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Variations in the rheology and penetrability of cement-based grouts—an experimental study

Magnus Eriksson[a,*], Martina Friedrich[b], Christoph Vorschulze[b]

[a]*Department of Civil and Architectural Engineering, Royal Institute of Technology, S-100 44 Stockholm, Sweden*
[b]*Institute of Geotechnics, Technical University Darmstadt, D-64287 Darmstadt, Germany*

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Abstract

To ascertain the most suitable grouting mixture to use in a specific project or to facilitate making predictions about grouting outcomes, laboratory tests are usually carried out to determine the properties of the particular grout. This paper presents a number of measurements of grout properties relating to the rheology and penetrability of fresh cement-based grout. The main purpose of this study is to investigate and describe variations that can be detected when measurements of these grout parameters are carried out repeatedly. Furthermore, a number of additional factors that can also influence these grout properties have been identified and examined. This study has shown that grout properties do vary and should therefore not to be regarded as uniform. The rheology-related properties of grout have been found to vary more than the penetrability-related parameters. Furthermore, it was found that the water–cement (w/c) ratio, the cement condition, and the mixing equipment could significantly influence the grout properties investigated in this study. Based on these experimental findings, it is therefore recommended that repeated testing be carried out on a specific grout mixture in preference to relying on the results of a single test.

Keywords: Penetrability; Rheology; Physical properties; Cement paste

1. Introduction

To adequately predict the outcome of grouting in rock and soil, a fundamental understanding of the grouting material is required [1–3]. If the grouting material is a cement-based grout, its rheology and penetrability are important properties to consider. How rheology influences the propagation of grout in rock and soil has been discussed by several authors. For example, equations for describing the flow of cement-based grouts in defined geometries have been presented in a number of publications [1–4]. Using grouting material that can penetrate fractures and soil easily and thereby facilitate a good sealing outcome has become the subject of increasing investigation [5–7]. This has been done especially to find those factors that govern grout penetrability as well as to promote grout development. The characterisation of grout rheology and penetrability are important steps in the ongoing improvement of grout. The rheology of cement-based grout is frequently investigated by means of viscometer measurements (see, e.g., Refs. [5] and [7]), whereas investigating the penetrability of grouts is carried out using various methods [6,9–12].

Characterising grout is difficult using deterministic parameters as the material exhibits pronounced variation in its properties. Most investigations done to date have reported on the properties of grout based on single tests. However, some authors have presented reports based on repeated testing of the same grout mix [5,8,13,14] and documented variation in the measured properties. For instance, measurements of parameters with coefficients of variation of 2–22% and 1–7%, respectively, have been reported [13,14]. This documented uncertainty in grout properties complicates predicting grouting outcomes, rendering them less precise. Variation occurring in the material may also explain contradictory experiences and findings relating to various grouting materials observed when the results from single tests have been compared.

This paper investigates variations in grout rheology and penetrability observed in a series of laboratory tests. This study has sought to gain an understanding of the variation in grout properties arising due to (a) the stochastic nature of the material and (b) inaccuracies in its handling and mixing.

* Corresponding author. Tel.: +46-8-790-6062; fax: +46-8-790-7928.
E-mail address: magnus.eriksson@byv.kth.se (M. Eriksson).

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Just how these variations affect grouting is not currently known, nor is the significance these have on predicting and achieving grouting outcomes. Numerical calculations indicate that the spread of grout could be a mean value process reflecting the rheological properties of grout but depend on the penetrability in terms of its weakest link [15]. If it can be concluded that these variations fall within a reasonable span and are symmetrically distributed, then a mean value could adequately represent rheological properties and could also be necessary for investigating the distribution of the penetrability. If it is found that large variations in properties should be expected, then different approaches to measuring and predicting grouting outcomes to those applied today could become a necessity.

2. Materials and methods

2.1. Outline of the method

To investigate the variation in properties of cement-based grout, a series of experiments was performed, with the main emphasis placed on variations in the rheology and penetrability of this type of grout. Several parameters were expected to significantly influence the rheology and penetrability of cement-based grout. These have been defined using the categories below:

- cement condition;
- water–cement (w/c) ratio;
- superplasticiser content;
- mixing time;
- mixer type; and
- water temperature.

