CHARACTERIZATION OF CEMENT TYPES USED FOR ROCK INJECTION

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Summary

This paper is a part of the research project “True Improvement in Grouting High pressure Technology for tunneling (TIGHT)" financed by the Research Council of Norway (project no. 236676/O30), in cooperation with Statens Vegvesen, Jernbaneverket, and industrial partners BASF, Mapei, Geovita, LNS, ITS, Normet, Bever Control, AMV and Veidekke. Research partners in TIGHT are NGI, NTNU and SINTEF, whilst KIGAM of Korea and Nanyang University in Singapore together with BeFo of Sweden are associated with TIGHT. The authors would like to thank Eivind Grøv (NTNU and SINTEF), Bahman Bohloli (NGI), Hans-Olav Hognestad (BASF), Bjarne Ruud and Espen Rudberg (Mapei), for significant contributions for the selection and preparation of cements, defining laboratory program and the comments on test results. The objective of the study was to characterize different cements used for rock injection in the Nordic countries, and provide basic data for the behavior of fresh grout prepared from these cements. A laboratory program was set up for characterization of the fineness for seven available cement types. The fineness was characterized by the use of Blaine, BET and particle size distribution. The results show good correlation between the obtained Blaine and BET values. It was also found that D_{95} from the particle size distribution curve is unsuited for characterization of cement fineness and most grout properties. Three of the most commonly used cements in Norway were selected for testing of flow properties, stability and initial set for fresh grout. Grout was prepared with four different water to cement ratios (w/c = 1.2, 1.0, 0.8 and 0.6) and tested at two different temperatures (8 and 20°C). The test methods applied were bleeding, consistency (ring and Marsh Cone), Vicat and temperature evolution in isolated cup. Additional tests with parallel plate rheometer were performed for mixes at 20°C. The results show that only one of the cements tested can be utilized at all w/c levels, while the other two have limitations both upwards (bleeding) and downwards (flowability) with respect to w/c applied. The results from this investigation should be evaluated together with additional penetrability tests in order to evaluate the suitability for grouting and filling of small cracks.

INTRODUCTION

The objective of the study was to characterize different cement used for rock injection in the Nordic countries, and provide basic data for the behavior of fresh grout prepared from these cements [1]. A total of seven cements were selected for initial testing; the two most commonly used in Norway and 5 other cements. It was emphasized to make initial tests on relevant cements within a wide fineness range, and then select 3 cements for testing in cement paste.

The following cements were tested

- Norcem Industrisement
- Cemex Industrisement
- BASF MasterRoc MP650
- BASF MasterRoc MP800
- BASF MasterRoc MP900
- Ultrafin 12
- Microfine 20

The cements are anonymized and referred to as cement A-G in random order in the present paper.

CEMENT CHARACTERIZATION

All cement samples were initially tested for

- Density by Accupyc 1330 helium pycnometer
- Particle size distribution by Colter LS Particle Size Analyzer
- Blaine fineness (EN 196-6)
- BET fineness by nitrogen absorption according to Braunauer, Emmett & Teller

The test results are given in Table 1 and the particle size distribution is given in Figure 1.

Table 1 Test results, characterization of cements

Cement	A	B	C	D	E	F	G
Density, g/cm^3	3.17	3.16	3.10	3.16	3.17	3.16	3.21
Blaine, cm^2/g	729	541	706	793	856	557	892
BET, m^2/g	1.88	1.58	1.93	1.95	2.16	1.49	2.11
D_{95}, µm	17	18	25	29	30	33	12

Testing of Blaine fineness is usually performed with a compacted cement bed porosity 0.5. For very fine cements the sample must be reduced in order to achieve a full compaction of the cement bed, and the porosity can thus be lower than 0.5. There is a good relation between specific surface values determined according to Blaine and BET, as shown in Figure 2

The relation between specific surface and D_{95} is rather poor, see Figure 2.

