Comparative Study of Testing Methods for Cement pastes

Helge Hodne[1], Svein O. Wick[1], Rolf A. Jakobsen[1] and Arild Saasen[2]

[1]Høgskolen i Stavanger, P.O.Box 2557, Ullandhaug, N-4004 Stavanger, Norway
[2]Statoil, N-4035 Stavanger, Norway

ABSTRACT

The initial and final setting times for cement slurries or pastes have been measured using various standard testing methods. A comparison have been made which indicates that the different methods highlights different important properties for flowing cement slurries.

INTRODUCTION

Various measuring methods for cement mixtures are used for different industrial applications. Each method has its own background and history of development. Some methods gives results in physical units while others measure relative values which means that the obtained results can only be used in comparing one cement paste with another as no conversion factor to other applicable units exists.

In this work we have tried to compare measuring methods used in two different types of industry: The construction industry and the oil-well drilling industry. Both industries have their own types of testing equipment and their own specifications for measurement procedures.

In the present study cement pastes have been tested in accordance with standard procedures for both the construction and the petroleum industry.

CONSTRUCTION INDUSTRY TESTS

Four different testing methods are used to evaluate properties of fresh cement pastes. Vicat testing in accordance with NS EN 196[1] Proctor testing in accordance with NS 3661[2], loss of consistence by means of a flow table as described in NS 3107[3] but with a modified cone having a height of 45mm an upper diameter of 60mm and a lower diameter of 90mm, and a table operation as described in BS 4551[4] and finaly a temperature measurement method.

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 300g. Measurements are given in mm with reference to the bottom of the sample. The 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.
The Vicat test requires a consistency of the slurry that is prescribed in the standard. This gives a rather stiff slurry normally with a water/cement-ratio well below 0.30. Higher ratios gives longer setting times with possibilities of segregation in the slurry. However, in the present study, we have used higher ratios in order to obtain comparative results from all measuring methods.

The Proctor testing is similar to the Vicat as a body is forced into the sample. Instead of applying a constant force on the needle the force used to press a piston 25 mm into the sample is measured. The piston can have different diameters. The measuring unit is MPa.

In the flow table testing a sample moulded as a cone is placed on a horizontal table. The table is upheaved by a camshaft and then allowed to fall free 9mm down. The table is allowed to fall 25 times with a frequency of 1.67 Hz. The average diameter of the cone is then measured. The diameter of the sample is then compared to the diameter measured on a fresh newly mixed sample which is said to have a consistency of 100%. Initial set is indicated to be reached when the diameter measured is reduced to 25-30%.

In the temperature test the hydration temperature development is logged by a thermocouple buried in the cement paste. Initial set is indicated when the temperature has risen 2 °C above the starting temperature.

OIL-WELL INDUSTRY TESTS

Specifications for well-cement testing is given by API[5]. Two types of measurements on the cement slurries is routinely carried out, viscosity and consistency evaluation.

In the present study a CHAN 35 viscometer was used for the viscosity measurements. This 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, 511 and 1022 s^{-1}. Gel strength was measured as the peak shear stress at a shear rate of 5.1 s^{-1} after 10 s. or 10 min. static delay.

The consistency was measured using an atmospheric consistometer. In this apparatus the torque exerted on a stationary paddle immersed in a rotating slurry-container is measured. This 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), see Eq. 1.

$$B_c = \frac{T - 78.2}{20.02} \qquad (1)$$

In Eq. 1 T is the torque in g·cm. The Bc unit is a dimensionless quantity relating to a viscosity value, however, with no direct conversion factor to more common 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 oil well application.

SAMPLE PREPARATION

Two types of cement material have been used to compare the different standard testing methods. One type is a construction cement named P-30 and the other is an oil well cement type Class-G. Both cements were produced by Norcem A/S (Table 2).

The mixing procedure for the cement paste tested by the Vicat, Proctor, flow table and temperature were in accordance with NS EN 196[1].

The samples tested in the consistometer and the viscometer were mixed in accordance with API Spec.10[5]. This gives that the samples are to be mixed in a high speed mixer at 4000 rpm for 20 seconds and then 12000 rpm for an additional 35 seconds prior to testing. This induces a similar amount of mixing energy into the sample as that applied during mixing on the drilling rig. The volume of each test sample was 600 ml. The standard procedure for preconditioning the samples for viscosity evaluation is to run the samples in the consistometer for 20 minutes prior to performing measurements. For the samples to be tested in the consistometer the testing started immediately after the high speed mixing.

