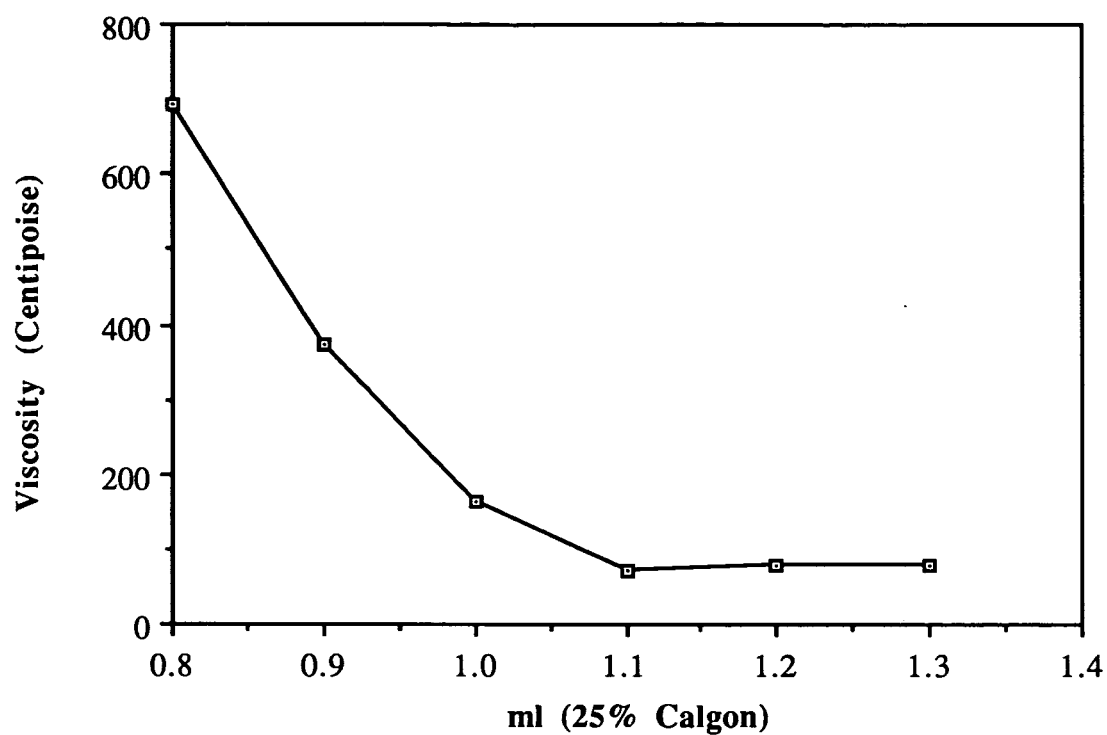
### Viscosity concentration



**Figure 10 Deflocculant demand and viscosity concentration of Muranga 12 and 14 