Grout with a w/c ratio of 0.8 and a superplasticiser content of 1% was selected to experimentally represent the basic cement-based grout in all the experiments conducted. This grout was used for comparisons of flow behaviour and penetrability and is referred to as the reference grout or RG. The reason behind this choice of RG was that this composition represents a frequently used mix in relevant grouting applications. Two loads of cement were required to adequately complete all the experiments. Each load consisted of 40 bags, each weighing 20 kg and covered with a plastic sheet. One of these loads was stored in the laboratory for a number of weeks, and the other was used to mix grout immediately after delivery. This allowed the immediate testing of new reference grout (NRG)-reflecting conditions in the new load of cement and the assessment of the influence of the condition of the cement over time on the properties of the grout. The storing conditions in the laboratory are considered to represent typical northern European indoor climates with a temperature around 20 °C.

Errors incurred in the relative quantities of the grout components can of course occur in the field. An additional aim of this study was therefore to show the influence of "field-plausible" mixing errors on the properties of grouts. Erroneous mixtures were represented by w/c ratios of 0.6 and 1.0, and superplasticiser contents of 0 and 0.5%. The deviations from the mixing instructions for the RG may seem relatively large; however, they were considered accidentally plausible in the field.

Mixing time, mixer type, and water temperature have all been identified in the literature as factors able to influence grout properties to a significant extent and have therefore been included in the testing schedule reported here. These potentially influential factors were compared with their respective RG, according to the cement load used in the particular test. This regime allowed the properties of the grout produced in the field mixer and the grout showing the effects of differing the water temperature to be related to the NRG, and all the other tests to the original RG.

Table 1 presents an overview of these series of experiments, including the respective grout compositions, mixing times, and water temperatures—note that the w/c ratios have been given by weight. The amount of superplasticiser has been specified as a percentage of the dry weight of cement. All the experiments were conducted using a high-speed laboratory mixer, except for the experiments investigating the effects of the field mixer, where a colloid-mill-type mixer was used.

Cem I 52,5 LA SR cement (especially formulated for grouting) was used in these experiments. This product is based on the same clinker minerals as ordinary Portland cement and is finely milled to a d_{95} of 30 μm (manufactured by Cementa). According to the BET method specifications in the manufacturer's information, its surface area is 1300 m^2/kg. This cement is also considered to be sulphate-resistant due to its low C_3A content (<3.5%). The superplasticiser in the cement acts both as a fluidifier and as a water reducer, and is based on sulphonated melamine and naphthalene polycondensates. It is available as a fluid with a particle content of 35%, a density of 1215 kg/m^3, and a pH of 7–8. According to the manufacturer's (Cementa) specifications, the mixing time should be in excess of 3 min, and the recommended quantity is 1% of the cement by weight. According to the specifications, it also has a slightly retarding effect.

Ordinary Stockholm tap water was used in the laboratory tests. The same general testing procedure was carefully observed in all the laboratory tests. Each day, the accuracy of both the Mud Balance and the Marsh Cone testing devices was checked using clear water. To attain comparable water conditions for all the tests, avoiding, for example, using water that had been stationary in the pipe for several hours. The tap water was allowed to flow freely first (this water used for other purposes) until a stable water temperature was reached. Then, 4 l of tap water were poured into the mixing container, and the mixer started at 8000 RPM. The cement was then added to the water gradually, and as soon as all cement was added, the stopwatch was started.

Table 1
Description of the laboratory tests performed on cement-based grout

Series of experiments	First load of cement	Second load of cement	w/c Ratio	Superplasticiser content (%)	Mixing time (min)	Water temperature (°C)
RG	✓		0.8	1	5	17
NRG		✓	0.8	1	5	17
w/c Ratio						
w/c Ratio 0.6	✓		0.6	1	5	17
w/c ratio 1.0	✓		1.0	1	5	17
Superplasticiser content						
Plasticiser 0%	✓		0.8	0	5	17
Plasticiser 0.5%	✓		0.8	0.5	5	17
Mixing time						
Mixing time 1 min	✓		0.8	1	1	17
Mixing time 10 min	✓		0.8	1	10	17
Mixer type						
Fieldmixer		✓	0.8	1	5	17
Water temperature						
Water temperature 10 °C		✓	0.8	1	5	10
Water temperature 25 °C		✓	0.8	1	5	25

The superplasticiser was then added as required, and after 5 (1 or 10, respectively) min, the mixer was stopped. The measurements using the Marsh Cone and the Mud Balance were then carried out, and the viscometer started at 7 (3 or 12, respectively) min after the first contact was made between the cement and water. The penetrability meter test was then carried out. The measurements using the Marsh Cone and the Mud Balance were carried out as quality control; the results are not presented in this paper but are included in Ref [15].

