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FUEL RESEARCH INSTITUTE
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ONDERWERP:
SUBJECT: INVESTIGATION OF VARIOUS PROPERTIES AFFECTING SETTLING RATES OF DENSE-MEDIUM SUSPENSIONS USING MAGNETITE.

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INVESTIGATION REQUESTED BY:	CHEMISTRY DEPARTMENT
DIVISION:	CHEMISTRY

FUEL RESEARCH INSTITUTE OF SOUTH AFRICA

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INVESTIGATION OF VARIOUS PROPERTIES AFFECTING SETTLING RATES OF DENSE-MEDIUM SUSPENSIONS USING MAGNETITE

1. SUMMARY

The settling rate of dense-medium suspensions of three different magnetite ore samples was investigated. The result of the investigation indicates that the settling rate increases with (i) the increase in temperature of the suspension and, (ii) with the increase of the diameter of the settling cylinder. The settling rate is affected by (iii) the procedure of creating a suspension and (iv) the quantity and fineness of the impurities in the ore.

The practical considerations, resulting from the above are:
since the behaviour of the medium in an actual plant is frequently judged on the basis of a settling rate determination, it is obviously of importance that the interpretation of the test result should rest upon a clear and well-defined basis. The study reported hereunder has indicated that the following points should be stressed:

1) The sedimentation rate is temperature dependent. To obtain a meaningful result, the sample should thus be at the same temperature as the liquid in the washer, i.e. it may be advisable to perform the test immediately after drawing the sample.

2) Particularly in magnetites which contain a large amount of non-magnetic impurities, the settling rate is much affected by the pre-treatment, i.e. the manner of shaking the sample. The shaking process should thus be standardised. Unfortunately, this is easier said than done, as no commercial shaking equipment suitable for this purpose is available - the human being surpasses all normal services in this respect.[*] Thus the same operators should preferably always perform this test and discipline themselves to do this in an acceptably reproducible manner.

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* Only the shaper (a metal planing machine with a quick return stroke) approaches the human being in efficacy. A fairly simple mechanism could obviously be built on such lines.

2. INTRODUCTION

The Fuel Research Institute frequently receives requests from colliery managements to determine the various properties of magnetite ores used for dense-medium suspensions of coal preparation plants.

Magnetite receives priority among various other minerals that may be used as dense media on account of its high solid density and the ease with which it can be recovered from the effluent for re-use by means of a magnetic separation drum. The recovered magnetite is deflocculated in a demagnetisation cell and densified before being returned to the circuit.

One of the important properties of dense-medium suspensions is their stability which depends, on the one hand, on the particle size distribution of the magnetite powder, and on the other hand on the completeness of demagnetisation of the recirculated powder.

With increasing particle size the difficulty of keeping the powder in suspension increases, and with decreasing particle size the difficulty of the demagnetisation of the powder (magnetised during separation) increases, which also causes unstable suspensions because of increasing flocculation.

During the washing process demagnetised magnetite from the effluent is continually recycled into the bath with the result that the bath quickly contains mostly demagnetised instead of original magnetite. This is slightly counter-acted by replacing lost magnetite with original magnetite. Therefore the stability of demagnetised suspensions should be determined besides that of original and magnetised suspensions.

The only practical method to determine the stability of a suspension is to measure its settling rate. When these determinations were started, it was realised that a special study of this subject was necessary.

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3. SETTLING RATE DETERMINATION

The settling rate is the speed with which the interfacial plane between the suspension and the supernatant descends and is measured in mm/s. The experiment starts as soon as the magnetite separates from the surface of the water and is completed when the interface has descended 25 mm, (1 inch) which yields the average speed over this distance.

In practice the operators at coal washing plants measure the time with a stopwatch (in minutes) required for the interface to pass the first 25 mm. This time is defined as settling time (minutes per 25 mm) and is reciprocal to the settling rate. Due to the fact that in practice the settling time is always used, the results of this study are also expressed in settling time. However, the expression "settling rate" is used in the discussion for ease of understanding.

4. SAMPLES USED

4.1 Description of the three magnetite samples

Phalaborwa 1

The Phalaborwa magnetite, which was wet milled in the big ball mill of the Pilot Plant of the Institute for three hours, was designated as Phalaborwa 1.

Phalaborwa 2

Phalaborwa magnetite, designated as Phalaborwa 2, was supplied by Martin & Robson (Pty) Ltd, who ground the raw Phalaborwa magnetite in a dry process where the finest particles were removed in an air-lift at Germiston.

Stanmore

Stanmore magnetite, designated as Stanmore, was received from General Mining and Finance Corporation Ltd.

4.2 Properties of the three magnetite samples

Properties obtained are shown in Table 1 and Fig. 1.

4.2.1 The amount of impurities (non-magnetics) was determined in the Davis tube. It consists of a glass tube which slopes at 45° and is shaken between the pole-shoes of a heavy electromagnet. When the sample is introduced into the tube filled with water, it is retained at the pole-shoes. Thereafter water is passed through the tube until it is clear. The magnetic material (magnetics) is retained by the magnet and the non-magnetic material (impurities) is collected at the effluence.

4.2.2 The densities of the original sample, the magnetic and the non-magnetic material, were determined.

4.2.3 The particle size distribution curve was obtained with the air-jet sieve, and in the sub-sieve range by water elutriation in the Cyclosizer[x] (consisting of a set-up of five inverted cyclones with decreasing diameter of the nozzles).