kaolinite**



### Deflocculant demand



### Viscosity concentration

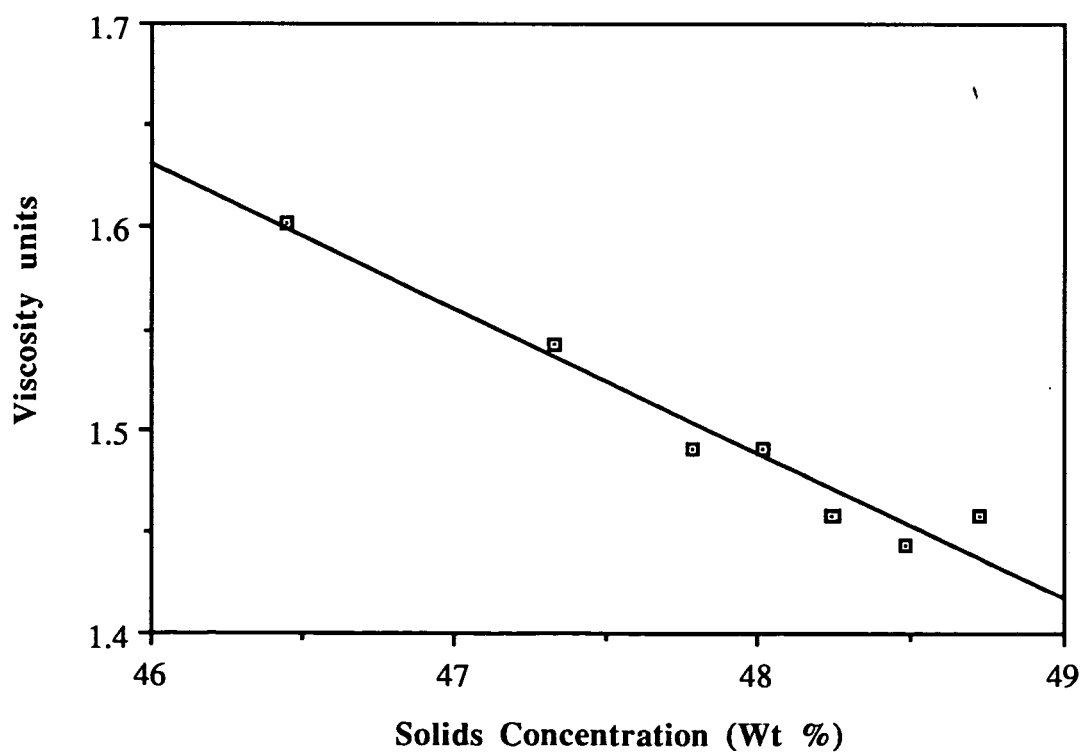
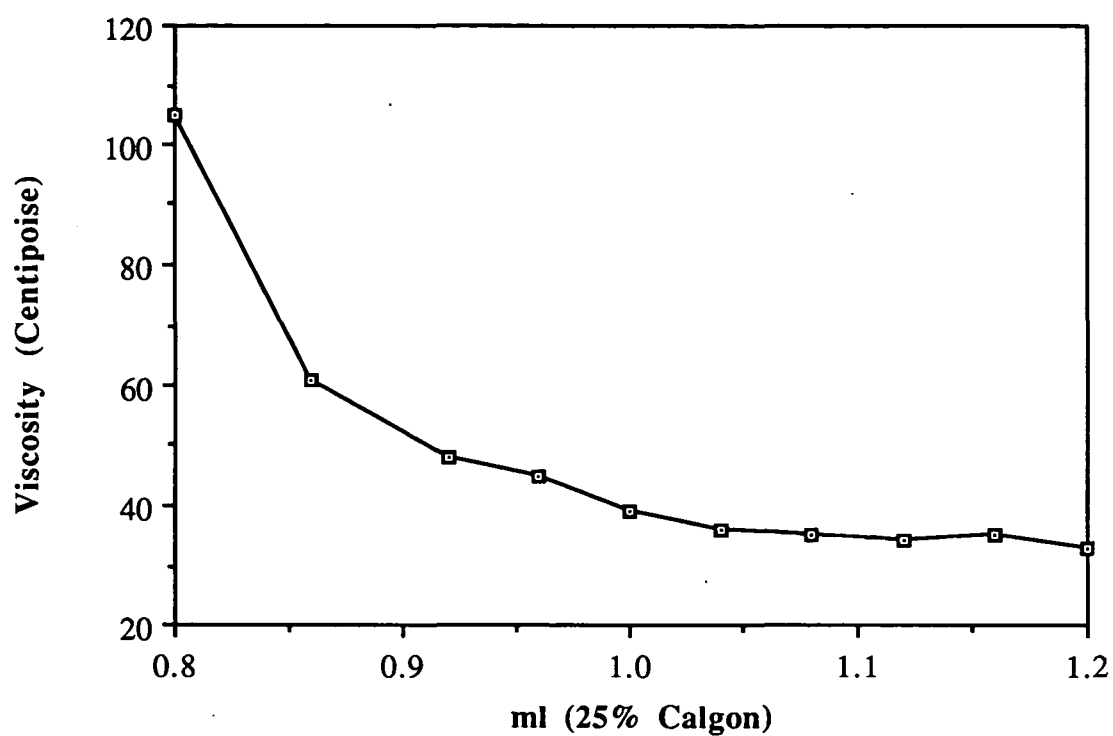