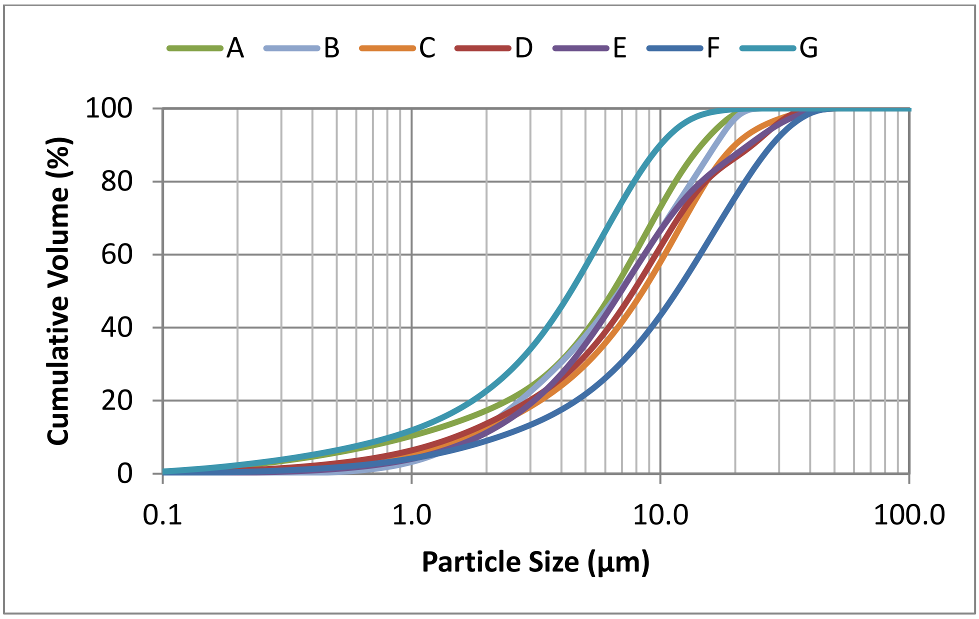

Figure 1 Particle size distribution for tested cements

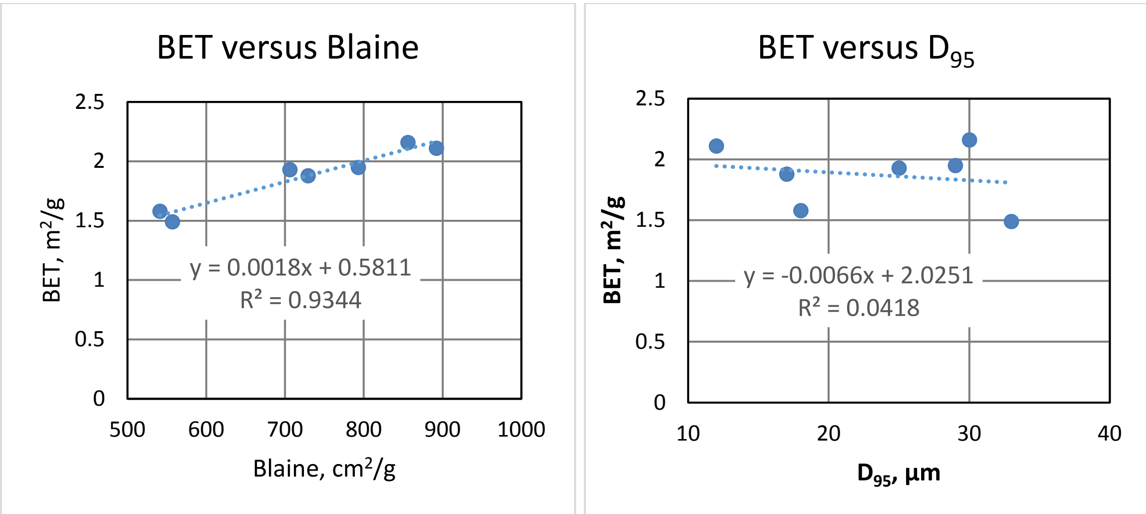

Figure 2 BET versus Blaine and D_{95}

TESTING IN CEMENT PASTE

The cements labeled A, B and C were picked for testing in cement paste. The testing was performed at 20 and 8^{0}C. Four mixes with w/c from 0.6 to 1.2 were made for each cement type. Mixing was performed by a 4 liter Waring high-speed mixer at 2000 rpm. Water was filled into the mixer and the mixer started before the cement was poured into the water during ½ min of mixing. The paste was thereafter mixed for 2 min.