Various mean particle diameters were obtained from the curve. The median (central value) is the particle diameter at 50% oversize; the upper and lower quartiles are the particle diameters at 75 and 25% oversize respectively. The two quartiles express the spread around the median[xx]. This spread, equal to the difference of the two quartiles, is also included in Table 1. Finally, the arithmetic mean particle diameter was calculated by means of the particle size distribution curve.

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x Only an air-jet sieve analysis was performed with Stanmore.

xx G. Herdan, Small particle statistics, Elsevier Publishing Company, Amsterdam 1953, p.25.

4.3 COMPARISON OF THE THREE SAMPLES (SEE TABLE 1)

The mean particle diameter (median and arithmetic) as well as the spread increases from Phalaborwa 1 to 2 to Stanmore.

Phalaborwa 1 and Stanmore have the same amount of impurities, whereas Phalaborwa 2 has much less. However, the impurities of Stanmore are much finer than those of Phalaborwa 1, because during washing only after the eighth 5-litre beaker, clear water was obtained with Stanmore, while three beakers were necessary for Phalaborwa 1.

The magnetic material of all three samples has a much higher density than their impurities.

5. EXPERIMENTAL CONDITIONS

5.1 Settling rate cylinder

In coal washing plants the settling rate determinations of dense-medium suspensions are made in one- or two-litre measuring cylinders at ambient temperature which varies considerably from winter to summer.

To obtain comparable results, the determinations in the laboratory have to be performed under conditions similar to those prevailing in practice. The amount of magnetite necessary for one-litre suspensions with a density of 1,5 g/cm^3 is approximately 625 g (depending on the actual density of the magnetite used). The preparation of such a mass of sample viz. washing, screening, and demagnetisation, is very time-consuming and the demagnetisation is almost always incomplete. It was therefore decided to perform one series of tests with measuring cylinders of 1 000, 500, 250 and 100 cm^3 capacity in order to determine whether the settling rate is affected by the size of the container in which the tests are made.

The series of measuring cylinders of decreasing volume to determine the settling rate was used for studying the wall effect which influences the settling rate. The settling rate should increase with increasing cylinder diameter, and therefore also with increasing volume.

The settling rate of magnetite suspensions was found to be strongly dependent on temperature. Therefore, the settling cylinders were equipped with cooling jackets in which water, at a constant temperature, was circulated. Experiments revealed that the bottoms of the cylinders should also be enclosed in the cooling jacket, otherwise a notable temperature gradient arises between the water surface and the settled magnetite ($5^{o}C$ was observed).

To simulate the conditions closest to practice the experiments were performed at $20^{o}C$, although the measurements were also undertaken at $30^{o}C$ for one series.

Normal measuring cylinders with a volume scale were used. To facilitate the reading of the distance for the settling rate determinations, a millimetre scale was additionally etched on the cylinders.

5.2 Shaking methods

To obtain a homogeneous magnetite suspension in water, the cylinder had to be shaken. As will be discussed later, it was found that the settling rate of some magnetite suspensions decreased with prolonged shaking.

Experience showed the following method to be the best. The closed cylinder was vigorously shaken horizontally for half a minute by hand. It was defined as "one shaking operation". In this way, the shaking necessary was subdivided into discrete shaking operations. A settling rate determination was performed after each shaking operation.

Various mechanical methods of shaking the cylinder were tried, e.g. shaking on a shaping machine and on a rotating wheel, or stirring the suspension with a propeller. Apart from the last one, these methods yielded good results, but the manual shaking, subdivided into shaking operations, was found to be by far the best method.

5.3 Magnetic treatment

After measuring the settling time of a suspension made out of original magnetite (i.e. a sample which has not undergone any magnetic treatment), the suspension was magnetised in the settling cylinder with a strong permanent powder magnet. Hereafter, the suspension was shaken, the settling time measured and the suspension was subsequently normally filtered in a Buchner funnel. The filtrate was tightly packed in a plastic cylinder which was introduced into the gap of a strong a.c. electro-magnet. The magnet was switched on and the cylinder was gradually removed out of the electro-magnetic field. To prevent the single magnetite particles from turning in the alternating field during demagnetisation, the powder must be packed very tightly into the cylinder, which takes some hours. The demagnetised sample was suspended again and the settling time measured.

6. PERFORMANCE OF INVESTIGATION

6.1 Establishment of the experimental conditions: dependence of settling time on the diameter of the settling cylinder and the temperature of the suspension

The relation between the settling time and the diameter of the measuring cylinder and the temperature of the suspension was determined respectively. The experiments were performed, using cylinders of 100, 250, 500 and 1 000 cm^3 with diameters of 25,8, 36,2, 49,1 and 63,0 mm, at 20 and 30 $^{\circ}C$, with Phalaborwa 1. Suspensions of 1,5 g/cm^3 density, which were shaken until the highest settling time was reached (see paragraph 5.2), were used. The results for 20 $^{\circ}C$ (average of ten determinations) and for 30°C (average of six determinations) are plotted in Fig. 2, showing that with increasing cylinder diameter and suspension temperature, the settling time decreases.

The curves indicate that with increasing temperature, i.e. with decreasing viscosity of water, the settling rate increases as expected according to Stokes' law. The settling rate increases with increasing diameter, showing that the influence of the wall which hinders the neighbouring suspension from settling, decreases (wall effect). The increase in settling rate slows down with increasing diameter. This is more pronounced at the lower viscosity (30°C).