Figure 11 Deflocculant demand and viscosity concentration of Pugu Hard kaolinite

### Deflocculant demand



### Viscosity concentration

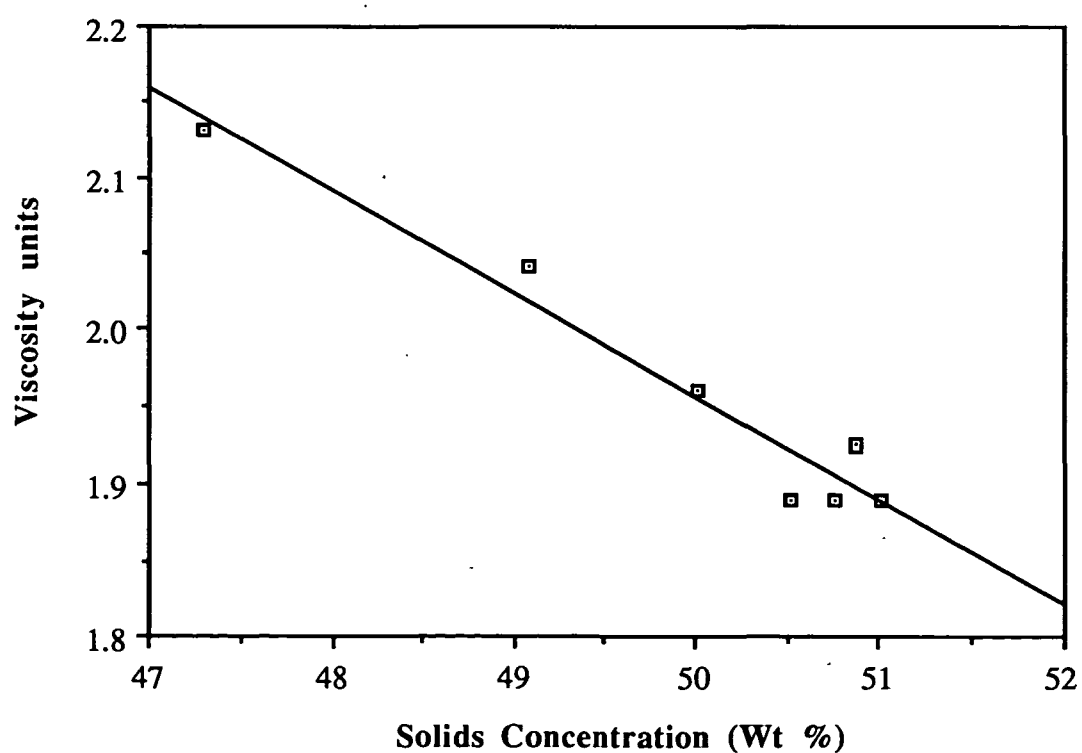