All tests were carried out at 20 ± 2 °C.

SENSITIVITY OF W/C-RATIO

Different water/cement-ratios were used ranging from 0.27 to 0.44. A ratio of 0.27 was too low to be used for well-cement testing and a ratio of 0.44 was found to be too high to be used in the static tests for construction cement due to development of free water. For most of the testing a w/c-ratio of 0.38 was used. The cement pastes were mixed with distilled water and no other additives were used. According to Taylor[6] for a Portland cement the w/c-ratio equal to 0.38 gives a paste consisting of only hydration products. Finally at a w/c-ratio equal to 0.44 the paste contains enough water to fill up the matrix pores completely after completed hydration. The w/c-ratio equal to 0.44 is widely used for oil-well application.

CEMENT DATA

The cements used are produced by Norcem. The P-30 cement meets the requirements to CEM I 42.5R[7] The Class G cement meets the requirements to CEM I 42.5-SR,LA[7] Both are in accordance with NS 3086[7]. Typical cement data from Norcem are given in Table 2.

MEASUREMENT RESULTS

Vicat testing

In Table 1 the results of the Vicat tests are shown. For low w/c-ratios there is a marked shorter setting time fore the P-30 cement compared to that of the Class-G cement. This difference is reduced as the w/c-ratio increases.

Table 1. Vicat testing. Values are given in minutes

	w/c	P-30	Class-G
Init. set	0.27	195	242
Final set	0.27	291	327
Init. set	0.38	331	350
Final set	0.38	385	425
Init. set	0.44	410	415
Final set	0.44	483	495

Proctor testing

The Proctor tests shows the same tendency as the Vicat, as w/c-ratio increases the difference in setting time between the two cements is reduced. These results are shown in Table 3.

Table 2. Cement data.

Type:	P-30	Class G
Fineness:	345	320
Specific surf. Blaine	m²/kg	m²/kg
Sieve residue 200 micron		0 %
Initial setting time	130 min	140 min
Soundness		
Le Chatelier expans.	1 mm	0 mm
Compressive strength		
1 day	21 MPa	
2 days	32 MPa	22 MPa
7 days	42 MPa	37 MPa
28 days	52 MPa	55 MPa
Chemical Analysis		
Loss on ignit.(L.O.I.)	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 %
PotassiumOx. (K_2O)		0.44 %
SodiumOxide (Na_2O)		0.21 %
Chloride (Cl)		0.02 %
Carb. Dioxide (CO_2)		0.15 %
WatersolubleChlorine	0.1%	
Carb. Dioxide (CO_2)		0.15 %
WatersolubleChlorine	≤0.1 %	
Watersol.Chromium	≤2.0 ppm	
Alkali (Na_2O) Eq.	0.95 %	0.50 %
(C3A)		1.2 %

Table 3. Proctor testing. Values are given in minutes.

	w/c	P-30	Class-G
Init. set	0.27	140	200
Final set	0.27	250	315
Init. set	0.38	295	290
Final set	0.38	415	440
Init. set	0.44	325	-*
Final set	0.44	460	-*

*not measured due to segregation

Flow table testing

The flow table tests were carried out at a w/c-ratio equal to 0.38. Results are shown in

Fig. 1. A measured consistency of 25-30% indicates initial set. The flow table test indicates an initial set for the P-30 cement after 345 minutes and between 360 and 390 minutes for the Class-G cement.