Regardless of the w/c ratio, the total volume of water per grout batch mixed in the laboratory tests was nominally constant at 4 l (and 16 l for the field mixer). To establish a suitable number of tests, the following two restrictions had to be considered and met: (a) the statistical demands and (b) the limitations on available time. Relevant statistical literature was reviewed to fulfil the first demand. It is found that four to eight measurements would be sufficient; therefore, 10 tests were carried out on the RG, the NRG, and the grouts with different w/c ratios. All the other grouts were tested five times each. The objective of this study involved investigating the properties of fresh cement paste. In this context, ‘‘fresh’’ means within the first 30 min after the cement first comes into contact with water. As it was already known that the characteristics and microstructure of grout change constantly due to the level of hydration, this phenomenon was not investigated here, and therefore, all the properties of the grout have been regarded as remaining constant over time.

2.2. Measuring equipment and data evaluation methods

The Bingham model [16] is commonly accepted as representing the rheological behaviour of cement-based grouts [5,8]. Viscosity and yield value results can be obtained using viscometer measurements, whereas a measuring device called a penetrability meter can be used to reveal further information about the penetration ability of a grout.

The viscosity and yield stress of the grouts used in these experiments were assessed using a coaxial cylinder viscometer VT 550, by Gebrüder Haake (Germany). A ‘‘large’’ cup was used (R_c=30 mm) throughout the experiment. The inner cylinder had a radius of 9.4 mm and was rotated at specific speeds by a highly accurate step motor. The required torque was measured via the deflection of a torsion element with a resolution of 1/1000 rad, and automatically recorded with a personal computer (PC). The program used in the PC allowed test parameters, such as the number of readings, the speed increments and decrements, and the sampling intervals between the readings to be adjusted. The PC was also used to run the tests, process the recorded data, and evaluate the rheological parameters. Each measurement cycle consisted of up to 16 sequences with alternately increasing and decreasing speeds, resulting in ‘‘up-curves’’ and ‘‘down-curves’’. The rotational speeds used were 13.81, 8.49, 4.97, 2.62, and 1.04 rad/s. When the system could not obtain a stable reading during a set interval of time, no measurement was recorded for the given speed. Flow curves were obtained from the recorded measurements, and the

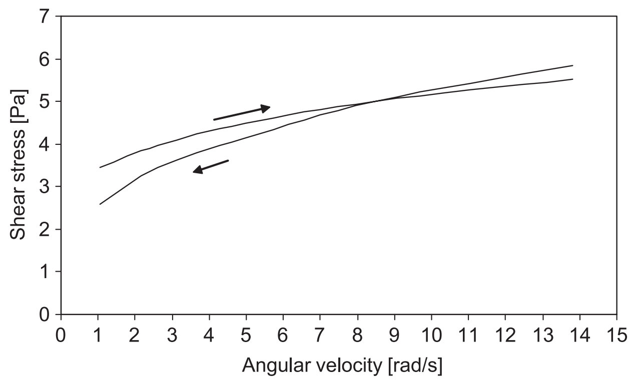

Fig. 1. Typical measurement showing up- and down-curves.

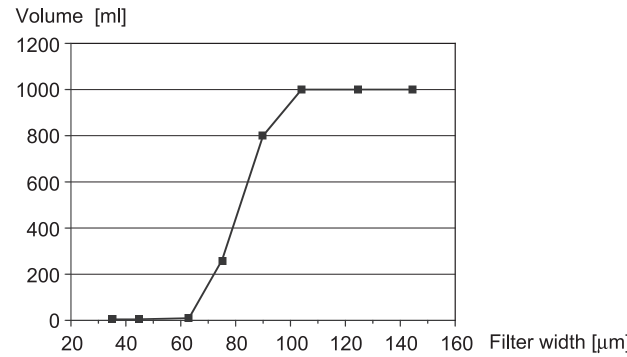

Fig. 3. Diagram showing measurements of the filter width versus the volume of grout passing through the penetrability meter.

parameters were evaluated according to the Bingham model. An example of a measurement cycle is presented in Fig. 1.

