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Effects of time and shear energy on the rheological behaviour of oilwell cement slurries

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Abstract

In the present study, it has been evaluated if the configuration of the high-speed mixer propeller blades used in cement slurry preparation done in accordance with API specifications [Specification for Materials and Testing for Well Cements, API Spec. 10, 5th edn., 1990.] does influence the input of mixing energy applied to the cement slurry. It has been shown that the term specific mixing energy (SME) is not valid for all types of slurries. It has also been shown through a comparison between a Vicat apparatus series of experiments and a consistometer study that there is no direct link between the consistometer data and the initial set of a cement slurry. Furthermore, the Vicat test was found not to give the initial set either. Finally, the study investigated the effect of mixing energy input on viscous properties and gel formation properties of the slurries. A drop in viscosity as a function of mixing energy has previously been considered to be a result of a structural breakdown. In the present study, it is suggested that this drop is a result of change in cement morphology.

Keywords: Rheology; Cement paste; oilwell cement

1. Introduction

In the primary cementing of an oil or gas well, a slurry is mixed on the rig before it is pumped down into the well and placed at the cementing interval between the casing and the formation. Cementing is one of the most important operations performed on a well. Without complete zonal isolation, the well will never reach its full potential as a gas or oil producer. Depending on the mixing procedure on the rig, the pumping time, pressure drop in the well, and well geometry, different amounts of shear energy are absorbed by the slurries before the slurries are left to cure. These parameters control the different rheological properties of a cement slurry. The success of a cementing job demands maximum control of these cement slurry properties.

The amount of absorbed energy is known to have a great influence on the slurry rheology and setting time and, thus, the success of the cementing job. Standard procedures suggest a method to simulate the amount of energy applied during a primary cementing operation in laboratory testing [1] prior to rheological measurements. This procedure includes an initial high-speed mixing followed by a slow-speed mixing prior to the use of a standard concentric viscometer to measure the viscosity profiles and gel strengths of the slurries as a function of energy input.

In practical cementing operations, the cement is left to cure after placement. To measure the early set development of the static slurries in the laboratory, a Vicat apparatus has been used to obtain additional information on the set development to the standard oil field methods. This is an apparatus used in civil engineering for the determination of initial and final set. The Vicat apparatus has no direct parallel in use for testing of oilwell slurries. Therefore, this apparatus was applied in the present study to see if applicable data could be collected.

2. Sample preparation and experimental method

2.1. Material

In North Sea oilwell cementing, it is customary to use API Class G cements [1]. It has also been suggested to use construction cement for top hole cementing. Therefore, two

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types of cements have been used in the present study. An ordinary Portland cement named P-30 [2] and an oilwell cement type Class G. Both cements were produced by Norcem. Typical data are shown in Table 1.

2.2. Sample preparation

The sample preparations were done in accordance with API [1]. This specification gives the standard procedure for sample conditioning prior to testing. For the initial mixing, a high-speed propeller-type mixer is used. The mixing intervals consist of an initial period of 15 s at 4000 rpm where dry cement is added to water. Then the speed is increased to 12 000 rpm for an additional period of 35 s. For our high-speed mixing, a Waring Blendor was used. After the high-speed mixing, the sample is placed in an atmospheric consistometer and stirred at 150 rpm for 20 min prior to viscosity or gel formation measurements. All samples were mixed using cement and distilled water with a W/C ratio of 0.38 by weight. No other additives were used. This W/C ratio gives a slurry readily mixable in the high-speed mixer combined with a low tendency to develop free water during the Vicat test [3]. The volume of each test sample was 600 ml. All tests were carried out at $20 \pm 2°C$.

2.3. Measurement of mixing energy

As the dimension of the propeller blade used for the initial high-speed mixing is not given in the API procedure [1], the Waring Blendor was fitted with three different types of propeller blades. Two of these types seem to be commonly in use in Scandinavian laboratories. The first being the original Waring Blendor propeller blade (WB-blade), the second being a propeller blade made of hard metal (HM-blade) similar in shape but having shorter wings compared to that of the original WB-blade. According to a local supplier [4], both types of blades are in use during testing. However, the original WB-blade is most frequently used. The third type of propeller blade used was smaller compared to the other two and of different shape. This latter blade was also used when mixing slurries for the rheological measurements and the Vicat testing in the experiments presented in this paper.