Figure 12 Deflocculant demand and viscosity concentration of Pugu Soft kaolinite

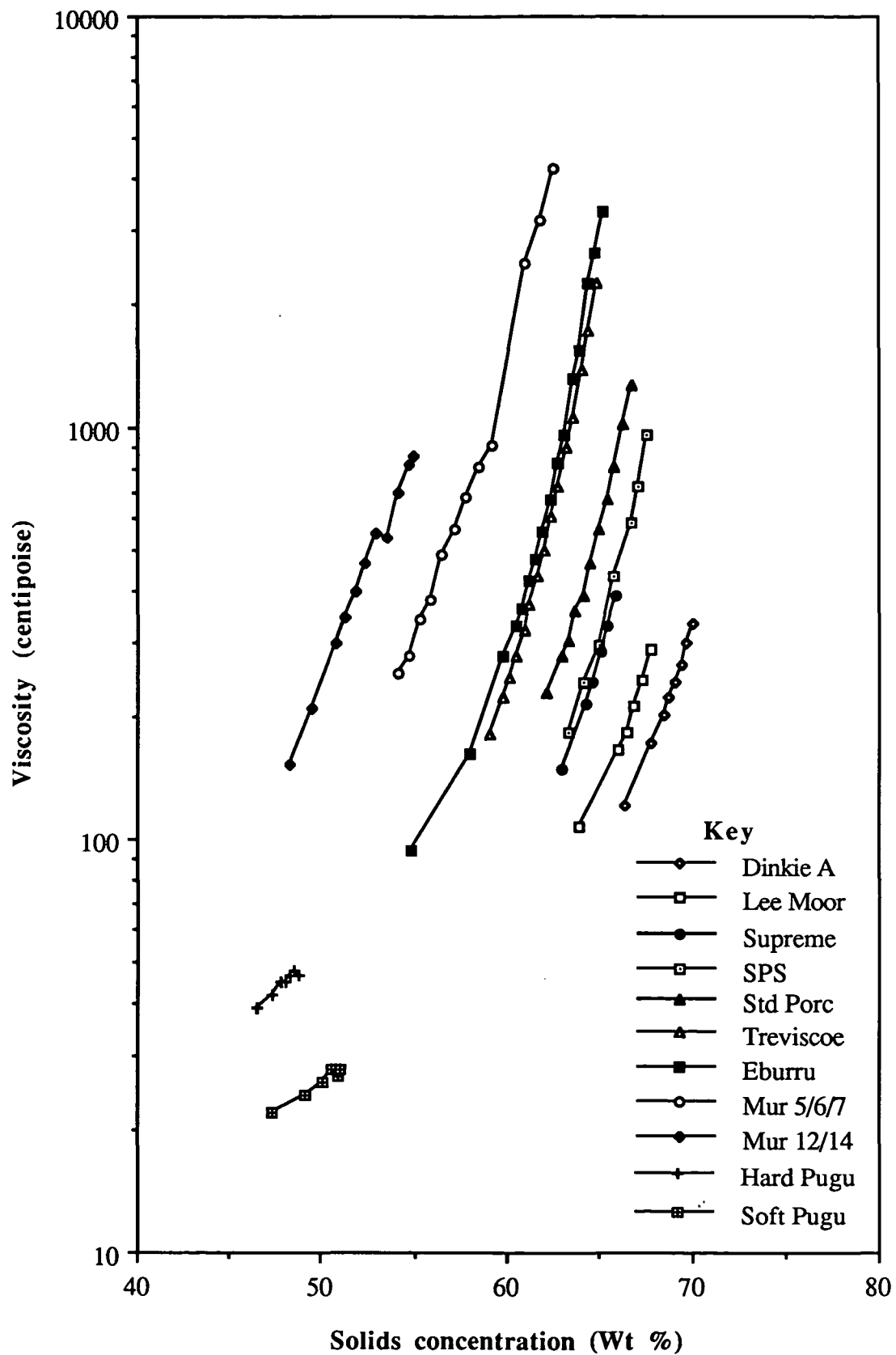
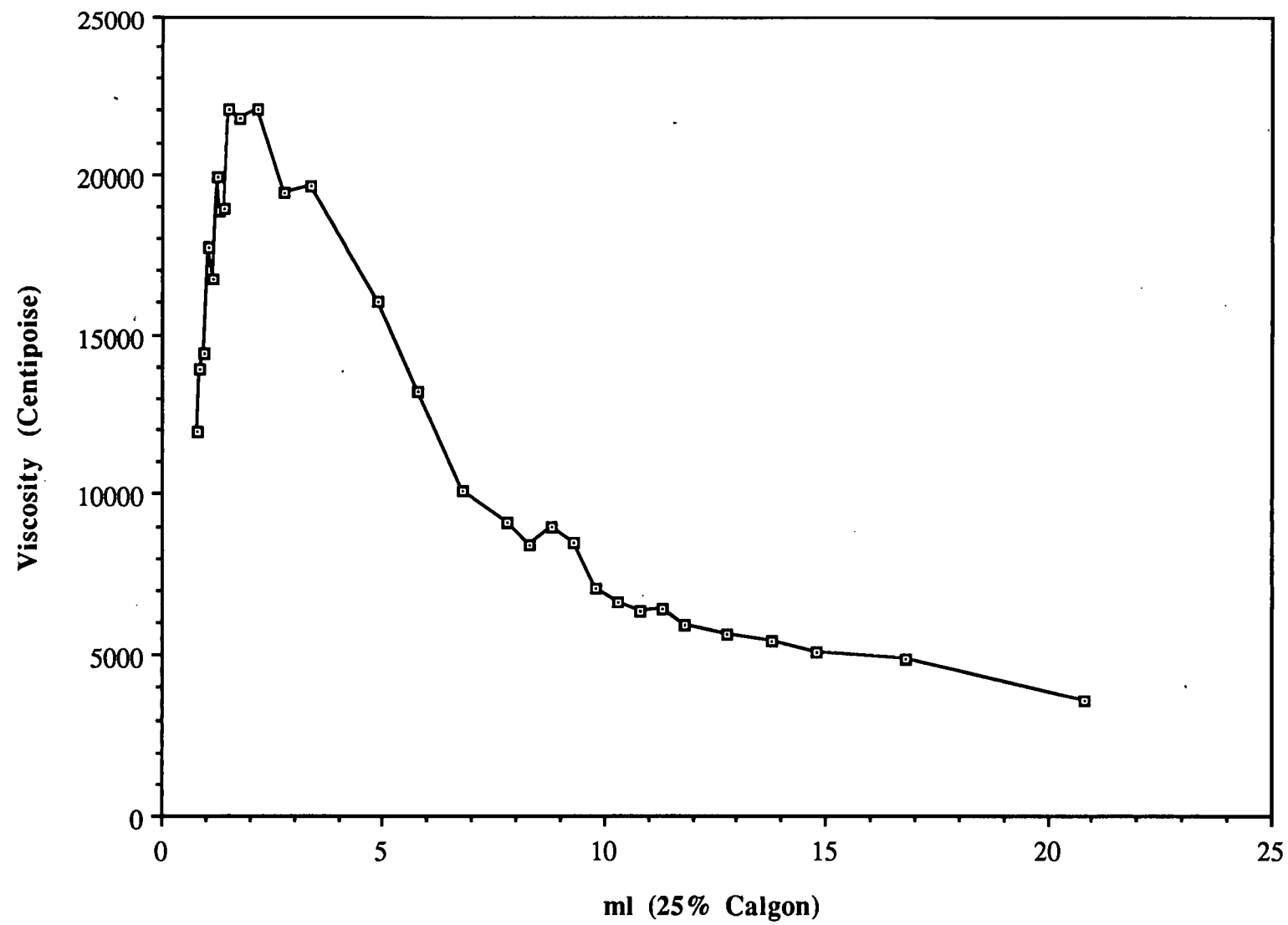