A special device that is called a penetrability meter (developed by the Division of Soil and Rock Mechanics at the Royal Institute of Technology, Stockholm [12]) was used to measure the penetrability of the grout (see Fig. 2). This device may have the potential to set a new standard for the measurement of penetrability of cement-based grouts. Using this device, the grout sample was first stored in a pressurised container fitted with an outlet pipe. The outlet pipe is equipped with a valve, a cap holder, and a cap housing a filter. The grout sample was then pressed through the filter, and the volume of grout filtrate, which passed through the filter before a filter cake was formed, blocking further flow, was registered. By using a series of caps fitted with a range of filters, the device can regulate the flocculation of particles and the flow of filtrate volumes through different apertures.

The filters used in the penetrability meter consist of a thinly threaded mesh forming rectangular openings $x{\cdot}x$ μm^2 in area, where x is assumed to represent an equivalent aperture in a grouting application. The amount of grout that passes through the filter is measured and then recorded in a diagram, showing filter size on the x axis and the volume of grout that has passed through the mesh on the y axis. Fig. 3 presents a recorded measurement of one of the tests of the RG.

The key values b_{min} and b_{crit} were calculated by means of the method of least squares for volumes between 0 and 1000 ml (0–100%), as shown in Fig. 4. If no reading of 10 ml was recorded with a filter, a theoretical aperture of 10 ml was calculated by linear interpolation between the two readings above and below. The minimum amount of 10 ml was chosen as the characteristic angle seen in the diagram that occurred most often around this value. Furthermore, it can be seen that below 10 ml, only a few drops of water permeated through the filter cake, but above this value, the fluid in the measuring cylinder looked like grout. The 1000-ml volume is the volume of grout found to be necessary to measure to ensure that no

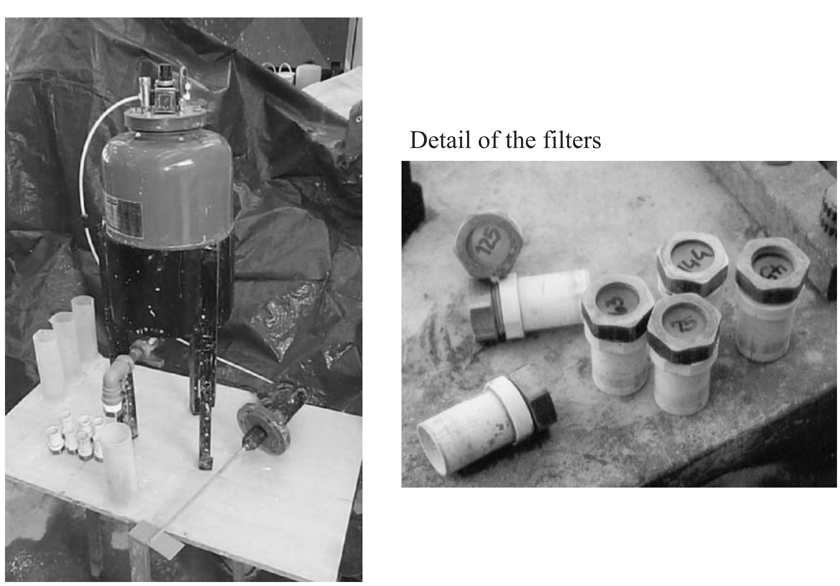

Fig. 2. The penetrability meter used in the experiments.

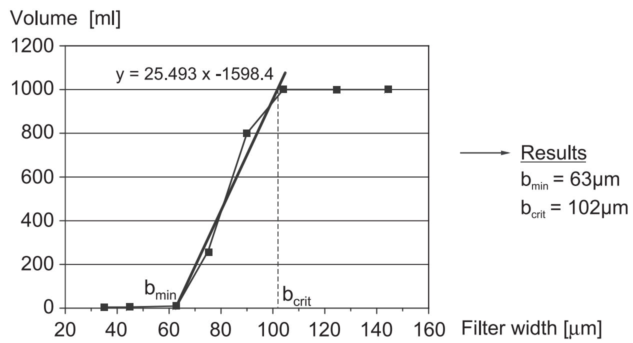

Fig. 4. Calculation of the parameters b_{min} and b_{crit} using the method of least squares.

filter cake formed (i.e., where an infinite volume of grout could theoretically pass through the filter). In accordance with the findings of Ref. [12], it can be difficult to define how large this volume needs to be due to the presence of oversized particles that can become stuck in the filter. These oversized particles make it difficult to verify whether a filter cake forms or not. This complicating factor and the 1000-ml volume used have not been addressed further in this study. Measuring the density and length of the filter cake was made possible by having a detachable filter cap but has also not been investigated further in this study.