The mixing energy was measured using all three different types of propeller blades. The input of mixing energy was measured by using a power meter. Only the power consumption during the 35-s mixing at 12 000 rpm was measured. This is in accordance with measuring methods used by Orban et al. [5,6] who measured the input of mixing energy for different types of cement slurries. The power consumption is given as an average reading during this period. The power consumption with an empty mixing container was measured and subtracted from the final reading thus giving the net input of mixing energy.

We also measured the input of mixing energy at lower shear rate by using a Hobart mortar mixer with a mixing bowl of 5 l. This mixing was done in accordance with NS-EN 196-3 [7]. The cement was added to the water during a period not less than 5 s nor more than 10 s. Then the mixer was run at low speed for a period of 90 s after which the mixing was stopped for 15 s during which any paste that adhered to the bowl outside the mixing zone was returned to the mix. The mixing was then continued at a low speed. Total volume of each test sample was 3 l.

The input of mixing energy from the Hobart mortar mixer was measured using a power meter. The initial 5–10 plus 90 s of mixing is not included in the measurement. Also here, the power consumption with an empty bowl was measured and subtracted.

Table 1
Cement data for a P-30 standard Portland cement and a Class G oilwell cement

Type	P-30	Class G
Fineness		
Blaine specific surface (m^2/kg)	345	320
Sieve residue, 200 μm (%)		0
Initial setting time (min)	130	140
Soundness		
Le Chatelier expansion (mm)	1	0
Limestone flour (%)	4	
Chemical analysis		
Loss on ignition (LOI, %)	2.5	0.4
Alumina (Al_2O_3, %)		3.5
Magnesia (MgO, %)		1.6
Sulphur trioxide (%)	3.0	1.8
Insoluble residue (%)	1.0	0.3
Potassium oxide (K_2O, %)		0.44
Sodium oxide (Na_2O, %)		0.21
Chloride (Cl, %)		0.02
Carbon dioxide (CO_2, %)		0.15
Water-soluble chlorine (%)	≤ 0.1	
Water-soluble chromium (ppm)	≤ 2.0	
Alkali (Na_2O, %) eq.	0.95	0.50
C_3A (%)		1.2

2.4. Consistency measurements

The consistency of the cement slurries was measured after the initial high-speed mixing using an atmospheric consistometer as specified by API [1]. In this apparatus, the torque exerted on a stationary paddle immersed in a rotating slurry container is measured. The torque defines the consistency of the slurry. The container rotates with 150 rpm during testing. The consistency is given in Bc (Bearden units of consistency). The Bc unit is a dimensionless quantity relating to a viscosity value, however, with no direct conversion factor to other units. The thickening time of the slurry is defined as the time required to reach a consistency of 100 Bc. Generally, 30 Bc is considered to be the maximum pumpable consistency for an oilwell application. The input of mixing energy to a slurry at a measured value of 30 Bc can be calculated to be approximately 0.72 W. The slurry in the consistometer experiences shear rates typically from 0 up to approximately 350 s^{-1}.

2.5. Rheological measurements

The rheological properties of the slurries were measured after high-speed mixing and preconditioning in the atmospheric consistometer as specified by API [1]. Viscosity measurements were done by use of a CHAN 35 viscometer in accordance with the API specification [1]. The viscometer is a rotational concentric viscometer with variable speed. The standard shear rates used were 5.1, 10.2, 17, 34, 51.1, 102, 170, 340 and 511 s^{-1}. Gel strength was measured as the peak shear stress at a shear rate of 5.1 s^{-1} after 10 s and 10 min static delay after a 10 s continuous rotation at 1022 s^{-1}.

