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Optimization of cement-based grouts using chemical additives

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ABSTRACT

Grout injection is used for sealing or strengthening the ground in order to prevent water entrance or any failure after excavation. There are many methods of grouting. Permeation grouting is one of the most common types in which the grout material is injected to the pore spaces of the ground. In grouting operations, the grout quality is important to achieve the best results. There are four main characteristics for a grout mixture including bleeding, setting time, strength, and viscosity. In this paper, we try to build some efficient grouting mixtures with different water to cement ratios considering these characteristics. The ingredients of grout mixtures built in this study are cement, water, bentonite, and some chemical additives such as sodium silicate, sodium carbonate, and triethanolamine (TEA). The grout mixtures are prepared for both of the sealing and strengthening purposes for a structural project. Effect of each above-mentioned ingredient is profoundly investigated. Since each ingredient may have positive or negative aspect, an optimization of appropriate amount of each ingredient is determined. The optimization is based on 200 grout mixture samples with different percentages of ingredients. Finally, some of these grout mixtures are chosen for the introduced project. It should be mentioned that grouting operations depend on various factors such as pressure of injection, ground structure and grain size of soils. However, quality of a grout can be helpful to make an injection easier and reasonable. For example, during the injection, a wrong estimated setting time can destroy the injected grout by washing the grout or setting early which prevents grouting. This paper tries to show some tests in easy way to achieve a desirable sample of grout.

1. Introduction

Grouting is one of the efficient ways to seal and strengthen the ground in geotechnical projects. It is used in both of the soil (e.g. Karol, 1990) and rock (e.g. Houlsby, 1990) environments with the same main purposes but different methods/technologies. There are many methods of grouting, and one of the most common methods is permeation grouting.

In permeation grouting, grout is injected to the pore spaces of the soil in order to fill them and create cohesion between the soil particles, thereby increasing the strength characteristics of the soil (Henn, 1996). Permeation grouting can be done using cement-based or chemical grouts. The decision about the type of grout depends on many factors, including the type of soil, purpose of grouting, or even financial matters.

In cementitious grouting, the main ingredients are cement, bentonite, and water (Gustin et al., 2007). Cement is the main ingredient of cement-based grouts and its type should be determined according to the grain size distribution in the site-specific project. This means that in coarse soils, Portland cement may be considered efficient, but as the soil grains become fine, other types of cement such as ultrafine, microfine, and superfine cements may be applicable. Portland cement is hydraulic cement mainly composed of hydraulic calcium silicate. This kind of cement is hardened by the chemical reaction known as hydration in touch with water (Warner, 2004). Bentonite is colloidal clay from montmorillonite group with high water absorption. Some bentonites absorb water up to five times their own volume. They are used to increase the viscosity and cohesion of the grout and cause the bleeding of the grout to be lessened (Brady and Clauser, 1986).

Chemical grouting can also be used when the target soil is cohesionless and its stability is the main aim of the grouting

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operation. The most conventional chemical grouts are sodium silicate-based grouts in which a chemical reaction occurs in between sodium silicate and acid. The result of this reaction is a jelly material. It is important to note that the setting time is highly dependent on the percentage of the ingredients in the grout mixture (Karol, 1990).

As the main scope of the paper is optimization of a cement-based grout, the most important characteristics of cementitious grouts are introduced. The main characteristics for cement-based grouts by which the efficacy of a grout is examined can be mentioned as follows:

(1) Bleeding. It is a phenomenon in which water is squeezed out from pores between cement particles into the ground. This process is similar to water drainage in soil consolidation. Bleeding has some consequences such as reducing the mobility and pumpability of the grout. Lambardi (1985) stated that a grout is considered stable when the final bleeding is less than 5% after 120 min. Tan et al. (2005) investigated the effects of bentonite, fly ash, and silica fume on the bleeding using Taguchi approach and found the silica fume as the most efficient additive among the above additives for bleeding reduction.
(2) Setting time. It defines the effective radius for the mobility of grout, and the ease of grouting operation. Depending on the individual conditions, either rapid or delayed setting may be desired. Rapid setting time is often desirable when injection is under water table (into moving or even not moving water), so that the grout will set before being excessively diluted or washed away. Conversely, where injection is to be made through a very long delivery system, extension of the setting time may be required. Likewise, where large linear void spaces require filling, delay of the initial set is usually desirable until filling is complete (Warner, 2004).
(3) Strength. It is a fundamental significance in the strengthening of rock or soil to enable it to withstand greater loads. It is highly crucial for strengthening of soils in slope stability and vertical trenches as well. In some grouting projects, however, especially those in connection with water control, strength is not of much importance.
(4) Viscosity. This characteristic refers to a fluid's resistance to flow, which is the result of internal molecular friction. The flow properties of a grout can be evaluated by the time in which a certain volume of grout is flowed out of a standard funnel. The pumpability of a grout mixture is primarily defined by its viscosity. Of course, based on the fact that there is a wide range of grout mixture with proper viscosity to pump, the in-range viscosity can easily be obtained.

It is important to note that the priority of importance between the above-mentioned characteristics is first belonged to bleeding, second to setting time, third to strength, and finally to viscosity. The reason for this fact is that one may build a grout mixture with noticeable strength but not acceptable bleeding or setting time. Therefore, apart from the high strength, this grout mixture cannot be considered. Besides, there are some projects with only purpose of sealing.

In this paper, we try to obtain the most optimized cement-based grouts, which have the least amount of bleeding, a proper setting time, an acceptable strength, and an in-range viscosity. For this purpose, chemical grouting ingredients in small percentages are used as additives and their effect is investigated profoundly and precisely.

2. Brief description of the project

Many urban buildings such as commercial complexes, skyscrapers, and tall civil structures inevitably need their foundations to be deep enough for two main reasons: the balance of the structure and the use of underground space for parking or other purposes. This research is basically done for Chaharbagh Official, Trade, and Parking Complex Center with a base size of 252 m × 27 m. The project needs the land to be excavated in different depths because of topographical height differences, i.e. 7.2 m in the northern part, 9.4 m in the middle part, and 8.6 m in the southern part. This Engineering, Production, and Construction (EPC) project is in progress by Zaminfanavaran Consulting Engineers.

In order to determine the geological layers of the study area, four exploratory boreholes were excavated up to a depth of 11 m and the recovered cores were tested in the laboratory. The project profile of the study is given in Fig. 1.

The permeability of the in situ soils has also been estimated using Lufran tests. The shear strength parameters have been estimated using consolidated undrained triaxial compression tests as well. The results for determination of the soil classes and their characteristics can be seen in Table 1.

In Table 1, there are four main classes of soils in the study area. It is evident that classes Nos. 3 and 4 are approximately cohesionless and grouting techniques are needed to improve their cohesion. Furthermore, these two classes are with high permeability and besides, under the water table to some extent. As a result, apart from creating cohesion for them, grouting is also important to seal the study area. Since both of the sealing and strengthening purposes are required, optimization of a cement-based grout in accordance with the above-mentioned characteristics is of crucial significance.

