Tunnelling and Underground Space Technology 91 (2019) 103011

Contents lists available at ScienceDirect

Tunnelling and Underground Space Technology

journal homepage: www.elsevier.com/locate/tust

Cements for tunnel grouting – Rheology and flow properties tested at different temperatures

Bahman Bohloli[a,*], Ola Skjølsvold[b], Harald Justnes[b], Roger Olsson[a,c], Eivind Grøv[b], Arnstein Aarset[a]

[a] Norwegian Geotechnical Institute, Sognsveien 72, 0855 Oslo, Norway
[b] SINTEF Building and Infrastructure, Rich. Birkelands vei 3, 7465 Trondheim, Norway
[c] Norwegian University of Science and Technology, Department of Geoscience and Petroleum, Sem Sælands vei 1, 7491 Trondheim, Norway

ARTICLE INFO

Keywords:
Bleeding
Cement grout
Hydration temperature
Rheology
Setting
Tunneling
Viscosity

ABSTRACT

This paper presents work being carried out in Work Package 3 of TIGHT (True Improvement in Grouting High pressure Technology for tunnelling) project. The objective is to investigate flow and mechanical properties of three cements (A, B and C) at actual tunnel- and room –temperatures using various laboratory methods. The cements were first characterized in terms of grain size distribution and specific surface area. Then the grouts made from the three cements were tested for flow properties and mechanical strength. Cement grouts were prepared at two different temperatures of 8 °C and 20 °C to represent the actual tunnelling temperature of projects in Scandinavia and the room temperature, respectively. The experimental program comprised of a total of 590 tests, including the tests presented in this paper and elsewhere. The experiments include grain size distribution, specific surface area, viscosity, bleeding, hydration temperature, setting time and strength of cured grout specimens. Four different water to cement (w/c) ratios of 0.6, 0.8, 1.0 and 1.2 were used for most of the tests. Results showed that the grouts prepared from the three cements had quite different behaviour in terms of rheology, flow and mechanical properties. Viscosity of various types of cement grouts is very different at low w/c ratios but the difference decreases with increasing w/c ratio. All three cements fulfil the requirements described by ASTM for bleeding at w/c ratios up to 1.0, but only cement A qualifies for w/c ratios of greater than 1.0. Also cement A sets much faster than cement B and C, as proved by the Vicat needle test and heat of hydration. Temperature evolvement and heat of hydration, during initial stages of setting, is both higher and faster for cement A than cements B and C. Results show that there is a negative correlation between the heat of hydration and the uniaxial compressive strength of cement grout specimens. It is also illustrated that fast temperature increase in a cement grout leads to a lower strength of the cured grout specimen.

1. Introduction

Grouting of rock mass is usually carried out to reduce water inflow to tunnels and underground excavations. Cement based grouts are the most commonly used material for rock grouting. Different types of cements; from ordinary Portland cement to very fine cements are used for tunnel grouting to seal fractures of different apertures. Cement grouts include Portland cement, aluminate cement or ground granulated blast-furnace slag called slag cement, among others (Dalmalm, 2004). To obtain a durable and high strength hardened cement grout, the grout should be stable in terms of bleeding and sedimentation. Excess bleeding and sedimentation may cause incomplete filling of cracks which in turn create seepage paths through a grouted crack (Eklund, 2003).

Characteristics of cement grouts appropriate for tunnel grouting, selection of grouting parameters and various grouting methods are described elsewhere (NFF, 2011; Tolppanen and Syrjänen, 2003; Dalmalm, 2004; ISRM, 1996; Byle and Borden, 1995). Properties of cement grouts such as rheology and flow behaviour are controlled by the grain size, water cement ratio (w/c), cement condition and the mixing equipment (Eriksson et al., 2004). In addition, curing temperature has remarkable influence on strength properties of cement grout specimens (Elkhadiri et al., 2009). Selection of appropriate grouting material is an important step for the success of grouting.

Several researchers have tested mechanical and flow properties of cement grouts in laboratory (e.g. Dalmalm, 2004; Eklund, 2005; Ortiz,

* Corresponding author.
E-mail address: bahman.bohloli@ngi.no (B. Bohloli).

https://doi.org/10.1016/j.tust.2019.103011
Received 1 November 2018; Received in revised form 17 March 2019; Accepted 13 June 2019
0886-7798/ © 2019 Published by Elsevier Ltd.

Table 1
Grain size and specific surface area of several cements commonly used in the Nordic countries for grouting (Tolppanen and Syrjänen, 2003).

Cement type*	Maximum grain size (μm)	Specific surface area, Blaine (m^2/kg)
Cementa Anlägningscement	d_{95} = 120, d_{100} = 128	300–400
Cementa Injekteringscement 64	d_{95} = 64, d_{100} = 128	600
Cementa Injekteringscement 30	d_{95} = 30, d_{100} = 32	1300 (BET)
MBT Rheocem 650	d_{95} = 16, d_{98} = 20	650
Cementa Ultrafin cement 16	d_{95} = 16, d_{100} = 32	800–1200
Orginy Spinor A16	d_{98} = 16	1200
Dykerhoff Mikrodur P-F	d_{95} = 16	1200
MBT Rheocem 800	d_{95} = 13, d_{100} = 20	820
Cementa Ultrafin cement 12	d_{95} = 12, d_{100} = 16	2200 (BET)
MBT Rheocem 900	d_{95} = 8, d_{98} = 10	875–950
Orginy Spinor A12	d_{98} = 12	1500
Dykerhoff Mikrodur P-U	d_{95} = 9.5	1600
Dykerhoff Mikrodur P-X	d_{95} = 6	1900

* Data in this table are from the manufacturers. The products from Cementa and MBT (Masterbuilders) are Portland cements, Orginy spinor is a slag cement and Dykerhoff contains both slag and Portland based cement.

2015). The focus of those studies was mainly on the characterization of cement grouts at standard room temperature. True tunnelling environment, particularly in the Nordic countries, has a temperature of about 8 °C to 10 °C. This paper focuses on the characterisation of cement grouts prepared and tested at both 8 °C and 20 °C. It presents results of several types of tests including grain size distribution, specific surface area, bleeding, rheology, setting time and hydration temperature. An effort has also been made to couple hydration temperature with the uniaxial compressive strength (UCS) of cured grout specimens. Strength and permeability of cured cement grout specimens along with the filtration stability of grouts at different w/c ratios have already been presented in Bohloli et al. (2018).

2. Background

Behaviour of cement grouts is controlled by key parameters including grain size distribution, specific surface area, w/c ratio, stability against grain separation and viscosity. Grain size of several cements commonly used for grouting in the Nordic countries is provided in Table 1. The maximum grain size of different cements varies over a wide range; from 6 μm to 120 μm.

Grain size distribution for some of the commonly used cements in Norway is provided in Fig. 1. These include a wide range of cements from ordinary Portland cement (OPC), microfine cements (MFC) and ultrafine cements (UFC) are used for grouting purpose. However, the

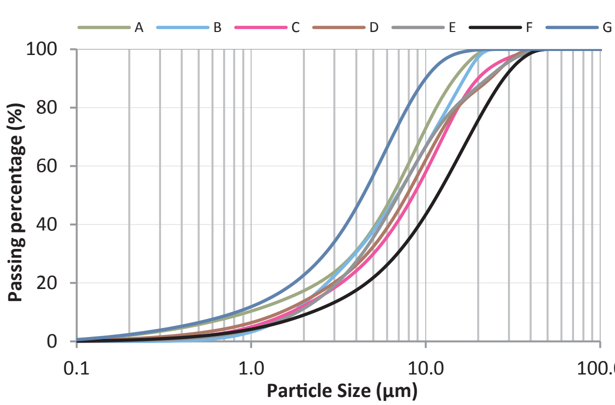


Fig. 1. Grain size distribution of cements commonly used in Norway for tunnel grouting (After Skjølsvold and Justnes, 2016).