Table 2 **Mixes performed for cement A, B and C**

Mix	1	2	3	4
Cement, kg	1.75	2.00	2.35	2.85
Water, kg	2.10	2.00	1.88	1.71
w/c	1.2	1.0	0.8	0.6

Figure 3 **Mixing in Waring high-speed mixer**

Paste consistency

The paste consistency was measured by a standard Marsh Cone with 4.5 mm outlet (27.6 sec for 1 liter water) and spread by the use of a cylinder (diameter 39 mm, height 60 mm) according to EN445.

Table 3 **Initial consistency at 20 and 8^{0}C**

Cement	Cement A				Cement B				Cement C			
w/c	1.2	1.0	0.8	0.6	1.2	1.0	0.8	0.6	1.2	1.0	0.8	0.6
Room temperature	20^{0}C											
Paste temperature	20	19	19	19	19	20	20	20	19	19	19	19
Spread (EN445), mm	290	255	229	143	284	263	222	127	295	255	220	150
Marsh Cone, sec	30.3	32.6	36.4	52.1	30.4	32.6	39.2	88.4	31.0	33.9	39.6	-*
Room temperature	8^{0}C											
Paste temperature	7	7	6	6.5	6	6	6	7	7	7	7	6
Spread (EN445), mm	295	290	250	205	280	280	255	190	285	260	215	160
Marsh Cone, sec	32.0	33.5	36.1	50.4	30.3	31.5	36.0	51.1	31.5	34.3	39.6	75.6

* The paste was too stiff for Marsh Cone testing

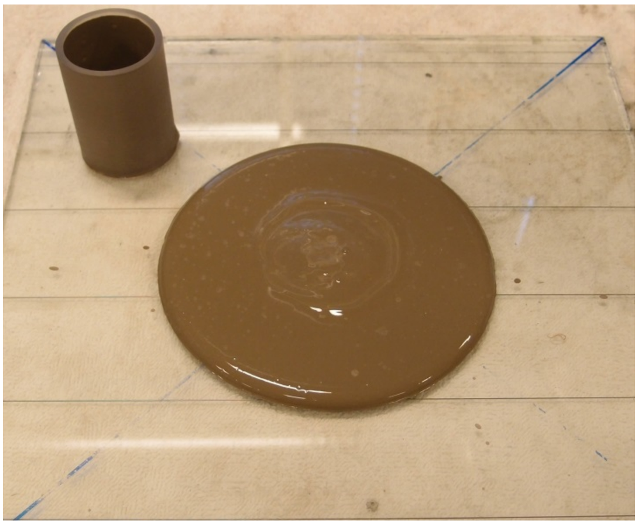

Figure 4 **Spread EN445 (left) and Marsh Cone with 1 litre graduated cylinder (right)**

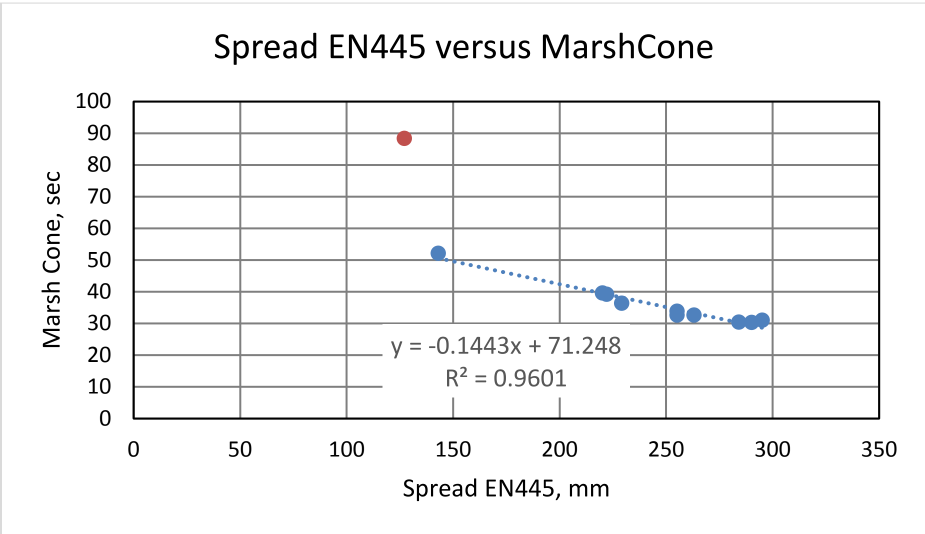

Figure 5 **Relation between spread according to EN445 and Mars Cone test results at 20^{0}C. The red point represents Cement B at w/c = 0.6 (w/c = 0.6 for cement C did not give any Marsh Cone result)**

Bleeding

Volume stability and bleeding were measured by 2 pcs. 250 ml graduated cylinders per mix. The volume of bleed water was recorded every ½ hour the 2 first hours and finally after 24 hours.