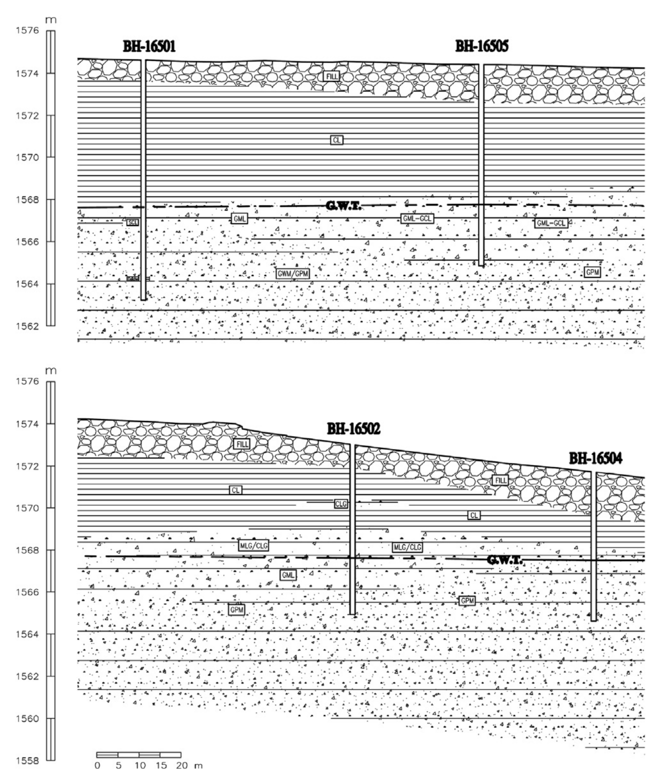

Fig. 1. Geological profile of the project area. G.W.T means ground water table.

Table 1
Physical and mechanical properties of geomaterials in the project.

Group	Soil symbol (BSCS)	Physical properties		Shear strength parameters	
		Permeability (m^2)	Density (g/cm^3)	c' (kPa)	φ (°)
1	Fill	10^{-4}	1.4	9.81	20
2	Cl, Ml, ClG, MlG, ClS, MlS	$<10^{-5}$	1.65	24.52	24
3	SCl, SMl, GCl, GMl	10^{-3}	1.9	4.9	30
4	GPM, GWM, SPM, SWM	$>10^{-2}$	2.1	0	35

In this paper, the effect of costs has not been considered because the cost of grouting is less than technical actions after excavation to prevent water entering into the area.

3. Materials used and their characteristics

In order to prepare some grout samples, cement, water, bentonite, sodium silicate, sodium carbonate, and triethanolamine (TEA) are used as the consisting ingredients/additives of samples. First, it is required to give a brief description about the materials used.

3.1. Cement and bentonite

Tables 2 and 3 show the specific characteristics of the used cement and bentonite in the designed grout samples. The grain size distributions for cement and bentonite are also plotted in Fig. 2.

One important point is that bentonite should be pre-hydrated a day before adding to the grout mixture (Huang, 1997). The reason for this fact can be shown by a simple test. Bentonite is mixed with water by an amount of 2%. Marsh viscosity is measured using a standard Marsh funnel every 5 min. The resulting graph is shown in Fig. 3.

As it is evident from Fig. 3, the viscosity of bentonite-water mixture increases with time. It shows that dry bentonite (with no preceding slake) mixed with the grout mixture may unnecessarily increase the viscosity. Of course, the viscosity–time curve (see Fig. 3) has a decreasing slope (downward concavity) which shows that the viscosity may become fixed after a few hours. However, bentonite is always slaked for 24 h before usage. In this way, bentonite will have enough time to absorb water and enlarge its volume. Thereafter, it can be mixed with the grout mixture without concern about its swelling effect.

Table 2
Chemical components of cement and bentonite used.

Component	Amount in cement (%)	Amount in bentonite (%)
Al_2O_3	7.35	14.5
SiO_2	22.5	54.3
MgO	1.75	3.7
Fe_2O_3	3.65	4.3
SO_3	1.5	3.5
Na_2O	0.43	2.7
K_2O	0.5	0.6
CaO	59.84	5.7
Mn_2O_4	0	0.2
P_2O_5	0	0.3
TiO_2	0.2	0.9
Other	2.28	9.3

Table 3
Physical properties of cement and bentonite used.

Material	Specific surface (m^2/kg)	Specific weight (kN/m^3)	Liquid limit (%)	Plastic limit (%)	Plastic index (%)
Cement	304	30.2			
Bentonite		27.3	167	51	116

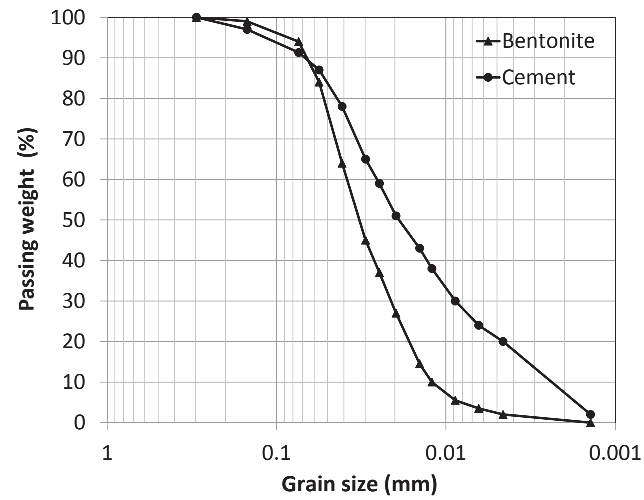

Fig. 2. Grain size distributions for the cement and bentonite used.

3.2. Water

The water used for the grout mixture preparation was also chemically analyzed. The analysis results are given in Table 4. The results indicated in Table 4 show that there is no harmful solution in the water used for grout mixtures.

3.3. Chemical additives

Some additives of the grouting mixtures in this paper are basic ingredients of chemical grouting. The used chemical ingredients are indicated as follows.

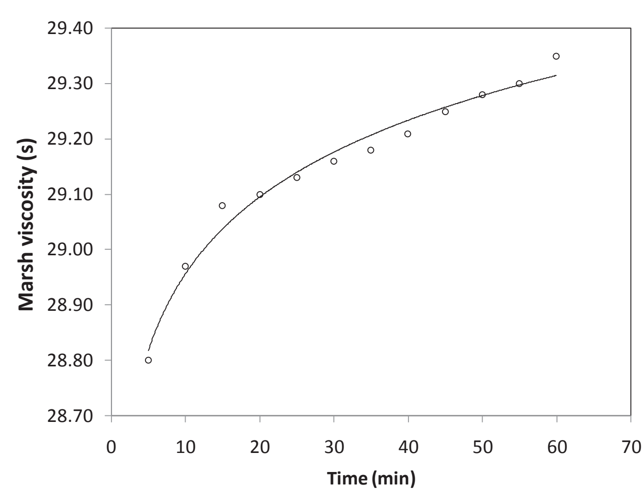

Fig. 3. Viscosity of bentonite-water mixture versus time.

Table 4
Chemical properties of the used water for grout mixtures.

Chemical property	Unit	Value	Limit	Standard test system
Total dissolved salt (TDS)	mg/L	940	2000	BS 3148 (1980)
Total hardness (TH)	mg/L	614	2000	USBR (1989)
HCO_3^-	mg/L	512	2000	Steinour (1960)
Cl^-	mg/L	225	500	ASTM D512-12 (2012)
SO_4^{2-}	mg/L	168	2000	ASTM D516-16 (2016)
Ca^{2+}	mg/L	192	200	Ryan and Samarin (1992)
Mg^{2+}	mg/L	38	150	Ryan and Samarin (1992)
$Na_2O + 0.658K_2O$	mg/L	140	1500	ASTM C114-15 (2015)

3.3.1. Sodium silicate

Sodium silicate is the common name for a compound sodium metasilicate, Na_2SiO_3, also known as water glass or liquid glass. It is available in aqueous solution in solid form. It is also used in cements. In industry, the different grades of sodium silicate are characterized by their SiO_2/Na_2O ratio, which can vary between 2:1 and 3.75:1. Grades with the ratio below 2.85:1 are termed alkaline. Those with a higher SiO_2/Na_2O ratios are described as neutral. Strength is raised with increasing ratio, whereas both viscosity and pH value are lowered (Warner, 2004). A sodium silicate with a ratio of 2.96 is used in this study. Cement-sodium silicate grouts are very effective where water is moving through the host deposit. It is common for the soil fines to be washed away. This kind of grout is used where a rapid set and a good strength are required (Warner, 2004). The main role of this chemical material in our study is to increase the strength of the grout. However, bleeding should be decreased by creating a gel in the mixed grout. A new research shows the effect of sodium silicate on mine backfill (Gelfill) strength. The uniaxial compressive strength (UCS) values increase by elevating the amount of sodium silicate up to 0.3% of the total dry weight (wt%). However, the UCS values decrease with any further increase of sodium silicate over this 0.3 wt% point. Moreover, the UCS values significantly decrease when the amount of sodium silicate surpasses 0.5 wt% and the specimens made with 0.7 wt% and 0.9 wt% sodium silicate have no measurable strength within the first 14 d of curing (Kermani et al., 2015).