Different filters have been used to represent the various apertures of constrictions in the rock of real grouting applications. It should be kept in mind that although these values fit the experimental setup used here, they may need to be adjusted when the specific circumstances, such as the pressure applied or type of cement used, are changed.

Basic statistics and significance tests have been applied in the study to facilitate the characterisation of the measurement data. Various key statistical values have been chosen to investigate the results of individual factors and to compare the outcomes of individual factors with their corresponding RG. These consisted of arithmetic mean values, standard deviations, and coefficients of variation. Furthermore, the samples have been used to calculate the confidence intervals within which the mean values of the sample populations were most likely to fall.

3. Results and discussion

3.1. General remarks

The overall results have been presented in the box plots in Figs. 5 and 6. These box plots show the positions of the mean and median values inside their respective quartiles and ranges of measurements, and how the mean values compare with their measurement ranges. These box plots also reveal that a rather large range was obtained in relation to the quartiles. It is also apparent that some results differ markedly from the mean, which could be defined as outliers. It has, however, been assumed that all the measurements represent a stochastic sample point of a true but unknown distribution. For this reason, all the measurements have been evaluated.

Relatively large variations were observed in the results of the RG, compared in particular with those of the NRG. Since as both grouts were produced using the same mixing instructions, approximately similar results had been anticipated. However, contradictory results were observed. Although these results appeared perplexing and unexpected at first glance, they nevertheless yielded valuable information regarding changes in properties when different loads of cement were utilised. Variations in the grout properties obtained from different supplies of cement (even from the same producer) were also observed in Ref. [17]. It has been shown that these differences can be in the same order of magnitude as those caused by changes in the grout mixing instructions and equipment.

How variations attributable to the stochastic behaviour of the cement relate to inaccuracies in mixing and other factors has also been presented in Figs. 5 and 6. It can be seen that especially large variations in the results were generally obtained in the RG and the grout with a w/c ratio of 0.6. As can be seen in Table 1, because these variations can be found in grouts produced from the same load of cement, they are therefore comparable in this respect. Therefore, these results indicate that lowering the w/c ratio increases the variation in rheological parameters and hence increases the uncertainty; which is supported by the results of the higher w/c ratio (w/c ratio=1.0). It can also be seen that large variations were obtained using the field mixer. These measurements should however be compared to those of the NRG.

3.2. Rheology results

When comparing the viscosity and yield value results for the RG and the NRG, it can be seen that the results were more even with the NRG (see Table 2). In terms of the coefficient of variation for viscosity, the values .38 and .22 were obtained for the RG and the NRG, respectively. These results are relatively high compared, for example, with those of Ref. [14], which reported a coefficient of variation of 12% for plastic viscosity.

Variations in cement quality are considered to be the main reason for the large spread of the RG results. This aberrant behaviour could be due to storage conditions despite storing the material in the laboratory, which is a relatively dry environment, and the production dates of the different batches of cement being recent. Nevertheless, a clear difference was seen in respect to grain size. The cement bags were stored in a pile, with the lowermost ones covered by a plastic sheet. Several bags (each with a weight of 20 kg) were used in the production of the RG, which means that bags from different layers were utilised