6.2 Dependence of settling time on the number of shaking operations

All the following tests were performed in 100 cm^3 cylinders (25,8 mm diameter), with suspensions of 1,5 g/cm^3 density at 20°C. The following five media of Phalaborwa 1 and 2 were first used: the original, the demagnetised and the magnetised sample, the demagnetised washed and the magnetised washed sample.* The samples were washed to remove the non-magnetic material (impurities).

The settling time was measured after each shaking operation. The settling times for Phalaborwa 1 are plotted against the number of shaking operations in Fig. 3, and in Fig. 4 for Phalaborwa 2.

From Fig. 3 it can be observed that with the three unwashed samples, the settling time increases with the number of shaking operations. With increasing number of shaking operations, the increase of settling time becomes slower, until it is zero. The two washed

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* The samples were still washed in a sloping launder over a set of permanent magnets in a slow stream of water, because the Davis tube was not yet available for these tests.

samples still showed a small increase, indicating that this effect is at least partially caused by the impurities.

With Stanmore only one test, using the original sample, was performed, from which the settling time against the number of shaking operations is plotted in Fig. 5. Again, an increase in the settling time with the number of shaking operations was observed, but the whole increase in settling time until it becomes zero is equal to approximately 16 min/25 mm in this case as opposed to 3,5 min/25 mm for the original sample of Phalaborwa 1 (Fig. 3).

As stated previously, the two samples compared here have the same amount of impurities, but it was also stated that the impurities of Stanmore are much finer than those of Phalaborwa 1. This leads to the conclusion that the increase in settling time becomes larger when the impurities decrease in particle size. It is interesting to note that this phenomenon was observed with Stanmore, which is much coarser than Phalaborwa 1. Attention could be given to the fact that to reach constant settling time, more shaking operations are necessary for Stanmore than for Phalaborwa 1.

Fig. 4 shows that all five samples of Phalaborwa 2 (with only two and a half times less impurities, and being slightly coarser than Phalaborwa 1 - see Table 1 and Fig. 1) settled much quicker than the samples of Phalaborwa 1. No increase in settling time with the number of shaking operations was observed. This result shows that the increase in settling time is not only dependent on the fineness of the impurities, but also on the amount of impurities.

From the settling test of Stanmore plotted in Fig. 5, it was observed that after approximately twelve shaking operations a separation of the magnetic from the non-magnetic material started to occur during settling, after each shaking operation. This could be observed because after each settling a separation line appeared between the dark magnetite below and the lighter-coloured impurities above. The fact that the density of the impurities of Stanmore is considerably lower than that of the magnetic material, helps to explain this phenomenon.

It was also found that the density of the impurities of Phalaborwa 1 is considerably lower than that of the magnetic material. Therefore, it is possible that after a certain number of shaking operations of the unwashed samples of Phalaborwa 1, a separation (at least partially) between the non-magnetic and the magnetic material also occurs, which is not so easily distinguishable as the two materials have nearly the same colour. However, it is possible that for this separation finer impurities, like those of Stanmore, are necessary.

6.3 Experiments to restore the initial settling time (i.e. after one shaking operation) of a well-shaken suspension of Phalaborwa 1

6.3.1 The question arose whether the settling time would remain the same if a suspension of Phalaborwa 1 which had been shaken until the settling time became constant, was left undisturbed in a settled condition for some time. It was found that after a maximum period of thirteen days the settling time was still unchanged.

6.3.2 After the stability of the settling time of a sufficiently shaken suspension of Phalaborwa 1 had been determined, the question arose whether a technique could be found to restore the initial settling time.

Accordingly a well-shaken suspension was dried, the recovered magnetite was magnetised, thereafter demagnetised, still in the dry condition, and then brought into suspension again. Settling time experiments with this suspension revealed that the number of shaking operations did not increase the settling time as can be seen from the broken line of Fig. 6. The initial settling time was not restored with this technique.

A second attempt was made by following the procedure at Phalaborwa Mining Company to recover the magnetite ore by separating it with a magnetic drum separator. Thereafter the magnetite is again demagnetised to pump it through pipes to dumps for storage, where it dries. This magnetite was finally obtained for the investigation, and has the properties according to the curve "original sample" of Fig. 3, viz. it is only partly demagnetised and the settling time increases with the number of shaking operations.

Therefore a well-shaken suspension was magnetised, shaken and filtered in a Buchner funnel. The filtrate was demagnetised in a damp condition, dried and suspended again. As can be seen from Fig. 6, solid curve, the experiment showed that approximately the initial settling time (see also Fig. 3) was restored after one shaking operation, and that with an increasing number of shaking operations the settling time increased until it reached that of the suspension where the medium was magnetised and demagnetised in the dry condition (Fig. 6, broken line).

The demagnetisation in a damp condition should be equivalent to the demagnetisation at Phalaborwa Mining Company, where the flocculated magnetite is pumped through a demagnetisation coil.

It is interesting to note that demagnetisation in a dry condition after the magnetisation of the suspension could not restore the initial settling time. Furthermore, the sample for demagnetisation in a damp condition must not be too small for a successful restoration of the initial settling time. For instance, the amount for a 100 cm^3 suspension as was used for this investigation was too small, but the amount used for 1 000 cm^3 was sufficient. However, the final test to prove whether the initial settling time had been restored, was performed in a 100 cm^3 settling cylinder.

7. RESULTS AND CONCLUSIONS

7.1 Experimental conditions

The experimental conditions of the investigation of the settling time of magnetite suspensions showed that with increasing diameter of the settling cylinder (wall effect) and of the temperature of the suspension, the settling time decreased, which is in accordance with Stokes' law.