Figure 13 Viscosity versus solids concentration



**Figure 14 Deflocculant demand of Goonvean kaolinite R9-55**



**Figure 15** TEM photomicrograph of hexagonal kaolinite crystals, Supreme Powder (ECC). Mag. x 15,000.



**Figure 16** SEM photomicrograph of kaolinite, Supreme Powder (ECC). Mag. x 6000.

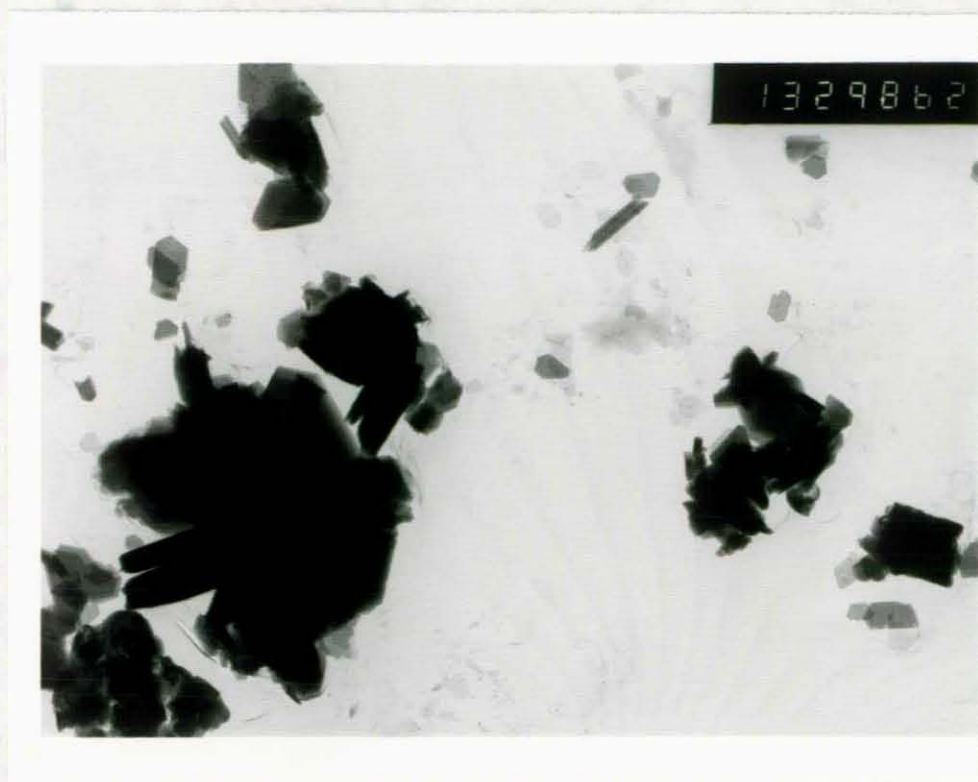


Figure 17 TEM photomicrograph of kaolinite, Dinkie A (ECC).  
Mag. x 19,500.



Figure 18 TEM photomicrograph of kaolinite, Eburru (Kenya).  
Mag. x 19,500.



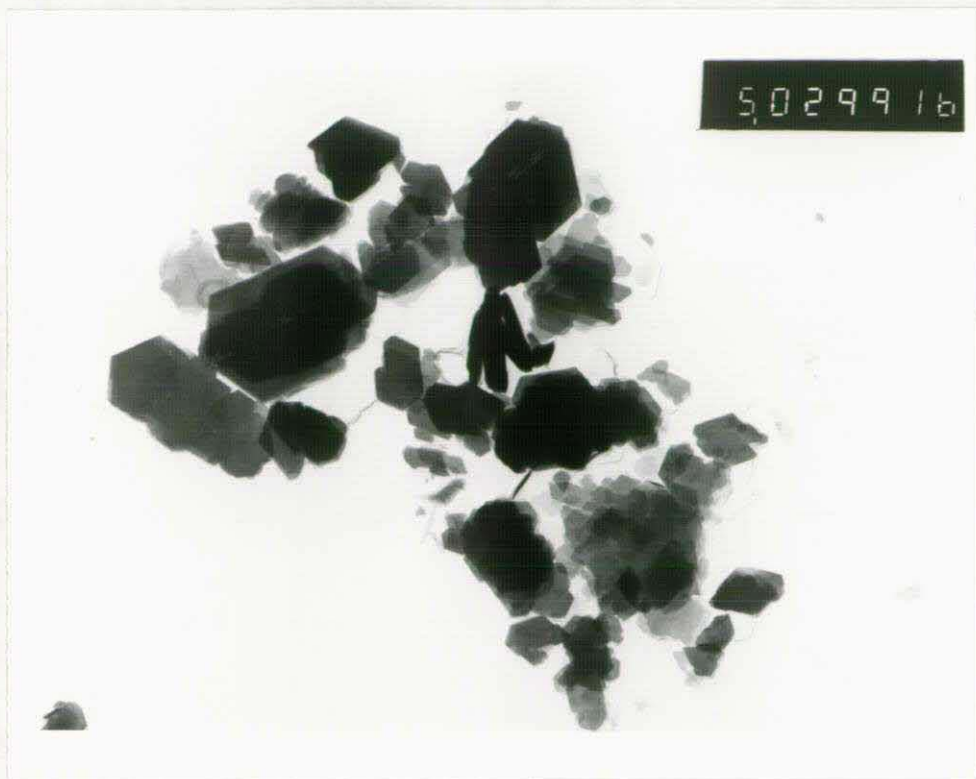


Figure 19 TEM photomicrograph of kaolinite, Lee Moor.  
Mag. x 7,500.