2.6. Vicat measurements

The Vicat testing was done in accordance with the industrial specification NS-EN 196-3 [7]. In the Vicat test, a cylindrical needle with a diameter of 1.13 mm is forced down into a 40 mm deep sample. The total load on the needle is 300 g. Measurements are given in millimeters with reference to the bottom of the sample. Initial set is said to be reached when the needle is no longer able to pierce the sample to 4 ± 1 mm from the sample bottom. The final set is measured with a needle of similar dimensions. This needle is fitted with a hollow attachment to leave a circular cutting edge 5 mm in diameter. The end of the needle projects 0.5 mm beyond this edge. Final set is said to be attained when the needle, gently lowered to the surface of the sample, makes an impression on it, but the circular edge of the attachment fails to do so. For comparison, in the Vicat test when final set is measured, the load could be said to rest on the end of the needle alone and a pressure of 2.9 MPa to be approached. According to API [1], the minimum compressive strength requirement for a Class G slurry after 8 h, at atmospheric curing pressure, and a curing temperature of 38°C, is 2.1 MPa. In oilwell drilling, a much-used procedure is to wait for a compressive strength development of at least 3.5 MPa (500 psi), before any further well operations are started up. Vicat final set should therefore comply with the API 8 h minimum compressive strength.

3. Results and discussion

3.1. Experiments

Three series of experiments were conducted. The objective of the first series was to measure the input of mixing energy during the high-speed mixing using different available propeller blades to see if the term standard mixing energy (SME) can be used without precaution. The second series with low shear rate mixing using construction cement equipment were done to verify the results obtained in the first series. The objective of the third series was to evaluate the effect of preconditioning and mixing energy input on rheological performance of cement slurries. This was measured using both oil field and construction cement testing methods.

3.2. Mixing energy

The effect the type of Waring Blendor propeller blade had on the input of mixing energy during preconditioning was investigated in the first series of experiments. This is shown in Table 2 where the measured input of mixing energy during the initial high-speed mixing when using three different types of propeller blades are given. The original WB-blade gives the highest input of mixing energy for both the P-30 and the Class G slurry. The value of 5.6 kJ/kg measured for the Class G slurry when using the WB-blade is slightly above the SME concept of 5.5 kJ/kg suggested by Orban et al. [5,6] for high-speed mixing of a neat Class G slurry with a W/C ratio of 0.44 by weight.

The input of mixing energy from the HM-blade is significantly less for both slurries compared to that of the WB-blade. The shape of the HM-blade is similar to that of the original WB-blade. The main differences being that the four wings of the HM-blade is of equal length and is shorter than that of the WB-blade. When applying the law of similarity on the two propeller blades, a ratio of 0.6 between the input of mixing energy from the HM-blade compared to that of the WB-blade could be calculated [8]. The law of similarity for hydraulic machines [8] states that, when other things are being similar, the energy delivered by propeller blades are related to the fifth power of the diameter. Comparing that with the measured ratios of 0.6 for the P-30 and 0.7 for the Class G slurry, we see that it gives a good indication of the expected ratio between the mixing energies for the two propeller blades. The input of mixing energy from the short-winged propeller blade is lowest for both slurries. However, as it is different in shape compared to the other two, the law of similarity cannot be used for comparison of the mixing energy.

It is seen from the data presented in Table 2 that the input of mixing energy is significantly larger when mixing the P-30 slurry than when mixing the Class G slurry using the original WB-blade. The mixing energy is approximately 36% larger in this case. When the HM-blade was

Table 2
Measured mixing energies for high-speed mixing of slurries in accordance with API [1] using different types of propeller blades applied on neat slurries with a W/C ratio equal to 0.38

Propeller type cement slurry	Class G (kJ/kg)	P-30 (kJ/kg)
WB-blade	5.6	7.6
HM-blade	3.9	4.6
Short-winged propeller blade	1.8	1.8

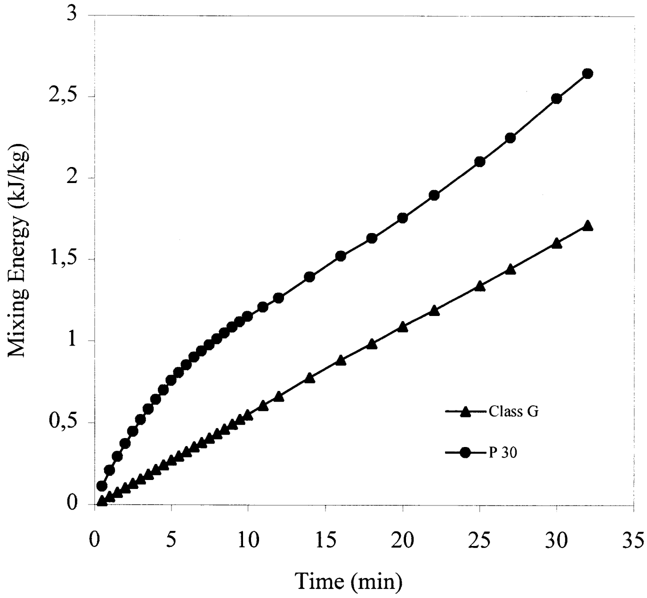

Fig. 1. The accumulated mixing energy measured as a function of time during mixing in the Hobart mixer.