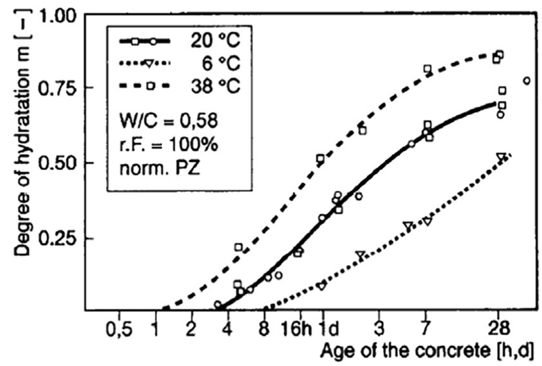


Fig. 2. Influence of temperature on degree of hydration (modified after ISRM, 1996).

use of microcements, together with high grouting pressure, shows an increasing trend in the recent years. This is due to the very satisfactory results that have been achieved by such an approach (Tolppanen and Syrjänen, 2003). In the Norwegian Public Road Administration's Code of Process 025, standard grouting cement is defined as cements with a particle size of about 20 μm < d_{95} less than 40 μm.

Cements are normally tested in room conditions by the manufacturers. Therefore, grout properties reported in product catalogues may appear different in tunnel conditions. Several authors have shown that grout material exhibit other characteristics when tested at settings different from room conditions (Håkansson, 1993). The degree of hydration of cement in suspensions, which determines the hardening, is known to depend largely on temperature (ISRM 1996). Fig. 2 shows the degree of hydration for grouts at different temperatures. It indicates a paste at 6 °C requires a longer time to set compared to others at 20 °C and 38 °C, since as a rule of thumb, most chemical reaction double the setting rate for every 10 °C increase. Furthermore, cement pastes cured at lower temperature (e.g. 4 °C) have a lower strength than those cured at higher temperature (e.g. at 22 °C and 40 °C) at a given time (Elkhadiri et al., 2009). Mirza et al. (2013) also reported that temperature variation has a significant effect on setting time of cement grouts and generally, a decrease in temperature results in a longer setting time for all types of cement grouts. Holt and Leivo (2004) in a study on concrete samples, showed that curing at low temperature may seriously hinder the rate of strength development in the early-age. For cement grouts used for tunnel grouting, early development of grout strength is important. A low strength grout may not function well when next tunnel face is excavated.

Uniaxial compressive strength of grout samples made of an ultrafine Type V cement versus w/c ratio is presented in Fig. 3-left (Ortiz, 2015). Compressive strength of the samples drops from about 15 MPa to 0.07 MPa when w/c ratio is increased from 1 to 2.5. Similarly, the uniaxial compressive strength of the three cements in this study; A, B and C drops from about 17 MPa to 5 MPa when w/c ratio increases from 0.6 to 1.2 (Fig. 3-right). The explanation is that excess water that is not bound chemically by hydration (i.e. water in excess of w/c ≈ 0.4) will lead to increased porosity, and the strength decreases with increasing porosity.

3. Experimental method

Three different types of cements (named A, B and C) were used in this study (Fig. 1). For preparation of grout samples, a 4-liter Waring high-speed mixer at 2000 rounds per minute (rpm) was used. Firstly, the prescribed amount of water was poured into the mixer, it was set on and the amount of cement was poured into the mixer during 30 s while mixing. Thereafter, mixing continued for two more minutes. For making grouts of 8 °C or 20 °C, the temperature of water and cement was chosen such that the ready mix had a temperature of about 8 °C or

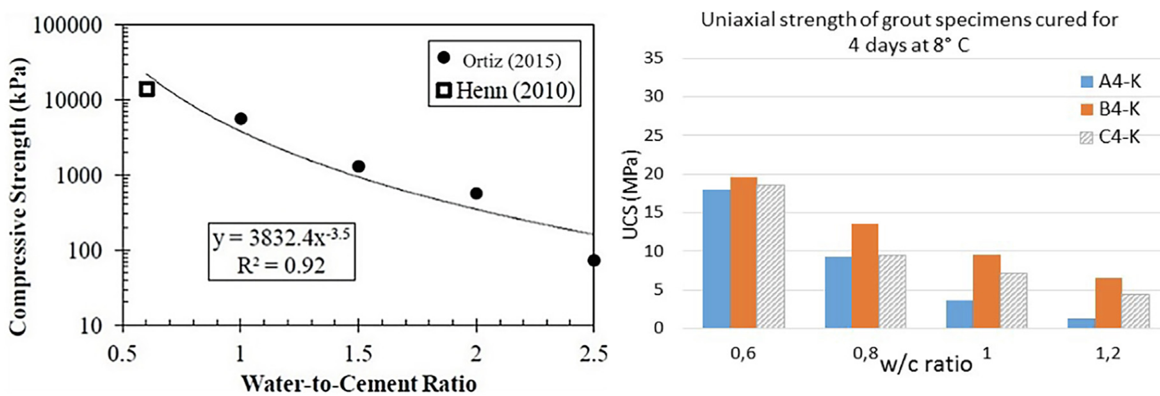


Fig. 3. Left: relationship between uniaxial compressive strength (UCS) and w/c ratio for Ultrafine Type V grout (modified after Ortiz, 2015). Right: uniaxial compressive strength of cured samples of the three cement; A, B and C, used in this study (after Bohloli et al., 2018).

20 °C. Different water-cement (w/c) ratios were used to explore its impact on mechanical and flow properties of mixtures. The fresh grouts were tested after mixing.

Grain size and specific surface area of cement powders were determined using d_{95}, Blaine and BET methods. Details on the Blaine and BET measurement methods can be found in ASTM C204 (ASTM, 2017) and Maryland and Azari (2013). The grain size of cement in liquid state (grout) was also measured using the Stoke's law and Fall drop method (for details see Moum, 1965). Viscosity of cement grouts was measured using Marsh cone, spread ring and parallel plate viscometer. Yield stress of the cement grouts was also determined by the Physica parallel plate viscometer. Experimental method for a few parameters of focus in this study are described in the following.

3.1. Stability of cement grouts

Stability of a cement grout is defined as the resistance against separation of water (filtration) from cement particles. Filtration occurs when particles of water and cement are separated from the grout such that cement particles accumulate and water penetrates pore spaces. The accumulated particles prevent further penetration of grout, which is required to seal-off pores and fractures. There are two established laboratory methods for determining stability of cement grouts; i) bleeding and ii) filtration stability. Bleeding is described in next section. Filtration stability for cement grouts A, B and C was measured and reported in Bohloli et al. (2018).

3.1.1. Bleeding

Bleeding, also called water separation, is the autogenous flow of mixed water into, or water that flows out of, fresh grouting material. The process of bleeding, in which cement grains separate from grout mix and clog the aperture of fine joints, does not occur with a stable grout mix (Holter and Hognestad, 2012). A comprehensive description of bleeding and parameters affecting bleeding of cement grouts can be found in Draganovic (2009). A maximum limit of about 2% to 5% bleeding after 2 h is recommended for tunnel grouting applications (Dalmalm 2004; NFF, 2011). In this study, a graduated cylinder was used to measure bleeding of the grouts after 60, 90 and 120 min.

3.2. Setting of grout (Vicat needle test)

Vicat Apparatus is a device used to determine the setting time of cement pastes. Vicat apparatus was originally a manual device that consisted of a metallic frame, graduated scale with index and a sliding probe of 300 g. For initial setting time, a needle of 1.13 mm is attached to the sliding probe. At fixed intervals, the 300 g probe and needle are dropped into the paste. The time when the needle stops 6 mm from the base plate is recorded as the time for initial setting. Final setting is defined as the time when the needle only makes a 0.5 mm mark on the surface. The Vicat apparatus is designed for cement paste of normal consistency (w/c ratio ≈ 0.3), and is therefore not so well suited for high w/c ratios. A new type of Vicat device, an automatic variant (ToniSet) that does eight parallel tests, was employed in this study.