Besides being used as an admixture for cement, soluble sodium silicate is also used for a number of applications in various industries or fields, such as the paper industry (e.g. for binding packaging), and mining (e.g. backfill). Sodium silicate has been used for various purposes including as an alkali activator of slag and fly ash, glue, cements, paints, detergents, and a hardening agent for natural and artificial stones (Shi et al., 2006). Many researchers believe that sodium silicate is the most effective alkali activator for most pozzolana including blast furnace slag and fly ash (Anderson and Gram, 1998; Bakhareva et al., 1999; Brough and Atkinson, 2002; Hilbig and Buchwald, 2006; Chen and Brouwers, 2007), soap and detergent manufacturing, textile processing, and foundries.

Soluble sodium silicates are silicate polymers. Furthermore, the view that compounds of alkali metals (sodium and potassium) stimulate the hydration of the main phases of Portland cement, at least in early stages of the hydration mechanism, and accelerate the setting of cement paste has long been recognized in cement research.

When sodium silicate is used in a grout mixture, a reactant may be added for the sodium silicate to work efficiently. There are some sodium silicate reactants mainly categorized in three classes: inorganic salts, organic/aliphatic esters and amides, and stabilizers. The proportion and dosage of different compounds can have a dramatic effect on both the setting time and the strength of grout. Of the first group, calcium chloride is the fastest acting reactant producing the strongest gel. Unfortunately, it is not possible to mix such a combination under the conditions common to grouting because of an excessively rapid gel time. The other member of the inorganic salts is sodium carbonate, which produces neither strength nor long-term durability as high as calcium chloride. Of course, the produced strength is still acceptable and the setting time is also approved as appropriate. Its fast deterioration after exposure to air can be remedied by a rapid action of spraying shotcrete (gunite) as well. Therefore, this reactant has been used in this study and is introduced in detail as follows.

3.3.2. Sodium carbonate

Sodium carbonate (also known as washing soda or soda ash), Na_2CO_3, is a sodium salt of carbonic acid. It most commonly occurs as a crystalline heptahydrate, which readily effloresces to form a white powder, i.e. the monohydrate. Sodium carbonate is domestically well known for its everyday use as a water softener. It can be extracted from the ashes of many plants. It is synthetically produced in large quantities from table salt and limestone in a process known as the Solvay process. The main purpose of using this additive is to simultaneously lessen the bleeding value and not reduce the strength of the grout.

3.3.3. Triethanolamine (TEA)

TEA is an organic chemical compound which is both a tertiary amine and a triol. A triol is a molecule with three alcohol groups. Like other amines, TEA is a strong base due to the lone pair of electrons on the nitrogen atom. TEA is used primarily as an emulsifier and surfactant. It is a common ingredient in formulations used for both industrial and consumer products. The TEA neutralizes fatty acids, adjusts and buffers the pH value, and solubilizes oils and other ingredients that are not completely soluble in water. TEA is also used as organic additive (0.1 wt%) in the grinding of cement clinker. It facilitates the grinding process by preventing agglomeration and coating of the powder at the surface of balls and mill wall. The main reason for its usage in grout mixtures is that it is a gas-producing material which generates very small air bulbs. These bulbs play a role in reducing the surface tension of the grout for being more easily injected. Moreover, the bulbs prevent any shrinkage after the grout is set. Besides, they cause the ingredients not to settle, thus resulting in a less bleeding value. It is considered as an accelerator for grout mixtures as well.

4. Experimental design

In this research, before any optimization, it is required to check the effect of each additive on the grout characteristics. Since the most important characteristic of the grout is considered as bleeding, the effect of each additive on bleeding is investigated. Some grout samples are designed, each of which consisting of only one additive. After obtaining the influence of each additive, two additives are added to the grout mixture in order to study the effect of one additive in the presence of the other. Then, three additives are added to the grout mixture for studying the tripling effect of the additives on bleeding. This trend is continued up to a level until all the additives are added and their effect on bleeding is determined.

For this, 14 grout samples are prepared to investigate whether or not the introduced additives are required to be added in the mixture. Their approximate amount is obtained as well.

After identifying the effect of each additive on bleeding, we try to determine their percentages for the grout characteristics (bleeding, setting time, strength, and viscosity). The optimization is done as follows.

Water to cement ratio (W/C) ranges from 1 to 3 with an interval of 0.5. Sodium silicate is from 2% to 5% with an interval of 1%. Sodium carbonate varies from 3% to 7% with an interval of 1%. TEA is the only material used with the fixed ratio of 0.1% of the cement weight. Bentonite is also added with only two different percentages of 2% and 3%. It is worth indicating that all the ingredients and additives are added to the main mixture by a weight ratio with respect to cement weight.

Using these ratios and considering a full factorial method, 200 samples are prepared for investigation of the grout characteristics (see Appendix). In the first step, bleeding of the 200 samples is considered and the best grout samples with the least value of bleeding are chosen. Thereafter, amongst the selected samples, three other characteristics are checked and the most optimized grouts are selected and introduced for the project.

Based on the project necessities (see Section 2), using a mixer with 5-L stainless steel bowl and wire whip blade, grout preparation is done in three stages. The first stage is mixing water, cement, and bentonite with water to cement ratio of unity (mixing time: 15 min). The second stage is adding water up to a level in which the desired water to cement ratio is obtained (mixing time: 10 min), and the final stage is adding the chemical additives (mixing time: 5 min). After the grout is prepared, the following tests are conducted:

(1) Density of the grout: Using volumetric method, a graduated cylinder, and a digital scale with precision of 0.01 g (ASTM C138/C138M-16a, 2016). Viscosity: Using a standard Marsh funnel according to API standard, right away after the mixing time (API, 1988). It should be mentioned that grouts with Marsh viscosities up to 60 s are groutable for moderate distance from the injection pump (Nonveiller, 1989).
(2) Temperature: Measuring the temperature using a digital thermometer (ASTM C1064/C1064M-12, 2012). Hot environments can have especially dramatic effects on all cementitious grouts. The hydration reaction will be significantly potent, and thus setting, curing, and drying time are lessened. The maximum allowable temperature for cementitious grouts is typically placed at a temperature level between 27 °C and 38 °C. Exceeding this temperature causes the setting time to become uncontrollably rapid and strength and durability will be negatively affected. Cool temperature, on the other hand, slows the reaction and causes the cementitious grouts not to set near the freezing temperature. From a practical standpoint, the minimum temperature of the grout should at no time descend to less than about 4 °C. Hence, we try to build all the grout mixtures in a constant temperature of 20 °C (T_1) and the temperature increase (T_2) is also measured to make sure that they are in the defined range.
(3) Bleeding: Using a graduated cylinder with a capacity of 500 cm^3, measured in percentage after 24 h of the mixing time (ASTM C940-16, 2016).
(4) UCS: Using 5 cm × 5 cm × 5 cm cubic molds, 3 d after the mixing time (ASTM C109/C109M-16a, 2016).
(5) Setting time: The standard for setting time of cementitious grouts is with water to cement ratio of 0.5 which cannot be applied here (ASTM C191-13, 2013). Because the water to cement ratio is changed in the range of 1–3, the real setting time for the proposed grout mixtures cannot be obtained using this standard. Therefore, as a rule of thumb, the Marsh viscosity of the grout mixtures were measured each 5 min up to a time in which the grout was not practically passed through the Marsh funnel. The time was used to represent the setting times of the produced grout samples.