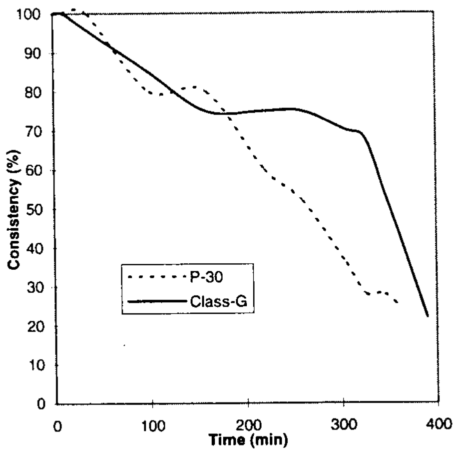

Figure 1. Flow table tests. The w/c-ratio is equal to 0.38

Temperature measurements

Hydration temperature development was measured for a P-30 and a Class-G cement paste, both with a w/c-ratio equal to 0.38. Initial set, i.e. a 2 °C increase in temperature is reached after 305 minutes for the Class-G cement paste and after 345 minutes for the P-30 cement paste. Temperature measurements are shown in Fig. 2 and 3.

Viscosity measurements

In Table 4 a comparison of the viscosity data obtained for the two cements are shown. The data shows the consistency of the slurry prior to hardening. The gel strength gives the workability of the floating slurry. For both a w/c-ratio equal to 0.38 and 0.44 the Class-G cement shows higher values for high shear rates. For lower shear rates there is no significant difference. At a w/c-ratio equal to 0.38 there is a faster gel formation than observed for the w/c = 0.44 slurries. This is anticipated to be a result of the higher solid fraction.

Table 4. Viscosity measurements for two different w/c-ratios. The values are average dial readings.

Cement:	P-30	Class-G	P-30	Class-G
w/c-ratio:	0.38	0.38	0.44	0.44
$1022 s^{-1}$	-	-	200	227
$511 s^{-1}$	275	285	134	167
$340 s^{-1}$	222	231	106	131
$170 s^{-1}$	161	160	77	93
$102 s^{-1}$	126	123	64	74
$51.1 s^{-1}$	87	82	50	55
$34 s^{-1}$	68	64	41	41
$17 s^{-1}$	47	44	28	27
$10.2 s^{-1}$	37	34	21	20
$5.1 s^{-1}$	22	26	15	15
10 s-gel	35	37	17	17
10min-gel	76	95	22	27

Consistometer measurements

The data for the consistometer measurements with a w/c - ratio equal to 0.38 and 0.44 are given in Table 5. Here the difference between the two cements is significant for both w/c-ratios as the time to reach 30 and 100 Bc for the Class-G cement is about doubled compared to the P-30 cement.

Table 5. Atmospheric Consistometer readings (minutes).

Cement:	P-30	Class-G	P-30	Class-G
w/c-ratio:	0.38	0.38	0.44	0.44
30 Bc	113	250	268	598
100 Bc	373	720	520	1323

DISCUSSION

In Table 6 the data for the indicated initial set from Vicat, Proctor, flow table and temperature measurements are compared to the 30 Bc values measured by the atmospheric consistometer. The Vicat, Proctor, flow table and hydration temperature measurements seem to correlate well for the P-30 slurry. The variation in the measured values for the Class-G slurry is greater. In none of these experiments the results correlate to the 30 Bc value from the atmospheric consistometer. The earlier 30 Bc measurement is anticipated to be a result of the additional mixing energy applied to these slurries. It is, however , questionable if 30 Bc does reflect the initial set in a cement slurry[8] .

In Fig. 2 and 3 the Vicat, Proctor, temperature and Bearden units of consistency are plotted vs. time in the same diagram for a w/c-ratio of 0.38. From Fig. 2 it is obvious that information about the development of the consistency of the slurry obtained from the Bc measurements before hardening is lost when measured with Vicat or Proctor. The consistency development vs. time is also indicated by the flow table measurements in Fig. 1.

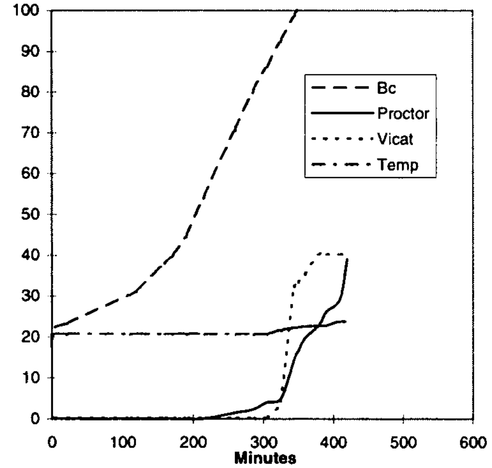

Figure 2. The Vicat, Proctor, temperature and Bearden units of consistency have been plotted vs. time in the same diagram for a P-30 cement slurry having a w/c-ratio equal to 0.38.