Setting of cement and its rate affects the development of paste strength over time. In a cement grout, several grains are dispersed in water. During hydration a soft gel is formed around a hard core of unhydrated cement grain. As long as the hydration products of different cement grains do not interact, the grout has no strength. When hydration products around grains interfere (initial setting) an internal structure forms and the strength of cement grout develops (De Schutter, 2002). During hydration the pores are progressively filled with hydration products and yield a stronger and stiffer structure (final setting) (Fig. 4). Cement slurry has the properties of a fluid immediately after addition of water, but it changes properties towards more plastic and finally to solid as hydration and setting processes progress (Fig. 4-left).

3.3. Hydration heat

When water is added to cement an exothermic reaction occurs that produces heat. The rate of heat generation is higher in the initial stages of setting and reduces gradually. In mass concrete structures, temperature gradient is generated between the core and the surface. Stresses induced by temperature change during hydration process depends also on the geometry and mechanical boundary conditions of the structure (Nagy and Thelandersson, 1994). In tunnel grouting, the mass of injected grout is not large thus it differs from the boundary conditions applied to mass concrete structures. However, a quick release of heat may affect mechanical and hydraulic properties of the cured grout within the rock mass.

Hydration heat is usually measured with a calorimeter. Here, the hydration heat was measured by a Thermometric TAM Air eight channel calorimeter, where small samples (approximately 10 g) were put into one of the device isolated cells, and maintained at constant temperature of 20 °C. The temperature in the sample was measured continuously, and the evolved heat, q, was calculated as:

$$q = C.\,\Delta t \tag{1}$$

where C is heat capacity of the device, and t is time.

A typical heat evolution curve is shown in Fig. 4-right. Initially, a heat evolution is observed (marked *initial reaction* in the figure) because of dissolution of ions and initial hydration. After that there is usually a dormant or *induction period* with low heat evolution and slow dissolution of silicates before the heat evolution starts due to silicate hydration (*acceleratory period*) followed by a slow down of heat generation (*deceleratory period*).

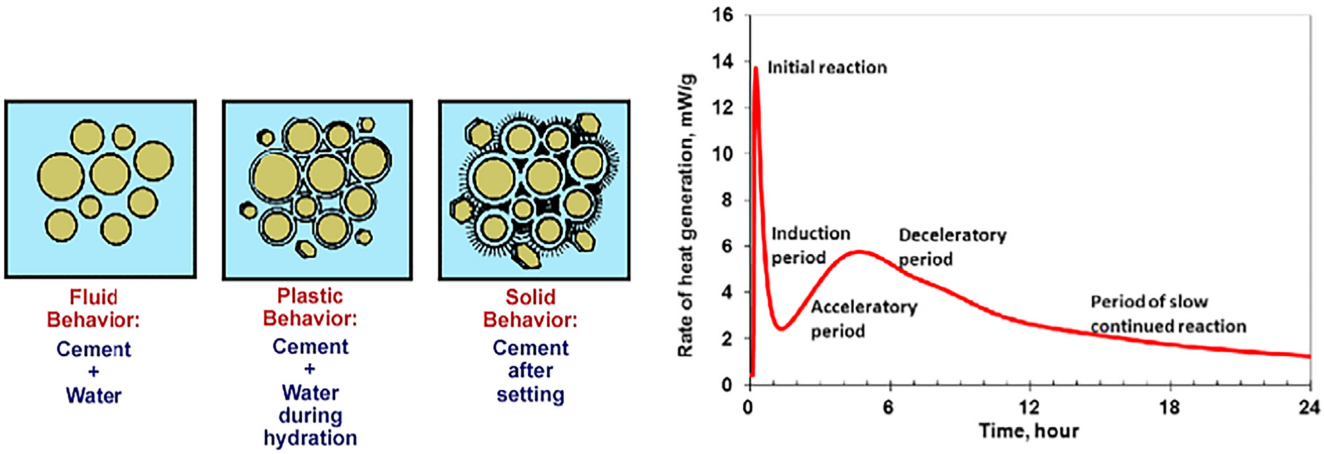


Fig. 4. Left: properties of cement grout during different stages of development (after Nguyen, 2012). Right: a typical heat evolution rate pattern for a cement grout (Hu et al., 2014).

4. Experimental results

4.1. Grain size distribution

The grain size distribution or fineness of cements was measured using Blaine Fineness. Grain size distribution and specific surface area for cements A, B and C are presented in Table 2 and Fig. 5.

4.1.1. Grain size distribution of cement in the grout (liquid state)

Size of cement particles when mixed with water (and additives) is more important for penetration into pores and fractures than the size of cement grain itself. Therefore, the size of cement particles in grouts prepared from all three cements was measured using a falling drop device (Fig. 5b).

Results of the falling drop shows that the size of particles in liquid state is between 20 and 70 µm. Grain size of the cement particles is between 0 and 40 µm. This shows that cement grains bind together in the grout and make larger particles, i.e. flocculation of grains occurs. In addition, there may be a surface hydration of the particles prior to setting that will increase the individual particle size and may reduce the effective w/c from the nominal.

It should be noted that all grout samples tested and reported in this paper were prepared from cement and water only and no additives were added. Impact of different additives and silica on flow behavior of grouts is an ongoing test program that will be reported elsewhere.

4.2. Specific surface area

Specific surface area for cement powders was determined using two methods; Blaine and BET (Fig. 6). Note that Fig. 6 presents properties of the seven cements, A-G, shown in Fig. 1. There is a strong correlation between the values of specific surface area measured with BET and Blaine. However, there is no clear correlation between the BET and d_{95}; specific surface area for cements with a d_{95} of about 15 µm is almost the same as that for cements with d_{95} of 30 µm. This implies that d_{95} may not provide comprehensive information about the specific surface area of a material and hence its flow behavior.

Table 2
Physical properties of cements A, B and C.

Cement	Density (g/cm^3)	Blaine fineness (m^2/kg)	Specific surface area, BET (m^2/kg)	d_{95} (µm)
Cement A	3.17	729	1880	17
Cement B	3.16	541	1580	18
Cement C	3.10	706	1930	25

4.3. Setting time

Setting time was estimated through measuring significant temperature rise in a 250 ml insulated styrofoam cup for grouts of 8 °C and 20 °C (Fig. 7a). The "significant temperature rise", shown in Fig. 7a, is an indication of cement setting and is roughly about 2 °C for low w/c ratios, but varies for high w/c ratios. Increase of temperature in a grout indicates initiation of hydration process which implies start of cement setting. This index (significant temperature increase) shows a trend that is almost independent of w/c ratio for grouts tested at 20 °C, full symbols in Fig. 7a. The time required for a major temperature rise for grouts with w/c ratio of 0.6 is almost the same as that for w/c ratio of 1.0. Cement A shows the shortest and cement B shows the longest time to reach such a temperature increase. For grout A, there is a very small difference between those at 8 °C and 20 °C implying that setting time of cement A will likely be about the same in standard room and in tunnel conditions. For cements B and C, there is a large difference between the time for significant temperature increase for grouts of 8 °C versus 20 °C. It indicates that cement grouts B and C need much longer time to set in temperatures close to tunnel conditions. This may be an important aspect for selection of cement type when early setting and strength of the grouted mass is essential.

