Nordic Concrete Research

Proceedings of the XXIII Nordic Concrete Research Symposium

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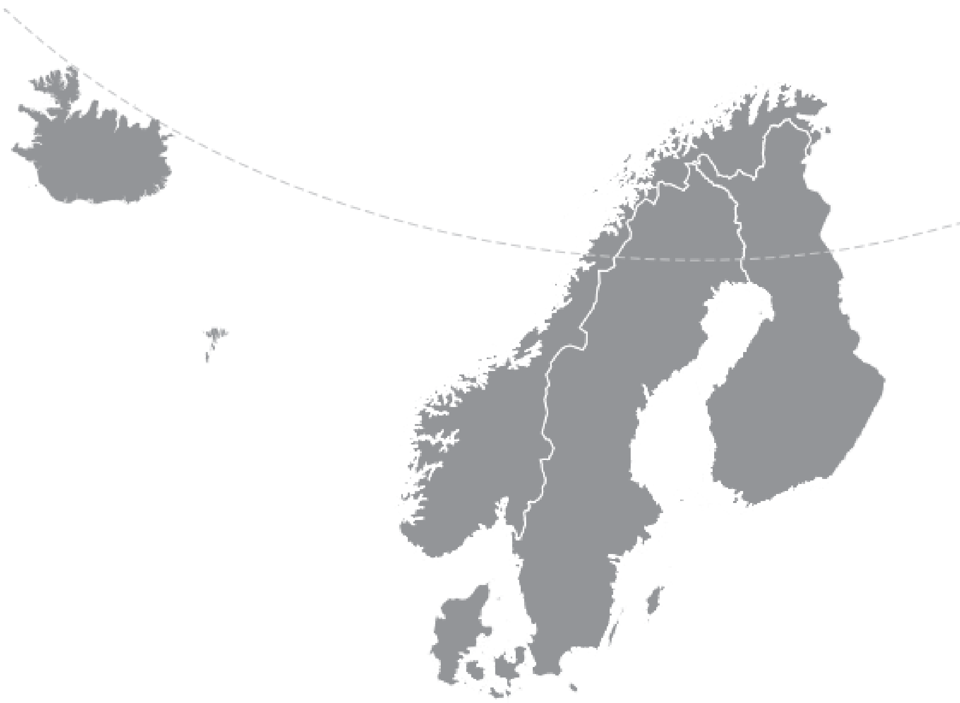

NCF Nordic Concrete Federation

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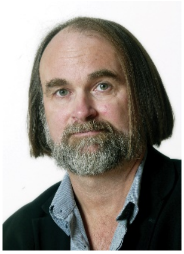
Characterization of cements for injection



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ABSTRACT
The three most common cements for rock injection in Norway were characterized in terms of grout flow properties, stability and initial set for w/c = 1.2, 1.0, 0.8 and 0.6 at 8 and 20°C. The fineness was characterized by Blaine, BET and particle size distribution (PSD). The test methods were bleeding, consistency (ring and Marsh Cone), setting by Vicat and temperature evolution in insulated cup. Additional rheology tests with parallel plate rheometer and isothermal calorimetry for hydration evolution were performed for mixes at 20°C only. Only one of the three cements tested could be utilized at all w/c levels.

Key words: Cement, Hydration, Injection, Rheology, Setting

1. 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. Limited data for the 3 selected cements on hydration and rheology are presented here, while additional data is published earlier by Skjølsvold and Justnes [2].

2. EXPERIMENTAL

2.1 Cement characterization

All cements were initially tested for 1) Density by Accupyc 1330 helium pycnometer, 2) Particle size distribution by Colter LS Particle Size Analyzer, 3) Blaine fineness (EN 196-6) and 4) BET fineness by nitrogen absorption . The test results are given in Table 1 and the particle size distributions are given in Figure 1.

Table 1 - Test results, characterization of cements

Cement	A	B	C
Density, g/cm^3	3.17	3.16	3.10
Blaine, cm^2/g	729	541	706
BET, m^2/g	1.88	1.58	1.93
D_{95}, µm	17	18	25

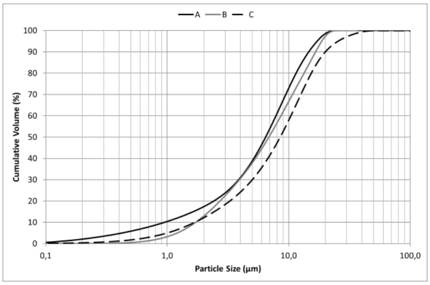

Figure 1 – The particle size distributions of the cements

2.2 Injection cement performance

The 3 cements were blended in a Waring blender with water to achieve 4 different w/c = 1.2, 1.0, 0.8 and 0.6. The resulting pastes were characterized in terms of rheology, setting and heat of hydration. 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. Bingham viscosity and dynamic yield point was also tested by a Physica MCR 300 parallel plate rheometer. Paste was placed between two parallel serrated plates in the rheometer where temperature is controlled to 20°C by a Peltier element. The speed was varied stepwise from 2 till 150 s^{-1} and back to 2 s^{-1} again. The Bingham viscosity (µ in mPa·s) and yield point (τ_{dyn} in Pa) was calculated from the ≈ linear part of the descending curve.

Setting time was determined at 20^{0}C by Vicat apparatus according to EN196-3 and by temperature evolution in insulated cups (i.e. semi-adiabatic) at 8 and 20°C taking a 2°C increase above base line as criterion for initial set. The hydration development was followed by a TAM Air isothermal calorimeter set at 20°C.