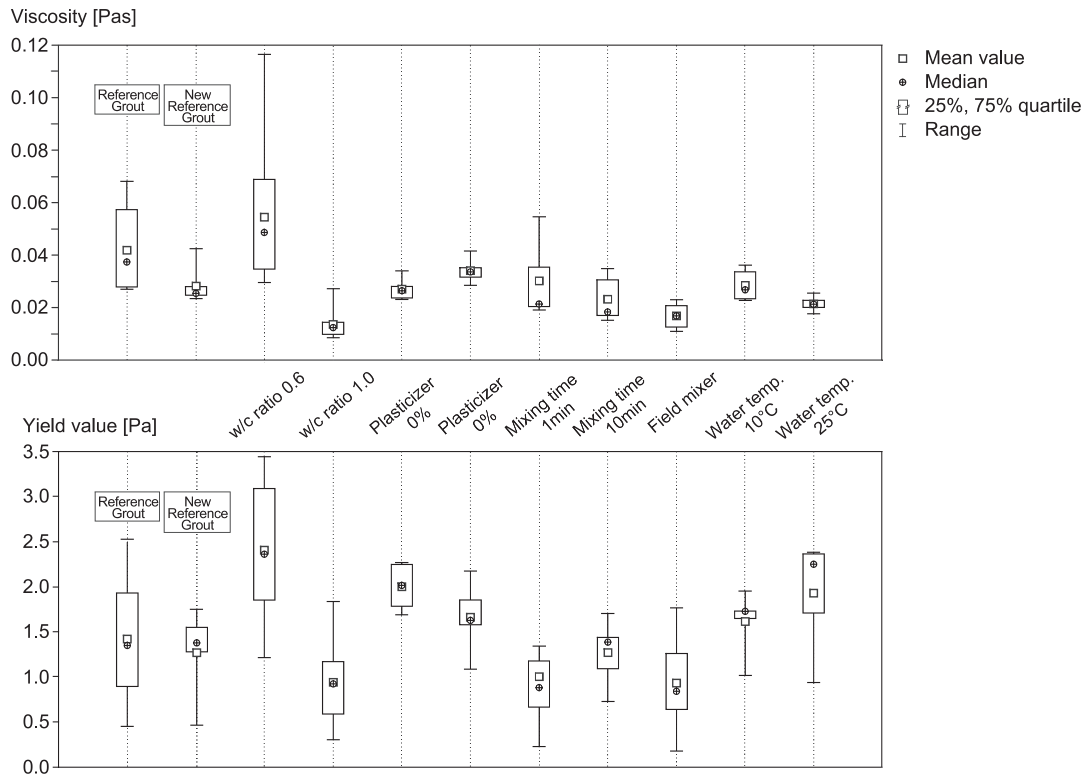

Fig. 5. Box plots of viscosity (upper graph) and yield value (lower graph) for all measurements.

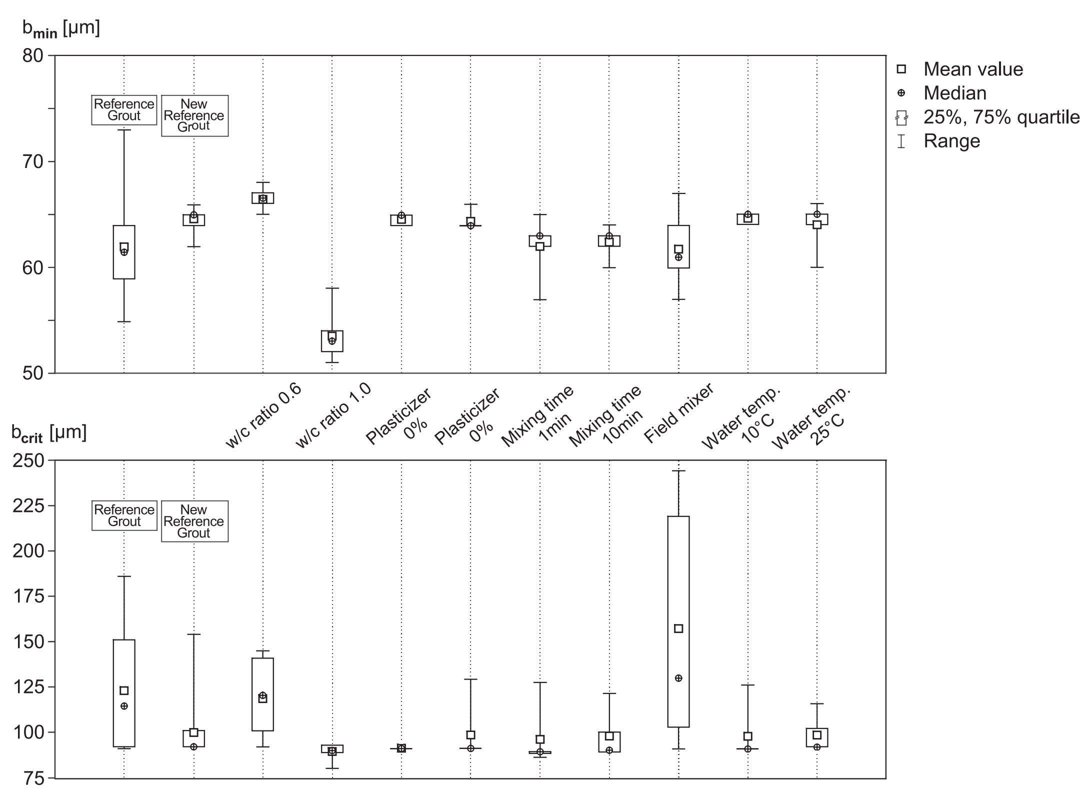

Fig. 6. Box plots of minimum (b_{min}; upper graph) and critical (b_{crit}; lower graph) apertures for all measurements.