Therefore the investigation was undertaken with only 100 cm^3 cylinders and at a temperature of 20°C.

To obtain a quantitative measure of the shaking method, the shaking was sub-divided into discrete well-defined shaking operations.

7.2 Behaviour of suspensions

With the number of consecutive shaking operations (i) the settling time increases and (ii) this increase becomes slower, until it is zero.

These two observed properties of suspensions increase with increasing amount and fineness of the impurity.

In the case of Phalaborwa 2 having a small amount of coarse impurities and a relative coarse magnetite, these two properties are zero.

During shaking operations a separation of the magnetic from the non-magnetic material may occur if the difference between the densities of the two materials is big enough as was observed with Stanmore.

From Fig. 3 and 4 it can be concluded that the original Phalaborwa 1 and 2 obtained from the storage dumps could still be demagnetised further under the laboratory conditions of the Institute.

The final settling time of a well-shaken suspension remains the same when left undisturbed under settled conditions.

The initial settling time (i.e. after one shaking operation) could only be restored when demagnetising a large quantity of filtered, magnetised magnetite in a damp condition which is according to the procedure followed at Phalaborwa Mining Company to separate magnetite. A demagnetisation in a dry condition had no success.

A possible explanation for the increase in settling time with the increase in the number of shaking operations is as follows: It is known that the demagnetisation of magnetite powders by means of a decreasing alternating magnetic field is always incomplete. Therefore, in a demagnetised suspension also small magnetic field line circuits are closed, forming floccules which settle relatively quickly. If non-magnetic material is present, it can be enclosed within the floccules. During the shaking, these floccules release some of the non-magnetic material, as was found with the Stanmore sample, diminishing the size of the floccules and thus decreasing their settling rate. The released non-magnetic material (with a lower density than the magnetic material) forms a suspension of a higher density and viscosity than water. In this suspension the floccules settle still more

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slowly. From this consideration it can be expected that the decrease of settling rate with increasing number of shaking operations is larger when the amount of non-magnetic material and/or the fineness is increased. These deductions agree with the presumptions made during the description of the results. Obviously, the floccules increase in size and settle more quickly when magnetised.

This theory explains why only a magnetisation of the concerned suspension itself and a demagnetisation of the flocculated wet magnetite restores the initial settling rate, because here the non-magnetic material can be enclosed in the big floccules formed by magnetisation and subsequent demagnetisation, which is not the case in a dry condition.

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TABLE 1

PROPERTIES OF THE THREE MAGNETITE ORES

Designation	Non-magnetics in %[1]	Number[2] of beakers used	Density in g/cm^3 of			Mean particle size diameter in micrometre				
			original[3]	magnetics[3]	non-magnetics[4]	median	upper quartile	lower quartile	spread[5]	arithmetic
Phalaborwa 1	15,3	3	4,51	5,12	3,00	14,6[6]	8,1[6]	21,2[6]	13,1	16
Phalaborwa 2	6,1	2	-	5,24	-	19,0[6]	9,0[6]	27,3[7]	18,3	20
Stanmore	16,0	8	3,95	4,48	2,79	70,5[7]	<20[7]	200[7]	>180	127

1) Determined by washing with the Davis tube.

2) Number of 5-litre beakers used during washing with the Davis tube to obtain clear water.

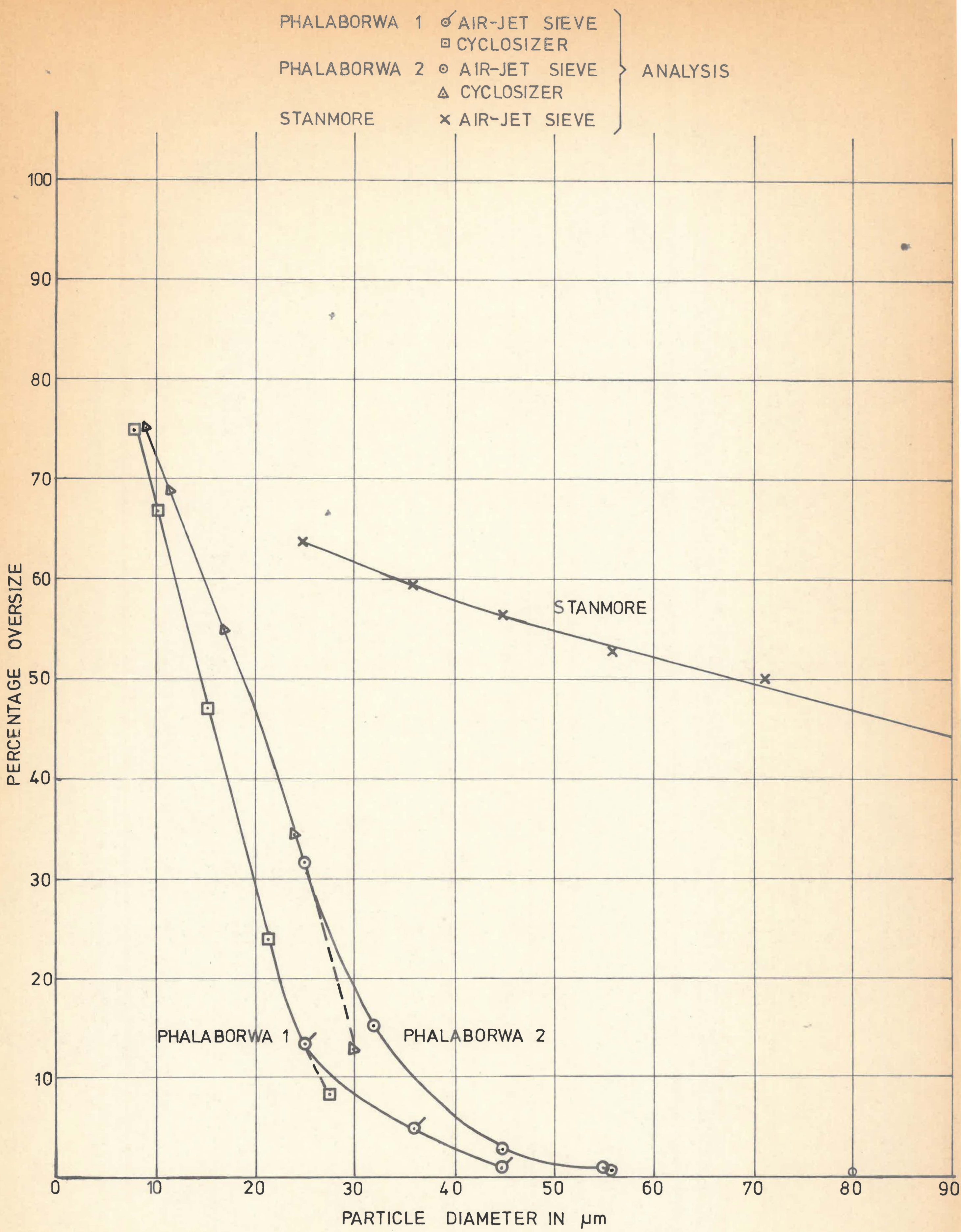
3) Determined with the normal density bottle method.