used, the mixing energy applied during mixing the P-30 slurry is 18% larger than when blending the Class G slurry. For the short-winged blade, no measurable difference in mixing energy was observed in the experiments with Class G and P-30 cement.

A complicating factor, when using a high-speed mixer, is that due to the volume of the sample and construction of the mixing cup, air is drawn into the slurries and this again reduces the input of mixing energy. This effect has not been evaluated in our study. It is, however, expected that the suction of air will increase with increasing viscosity.

The differences in mixing energies observed when mixing the P-30 and the Class G slurries are anticipated to be mainly a result of the different viscous properties of the partially hydrated slurries. Since the Blaine surface area of the P-30 cement is larger than that of the Class G cement as shown in Table 1, the P-30 slurry must consist of finer particles than the Class G cement. The water concentrations are equal in the two slurries. A particle slurry built using finer particles will be more viscous than a slurry built with coarser particles. Therefore, at any time during mixing, the viscosity of the P-30 slurry will be larger than the Class G slurry viscosity, as will be shown in the third part of the present study. To mix a more viscous slurry will require more energy than mixing a less viscous slurry. Therefore, the concept of SME is not well defined and should be used with great caution.

3.3. Evaluation of the effect of mixing energy using the Hobart mixer

The input of mixing energy to the two slurries has been compared when using a low shear rate Hobart mortar mixer. It is expected that the effect of air mixed into the slurries will not vary so much with this type of mixer. The results are shown in Fig. 1. Here, the accumulative input of mixing energy in J/kg has been plotted as a function of time. From the curves, it is seen that the accumulative input of mixing energy is highest for the P-30 slurry. The difference is most profound during the first 7 min of mixing. The slope of the P-30 curve is higher than that of the Class G slurry during the first 7 min. A possible explanation for this higher slope may be that it is more difficult to disperse all the cement particles with the applied mixing energy when working on the P-30 slurry. Therefore, roughly 1 kJ/kg may be needed before a sufficient number of particles have been hydrated to create stable agglomerates. Since the Class G cement consists of larger particles, the cement slurry may be sufficiently hydrated nearly immediately during this type of mixing to reach the similar agglomeration level. It is unlikely, however, that total hydration of the cement grains have occurred during this mixing. If this were the case, the cement hydration would not be as sensitive for high-speed mixing as have been observed in the past [9].

3.4. The effect of consistometer mixing energy on rheological properties of the cement slurries

The consistency development measured with the atmospheric consistometer is shown in Fig. 2. Here, the development of the consistency is given as a function of time for the P-30 and the Class G slurries. For these tests, the propeller blade used for high-speed mixing gave no measurable difference in the input of mixing energy, see Table 2. The curves show the torque and thus, the input of mixing energy per unit time to the different slurries can be calculated. The P-30 slurry demonstrates a higher torque value

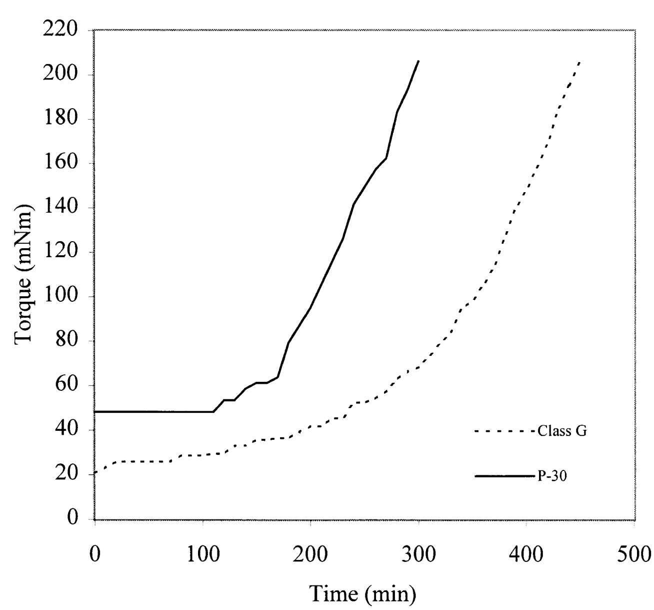

Fig. 2. The consistency development measured as a function of time in the atmospheric consistometer.