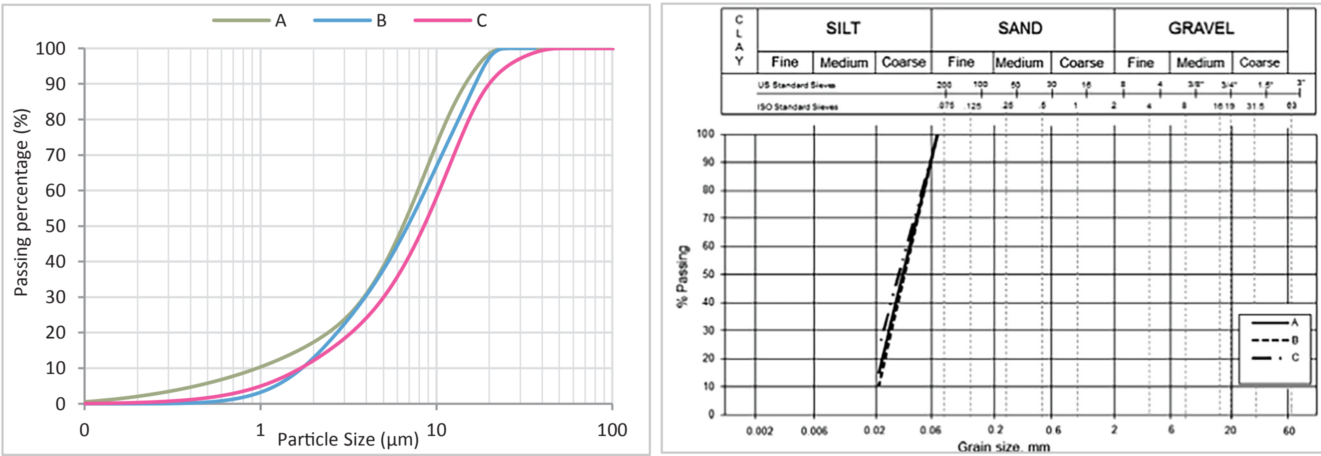
Setting time was also determined for grouts at 20 °C by Vicat device according to EN196-3 (Fig. 7b). A general observation from the results of Vicat needle is that setting time increases with increasing w/c ratio for all cement grouts at 20 °C, although the increasing trend is different. Cement A sets very quickly while cements B and C need several hours before the start of setting.

The setting time obtained from both methods (Vicat needle and significant temperature increase) agrees quite well for cement A. However, it provides very different setting times for cements B and C. The index of significant temperature rise gives a shorter time value than Vicat. Both methods are not very accurate, especially at high w/c ratios. It should be noted that the "significant temperature rise" or "2 °C temperature rise" criterion is based on the experience from Portland cement type concrete in insulated boxes, and cannot be transferred to other cement types like slag cement and aluminate cement.

4.4. Grout viscosity (consistency)

Viscosity of the three cements was measured at both 8 °C and 20 °C. Four mixes with w/c ratios of 0.6, 0.8, 1.0 and 1.2 were made from each cement type. Results of the viscosity measurements are presented in Fig. 8.

The grouts were tested immediately after mixing. Results show that initial viscosity decreases with increasing w/c ratio, as expected.



(a) (b)

Fig. 5. Left: grain size distribution of cements A, B and C in dry condition (a), and the size of particles in a grout prepared from the same cements and measured with a falling drop (b).

Viscosity of the grouts ranges from 20 to about 300 mPa s depending on the type of cement and w/c ratio (Fig. 8a). It decreases sharply with increasing w/c ratio from 0.6 to 0.8, but at a lower rate thereafter. Viscosity difference between the cements is more obvious at lower w/c ratios but is very small at higher w/c ratios. Grout of cement C shows the highest viscosity at w/c ratio of 0.6 followed by cement A and B. Cement B has the lowest viscosity, measured with rheometer, at all w/c ratios compared to cements A and C, despite that cement B has slightly coarser grain size and lower specific surface area (Fig. 5 and Table 2).

Marsh cone time shows cement A has the lowest and cement C has the highest viscosity at 20 °C and w/c ratio of 0.6 (full symbols in Fig. 8b). Note that cement C at 20 °C and w/c ratio of 0.6 was so viscous that it could not be measured with Marsh cone; only 480 ml of the grout passed through the cone in 90 s. It has a positive correlation with viscosity; the higher the viscosity the greater the time for a specific volume to pass through the Marsh cone. Marsh cone time for colder and warmer grouts at other w/c ratios are quite close for all cements. Similar to viscosity from rheometer, Marsh cone time also shows a decreasing trend with increasing w/c ratio. For cement grouts B and C at 8 °C and 20 °C, it varies significantly for w/c ratio of 0.6. The difference is marginal for cement A at w/c = 0.6. Cement grouts with a lower temperature (8 °C) have lower viscosity at w/c ratio of 0.6, possibly because of lower extent of surface hydration at lower temperatures. This is a positive aspect for grouts in tunnel conditions; lower viscosity will lead to a better workability.

Result of the Spread Ring measurements show that the diameter of spread circle increases with increasing w/c ratio. The spread circle of grouts at 8 °C is generally larger than those of 20 °C (Fig. 8c). This means that colder grouts have lower viscosity. This agrees with the results of Marsh cone time presented in Fig. 8b that grouts of 8 °C had lower viscosity than those at 20 °C. Diameter of spread circle at w/c ratio of 1.2 is very close for all cements and for colder and warmer grouts.

Comparison of viscosity measurements from these three methods shows that the difference between viscosity of cements A, B and C is more pronounced at low w/c ratios but decreases with increasing w/c ratio. Considering viscosity, selection of the type of cement is more important when using low w/c ratios of about 0.6. Another observation is that different methods provide slightly different viscosity values. Rheometer shows the highest viscosity for cement grout C but the lowest for cement grout B at w/c ratio of 0.6 at 20 °C (Fig. 8a). Marsh cone time, on the other hand, shows the lowest viscosity for cement grout A, at the same conditions (Fig. 8b). At higher w/c ratios, the measured viscosities are quite close and difficult to differentiate. Therefore, determining viscosity with one method may be misleading. Using a combination of methods and carrying out several measurements for every grout type is recommended to provide a representative viscosity value. Since viscosity affects workability of grout and penetrability to rock fractures and voids, selection of the type of cement for low w/c ratios may be more important than that for higher w/c ratios. Furthermore, viscosity of cements B and C seems to be more sensitive to temperature than cement A when using Marsh cone (Fig. 8b). An overall conclusion based on the viscosity measurement is that cement A may be more favorable for grouting in true tunneling temperatures of about 8 °C than cements B and C.

4.5. Bleeding

Bleeding is the amount of water separated from grout after certain time and is expressed as the percentage of initial grout volume.

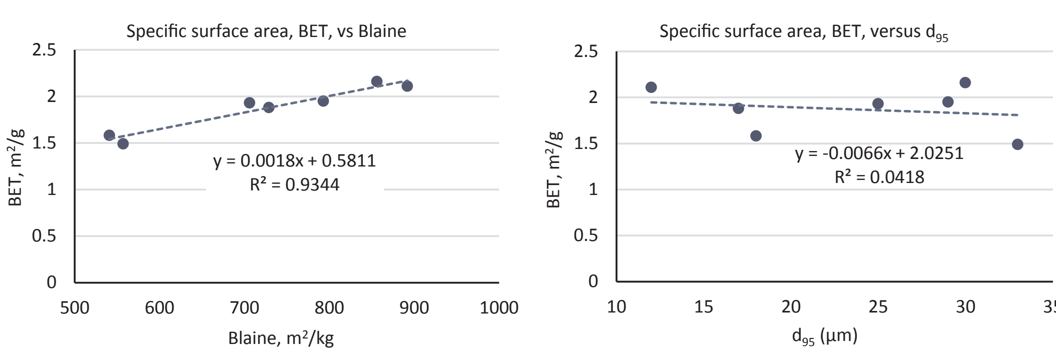


Fig. 6. Correlation between the specific surface area and grain size of cements A, B and C.