4) Determined with the Air Comparison Pycnometer.

5) Spread is the difference between lower and upper quartile.

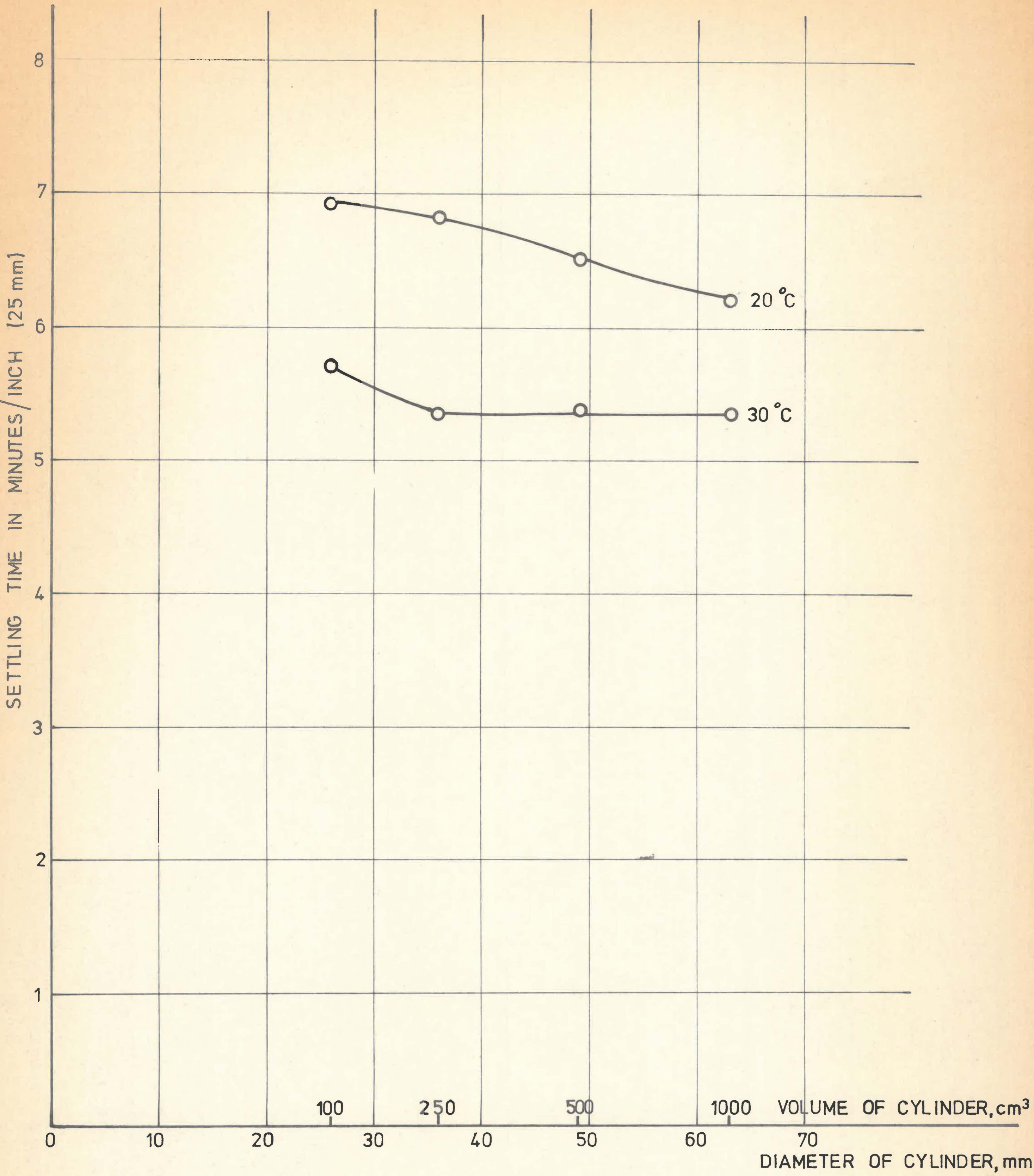
6) The values were obtained with the washed samples (Cyclosizer analysis).