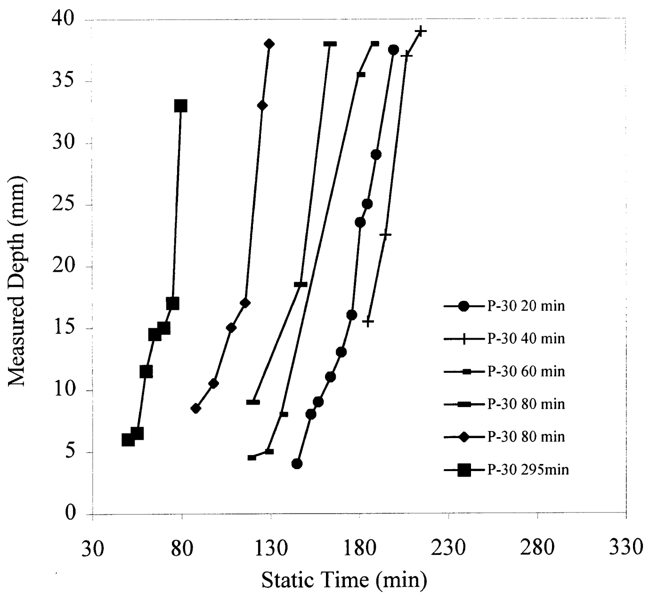

Fig. 3. Measured height in a Vicat test as a function of time for the P-30 slurry. The different curves represent different preconditioning times in the consistometer prior to Vicat testing.

from the start of the test indicating a higher input of mixing energy. This is in accordance with the measured input of mixing energy shown in Fig. 1. The energy input during the first 20 min, taken as the area below the curves, is approximately 0.86 kJ/kg for the P-30 slurry and 0.48 kJ/kg for the Class G slurry. The first 20 min corresponds to the standard API [1] conditioning time prior to viscosity measurements and other laboratory evaluations. Fig. 2 also shows that, although at every time the input of mixing energy to the P-30 slurry is the highest, the total input of mixing energy before reaching 30 Bc is higher for the Class-G slurry, reflecting the coarser grain sizes of the Class G cement.

The value of 30 Bc, which generally is considered to be the maximum pumpable consistency, is reached after 285 min for the Class-G slurry. The measured consistency of the P-30 is above 30 Bc from the start. It has been proposed [10] that the time to reach the 30-Bc value reflects the time to reach initial set for oilwell cement slurries. However, this has not been confirmed [3]. It may also be suggested that initial setting of the cement occur when the slope of the consistency curves increase to a higher value as shown on the right-hand side in Fig. 2. If this would be the case, initial setting should occur approximately 160–170 min after blending with water for the P-30 slurry and after 310–330 min for the Class G slurry.

To investigate if initial setting would occur at 30 Bc or at the point where the curves of the consistency measurements change slope, a series of Vicat experiments were performed. The results from these Vicat experiments are shown in Figs. 3 and 4. The measured values are given in millimeters with reference to the bottom of the samples as a function of time in the Vicat apparatus. The input of mixing energy to the slurries given as the time spent in the consistometer prior to testing is shown in the legend for each curve. Each curve is based on data from one slurry. Only for the P-30 slurry with 80 min in the consistometer, the two slurries have been tested.

As can be seen in Fig. 4, the initial set of the Class G slurry when measured using the Vicat apparatus occur after approximately 170 min in the test apparatus when the preconditioning time was 20 min. This gives a shorter setting time than that observed in the consistometer thickening time test. If the slurry had experienced a 240-min-long preconditioning, a Vicat set time of 50 min was observed. This set time could reflect the suggested initial setting time measured with the consistometer. However, for slurries being preconditioned for more than 500 min, the Vicat set time is still 50 min. Furthermore, less mixing energy has been applied to the slurries preconditioned for 20–80 min. All these slurries obtain a Vicat set time in less than 170 min with a total time after blending with water significantly less than 285 min. These measurements indicate a shorter set time if the amount of mixing energy is increased, which is in contradiction to expected results.