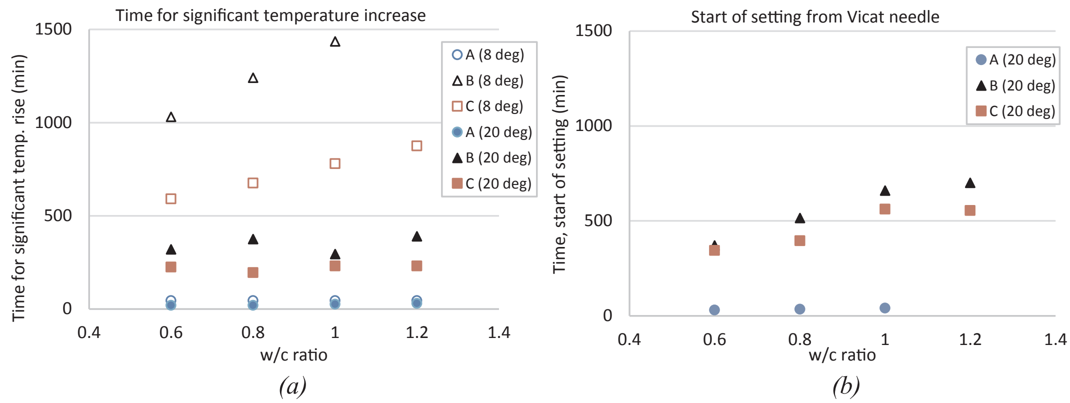


Fig. 7. Time (since mixing) for significance temperature increase measured in a styrofoam cup as an indication for cement setting (a) and start of setting time for different cements measured with Vicat apparatus (b).

Bleeding was measured for two samples from every type of cement grout. The volume of separated water was read after 30, 60, 90 and 120 min and 24 h (Fig. 9). Bleeding increases non-linearly with increasing w/c ratio for all cements, as expected. Cement A shows minimum bleeding and cement B shows maximum bleeding for all w/c ratios. Generally, bleeding for 8 °C grouts is lower than that for 20 °C mixes. This is a positive result since it implies that bleeding of grouts in tunnel conditions may be less than that measured in room temperature.

Cement grouts used for tunnel grouting are required to have bleeding less than about 5% (ISRM, 1996). All the cement grouts with w/c ratios of up to 1.0 may pass that criteria, except cement B which has a bleeding of 5.5% at 8 °C. At w/c ratio of 1.2, only cement A qualifies (Fig. 9b). Thus cement B and C need some additives to reduce their bleeding should they be used at w/c ratios of about 1.2 or higher.

4.6. Heat of hydration for grouts at 20 °C

Heat generation rate for all cement grouts was measured at 20 °C using calorimetry (Fig. 10). It is expressed in terms of W/g cement (heat of hydration per gram of cement). The first peak observed is due to dissolution ions and initial hydration. The rate of hydration heat depends on the percentage of cement in the grout and increases with decreasing w/c ratio. This is true for all three cements (Fig. 10). For cement A, this initial heat evolvement is surprisingly high, while for cement B and C the initial heat development is normal for Portland cements. Therefore, for cement A, a zoom-in of the first two hours of cement grout development is shown in Fig. 10a and a complete curve for 30 h is shown in Fig. 10b. The heat rate for cement A shows double peaks; the first peak appears about 30 min (Fig. 10a) and the second one occurs about 25–30 h after mixing (Fig. 10b). The first peak is much greater; about 25 times the second peak. In the first peak, there are

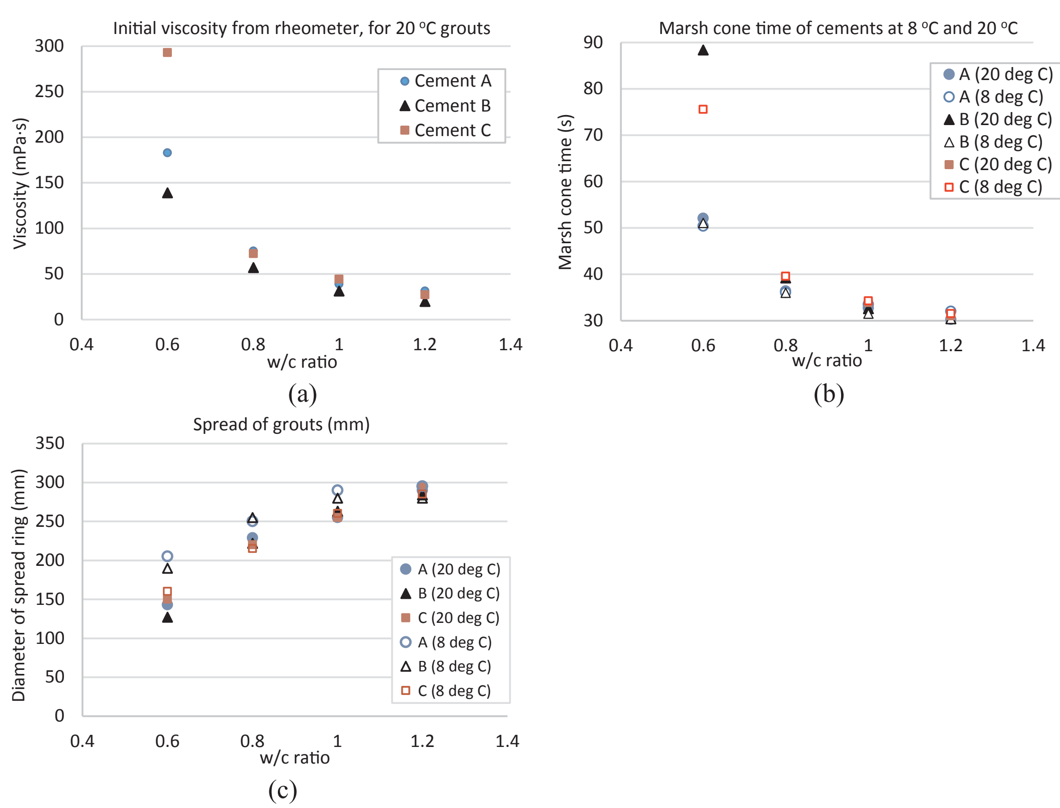


Fig. 8. Viscosity of cement grouts: (a) viscosity (in mPa s) versus w/c ratio at 20 °C measured with a rheometer, (b) Marsh cone time versus w/c ratio for grouts prepared and tested at 8 °C and 20 °C, and (c) spread of grouts measured at 8 °C and 20 °C after EN445.

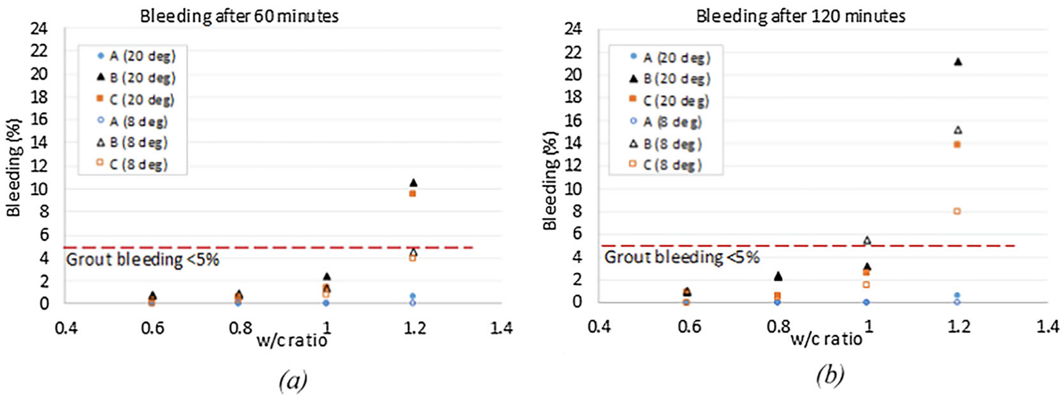


Fig. 9. Bleeding for all cements at 8 °C and 20 °C after 60 min (a) and 120 min (b) since mixing.