7) The values were obtained with the unwashed samples (air-jet analysis).



PARTICLE SIZE ANALYSIS OF THE THREE MAGNETITE SAMPLES

FIGURE 1



SETTLING TIME OF PHALABORWA 1 VERSUS DIAMETER OF CYLINDER AT 20 AND 30 °C

FIGURE 2

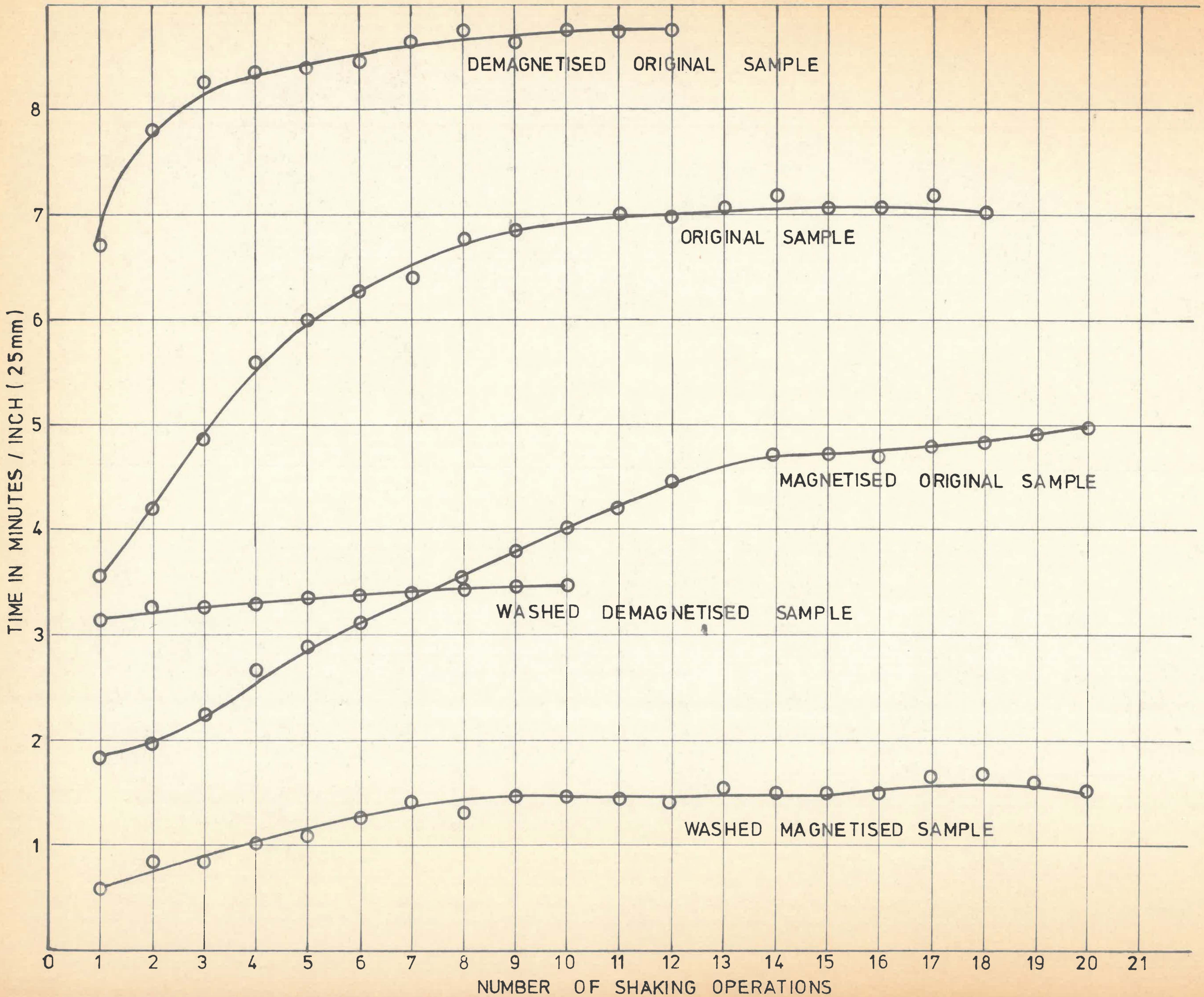
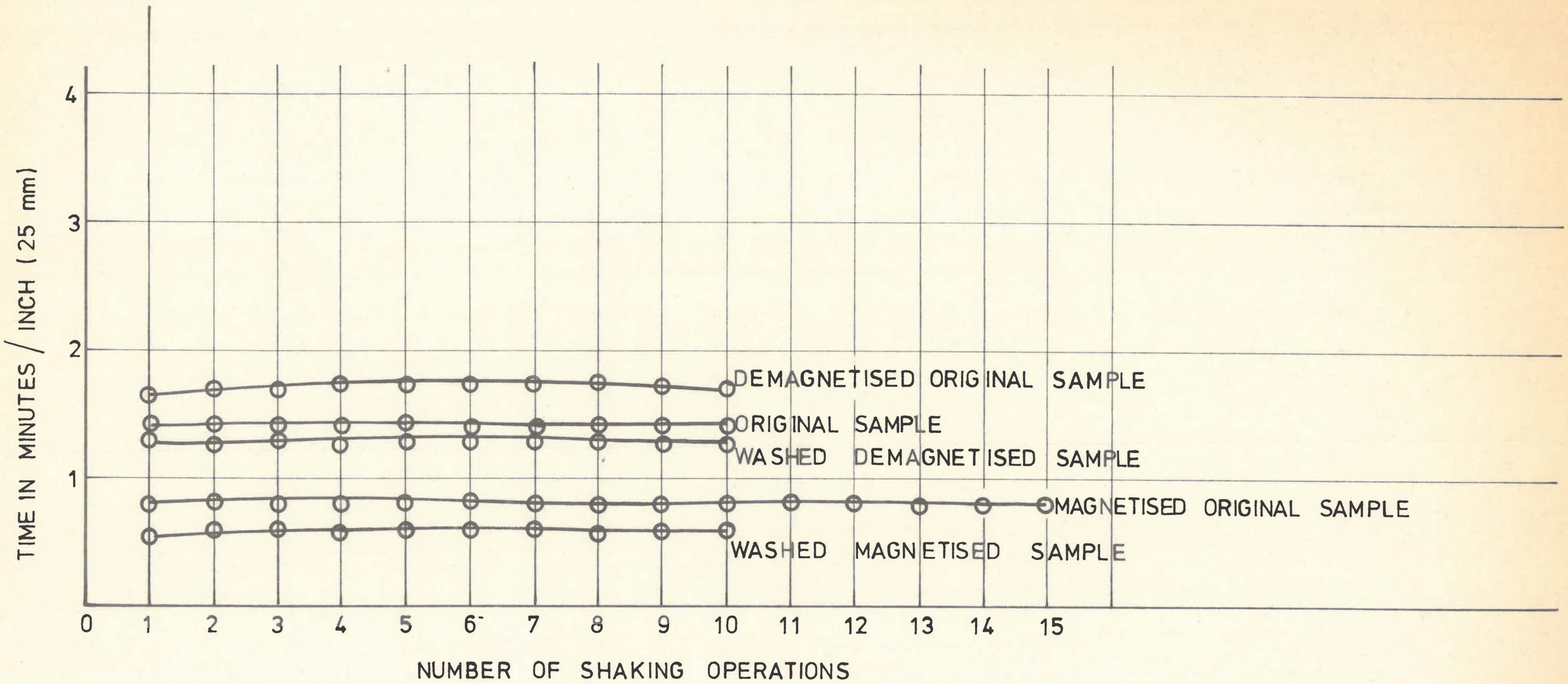