Table 2
Viscosity and yield values for the RG and the NRG

	RG		NRG	
	Viscosity	Yield value	Viscosity	Yield value
Test 1	0.041	2.50	0.042	0.47
Test 2	0.036	2.11	0.036	0.62
Test 3	0.066	0.45	0.024	1.40
Test 4	0.057	1.93	0.025	1.58
Test 5	0.027	1.25	0.025	1.32
Test 6	0.028	1.37	0.025	1.75
Test 7	0.038	0.74	0.027	1.39
Test 8	0.031	1.33	0.023	1.28
Test 9	0.027	1.63	0.028	1.55
Test 10	0.068	0.90	0.026	1.37
Mean value	0.042	1.42	0.028	1.27
S.D.	0.016	0.637	0.006	0.411
Coefficient of variation	.38	.45	.22	.32

Table 4
Values of b_{min} and b_{crit} in the RG and the NRG

	RG		NRG	
	b_{min}	b_{crit}	b_{min}	b_{crit}
Test 1	59	110	62	154
Test 2	73	186	65	92
Test 3	60	126	66	92
Test 4	59	162	65	92
Test 5	64	91	65	92
Test 6	64	92	64	101
Test 7	64	91	65	92
Test 8	63	102	65	92
Test 9	55	119	64	101
Test 10	59	151	65	92
Mean value	62	123	65	100
S.D.	5	33	1	19
Coefficient of variation	.08	.27	.02	.19

throughout the tests. The high standard deviations seen with the slightly older cement could therefore be due to different degrees of exposure to environmental influences, such as varying humidity during storage. Although the air in the laboratory is considered to be rather dry (typical of indoor climates in northern European buildings), there is always some moisture present, which might cause slight reactions in the cement during storage. Under field conditions, this problem would be expected to be even more significant.

Table 3 presents the coefficients of variation for the yield values and the viscosities for all the tested cases. Comparing the different results in Table 3 shows that general conclusions are rather difficult to draw. The clearest trends in the results can be seen when changing the amount of superplasticisers and mixing times. Increasing the amount of superplasticisers can be seen to increase the variation in both viscosity and yield value, which agrees well with the findings of Hanehara and Yamada [18]. Increasing the mixing time was found to decrease the variation. Overall, the results suggest that the inherent variation in the different cement batches is greater than the induced changes in the mixing proportions.

3.3. Penetrability results

Large variation was also seen in the penetrability parameter results. These variations, however, seem generally smaller than those in the rheological parameters. The variations seen in the key values of the NRG (the second load of cement) were low compared with those in the rheological parameters. Comparisons showed that the RG exhibited larger variations than the NRG, agreeing with the rheological study. It was also noted that in both cases, the variation in b_{min} was considerably smaller than in b_{crit} (Table 4).

In Table 5, it can be seen that the main factors influencing the variation in penetrability are not at all the same as those affecting the rheology. For example, the w/c ratio results are ambiguous, as both increasing and decreasing the w/c ratio results in smaller variations in b_{crit} and b_{min}.. As with the rheology, reducing the amount of superplasticiser results in a decrease in the coefficient of variation. Likewise, the results for different mixing times also show lower

Table 3
Coefficients of variation for the parameter measurements

	Coefficient of variation	
	Viscosity	Yield value
RG	.38	.45
w/c Ratio 0.6	.48	.32
w/c Ratio 1.0	.38	.49
Plasticiser 0%	.16	.13
Plasticiser 0.5%	.14	.24
Mixing time 1 min	.50	.50
Mixing time 10 min	.38	.29
NRG	.22	.32
Field mixer	.30	.64
Water temperature 10 °C	.21	.22
Water temperature 25 °C	.14	.32