Table 4 Bleeding test results

Cement		Cement A			Cement B			Cement C		
Room temperature		20^oC								
w/c		1.2	1.0	0.8	1.2	1.0	0.8	1.2	1.0	0.8
Bleed water in % of original paste volume	0 min	0	0	0	0	0	0	0	0	0
	30 min	0.6	0	0	4.8	1.6	0.8	4.3	1.0	0.4
	60 min	0.6	0	0	10.6	2.4	0.8	9.6	1.4	0.6
	90 min	0.6	0	0	17.8	2.8	1.2	13.0	2.4	0.6
	120 min	0.6	0	0	21.2	3.2	2.2	13.8	2.5	0.6
	24 timer	0	0	0	18.4	1.2	0	11.4	0	0
Room temperature		8^oC								
Bleed water in % of original paste volume	0 min	0	0	0	0	0	0	0	0	0
	30 min	0.8	0.4	0.4	2.2	0.8	0.4	2.0	0.4	0
	60 min	0	0	0	4.6	1.4	1.0	4.0	0.8	0.4
	90 min	0	0	0	6.4	3.6	1.6	5.6	1.0	0.4
	120 min	0	0	0	15.2	5.4	2.4	8.0	1.4	0.4
	24 timer	0	0	0	24.8	11.6	1.8	11.4	0	0

Cement B and C show unacceptable bleeding at w/c = 1.2, while cement A shows insignificant bleeding at all w/c levels. For paste with w/c = 0.6 only insignificant bleeding was observed for all cements tested.

Setting time

Setting time was determined at 20^oC by Vicat apparatus according to EN196-3. Tests were also performed by measurements of 2^oC temperature rise in 250 ml insulated (polystyrene) cups. Temperature measurements were performed at both 20 and 8^oC. Both methods are rather inaccurate for paste with high w/c, but a rough estimation of initial set can be made.

Table 5 Initial set results

Cement	Cement A				Cement B				Cement C			
w/c	1.2	1.0	0.8	0.6	1.2	1.0	0.8	0.6	1.2	1.0	0.8	0.6
Room temperature	20^oC											
Vicat, initial set, hours-min	-*	0-40	0-35	0-30	11-40	11-00	8-35	6-10	9-15	8-10	6-35	5-45
Time till 2°C temperature rise, hours-min	0-30	0-25	0-20	0-20	6-30	4-55	6-15	5-20	3-50	3-50	3-15	3-45
Room temperature	8^oC											
Time till 2°C temperature rise, hours-min	0-45	0-45	0-45	0-45	-**	23-55	20-40	17-10	14-35	13-00	11-15	9-50

* Initial set occured quickly, but was not recorded as the paste did not achieve enough strength to withstand the needle
** 2^oC temperature rise was not achieved

Cement A sets within 30-40 min, and is very little influenced by the w/c. For this cement, the set is only minor influenced also by the paste temperature (20 and 8^oC). Cement B and C set after several hours, and the Vicat set value seems to be dependent on the w/c. Temperature recordings indicate, however, that the paste sets a bit quicker and rather independent of the w/c. The cement reaction starts in the paste, but it takes time to develop sufficient strength to withstand the needle from penetrating. The set for Cement B and C also seems to be significant slower at lower temperatures.

RHEOMETER MEASUREMENTS

Immediately after mixing a sample was drawn for testing by Physica MCR 300 parallel plate rheometer. The rheometer consist of two serrated metal plates1 mm apart where the upper plate rotates, and the lower plate is fixed and maintained at 20°C by cooling water and a Peltier element for heating. Paste is placed between the plates and the upper plate rotates for 1 min at a speed corresponding to 100 s^{-1}. The speed is thereafter varied stepwise from 2 till 150 s^{-1} and back till 2 s^{-1} again. The Bingham viscosity (μ in mPa·s) and yield stress (τ_{dyn} in Pa) is calculated from the ≈ linear part of the descending curve.

Initial viscosity results are shown in Table 6 and Figure 6, and the relation between initial Marsh Cone and viscosity values is shown in Figure 7.