FIGURE 3

SETTLING TIME OF PHALABORWA 1



SETTLING TIME OF PHALABORWA 2

FIGURE 4

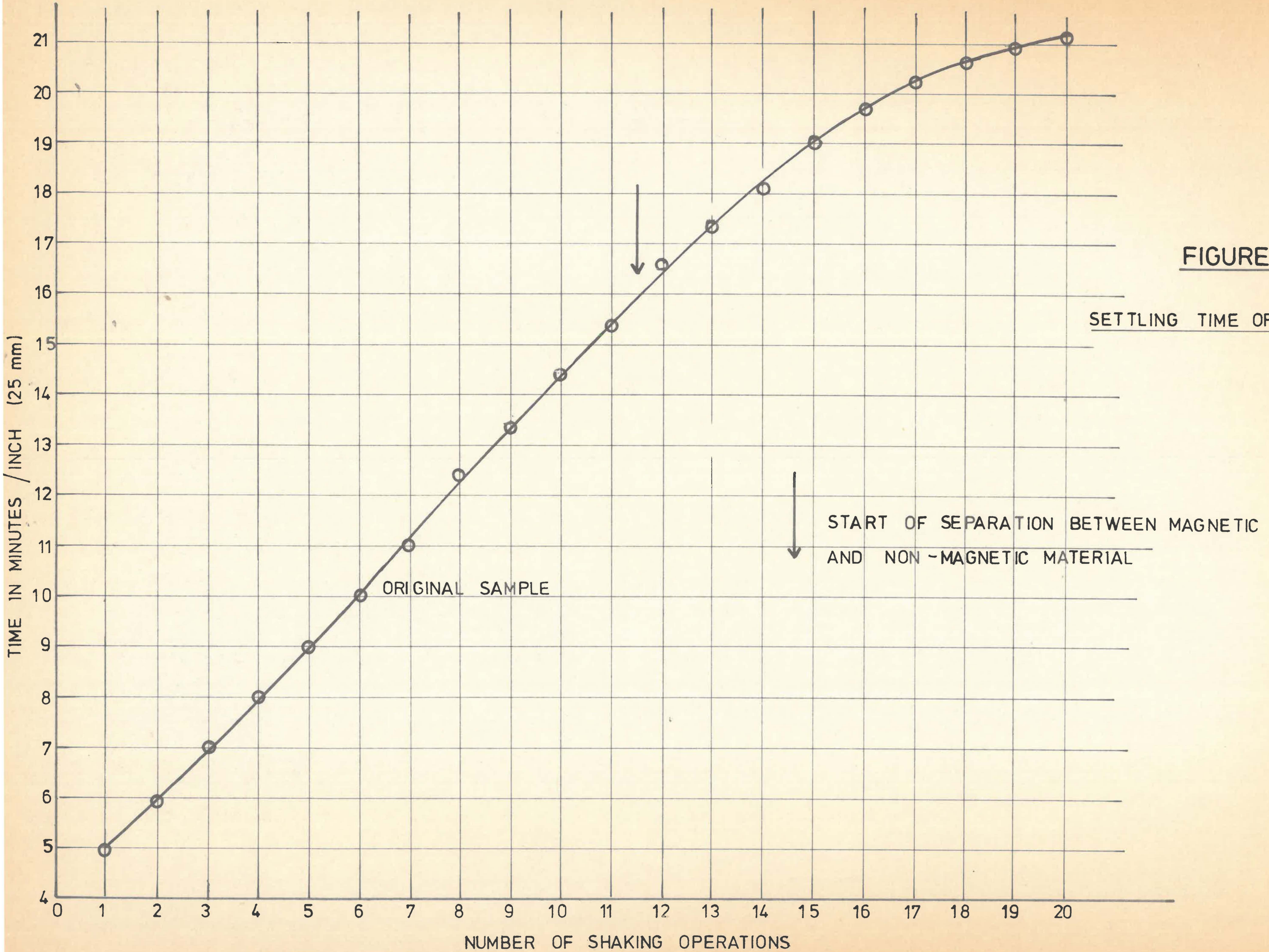


FIGURE 5