Figure 20 SEM photomicrograph of kaolinite, Lee Moor. Mag. x 6000.



Figure 21 TEM photomicrograph of kaolinite, Standard Porcelain (ECC). Mag. x 7,500.



Figure 22 SEM photomicrograph of kaolinite, Standard Porcelain (ECC). Mag. x 6000.





Figure 23 TEM photomicrograph of kaolinite, Treviscoe (ECC).  
Mag. x 5,000.



Figure 24 SEM photomicrograph of kaolinite, Treviscoe (ECC).  
Mag. x 6000.

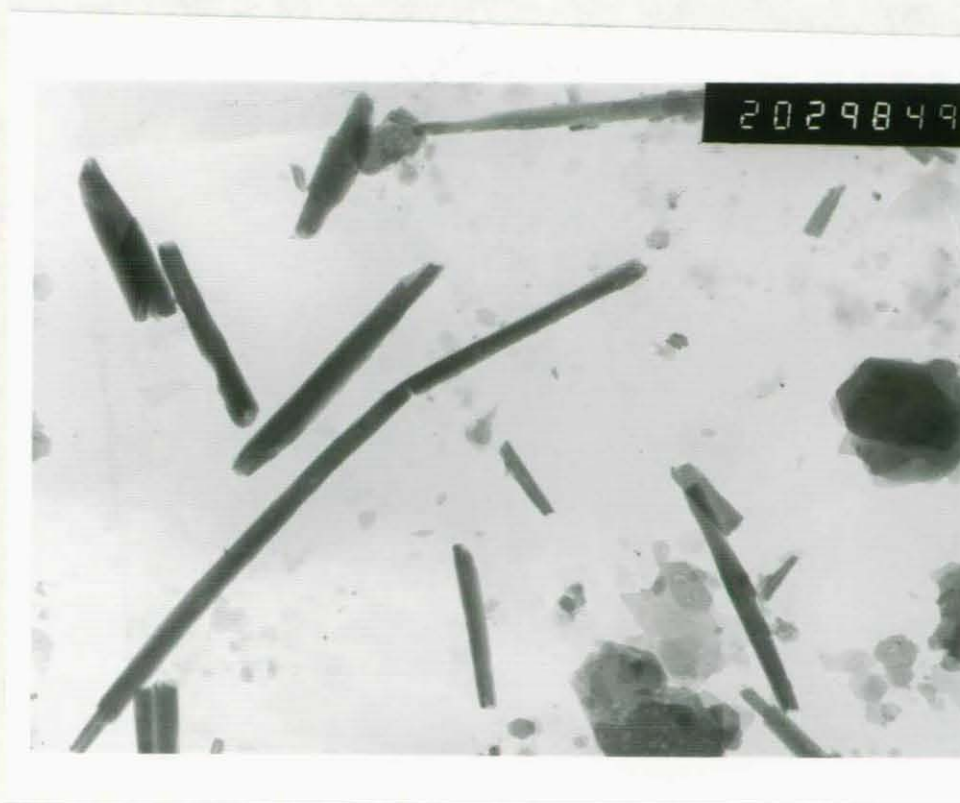


Figure 25 TEM photomicrograph of halloysite rods and kaolinite plates, Muranga 5, 6 and 7 (Kenya). Mag. x 20,000.

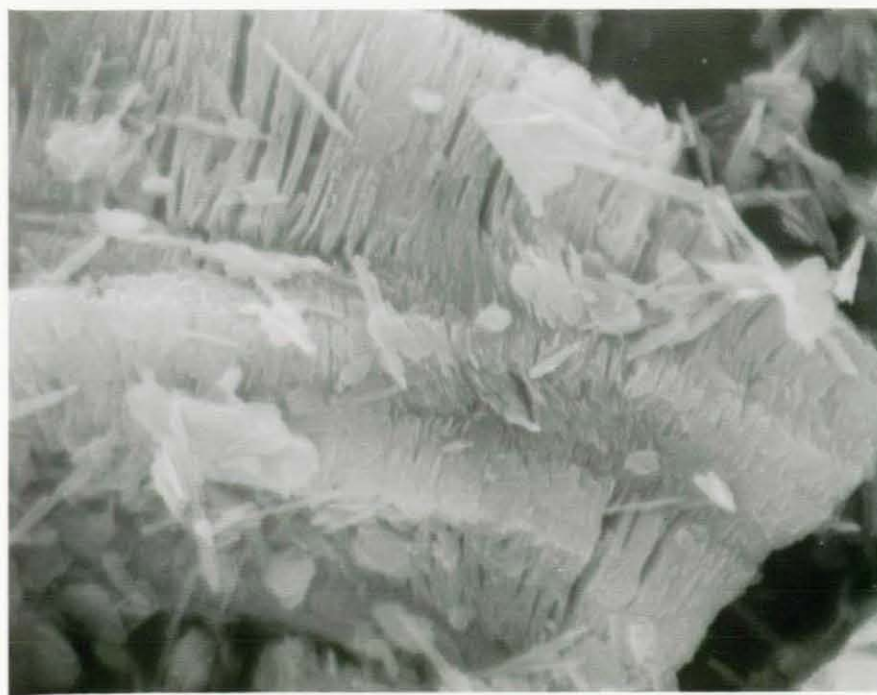


Figure 26 SEM photomicrograph of halloysite rods on weathered feldspar, Muranga 5, 6 and 7 (Kenya). Mag. x 6000.