Table 6 Initial Bingham viscosity

	Viscosity 15 min after water addition, μ, mPa·s,			
w/c	0.6	0.8	1.0	1.2
Cement A	183	75.1	38.9	31.4
Cement B	139	56.9	31.3	19.7
Cement C	293	72.2	44.4	27.1

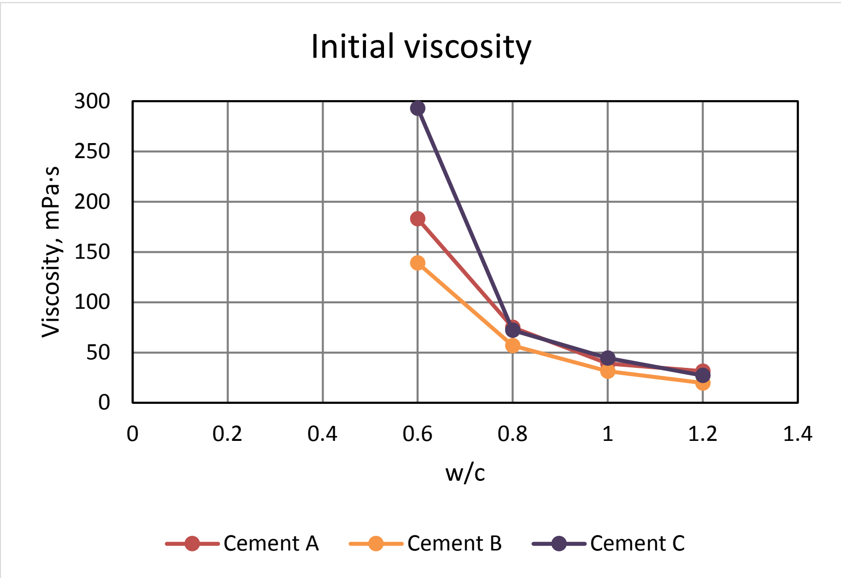

Figure 6 Initial viscosity

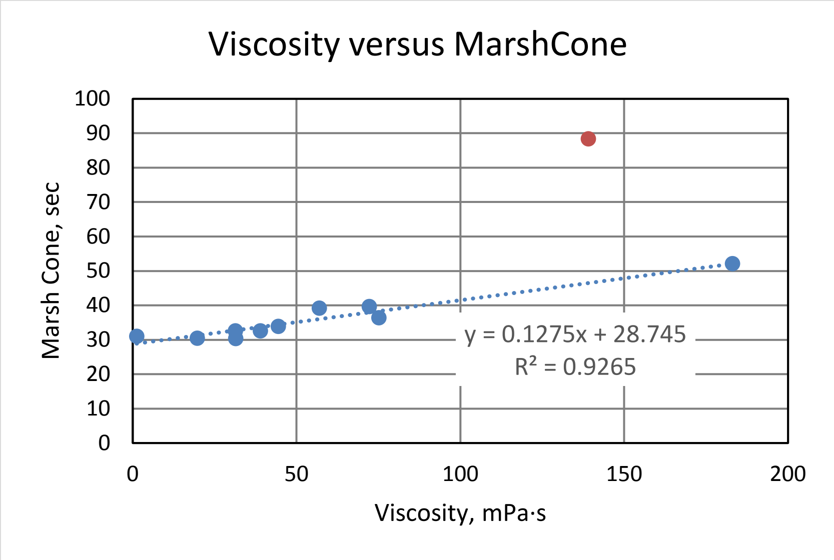

Figure 7 **Viscosity after 15 min versus Marsh Cone values. The red spot is w/c = 0.6 for cement B (w/c = 0.6 for cement C did not give any Marsh Cone result)**

The viscosity was determined after 15, 30, 45 and 60 min. Cement B and C showed very stable results during the whole period, while cement A also showed constant values until set occurred at about half an hour. The development for Cement A and B is given in Figure 8 and 9. The viscosity development for cement C (Figure 10) is more or less identical to the development for Cement B (the viscosity values are on a higher level for Cement C than for Cement B as shown in Table 6).