5. Efficacy of the additives

In order to study the effect of the additives on the bleeding value, 14 grout samples numbered from I to XIV have been built. In the first three samples (I–III), only bentonite is added to the grout mixture. In the second three samples (IV–VI), bentonite together with sodium silicate is added to the grout mixture. In the third three samples (VII–IX), bentonite together with sodium carbonate is added to the grout mixture. In the tenth sample (X), bentonite, sodium silicate, and sodium carbonate are added to the grout mixture, and in the final four samples (XI–XIV), apart from these 3 additives, TEA is also added.

Table 5 shows the grout samples with water to cement ratio of 2.5 and the resultant bleeding, viscosity, and density. As mentioned previously, the most important parameter in this stage of study is bleeding which is going to be minimized.

As it is evident in Table 5, samples Nos. I–III are grout samples without chemical additive. The only variant material in these three samples is bentonite with values of 3%, 6% and 10%. It can be seen that the amount of bleeding is noticeable for all the three samples (changing from 18% to 10%). The other important point is that the amount of bleeding for these three samples decreases with raising the percentage of bentonite. However, the amount of bleeding is still considerable with even 10% bentonite.

By looking at samples Nos. IV–VI (see Table 5), it can be seen that in these grout samples, only bentonite and sodium silicate are added to the grout mixtures. In this stage, bentonite percentage is fixed to 3% of the cement weight but sodium silicate is changed from 2% to 6% by an interval of 2%. Comparing samples Nos. IV and I, one can see that bleeding value decreases from 18% to 15% by adding 2% sodium silicate to the grout mixture. Moreover, it is obvious that the bleeding of the grout samples decreases as the percentage of sodium silicate rises. The grout sample No. VI with 3% bentonite and 6% sodium silicate has a bleeding of 10%. Of course, this value of bleeding is still not acceptable as a minimum value.

By again looking at samples Nos. VII–IX in Table 5, it can be seen that in these grout samples, only bentonite and sodium carbonate are added to the grout mixtures. In this stage, bentonite percentage is again fixed to 3% of the cement weight, but three different sodium carbonate percentages of 3%, 6% and 10% are used. By making a comparison between samples Nos. VII and I, it can be seen that bleeding value decreases from 18% to 15% by only adding 3% of sodium carbonate to the grout mixture. Of course, the value of bleeding rises when the amount of sodium carbonate is 6% and 10%. This means that sodium carbonate causes the bleeding to decrease up to a boundary value, beyond which bleeding of the grout is negatively affected. This boundary value will be obtained and discussed in the following sections.

Continuing with Table 5, a comparison can be made between three samples Nos. V, VII and X. It can be seen that the amount of bentonite is fixed to a value of 3% in all these three samples. Moreover, it is observed that in sample No. V, only sodium silicate (4%) is added as a chemical additive to the grout mixture. In sample No. VII, on the other hand, only sodium carbonate (3%) is added as a chemical additive to the grout mixture. The bleeding in the former sample (No. V) is 12% and in the latter (No. VII) is about 15%. The point is that when both of the sodium silicate and sodium carbonate with the same amount are added to the grout sample No. X, the bleeding value surprisingly decreases to a value of 8%. This means that the presence of both of the sodium silicate and sodium carbonate causes the bleeding to be less than that when only one of them is presented in the grout mixture. The reason for this fact is that sodium carbonate acts as a reactant for sodium silicate and a gel-like material is more efficiently produced.

Considering samples Nos. X and XI, one can see that the bleeding value is again reduced from 8% to 5% by only adding 0.05% TEA to the grout mixture. This value of bleeding can become less (3%) by increasing the amount of bentonite to 5% (see sample No. XII, Table 5).

Table 5
Investigation of the effect of additives (samples with and without additives).

Sample No.	T_1 (°C)	T_2 (°C)	Content (%)				W/C ratio	Viscosity (s)	Density (g/cm^3)	Bleeding (%)
			Bentonite	Sodium carbonate	Sodium silicate	TEA				
I	20	21	3	0	0	0	2.5	33	1.22	18
II	20	21	6	0	0	0	2.5	34	1.22	14
III	20	21	10	0	0	0	2.5	34	1.22	10
IV	20	21	3	0	2	0	2.5	35	1.21	15
V	20	21	3	0	4	0	2.5	35	1.21	12
VI	20	21	3	0	6	0	2.5	37	1.22	10
VII	20	21	3	3	0	0	2.5	35	1.22	15
VIII	20	21	3	6	0	0	2.5	35	1.23	16
IX	20	21	3	10	0	0	2.5	35	1.25	17
X	20	21	3	3	4	0	2.5	37	1.23	4
XI	20	23	3	3	4	0.05	2.5	39	1.23	3
XII	20	22	5	3	4	0.05	2.5	39	1.23	<1
XIII	20	21	3	3	4	0.1	2.5	40	1.23	1
XIV	20	21	5	2	4	0.1	2.5	40	1.23	<1

Note: The percentages of bentonite, sodium carbonate, sodium silicate, and TEA are the weight percentages with respect to cement.

The final stage is to check whether or not raising the amount of TEA influences the bleeding value. Hence, TEA is raised to 0.1% in samples Nos. XIII and XIV. It is evident in Table 5 that the bleeding value is reduced to 1% in sample No. XIII. Increasing the amount of bentonite in sample No. XIV in comparison with sample No. XIII has even caused the bleeding to become less than 1%.

Considering all the grout samples indicated in Table 5, one can conclude that all the three chemical additives together with a small percentage of bentonite can cause the bleeding to almost vanish in the grout mixture.

6. Experimental results and discussion

In the previous section, it was proven that all the four additives (bentonite, sodium silicate, sodium carbonate, and TEA) are required for an efficient grout mixture to have the least bleeding value. In order to check the required amounts in a more precise way, 200 grout mixtures are designed, built, and examined. Table A1 in Appendix shows all the results for the 200 grout samples.

First, it should be indicated that all the grout samples were prepared with 0.1% TEA and this percentage was fixed for all the samples. It is worth mentioning that almost all the producers of this product have recommendations about the proper percentage of this additive for production of sufficient amount of air bulbs.

Secondly, 100 grout samples from 200 samples were prepared with 2% bentonite and the other 100 samples were prepared using 3% bentonite. The bleeding value was measured for all the grout samples with water to cement ratio from 1 to 3 with an interval of 0.5.

All the four major characteristics (bleeding, setting time, strength, and viscosity) of the grout mixtures are to be investigated in detail using these additives. Since it was really time-consuming to measure all these characteristics for all the grout samples, only bleeding and viscosity of all the 200 grout samples were obtained. Thereafter, some key grout mixtures were selected for the setting time and strength to be obtained. It is worth mentioning that key grout mixture means grouts with different amounts of additives for their effect to be observable.