3. RESULTS

The initial consistency data from Marsh funnel and spread are given in Table 2, while Bingham viscosity and yield point are listed in Table 3. The setting times from Vicat needle tests and cups are reported in Table 4. The hydration heat development curves are depicted in Figures 2 and 3.

Table 2 - Initial consistency at 8 and 20 °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

Table 3 - Initial Bingham viscosity and yield stress 15 minutes after water addition at 20℃

w/c	1.2	1.0	0.8	0.6	1.2	1.0	0.8	0.6
	Viscosity, μ, mPa·s				Yield stress, τ_{dyn}, Pa			
Cement A	31.4	38.9	75.1	183	0.4	1.4	3.7	17.0
Cement B	19.7	31.3	56.9	139	0.0	1.6	4.9	23.2
Cement C	27.1	44.4	72.2	293	1.2	2.9	7.3	24.9

Table 4 - Initial set results by Vicat needle at 8 and 20℃

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	20^0C											
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 to 2°C T-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	8^0C											
Time to 2°C T-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 occurred quickly, but was not recorded as the paste did not withstand the needle
** 2^0C temperature rise was not achieved

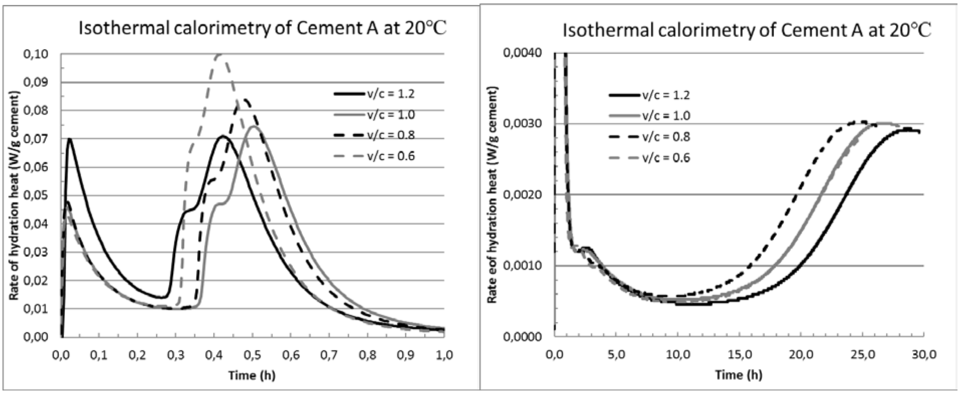

Figure 2 – Rate of hydration heat for cement A as a function of w/c (left; unusual early peak).

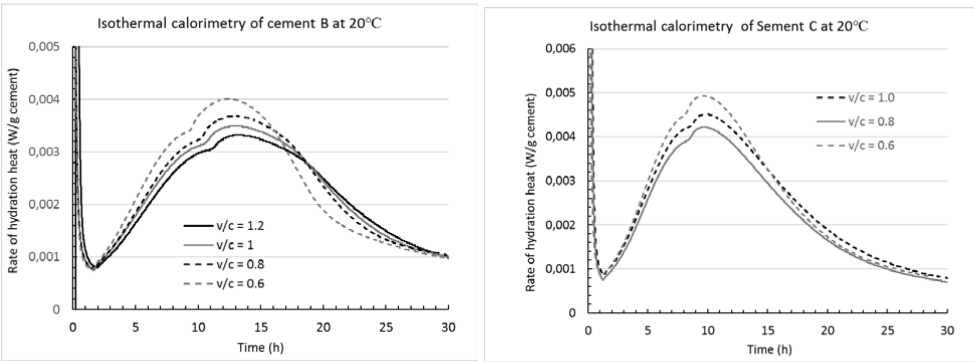

Figure 3 – Rate of hydration heat for cement B (left) and cement C (right)

4. DISCUSSION

All cements had similar initial spread and Marsh funnel flow, which correlated well with the viscosity measured by the rheometer as shown in Figure 4. All cements gave poor flow at the lowest w/c and unsatisfactory bleeding at the highest w/c (exception for cement A). The reason for reduced bleeding for Cement A can be understood from the early hydration development curve in Figure 2 showing an unusual early hydration heat between 0.25 and 0.75 h that would lead to a premature weak set before the second major hydration start at around 11 h at 20°C. This is also reflected in the very early setting by vicat and semi-adiabatic cup for this cement. The heat of hydration curves of cements B and C in Figure 3 looks like what one would expect from a fine portland cement and their difference reflects the difference observed for the setting times. 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.

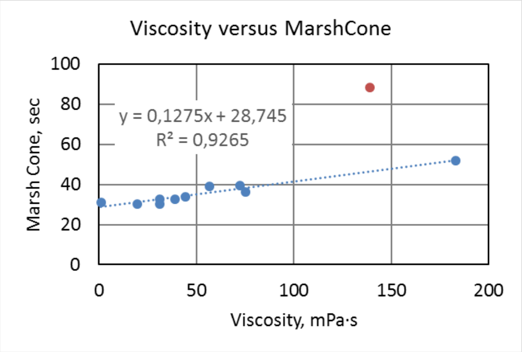

Figure 4 - Viscosity after 15 min versus Marsh Cone values. The open circle is w/c = 0.6 for cement B (w/c = 0.6 for cement C did not give any Marsh Cone result)

5. CONCLUSION

Only cement A among the three tested cements were suitable at the whole w/c range in terms of rheological properties, but had a very early setting and unusual hydration heat evolution.
The setting time roughly triples when going from 20 to 8°C (typical rock temperature), which is worth noting for tunnel injections. The tests performed do not indicate the suitability for grout penetration into small cracks and therefore additional tests on penetrability properties (e.g. fluid loss by filter pump) are performed by other project partners and will be published elsewhere.

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