The Bc-curves for the two different cements shown in Fig. 2 and 3 shows two cements that behave quite different when pumped in place. The period of about 200 minutes with constant Bc for the Class-G cement in Fig. 3 shows a cement that is pumpable for a long period. The same curve in Fig. 2 indicates that the P-30 cement is not suitable for slurry pumping operations.

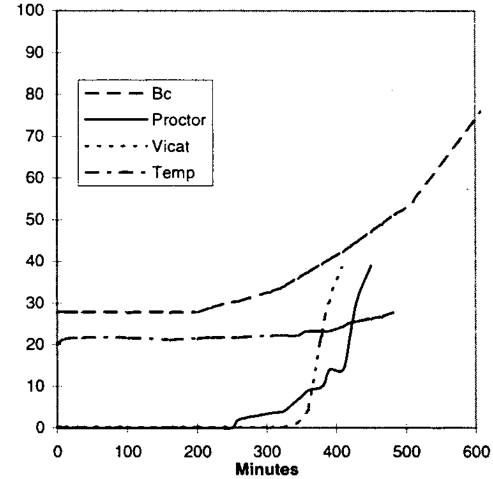

Figure 3. The Vicat, Proctor, temperature and Bearden units of consistency have been plotted vs. time in the same diagram for a Class-G cement slurry having a w/c-ratio equal to 0.38.

It is seen in Table 7 that no relation can be found between values for the final set from Vicat or Proctor tests and the 100 Bc consistency value. According to API Spec. 10[5] Bc-values higher than 30 should not be quoted from the atmospheric consistometer. However, the atmospheric consistometer is more applicable than Vicat or Proctor for showing the development of consistency for flowing slurries.

The viscosity and gel development as a function of time cannot be measured with Vicat, Proctor or atmospheric consistometer. The experiments shown in Table 4 has indicated that a particle gel is likely to be formed in a cement slurry. A viscometer or a more advanced rheometer is needed to measure such gel properties.

If a cement slurry has to be pumped, it is important to have detailed knowledge about the slurry viscosity. This information can not be obtained from Vicat, Proctor or atmospheric consistometer tests.

Table 6. Indicated time for initial set or 30 Bc for w/c-ratio of 0.38

	P-30	Class-G
Vicat	331	350
Proctor	295	290
Flow table	345	360-390
Temperature	345	305
Atm. Consistom.	113	250

Table 7. Indicated time for final set or 100 Bc for w/c-ratio of 0.38

	P-30	Class-G
Vicat	385	425
Proctor	415	440
Atm. Consistom.	373	720

CONCLUSION

For slurries without dispersion agents an atmospheric consistometer can be used for w/c-ratios ≥ 0.38. For slurries with a w/c-ratio < 0.38 the Vicat, Proctor, flow table or hydration temperature must be used.

Consistometer data using atmospheric consistometer does not correlate well to initial and final set data from Vicat or Proctor.

It is also shown that the atmospheric consistometer is more applicable than Vicat or Proctor for showing the development of consistency for flowing slurries.

The viscosity and gel development as a function of time can not be measured with Vicat or Proctor.

REFERENCES

1. NS-EN-196: Methods of testing cement. 1995

2. NS 3661: Concrete testing. Fresh concrete. Hardening. February 1987

3. NS 3107: Mortars for brick work and plaster. Testing. Consistency. 1988.

4. BS 4551:Methods of testing. Mortars, screeds and plasters. 1980.

5. American Petroleum Institute, *Specifications for Materials and Testing for Well Cements,* API Spec. 10, Fifth ed., Washington DC, July 1, 1990.

6. Taylor, H.F.W., "Cement Chemistry", Academic Press Ltd., London (1990), pp. 249-250.

7. NS 3086:1995: Portland cements. Specifications of composition and properties. September 1995.

8. Kleef, R.P.A.R.van. and Vllt, J.P.M.van, "Improving the Reliability of Cement-Setting-Time Tests by Taking Into Account the Influence of Shear" SPE Drilling & Completion, March 1993, pp. 51-56.