6.1. Bleeding

It was proven in Section 5 that all the four additives should be presented in a grout mixture for the bleeding value to be minimized. Now, the only remaining point is the required amount of additives for the grout mixture to be optimized.

In Table A1 in Appendix, it can be seen that samples Nos. 1–8 and Nos. 101–112 have no bleeding values. It should be indicated that all these samples are with $W/C = 1$. The maximum bleeding value for the prepared samples is 8% which has been measured for samples Nos. 89–100. Comparing samples Nos. 89–100 with Nos. 189–200, one can easily understand that the bleeding value has decreased from 8% to 3%. Since the only difference between these two sample groups is the amount of bentonite (samples Nos. 89–100 have 2% bentonite and samples Nos. 189–200 have 3% bentonite), it is concluded that the only reason for such decrease in bleeding value is raising the bentonite amount. One of other conspicuous points in the results of these grout samples is that the value of bleeding increases as water to cement ratio increases. Hence, the more the W/C value is, the more the bleeding value becomes.

Taking a look at Table A1 in Appendix, one can see that there are five different W/C ratios and two varied percentages of bentonite. Therefore, there are 10 situations in which the boundary value for sodium carbonate can be obtained. The procedure is to see, up to which amount of sodium carbonate, the bleeding value will not increase (Table 6).

In Table 6, the boundary value of sodium carbonate differs by bentonite percentages. As the bentonite amount rises, the boundary value of sodium carbonate is raised. This is because higher percentage of bentonite prevents the bleeding to be negatively influenced. Therefore, based on the boundary values reported in Table 6, 4% sodium carbonate for samples Nos. 1–80 (W/C up to 2.5 and 2% bentonite) and 5% sodium carbonate for samples Nos. 101–180 (W/C up to 2.5 and 3% bentonite) are suggested as estimated boundary values of sodium carbonate beyond which bleeding value

Table 6
Boundary value of sodium carbonate based on the bleeding value.

W/C ratio	2% Bentonite		3% Bentonite	
	Sample No.	Sodium carbonate boundary value	Sample No.	Sodium carbonate boundary value
1	1–20	4	101–120	5
1.5	21–40	5	121–140	7
2	41–60	4	141–160	5
2.5	61–80	4	161–180	5
3	81–100	3	181–200	4

is negatively affected. Of course, for the cases of $W/C = 3$, this boundary value will be 3% and 4% for 2% bentonite (samples Nos. 81–100) and 3% bentonite (samples Nos. 181–200), respectively.

6.2. Strength

It is needless to say that the strength of a grout mixture is really important since a main application of grouting is stabilizing loose sands and gravels. The grout penetrates in between soil particles, bonds the soil particles, and creates cohesion for the entire material. As a result, in this section, we try to investigate which additive has what effect on the strength of the set grout samples. Thus, some grout samples were molded in 5 cm × 5 cm × 5 cm standard molds for the compressive strength to be measured after three days.

6.2.1. Effect of sodium silicate on the strength of grout

In order to study the effect of sodium silicate on the strength of grout mixtures, some grout samples were considered with constant ratio of $W/C = 1$, fixed value of sodium carbonate (3%), but varying sodium silicate percentage from 2% to 5% with an interval of 1% (see samples Nos. 1–4 in Table A1). The results of uniaxial compression tests on the molded grout samples are shown in Fig. 4.

In Fig. 4, the compressive strength of a grout sample increases by raising the sodium silicate amount. In this study, the value of compressive strength rises from 337.3 kPa to 425.6 kPa by raising the amount of sodium silicate from 2% to 5%. Hence, by considering the necessities of the project and the required strength for the grout, the amount of sodium silicate should be determined and suggested for use.

6.2.2. Effect of sodium carbonate on the strength of grout

Similar to the study of the effect of sodium silicate, some grout samples were prepared with constant ratio of $W/C = 1$, fixed value of sodium silicate (5%), but varying sodium carbonate percentage from 3% to 7% with an interval of 1% (see samples Nos. 4, 8, 12, 16 and 20 in Table A1). The results of uniaxial compression tests on the molded grout samples are shown in Fig. 5.

As it is evident from Fig. 5, the value of compressive strength remains unchanged by raising the sodium carbonate value from 3% to 4% but it starts to decrease from 429.5 kPa to 317.7 kPa by raising the sodium carbonate percentage from 4% to 7%. As a result, raising the amount of sodium carbonate beyond 4% may be harmful when high compressive strength is required.

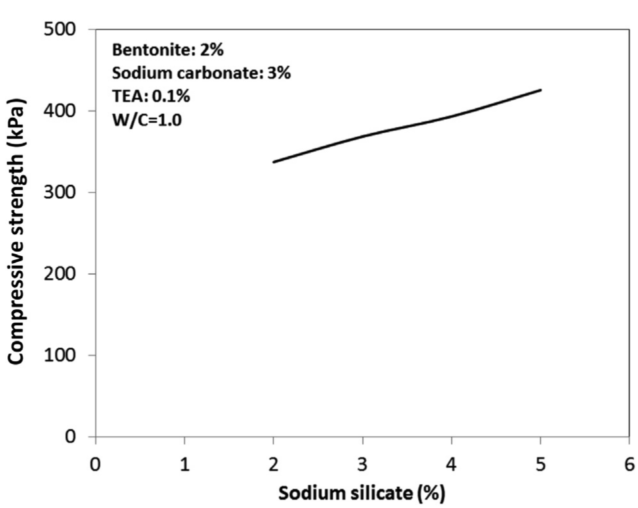

Fig. 4. Effect of sodium silicate on the compressive strength of the grout.

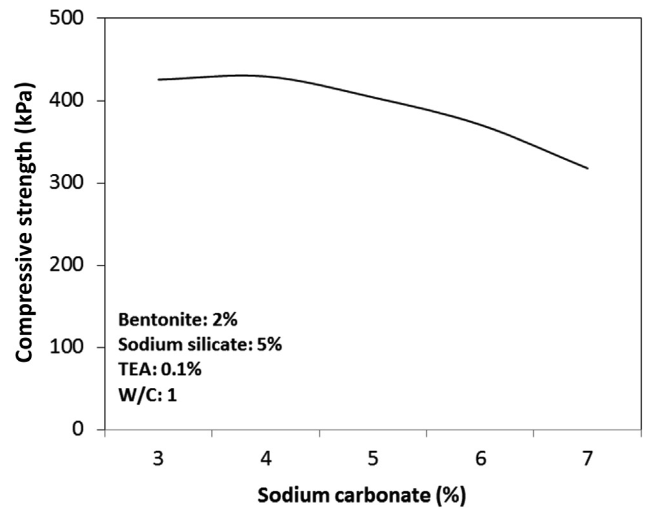

Fig. 5. Effect of sodium carbonate on the compressive strength of the grout.

6.2.3. Effect of water to cement ratio on the strength of grout

In the two previous sections, all the grout samples were prepared with W/C ratio of unity. Thus, it is also required to investigate the effect of W/C ratio on the compressive strength of grouts. In order to do so, some grout samples with fixed values of sodium silicate, sodium carbonate, and bentonite but different W/C ratios were prepared and tested (see samples Nos. 1, 4, 21, 24, 41, 44, 61, 64, 81 and 84 in Table A1). The results of these tests are shown in Fig. 6 for two different amounts of sodium silicate (2% and 3%).

It is shown in Fig. 6 that the compressive strength of a grout sample decreases when its water to cement ratio increases. The interesting point is that raising the sodium silicate causes the strength-W/C ratio curve to be entirely shifted up but the trend is the same as the water to cement ratio increases.