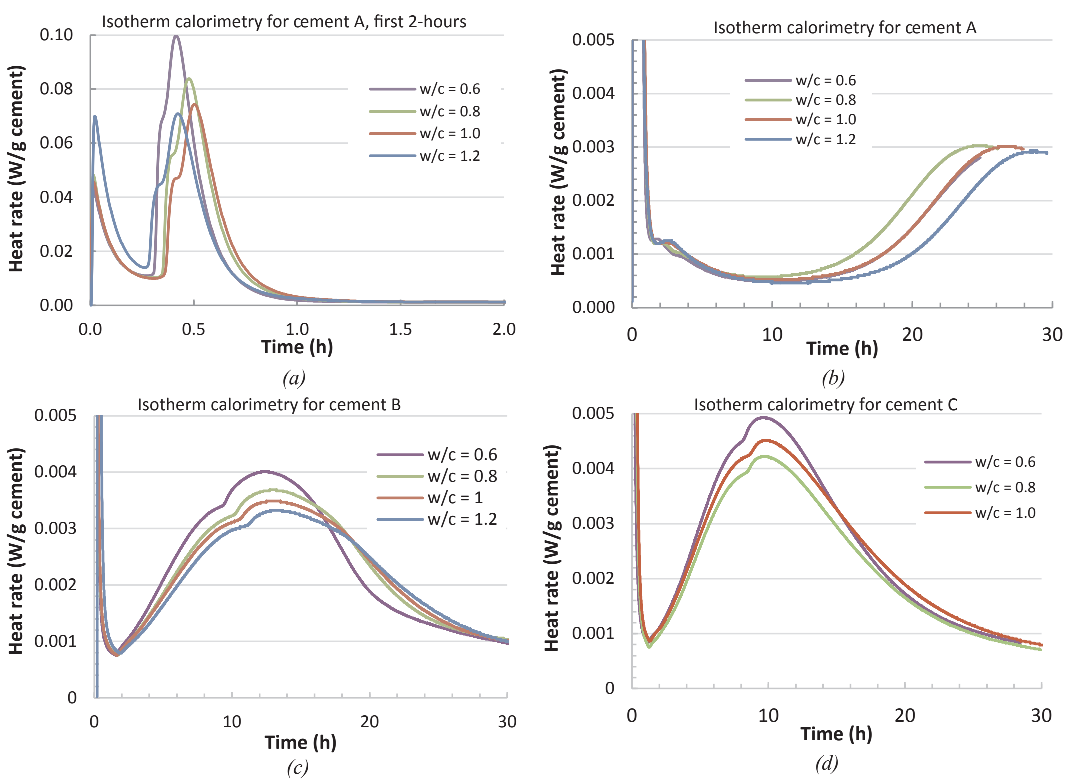


Fig. 10. Heat of hydration for all cements at 20 °C. Development of heat for cement A during the first 2 h (a) and during 20 h (b) since mixing. Heat rate versus time for cement B (c) and cement C (d). Note that plot (a) is a zoom-in of the initial two hours of plot (b).

some differences between the heat rate for various w/c ratios. In order to accurately quantify those differences, more tests and larger statistics may be needed. Cements B and C show one peak of heat rate at about 10–15 h after mixing (Fig. 10c and d). Heat rate of cement C is slightly higher than that for cement B.

In addition to heat rate, temperature of cement grouts was also measured during initial stages of setting (Fig. 11). Grouts with a low w/c ratio produce higher temperatures for all cements simply because of more cement per unit volume. All cement grouts show a major peak. Cement A shows a range of maximum temperatures from 12 °C to 25 °C for w/c ratios of 1.2 and 0.6, respectively. These peak temperatures are reached about 30 min after mixing. For Cement B, the range is much smaller; between 5 °C and 10 °C, and appears about 650–750 min. Similarly, the peak temperatures for cement C are between 7 °C and 15 °C and appears between 450 and 650 min. Comparing the three cements tested, mixes of cement B have both lower peak temperatures and the peak temperatures occur later during the setting process.

5. Discussion on strength development

Heat generation and especially a high heat rate in the initial stages of setting increases differential stresses between the core and the surface of cement mass and may create micro-cracks inside sample. If this is true, heat generation of grout should have an inverse correlation with the uniaxial compressive strength (UCS) of samples. Cement A showed the highest heat rate, followed by cement C and cement B, as shown in Fig. 10. Furthermore, maximum temperature during setting was highest for cement A, followed by cement C, then cement B. Thus, the cured grouts of cement A may contain more defects or micro cracks than those of cements B and C. This may influence compressive strength of cured grout samples.

Uniaxial compressive strength (UCS) of cured grout samples prepared from the same cements at similar w/c ratios was measured in the course of TIGHT project. Fig. 12 presents results of the UCS on samples prepared and cured at 20 °C and room relative humidity for either

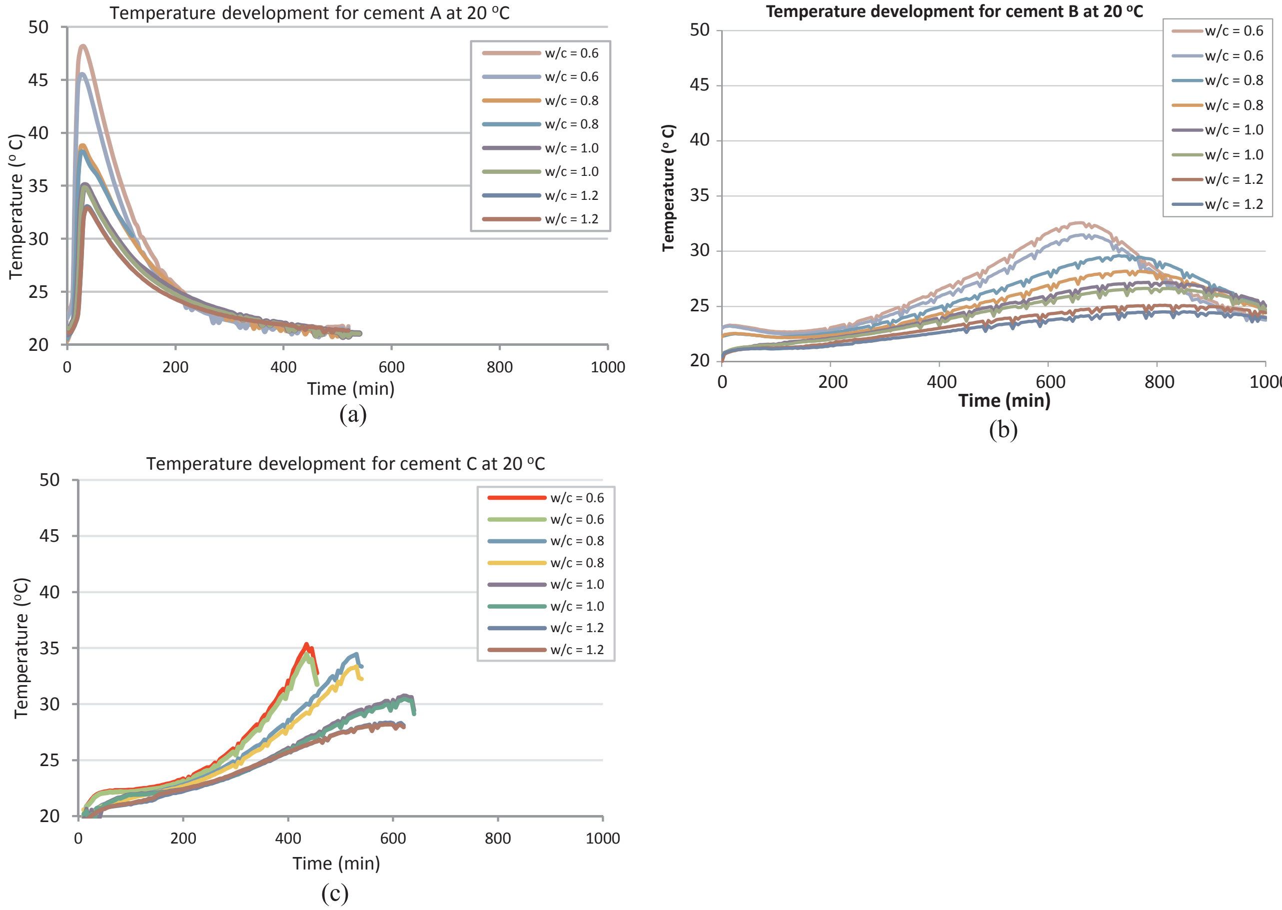


Fig. 11. Initial temperature development for grouts of cements A, B and C at 20 °C measured in a styrofoam cup. Note that for cement A, another peak will likely appear in a longer time span but the measurement time has not been long enough to record that.