The total set time for the P-30 slurries compare more accurately to the suggested change in slope of the consistometer curve for the slurries being preconditioned for 20–80 min. This can be seen in Fig. 3. The slurry, which was preconditioned for 40 min, had a longer Vicat set time than the slurry preconditioned for 20 min. This is expected to be an experimental inaccuracy. Most likely, only minor differences exist between the slurries after this short preconditioning time. The effect on Vicat set time may therefore only be insignificant. This type of argument also seems valid for the Class G slurry measurements as is shown in Fig. 4.

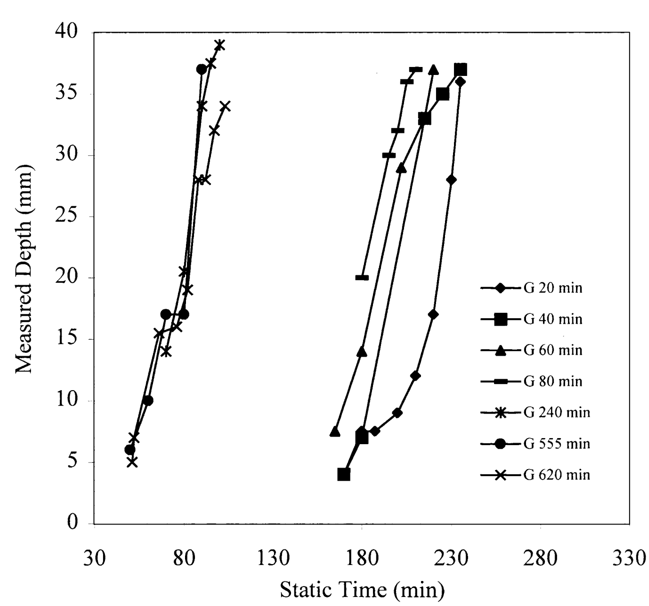

Fig. 4. Measured height in a Vicat test as a function of time for the Class G slurry. The different curves represent different preconditioning times in the consistometer prior to Vicat testing.

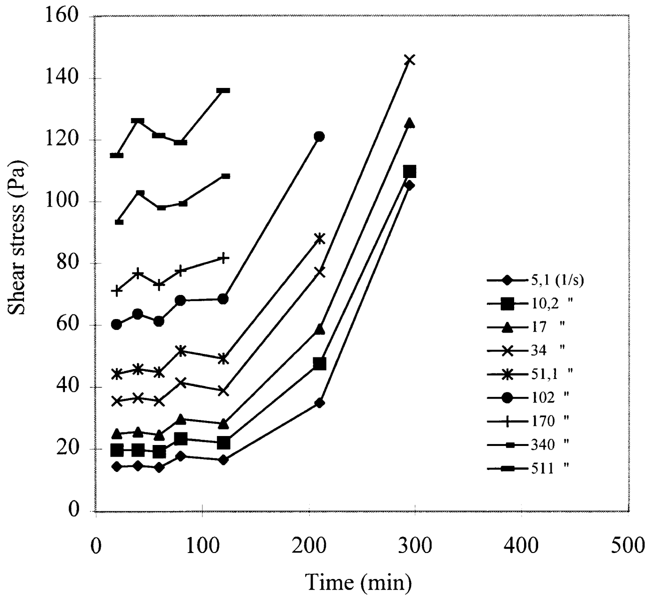

Fig. 5. Shear stress measured at different shear rates as a function of preconditioning time in the consistometer for the P-30 slurry.