6.2.4. Effect of bentonite on the strength of grout

Since bentonite is hydrophilic and increasing its percentage causes the bleeding to decrease, its effect on the strength of grout has to be investigated as well. For this purpose, some grout samples were also prepared to investigate the effect of bentonite on the strength of grout (see samples Nos. 4, 24, 44, 64, 84, 104, 124, 144,

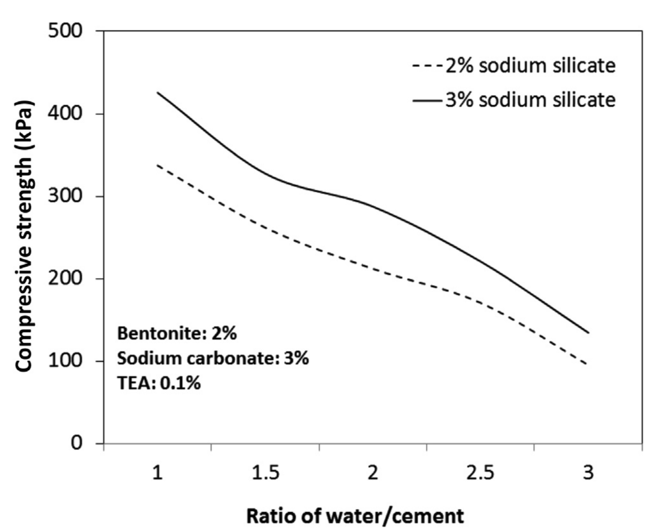

Fig. 6. Effect of water to cement ratio on the compressive strength of the grout.

164 and 184 in Table A1). The results of these tests are shown in Fig. 7.

It is shown in Fig. 7 that any increase in bentonite amount causes the strength to decrease. In this figure, increasing the amount of bentonite has shifted the strength-*W*/*C* ratio curve down considerably.

6.3. Setting time

The grout mobility is of paramount importance. The reason for this fact is that in case the grout setting time is higher than the time required, it goes beyond the desired area and causes the expenses of the project to rise noticeably. When the setting time is lower than the time required, the grout cannot be injected. This consequence can be dangerous, because it can generate large explosions in which grouting equipment breaks apart and causes severe injuries or even death for the grouting personnel.

The same grout samples, the strength of which was tested, are chosen to measure the setting time. Table 7 shows the setting time of these grout samples and their corresponding compressive strengths.

Many parameters influence the required setting time such as the largest distance to grout holes, mixing time, and temperature. Based on the conditions of the introduced project, a setting time of 45–75 min was accepted as appropriate.

6.3.1. Effect of sodium silicate on setting time of grouts

Setting time of a grout mixture can be highly influenced by adding only a small percentage of sodium silicate to the grout mixture. Fig. 8 shows the effect of sodium silicate on the setting time of a grout mixture. As it is evident in Fig. 8, the setting time of the grout mixtures is significantly reduced by increasing the amount of sodium silicate.

6.3.2. Effect of sodium carbonate on setting time of grouts

In this stage, sodium carbonate amount is changed in order to investigate its effect on the setting time. The results show that the setting time of the grout mixtures is gently reduced by raising the sodium carbonate. Fig. 9 shows the effect of sodium carbonate on the setting time of a grout mixture.

It is worth mentioning that this decrease in the setting time (as a result of adding sodium carbonate) has only been observed in a grout mixture with 2% bentonite, 5% sodium silicate, and 0.1% TEA.

Table 7
Setting time and compressive strength of some grout samples.

Sample No.	Compressive strength after 3 d (kPa)	Setting time (min)
1	337.3	60
2	368.7	45
3	393.2	35
4	425.6	30
8	429.5	30
12	404	25
16	370.7	25
20	317.7	25
21	261.8	60
24	327.5	50
41	211.8	70
44	287.3	60
61	170.6	85
64	220.6	70
81	95.1	100
84	134.4	85
104	347.2	30
124	261.8	45
144	211.8	55
164	168.7	65
184	112.8	85

Note: Sample No. refers to sample numbers mentioned in Appendix.

Comparing Figs. 8 and 9, one can deduce that the effect of sodium carbonate is much less than that of sodium silicate on the setting time. Of course, it is possible that the influence of sodium carbonate on the setting time becomes more tangible with lower percentage of sodium silicate.

6.3.3. Effect of water to cement ratio and bentonite on setting time of the grout

It is reasonable that a thinner grout mixture, with higher water to cement ratio, has higher setting time in comparison with a thicker grout mixture with smaller water to cement ratio. Fig. 10 shows how setting time increases by raising the water to cement ratio.

It can be easily understood from Fig. 10 that the difference between setting times of grout mixtures with 2% and 3% bentonite is maximally 5 min. Therefore, we can see that bentonite change in small percentages cannot cause noticeable changes in setting time of grout mixtures.

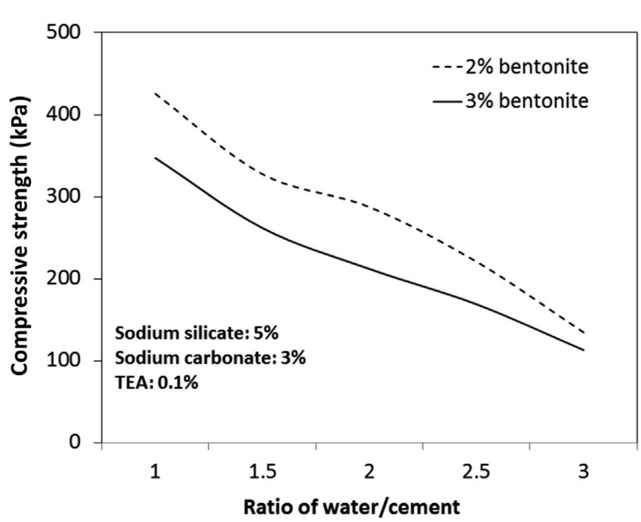

Fig. 7. Effect of bentonite on the compressive strength of the grout.

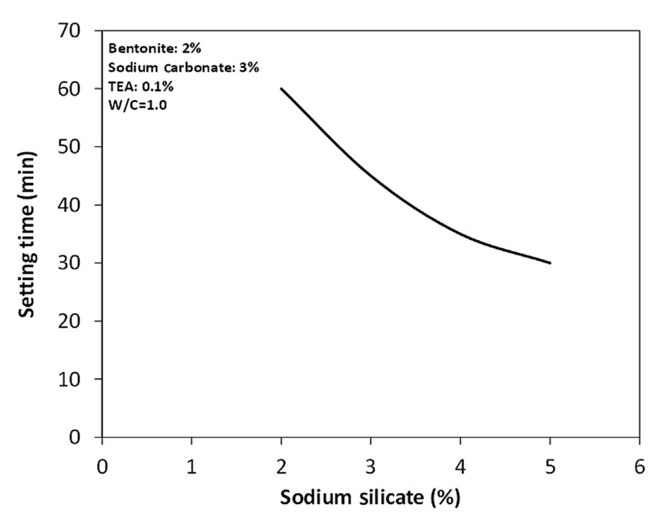

Fig. 8. Effect of sodium silicate on setting time of the grout.

6.4. Viscosity

In order to show the effect of each ingredient on the viscosity, Marsh funnel test was done for all the 200 grout samples. As it is evident from Appendix, the results demonstrate that all the measured viscosities are in-range (less than 60 s) based on Nonveiller (1989). At this stage, based on the observed results and considering the varying ingredient in the grout mixtures, the effect of each ingredient on the viscosity can be demonstrated.