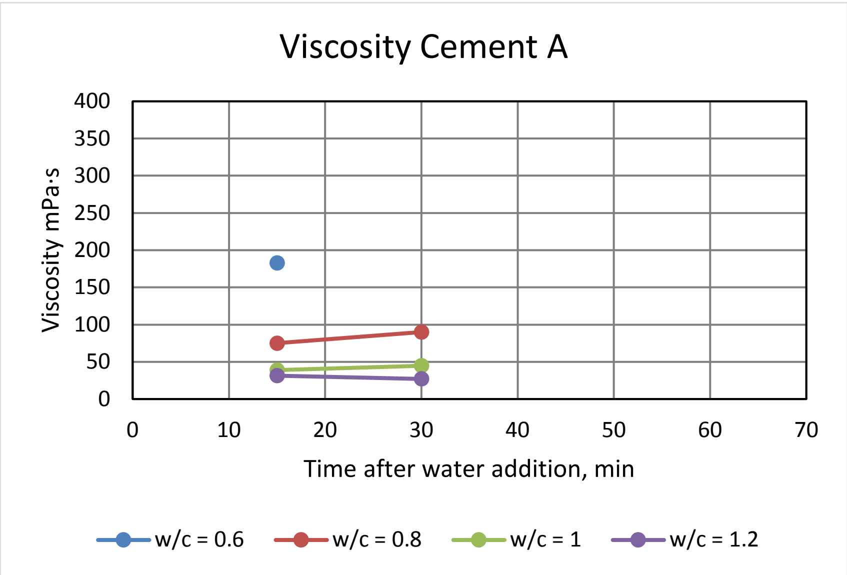

Figure 8 **Viscosity development for Cement A**

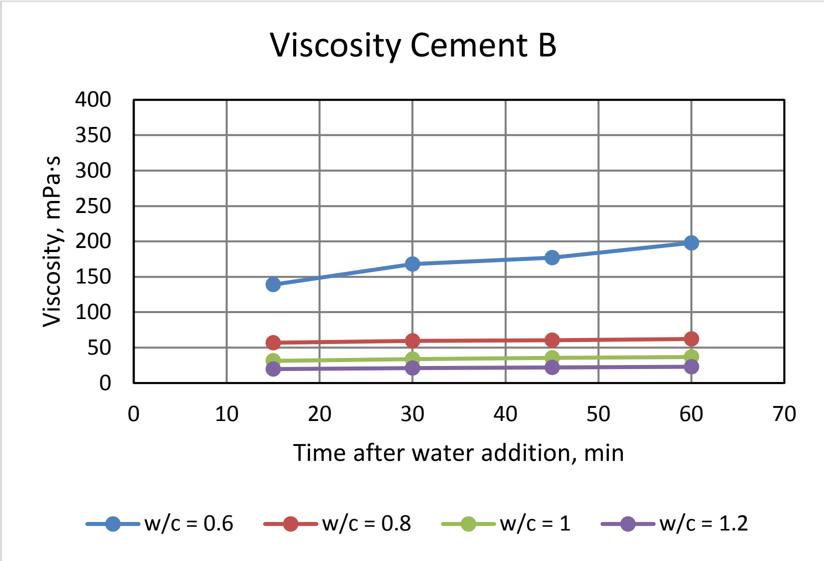

Figure 9 Viscosity development for Cement B

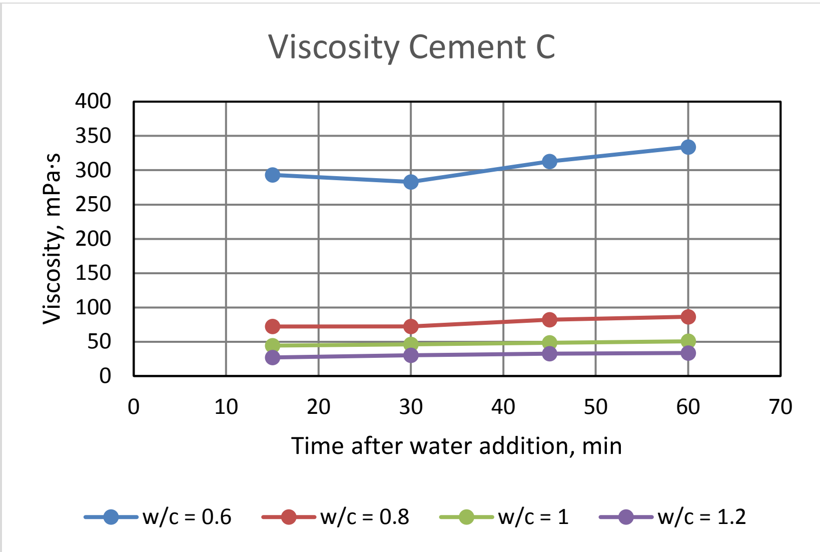

Figure 10 Viscosity development for Cement C

The initial yield stress values are shown in Table 7 and Figure 11. The yield stress results show a similar development over the first hour as the viscosity values, see Figure 12-14.