4 days or 7 days. Each histogram bar in this figure presents an average UCS value from 4 to 6 specimens. It shows that (i) UCS of all grouts decreases with increasing w/c ratio, (ii) grout of cement B has the highest strength at all w/c ratios and ages, and (iii) strength of grouts from cement A decreases more sharply with increasing w/c ratio.

Grout of cement A shows the lowest compressive strength at all w/c ratios, expect for w/c = 0.6 of 7 days. In general, grout with high w/c ratio needs longer time to obtain strength than grout with low w/c ratio. This agrees well with the observations from heat rate and maximum temperature developments presented in Figs. 10 and 11. Such relationships are presented in Fig. 13. Compressive strength decreases with increasing peak temperature and heat rate during cement setting (Fig. 13a and b). Another heat indicator during setting is the time required for a grout to reach a significance temperature increase. This was also correlated with the compressive strength (Fig. 13c and d). It shows a positive correlation with strength; a longer time required for reaching a significant temperature increase leads to a higher strength. In other words, quick temperature increase of a grout leads to a lower strength, which is in agreement with the results mentioned above.

Among the three cements tested, Cement A shows more favorable characteristics for rock grouting based on bleeding, setting time and viscosity. However, it shows a lower compressive strength that cements

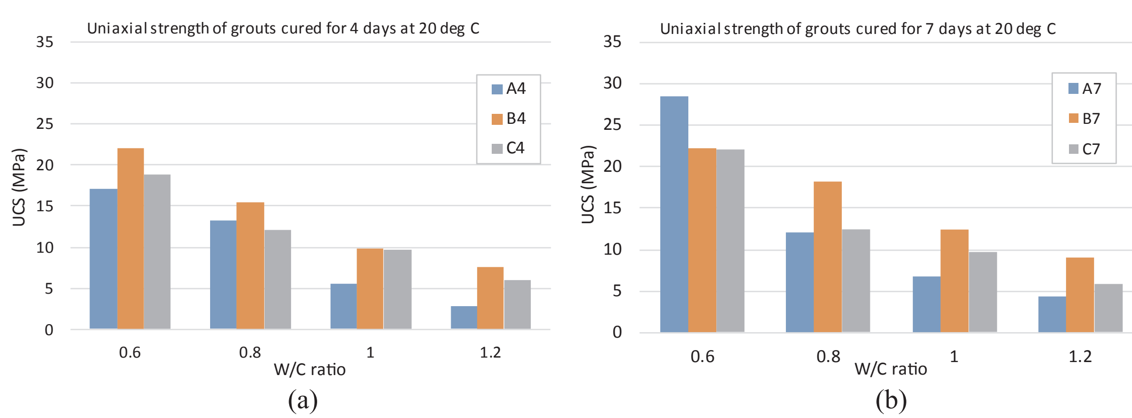


Fig. 12. Uniaxial compressive strength (UCS) of cured grout samples prepared from cements A, B and C. Grouts of cement B show the highest strength and grout of cement A shows the lowest strength, except sample of 7 days age at w/c ratio of 0.6. Each histogram bar presents an average value for 4–6 tests.

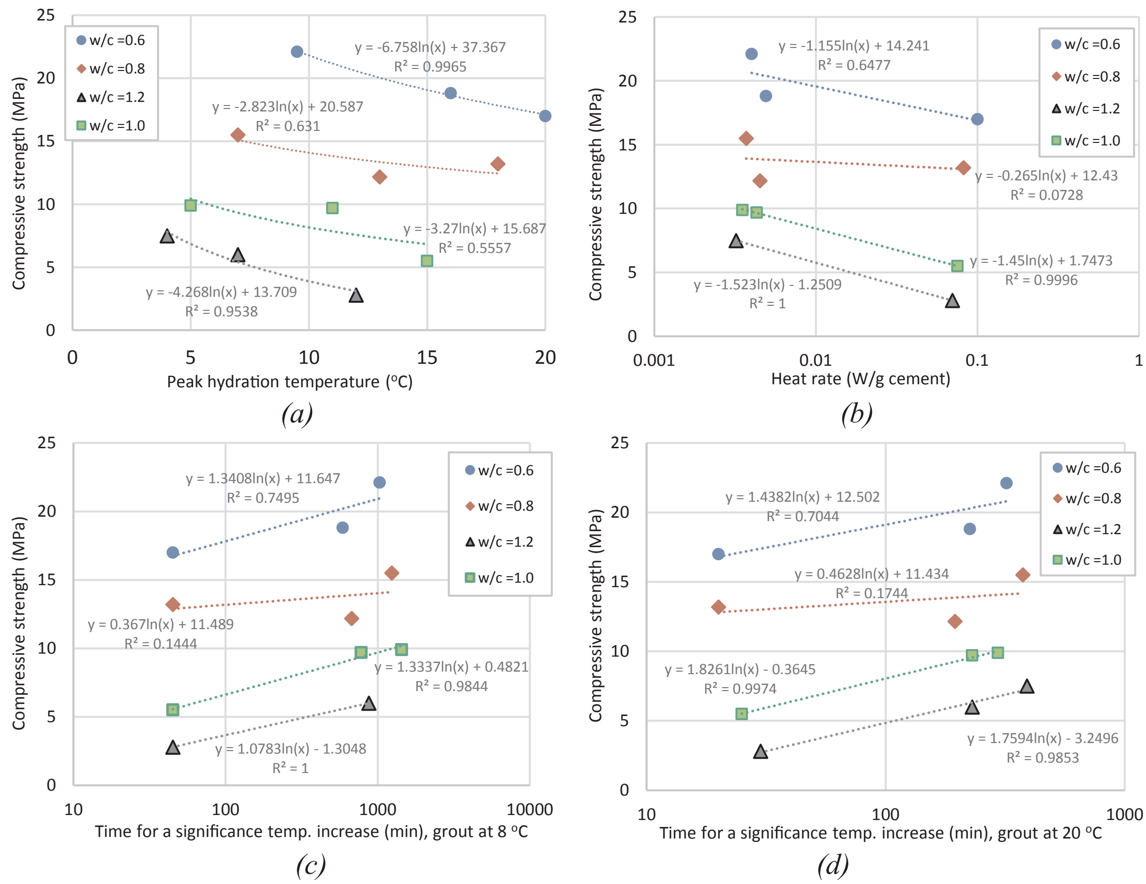


Fig. 13. Relationship between uniaxial compressive strength (of samples prepared and cured at 20 °C) and maximum hydration temperature (a), heat rate (b) and time for a significance temperature increase (c and d) for grout of cement A, B and C.

B and C. A possible method for increasing the strength of cement grouts A is to retard its temperature rise during setting. This may be done through adding specific retarders or modifying its chemical properties.

The correlations presented in Fig. 13 may not directly be applicable to grouting in tunnels. The samples used for UCS test were cylinders of 40 mm in diameter and 80 mm in length while the cement grout in the joints has a much smaller mass. Therefore, the heat generation and the consequent stress development within the injected grout mass in small fractures may not be significant. Hence, the impact of hydration heat on the strength of cement grout in-situ is a question yet to be investigated.

6. Conclusions

Three commonly used cements for tunnel grouting were selected for a comparative laboratory testing. The d_{95} of the cements ranges from 17 to 25 μm. Their Blaine fineness varies from 541 to 729 m^2/kg and their specific surface area, expressed in BET, ranges between 1580–1930 m^2/kg. Various mixes of the three cements, with w/c ratios of 0.6, 0.8, 1.0 and 1.2 were prepared. The mixes of each cement were divided into two batches; one mixed and cured at 8 °C (which is supposed to be the in-situ temperature in tunnels in the Nordic countries) and the other at 20 °C. The grout samples were studied for rheology and mechanical testing. The tests carried out on the cement grouts include: setting time, viscosity, bleeding, heat of hydration, maximum temperature during setting, and compressive strength of cured samples.