Table 5
Coefficients of variation for b_{min} and b_{crit} measurements

	Coefficient of variation	
	b_{min}	b_{crit}
RG	.08	.27
w/c Ratio 0.6	.01	.17
w/c Ratio 1.0	.04	.04
Plasticiser 0%	.01	.00
Plasticiser 0.5%	.01	.17
Mixing time 1 min	.05	.18
Mixing time 10 min	.02	.14
NRG	.02	.19
Field mixer	.06	.44
Water temperature 10 °C	.01	.16
Water temperature 25 °C	.04	.11

coefficients of variation. Although ambiguous, these results still indicate that the coefficient of variation of the RG is high, as was the case for the rheological parameters. In the second round of testing, there are indications that changes in temperature exert little influence. The results from the field mixer show a pronounced variation in b_{crit}.

3.4. Differences in the mean values

Although the main objective of this paper was to discuss the variation in the results, it was also considered to be of some interest to come up with a brief overview of how the mean values change in response to various factors. The mean values of all the results have been presented in Table 6.

The viscosity and yield values showed the most marked change in their mean values with different w/c ratios and amounts of superplasticisers. Only moderate changes in the mean values of viscosity and yield values were seen in relation to the other factors. There were also some unexpected results seen in the mean values of viscosity with different amounts of superplasticiser. It was observed that viscosity increased with increasing amounts of superplasticiser, which is contradictory to the original rationale for adding superplasticiser. Similar results for viscosity using the same mixtures have also been reported by Eklund [19] and on other mixes, for example, by Roy and Asaga [20]. As expected, however, the yield value decreased with increasing amounts of superplasticiser [21].

The penetrability of the grout (indicated by b_{min} and b_{crit}) showed the greatest sensitivity to changes in the w/c ratio and the type of mixer. The b_{min} values were affected somewhat less significantly (54–66 μm) than the b_{crit} values (90–157 μm). The RG b_{crit} results were somewhat surprising, showing an illogically larger value for the RG than for most of the alternative tests, with the exception of the field mixer test. The addition of superplasticiser results in a reduced penetrability, disagreeing with most published results. This finding should not be taken as valid for all grouts and admixtures.

Table 6
Mean values of the parameters for all the tests

	Mean value			
	Viscosity	Yield value	b_{min}	b_{crit}
RG	0.042	1.42	62	123
w/c ratio 0.6	0.054	2.41	66	119
w/c ratio 1.0	0.014	0.94	54	90
Plasticiser 0%	0.027	2.00	65	91
Plasticiser 0.5%	0.034	1.67	64	99
Mixing time 1 min	0.030	0.88	62	97
Mixing time 10 min	0.023	1.27	62	99
NRG	0.028	1.27	65	100
Field mixer	0.017	0.94	62	157
Water temperature 10 °C	0.028	1.62	65	98
Water temperature 25 °C	0.021	1.93	64	99

4. Concluding comments

The investigation of grout properties presented in this paper has revealed significant variations in the measurements of these properties. These findings are especially interesting with regard to the following: (a) the potential that these variations in grout properties have to affect grouting outcomes and (b) how measurements should be performed to adequately investigate grout properties.

The grouting of rock involves both the penetration of grout into small aperture fractures and the capacity for the grout to flow a minimum distance into the rock to seal it. Therefore, both the penetrability of the grout (as defined in this paper) and the rheology of the grout are important for grouting outcomes. When grout has poorer quality than expected in relation to these parameters, this naturally results in a poorer grouting outcome than expected.

The scope of this paper did not cover investigating how various aspects of variations in grout properties should be managed; it merely covered how these variations can be measured and described. It has become clear here that a number of measurements are required to adequately determine grout properties, which address both its rheology and penetrability. Importantly, when only single measurements are relied on, incorrect conclusions may be drawn regarding these properties. The main conclusions of this study have been summarised below.

- It was found that the properties of cement-based grouts vary significantly, and these properties cannot be reliably determined using single values.
- The inherent variations within a single load or batch of cement can be of the same order of magnitude as the changes induced through varying the mixing proportions and technique.
- Variations in grout rheology were found to increase with decreased w/c ratios and an increased amount of superplasticiser. Small effects were observed with changes in the temperature of the mixing water.
- The greatest effects on grout penetrability were found to occur when the field mixer was used (compared with the laboratory mixer). Variation in the penetrability parameters was less than that in the rheology parameters.
- It was found that a proper investigation of grout properties requires a number of tests to be conducted due to the inherent variation in the material.

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