Table 7 Initial yield stress

	Yield stress 15 min after water addition, τ_{dyn}, Pa			
w/c	0.6	0.8	1.0	1.2
Cement A	183	75.1	38.9	31.4
Cement B	139	56.9	31.3	19.7
Cement C	293	72.2	44.4	27.1

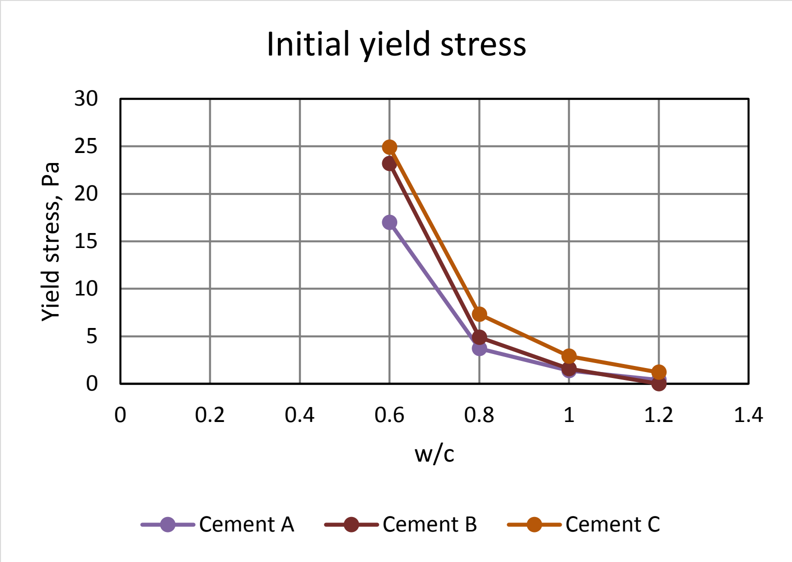

Figure 11 Initial yield stress

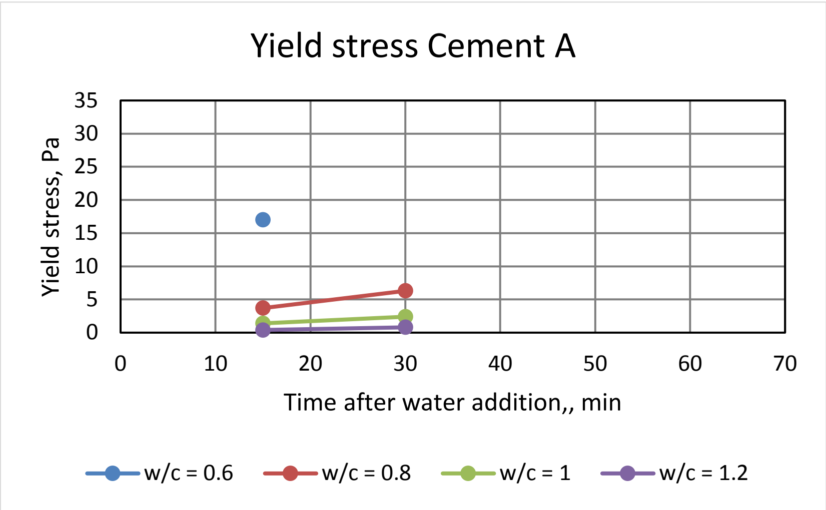

Figure 12 Yield stress over time for Cement A

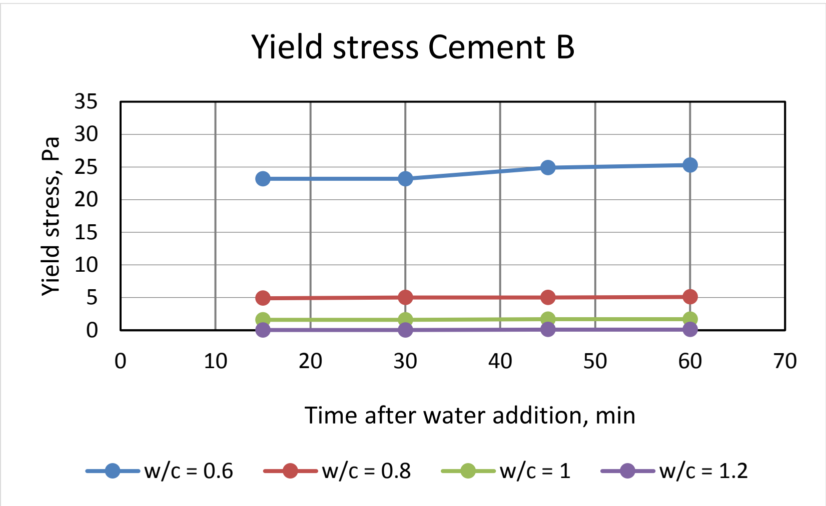

Figure 13 Yield stress over time for Cement B

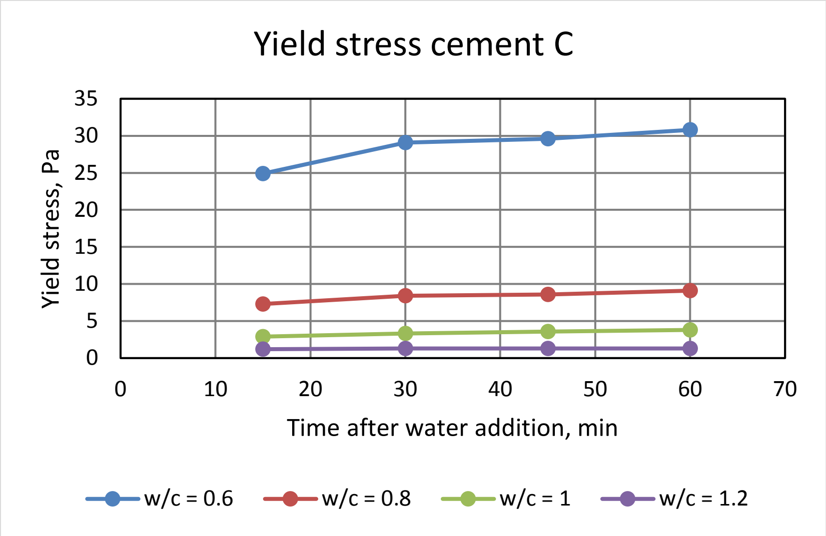

Figure 14 Yield stress over time for Cement C

CONCLUSION

Seven cements relevant for rock injection were tested for fineness according to three different methods. A good correlation was found between fineness determined according to Blaine and BET, while the correlation between D_{95} from the particle size distribution analysis and fineness is poor.

Three commonly used cements were selected for testing in cement paste. The paste flowability was determined according to three different method. A good correlation was found between Marsh Cone and spread according to EN445 as well as between Marsh Cone and viscosity determined by rheometer. All cements gave poor flowability at w/c = 0.6.