SETTLING TIME OF STANMORE

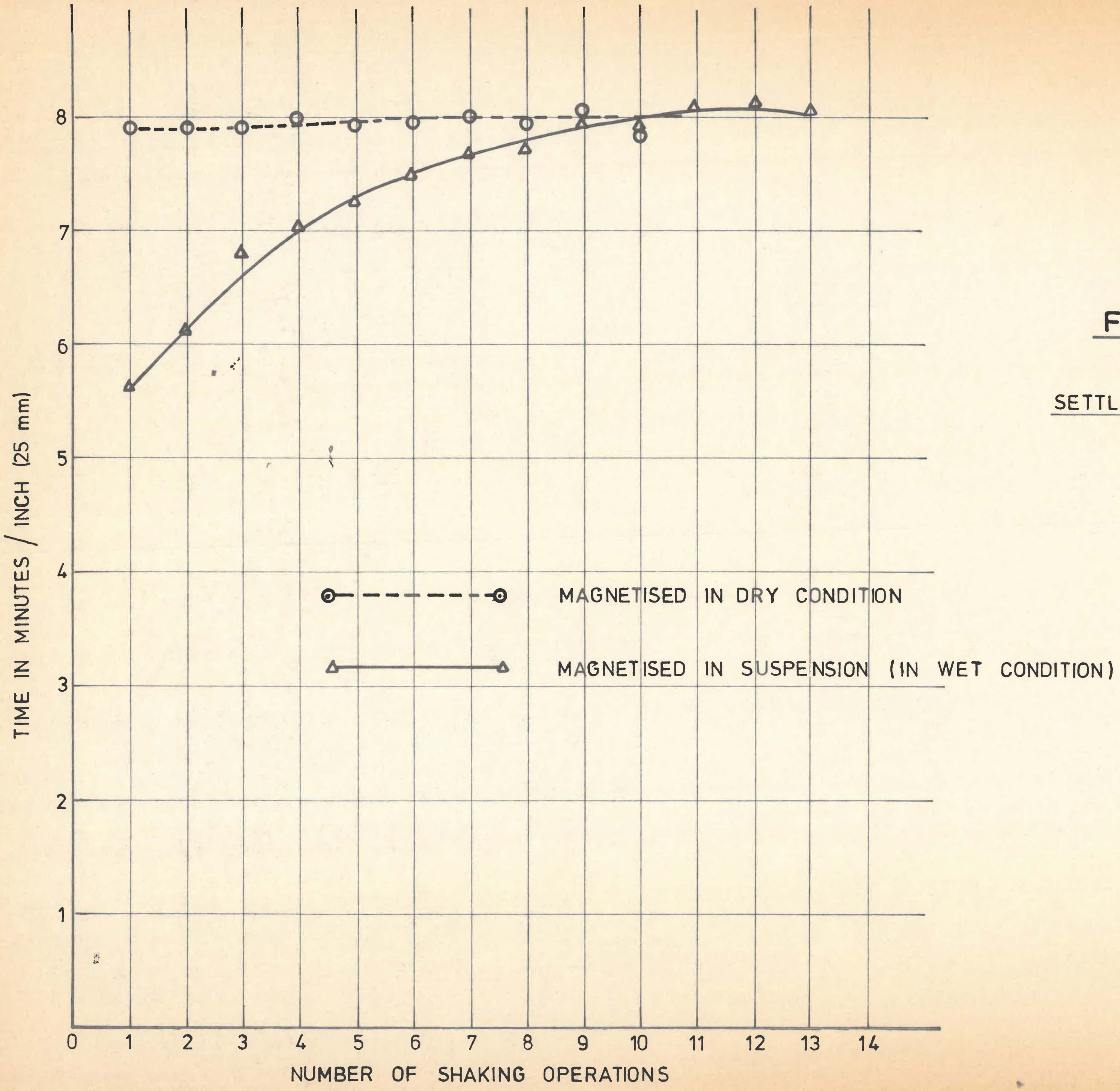


FIGURE 6

SETTLING TIME OF PHALABORWA 1

(ORIGINAL SAMPLE)