**Figure 27** TEM photomicrograph of halloysite rods and books of kaolinite, Muranga 12 and 14 (Kenya). Mag. x 8,800.



**Figure 28** SEM photomicrograph of halloysite rods on the surface of a kaolinite book, Muranga 12 and 14 (Kenya). Mag. x 6000.

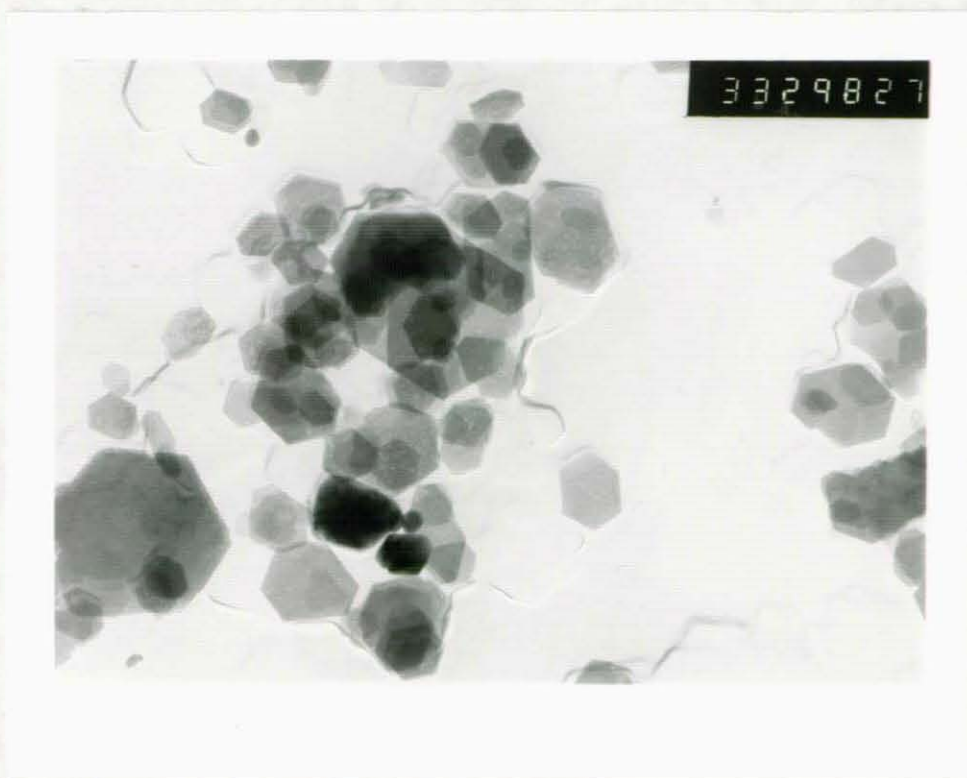


Figure 29 TEM photomicrograph of hexagonal kaolinite crystals, Pugu Hard (Tanzania). Mag. x 30,900.



Figure 30 TEM photomicrograph of hexagonal kaolinite crystals, Pugu Soft (Tanzania). Mag. x 24,900.

## **APPENDIX 1. Viscosity measurement**

1. Gently grind the equivalent of 200 g dry weight of clay in a pestle and mortar, so that it all passes through a 500  $\mu\text{m}$  sieve.
2. Determine the moisture content of the clay by drying 20 g at 55°C overnight in a moisture extraction oven.
3. **Flowability test** - solids content at which the slurry just flows.
  - 3.1. To a 100 ml beaker add 4 ml of distilled water (include the water, if any, present in the sample), 0.75 ml of 10% Calgon solution and 0.2 ml of 10% NaOH solution.
  - 3.2. Add the equivalent of 20 g dry weight of clay and mix with a glass rod.
  - 3.3. Add 0.2 ml of distilled water and stir. Continue to add water until the slurry just flows from the glass rod. The solids content at this point is the flowability. (NB Ensure that the slurry is thoroughly mixed between additions of water and that a reasonably thick glass rod is used, about 6 mm diameter.)
4. **Deflocculant demand** - the amount of deflocculant required to obtain minimum viscosity of a slurry.
  - 4.1. To a tall 125 ml beaker add 2 ml of 10% Calgon solution and 1.0 ml of 10% NaOH solution.
  - 4.2. Add enough distilled water such that when the dry equivalent of 100 g of clay is added the solids content is the flowability plus 3% (i.e. if the flowability is 60% then start at 63% solids content). (NB Volume of slurry required is that which will cover the shaft of the viscometer spindle past the indentation, with at least 5 mm gap between the spindle and the bottom of the beaker. 125 ml of slurry is usually enough. The slurry volume is roughly equivalent to the dry volume of the kaolinite.)
  - 4.3. Add 70 g of clay to the beaker, mix thoroughly using a low-speed, high-torque mixer and then gradually mix in the remainder of the clay. At this point the solids content will exceed the flowability, making the slurry a thick paste and difficult to stir. Fluidity of the slurry is maintained by 1 ml additions of distilled water until the slurry is fluid. Note the total addition of water.
  - 4.4. The slurry should be mixed for a total of 25,000 revolutions from the start of 4.3. onwards, i.e. 25 minutes using a speed of 1000 rpm.
  - 4.5. Cool the slurry to  $22 \pm 1^\circ\text{C}$ ; after mixing, the temperature of the slurry commonly exceeds 30°C. Partially immerse the beaker in cold water to lower the