Initial set was determined by standard Vicat test at 20°C and temperature rise in insulated cup at both 20 and 8°C. Vicat is not well suited for testing at much higher w/c than 0.5 as the cement paste sets without development of sufficient strength to withstand the needle from penetration. Determination of temperature development at low temperature was also difficult at high w/c as the heat development is too slow compared to the temperature loss from the cup. Cement labelled A sets within 30-45 minutes, also at low temperatures. Cement B and C set within 3 till 6 hours at 20°C, and the set time is about doubled at low temperatures.

Use of w/c 1.2 gave unsatisfactory bleeding for cement B and C while cement A gave only insignificant bleeding at all w/c tested.

Initial viscosity and yield stress is at about the same level for all cements tested. The development of these parameters the first 60 minutes after water addition is also similar for all three cements disregarded that set occurred at about ½ hour for cement A and that the use of w/c 0.6 gave flowability problems for all three cements.

The tests performed do not give any information about the suitability for rock grouting and grout penetration into small cracks. The results should therefore be evaluated together with additional tests on penetrability properties e.g by filter pump (fluid loss). Such tests are planned to be performed at NGI as part of the TIGHT project.

REFERENCES

1. Skjølsvold, O., Justnes, H. "TIGHT – Prøving av injiseringssementer" SINTEF Report No SBF 2015 A0413, Trondheim, 2016