The results show that the sodium silicate increase causes the viscosity to rise. Of course, the increase is less than 1 s in Marsh viscosity. Fig. 11 shows the rise of viscosity for varying sodium silicate from 2% to 5%. The sodium carbonate also raises the viscosity of grout. Based on the viscosity results of all the 200 samples (see Appendix), the viscosity increase caused by sodium carbonate is more tangible than that produced by sodium silicate. Fig. 12 is an example showing the increase of viscosity by raising the sodium carbonate.

Comparing the viscosity results of grout samples Nos. 1–100 with those of grout samples Nos. 101–200, one can see that raising the bentonite percentage in a grout mixture results in a more viscous grout. It is also obvious that raising the water to cement ratio causes the viscosity of a grout to fall down. Fig. 13 has been drawn for grout mixtures with 2% sodium silicate, 3% sodium carbonate, but varying *W*/*C* ratio and bentonite percentage. The figure shows that all the grout samples with 3% bentonite are more viscous in comparison with their corresponding mixture but with 2% bentonite.

6.5. Choosing the best optimized grout mixtures

Choosing an appropriate grout mixture is entirely dependent on the project and its necessities. Some projects are only done for sealing of the ground. Therefore, strength is not much considered in grout mixture design. In some other projects, on the other hand, strengthening of the ground may be the main purpose of grouting.

Bleeding is a characteristic that should be minimized all the time. Of course, the allowable bleeding value differs based on the available literature. In the project mentioned in Section 2, bleeding values less than 5% were considered as acceptable.

Setting time is also a characteristic that should be determined based on the specifications of the project. It is worth mentioning that considering the project described in Section 2, a setting time range between 45 min and 75 min was experimentally estimated as logical for this project because it allowed the grout to travel in ground far enough to generate the desired grouted area and prevented its escaping from the optimized length of the grouted zone as well.

Since strengthening of the surrounding ground was also an important aim of grouting in the project mentioned in Section 2 based on the project specifications, a compressive strength of the set grout not less than 196.1 kPa (3-d strength) was considered as acceptable.

Finally, all the measured viscosities were found in-range and could be considered as pumpable.

In this study, four additives have been added to the grout mixtures in order to improve the above-mentioned characteristics. Even though each additive has some advantages, it has also some disadvantages that should be considered when determining its percentage.

Considering the previous observations, one can see that all the four additives reduce the bleeding value but each with its own simultaneous consequence. For instance, bentonite reduces the strength tangibly, while sodium carbonate does not reduce the strength up to a boundary percentage.

Likewise, sodium silicate reduces the bleeding desirably but also lessens the setting time sharply. Similarly, TEA also causes the bleeding value to fall down but its excessive amount may cause air bulbs in the grout mixture much more than required, thereby lessening the strength of the grout. Moreover, it may cause the setting time to decrease because of its accelerating effect.

This means that the percentage for each of these additives should be chosen carefully in order to optimize their effect on each characteristic of the grout. Table 8 shows the effect of each ingredient on the four main characteristics of the grout mixtures. There are some symbols used in Table 8 that have the following meaning: ↓ means decrease, ↑ means increase, ↓ means small decrease, ↑ means small increase, – means no sensible effect, and ↑↓ means no sensible effect up to a boundary percentage, beyond which significant reduction is observed.

According to the experimental results, it was demonstrated that less amount of bentonite is preferred since higher percentages of bentonite reduce the strength of the resultant grout. Hence, 2% bentonite is more acceptable than 3% of it.

It was also observed that sodium carbonate has two boundary values for bleeding to decrease (3%–4% for 2% bentonite, and 3%–5% for 3% bentonite). It was clear that strength of the grout mixtures is negatively affected for sodium carbonates above 4% as well. Therefore, the optimized value of sodium carbonate should be used up to 4%. It is also noted that there was no tangible difference between the strength and bleeding of grout samples with 3% sodium carbonate and those of grout samples with 4% sodium carbonate. As a result, 3% sodium carbonate would be enough for the bleeding to decrease and strength not to decrease.

After that, it is realized that sodium silicate is a strong chemical additive, by which strength of the grout can be raised noticeably and setting time can be simultaneously controlled as well. In this research, sodium silicates in the range of 2%–5% were understood to be enough for the above-mentioned purposes. In this range, lower percentages of sodium silicate are used for lower *W*/*C* ratio and bentonite percentage (*W*/*C* ratio = 1–1.5; bentonite: 2%), but higher percentages of it are used for higher *W*/*C* ratio and bentonite percentage (*W*/*C* ratio = 2–2.5; bentonite: 3%).

Based on the observed strength (lower than 196.1 kPa) and setting time (higher than 75 min) for the *W*/*C* ratio = 3 (see Table 7), it was concluded that this *W*/*C* ratio may not be applicable for the project (see Section 2). Therefore, *W*/*C* ratio = 3 was not

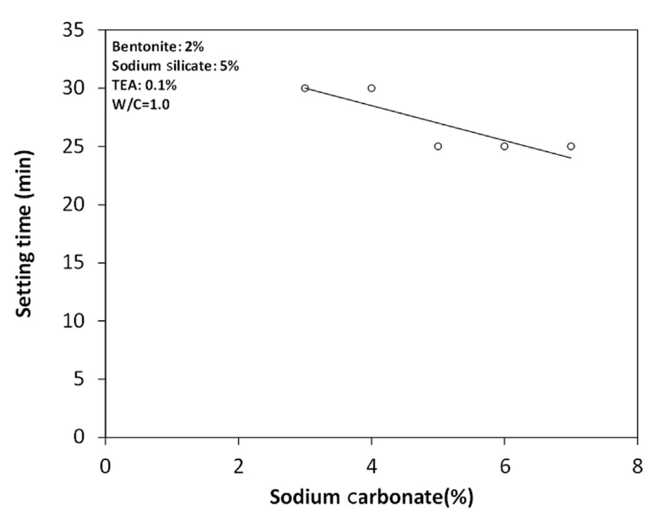

Fig. 9. Effect of sodium carbonate on setting time of the grout.

recommended for the project and all the suggested grouts were chosen amongst *W*/*C* ratio = 1–2.5.

In this way, the final optimized grout mixtures for the presented project (see Section 2) can be categorized based on the required water to cement ratio as shown in Table 9. It is worth mentioning that all the grout mixtures presented in Table 9 are suggested for the project introduced in Section 2. It means that these grout mixtures have been selected based on the necessities of the project (bleeding less than 5%, strength not less than 196.1 kPa, a setting time between 45 min and 75 min, and an in-range viscosity).

One important point about the designed grout mixtures (see Appendix) is that the bleeding value rises when higher water to cement ratio is used. Therefore, in order to control the amount of bleeding, the decision about the type of the additive is made based on the purpose of grouting operation. Whenever the purpose of grouting is only sealing of the ground, bentonite can be used with no limitation but for strengthening the surrounding ground, sodium silicate is used to strengthen the grout mixture, lessen the bleeding, and control the setting time. Of course, its effect on setting time can sometimes be problematic. This means that a

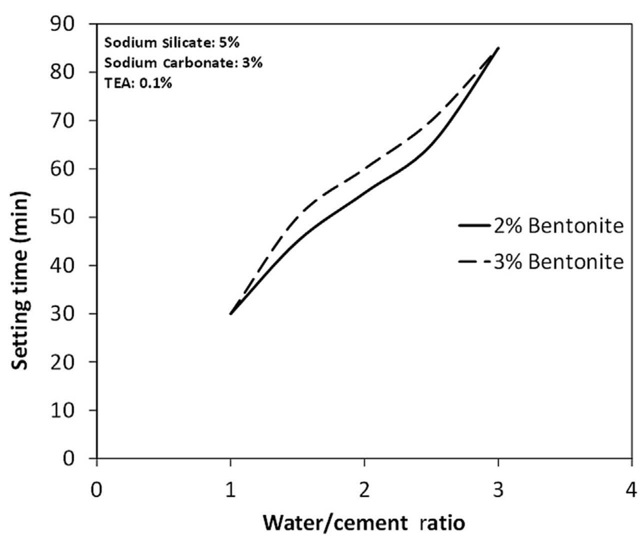

Fig. 10. Effect of water to cement ratio and bentonite on setting time of the grout.