temperature of the slurry. Tap the beaker down on a work surface repeatedly in order to remove any bubbles entrained during mixing.

4.6. Viscosity measurement. Measure the viscosity of the slurry using a Brookfield viscometer RVF 100 at 100 rpm and 22°C. The choice of spindle depends on the viscosity of the slurry; the lower the viscosity the lower the spindle number (see Appendix 2 for the viscosity ranges of spindle/speed combinations). The spindle should be carefully screwed into place by holding the spindle coupling (the spindle has a left-hand thread to prevent it loosening during measurement). Do not use the spindle guard. The spindle is immersed up to the middle of the spindle shaft, in the indentation, above the disc. Position the spindle centrally, away from the sides and bottom of the beaker.

After starting the motor allow the slurry to reach equilibrium - two minutes is sufficient - and take a reading. Depress the clutch lever and flick the on/off switch a few times until the dial needle is visible in the window. If the reading is over 100 use a higher spindle number and vice versa if the reading is under zero. Take three consecutive readings; if they coincide to within  $\pm 1\%$ , average them and multiply by the appropriate factor to convert to viscosity in centipoise. Viscosity measurements are accurate to within  $\pm 1\%$  of the range maximum of the spindle /speed combination used, therefore readings from the Brookfield are more accurate the closer they are to 100 on the dial.

4.7. Add 0.08 ml 25% Calgon from a burette, stir with a glass rod until homogeneous and measure viscosity. Continue adding calgon and measuring viscosity until the viscosity stops falling and starts to rise again. Plot the weight of deflocculant added per 100 g of clay against viscosity. The amount of deflocculant required to produce the minimum viscosity is the deflocculant demand. (NB During the test the viscosity may fall below the viscosity range of the spindle being used, measurements below 10 on the dial are unreliable. Record the viscosity using both the old and new spindles. It is preferable to use one spindle throughout the test.)

5. **Viscosity concentration test** - this determines the solids concentration of a deflocculated clay which has a viscosity of 5 poise at 22°C.

5.1. Follow stages 4.1. to 4.6., except that enough calgon to meet the deflocculant demand is added.

5.2. Add 2 ml of distilled water, stir with a glass rod until homogeneous and measure the viscosity. Keep adding water and measuring the viscosity until the viscosity has fallen below 5 poise. Plot the reciprocal of the square root of viscosity (in poise) against the solids concentration. The viscosity concentration is the solids content at 5 poise.

**APPENDIX 2. Factors to convert Brookfield readings to viscosity in centipoise.**

Spindle Number	Factor	Speed of rotation (rpm)			
		100	50	20	10
1	100/N	1 (10-100)	2 (20-200)	5 (50-500)	10 (100-1000)
2	400/N	4 (40-400)	8 (80-800)	20 (200-2000)	40 (400-4000)
3	1000/N	10 (100-1000)	20 (200-2000)	50 (500-5000)	100 (1000-10,000)
4	2000/N	20 (200-2000)	40 (400-4000)	100 (1000-10,000)	200 (2000-20,000)
5	4000/N	40 (400-4000)	80 (800-8000)	200 (2000-20,000)	400 (4000-40,000)
6	10,000/N	100 (1000-10,000)	200 (2000-20,000)	500 (5000-50,000)	1000 (10,000-100,000)
7	40,000/N	400 (4000-40,000)	800 (8000-80,000)	2000 (20,000-200,000)	4000 (40,000-400,000)

The conversion factor for each spindle/speed combination is calculated by dividing a factor by the speed of rotation (N). To convert to viscosity, in centipoise, multiply the Brookfield reading by the appropriate spindle/speed conversion factor. The figures in brackets underneath the factors are the ranges of viscosity (in centipoise) that a specific spindle/speed combination covers. The conversion factor is also the accuracy, in centipoise.