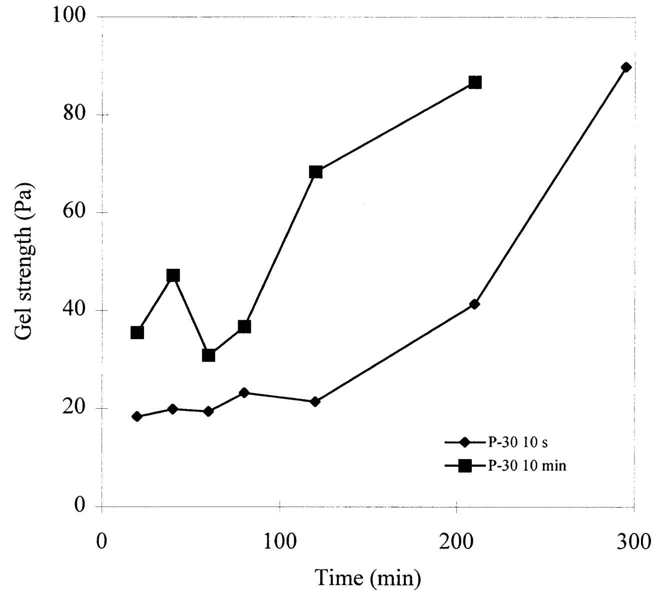

Fig. 7. Peak gel strength measured on the P-30 slurry.

It was possible to precondition the P-30 slurry for 295 min. Still, the Vicat measurement gave a Vicat set time of approximately 50 min. This gives a total set time, including the precondition time, in excess of 340 min, far longer than indicated during any other measurements.

The Vicat set time measurements and the initial set measurements using the atmospheric consistometer gave a large variation in set time. A large discrepancy exists even within the Vicat measurements. It is therefore natural to conclude that in neither of these measurements the set point

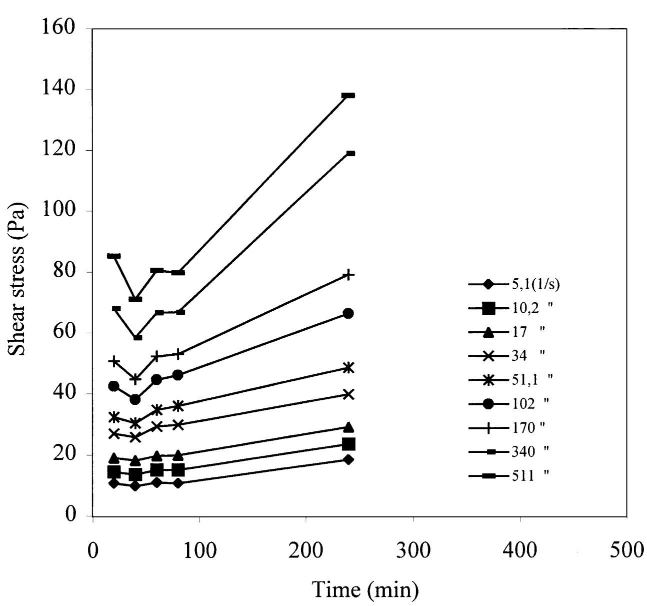

Fig. 6. Shear stress measured at different shear rates as a function of preconditioning time in the consistometer for the Class G slurry.

was measured. The consistometer measurements will most probably yield data regarding viscosification of the cement slurry in the early stages of cure. This viscosification is most likely a result of creation of small precipitated particles and change in morphology of the cement itself. The Vicat test, however, would not depend on the viscosity of the cement slurry to the same degree. In this case, it is more likely that the method is vulnerable to gel formation or structure build up in the slurries. Therefore, since cement slurries do form a progressive gel, it is possible to see similar data for different degrees of preconditioning. The necessary condition to obtain a Vicat result is to wait for a sufficient gel strength to be formed.

The development of viscous properties and gel strength of the P-30 and Class G cements has not yet been compared. The viscosity and gel strength of the slurries were measured as a function of shear rate and input of mixing energy, the latter was done by letting the slurries spend different amounts of time in the atmospheric consistometer. In Figs. 5 and 6, each curve represents the shear stress measured at a particular shear rate and is plotted as a function of the time spent in the atmospheric consistometer. The first data plotted after 20 min represents the standard API [1] testing. For all shear rates and times, the measured shear stresses are highest for the P-30 slurry. In Table 1, we see that the P-30 cement has the highest surface area and thus this is in accordance with Shaugnessy and Clark [11] who reports increasing viscosity with increasing surface area. The higher viscosity of the P-30 slurry is also in accordance with the higher input of mixing energy measured both in the Hobart mixer and in the consistometer.