The measurements showed that viscosity of the grouts ranges from 20 to about 300 mPa s depending on the type of cement and the w/c ratio used. The viscosity difference between the cements is more obvious at lower w/c ratios but is very small at higher w/c ratios. Cement grouts with 8 °C have lower viscosity than those at 20 °C. Test results showed that setting time (measured with Vicat needle) increases with increasing w/c ratio for all cement grouts at 20 °C, although the increasing trend is different for different cements. Cement A sets very quickly while cements B and C need several hours before setting starts. All cement grouts with w/c ratios of up to 1.0 have a bleeding less than or about 5% and thus may be suitable for rock grouting. At w/c ratio of 1.2, only cement A has a bleeding less than 5%. Other cement types may need additives to reduce bleeding.

Compressive strength of cement grouts with a lower w/c ratio is higher than those with a higher w/c ratio and the strength increases with increasing age of cement grout specimens, as expected. Cement A has the lowest and cement B has the highest compressive strength at most w/c ratios employed. Strength of the cement grouts with w/c ratio of 0.6, cured at 20 °C for 4 days, is about 16–23 MPa while it is between 3 and 7 MPa with a w/c ratio of 1.2. Similar trend was also observed for grouts of 7 days age. Reduction of strength with increasing w/c ratio is more pronounced for cement A than the other two.

Correlation between the heat produced during setting of cement grouts indicates a negative correlation between the amount of heat produced and the strength of cement. Uniaxial compressive strength decreases with increasing the heat rate and maximum temperature produced during cement setting. Furthermore, the index of "significant temperature increase" shows a positive correlation with the strength of samples; the longer it takes for a sample to gain temperature, the higher is its strength.

The results above suggest that there may be a need to evaluate additional parameters for an effective characterization of cement grouts for rock grouting purpose. If strengthening of rock mass is considered, an appropriate cement with a high strength should be used, for instance cement B tested in this study. By selecting cement B you may compromise other aspects, for instance have high bleeding or obtain a late setting. The results of this study and the correlations suggested between various parameters may also be used to develop new types of cement products that are more favorable for tunnel grouting.

Acknowledgement

The authors would like to acknowledge Hans Olav Hognestad and Bjarne Ruud (Mapei), Karl Gunnar Holter (BASF), Aril Neby (SVV) and Hanne Elisabeth Wiig (BANE NOR) for support, comments and contributions made to this study. First author thanks Frode Pettersen, Magnus Soldal and Heidi Wilkinson (Norwegian Geotechnical Institute) for their assistance in cement testing. 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.

Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tust.2019.103011.

References

ASTM, 2017. ASTM C204-17 Standard Test Methods for Fineness of Hydraulic Cement by Air-Permeability Apparatus, ASTM International, West Conshohocken, PA, 2017, http://www.astm.org.

Bohloli, B., Morgan, E.K., Grøv, E., Skjølsvold, O., Hognestad, H.O., 2018. Strength and filtration stability of cement grouts at room and true tunneling temperatures. Tunnel. Underg. Space Technol. 71, 193–200.

Byle, M.J., Borden, R.H., 1995. Verification of Geotechnical grouting. A report from the ASCE Committee on Grouting of the Geotechnical Engineering Division and papers presented at the ASCE Convention in San Diego, California, October 23-27, 1995. ASCE Geotechnical Special Publication No. 57, 183 p.

Dalmalm, T., 2004. Choice of Grouting Method for Jointed Hard Rock based on Sealing Time Predictions. PhD dissertation, Division of Soil and Rock Mechanics, Royal Institute of Technology, Stockholm, Sweden.

De Schutter, G., 2002. Influence of hydration reaction on engineering properties of hardening concrete. Mater. Struct. 35, 447–452.

Draganovic, A., 2009. Bleeding and filtration of cement-based grout. Doctoral Thesis, Department of Soil and Rock Mechanics, Royal Institute of Technology, Stockholm, Sweden.

Eklund, D., 2003. Penetrability for cementitious injection grouts. Licentiate Thesis, Division of Soil and Rock Mechanics. Royal Institute of Technology, Stockholm, Sweden.

Eklund, D., 2005. Penetrability due to filtration tendency of cement-based grouts. Doctoral Thesis. Royal Institute of Technology, Stockholm, Sweden.

Elkhadiri, I., Palacios, M., Puertas, F., 2009. Effect of curing temperature on cement hydration. Ceram.-Silik. 53 (2), 65–75.

Eriksson, M., Friedrich, M., Vorschulze, C., 2004. Variations in the rheology and penetrability of cement-based grouts-an experimental study. Cem. Concr. Res. 34, 1111–1119.

Håkansson, U., 1993. Rheology of fresh cement-based grouts, PhD dissertation. Department of Soil and Rock Mechanics. Royal Institute of Technology, Stockholm, Sweden.

Holt, E., Leivo, M., 2004. Cracking risk associated with early age shrinkage. Cem. Concr. Compos. 26, 521–530.

Holter, K.G., Hognestad, H.O., 2012. Modern pre-injection in underground construction with rapid setting microcements and colloidal silica- applications in conventional and TBM-tunnelling. Geomechanik und Tunnelbau 02/2012; 5(1). https://doi.org/10.1002/geot.201200001. Accessed (1/11/2018) at: http://www.researchgate.net/publication/264528451.

Hu, J., Ge, Z., Wang, K., 2014. Influence of cement fineness and water-to-cement ratio on mortar early-age heat of hydration and set times. Available from. Constr. Build. Mater. 50, 657–663. https://www.researchgate.net/figure/Example-of-heat-of-hydration-curve_fig2_274050852.

ISRM, 1996. International society for rock mechanics commission on rock grouting. Int. J. Rock Mech. Min Sci. & Geomech. Abstr. 33 (8), 803–847.

Maryland, F., Azari, H., 2013. Research Results Digest 382: Measuring Cement Particle Size and Surface Area by Laser Diffraction. National Cooperative Highway Research Program, pp. 1–10.

Mirza, J., Saleh, K., Langevin, M.A., Mirza, S., Rafique Bhutta, M.A., Tahir, M.M., 2013. Properties of microfine cement grouts at 4 °C, 10 °C and 20 °C. Constr. Build. Mater. 47, 1145–1153.

Moum, J., 1965. Falling drop used for grain-size analysis of fine-grained materials. Sedimentology 5 (4), 343–347.

Nagy, A., Thelandersson, S., 1994. Material characterization of young concrete to predict thermal stresses. Cracking in concrete at early ages, Proceedings of the international RILEM Symposium, 10-12 October 1994, Munich, Germany. E & FN Spon, London.

NFF, 2011. Norwegian Tunnelling Society (NFF). Rock Mass Grouting in Norwegian tunnelling. Publication No. 20, 105 p.

Nguyen, T., 2012. Well design PE 413. Available (17/04/2015) at: http://www.nmt.edu/search-results?cx=010643251835841216931%3Axdpjttfxqhk&cof=FORID%3A11&ie=UTF-8&q=Tan+Nguyen.

Ortiz, C.R., 2015. Mechanical Behavior of Grouted Sands. Thesis for Master of Science in Civil Engineering, College of Engineering at the University of Kentucky, Lexington, KY, USA.

Skjølsvold, O., Justnes, H., 2016. TIGHT-Prøving av injiseringssementer, laboratorieprøving. SINTEF Byggforsk, Rapport SBF 2015 A0413, 22 p.

Tolppanen, P., Syrjänen, P., 2003. Hard Rock Tunnel Grouting Practice in Finland, Sweden, and Norway - Literature Study. Finnish Tunnelling Association, Raportti Elokuu 2003, 86 p.