However, for both slurries, there is a reduction in the measured shear stress followed by an increase as a function of time. For the Class G slurry, this local minima occurs

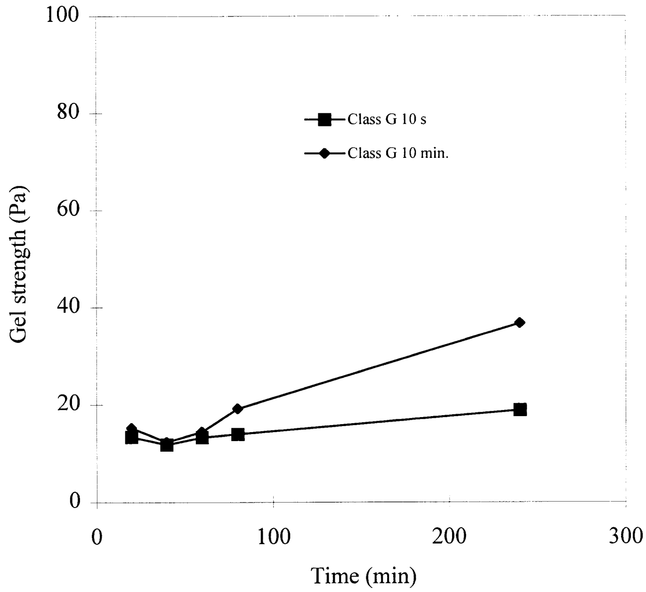

Fig. 8. Peak gel strength measured on the Class G slurry.

after 40 min in the consistometer and for the P-30 slurry after 60 min, respectively. Local minima after 40 min preconditioning in the consistometer has been observed earlier [12]. This drop in viscosity as a function of input of mixing energy is in accordance with results obtained by Banfill and Kitching [13] who refer to it as a result of structural breakdown. Vlachou and Piau [14,15] also measured a local minima at low shear rates on a retarded Class G slurry with a W/C ratio of 0.44 after 8 h preconditioning in a consistometer. It is less likely, however, that a structural breakdown can be observed after 8 h. A speculation, therefore, is if the reduction in viscosity is a result of change in morphology of the cement as a result of chemical effects. Can some of the cement particles have grown to sizes allowing for a fluidity with a lower water content? After the local minima, the viscosity increases again as a function of time and increasing input of mixing energy. According to Banfill and Kitching [13], this thickening effect is due to the onset of an early hydration.

The measured gel strengths of the two slurries are shown in Figs. 7 and 8. The curves show the gel strength development after a 10 s and a 10 min static period, respectively, as a function of the input of mixing energy in the consistometer. The P-30 slurry data shown in Fig. 5 obtain the highest values for both the 10 s and 10 min gel compared to the data from the Class G slurry shown in Fig. 6. The rate of gel formation given as the difference between the 10 s and the 10 min reading is also higher for the P-30 slurry compared to that of the Class G slurry. Generally, the measured gel values show an increase with increasing input of mixing energy. The local minima in the measured shear stresses observed in the viscosity measurements shown in Figs. 5 and 6 are also reflected in the measured gel strengths shown in Figs. 7 and 8. These minima occur after the same input of mixing energy in the consistometer as the minima observed in the viscosity measurements: 40 min for the Class G slurry and 60 min for the P-30 slurry.

A similarity with the viscosity measurements was also reflected in the gel strength measurements where the ability of the Class G slurry to form a strong gel is lower than that of the P-30 slurry. This is clearly illustrated in Figs. 7 and 8.

4. Conclusion

The input of mixing energy has a measurable influence on the rheological properties of cement slurries. It has been shown that the input of mixing energy per time unit both in the mixers and in the atmospheric consistometer depends on the viscous properties of the slurry. The term SME introduced by Orban et al. [5,6] has been found to be valid for a neat Class G slurry only when using a Waring Blendor high-speed mixer with an original Waring Blendor propeller blade. The SME concept should therefore be used with caution.

As various types of propeller blades are in use in the laboratories of the oil industry, there is a need for stating the type of propeller blade used during the high-speed mixing of the cement slurries. Alternatively, the input of mixing energy could be stated as experimental conditions.

It has been shown that neither consistometer measurements nor Vicat measurements can be used to determine the initial set of a cement slurry. The consistometer most likely reveals an increase in viscosity as a result of cement hydration. Similarly, the Vicat test depends most likely on gel formation processes in the cement slurry. These gel formation processes will again depend on cement hydration.

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