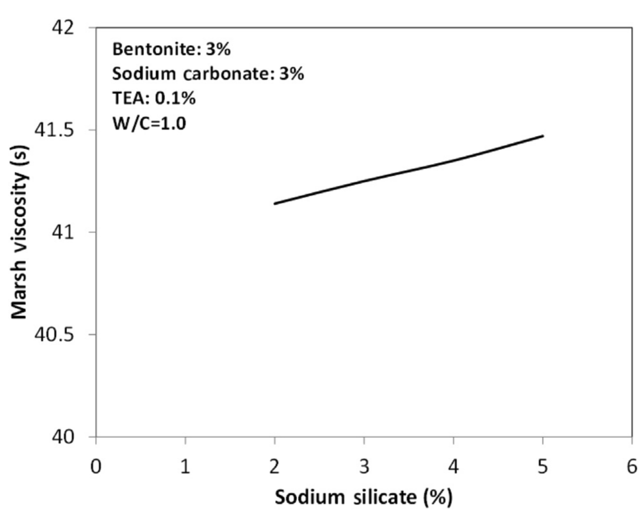

Fig. 11. Effect of sodium silicate on viscosity of the grout.

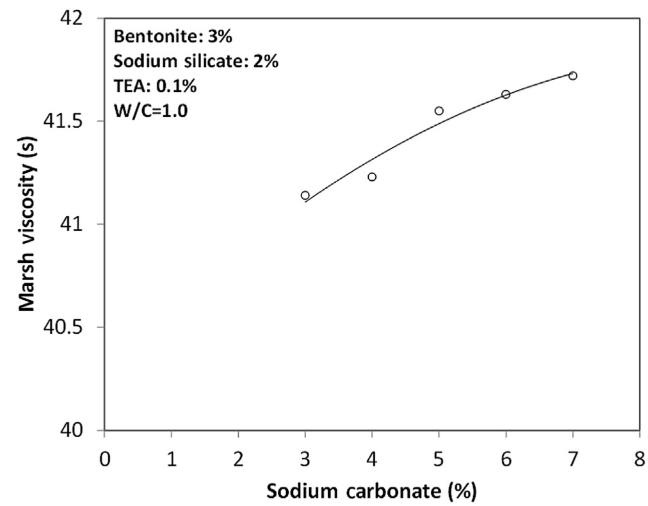

Fig. 12. Effect of sodium carbonate on viscosity of the grout.

desired bleeding value and high strength may be achieved by high percentages of sodium silicate but in such a case, rapid set of the grout can cause the grout not to be injectable. Apart from this point, it was observed that simultaneous usage of sodium silicate and sodium carbonate can reduce the bleeding value much more efficiently compared to the case of using only one of them (see Table 5). Therefore, using sodium carbonate as an effective reactant for sodium silicate is highly recommended. In this way, setting time of the grout mixture can be easily controlled as well.

In the end, it is important to note that the recommended percentages for the additives used cannot be changed since the aim is to have a certain value for all the four characteristics (bleeding, setting time, strength, and viscosity). In some projects, on the other hand, not all the four mentioned characteristics are important to be

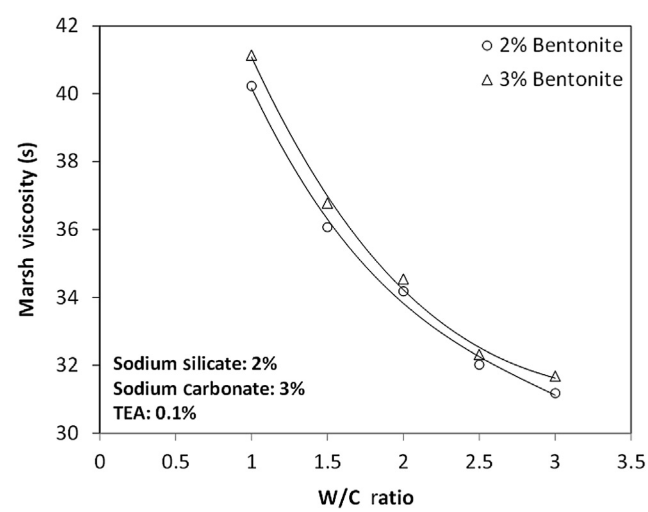

Fig. 13. Effect of water to cement ratio and bentonite on viscosity of the grout.

Table 8
Effect of each ingredient on the main characteristics of grout mixtures.

Material	Effect on grout characteristics			
	Bleeding	Strength	Setting time	Viscosity
Bentonite	↓	↓	–	↑
Sodium silicate	↓	↑	↓	↑
Sodium carbonate	↓	↑↓	↓	↑
TEA	↓	↓	↓	↓

Table 9
Optimized grout samples with different water to cement ratios.

Sample No.	T_1 (°C)	T_2 (°C)	Content (%)				W/C ratio	Viscosity (s)	Density (g/cm^3)	Bleeding (%)	Strength (kPa)	Setting time (min)
			Bentonite	Sodium carbonate	Sodium silicate	TEA						
1	20	21	2	3	2	0.1	1	40.23	1.48	0	337.3	60
2	20	21	2	3	3	0.1	1	40.27	1.48	0	368.7	45
21	20	21	2	3	2	0.1	1.5	36.07	1.37	<2	261.8	60
24	20	21	2	3	5	0.1	1.5	36.37	1.37	<2	327.5	50
44	20	21	2	3	5	0.1	2	34.02	1.28	<3	287.3	60
144	20	21	3	3	5	0.1	2	34.47	1.28	<1	211.8	55
64	20	21	2	3	5	0.1	2.5	33.3	1.23	4	220.6	70

considered in grout designation. In this situation, the designer has more choices to make about the percentage of each additive and the flexibility of design will be raised.

Therefore, all the 200 grout samples mentioned in Appendix should be considered by the designer when choosing an appropriate grout sample. Each grout sample may be appropriate for a project with its own specifications.

7. Conclusions

The main purpose of this paper was to create some optimized grout mixtures with minimized values of bleeding, appropriate setting time, maximum compressive strength, and in-range viscosity for a grouting project. This aim was achieved by using four different types of additives as bentonite, sodium silicate, sodium carbonate, and TEA. Each of these additives has its own advantage and disadvantage for being used in grout mixtures.

Bentonite is a known clay from montmorillonite group which has always been used for reducing the bleeding. However, it causes the strength of grout to decrease. Therefore, for cases where strength is important, an appropriate percentage of bentonite should be used in order to lessen the bleeding value, but not tangibly reduce the strength. In this study, using 2%–3% bentonite has been obtained as adequate for such purpose.

Sodium silicate is one of the main ingredients of chemical grouting which is also used for increasing the strength of cement-based grouts. It reduces the bleeding value as well. Of course, this additive may reduce the setting time up to a level in which the grout is set before being injected. Hence, high percentages of this additive cannot be recommended for a cementitious grout mixture. Appropriate percentages of this additive have been obtained in the range of 2%–5%. It is worth noting that in this proposed range, higher percentages of sodium silicate are more appropriate for grouts with higher water to cement ratios.

Sodium carbonate is a sodium salt of carbonic acid which can play the role of a reactant for sodium silicate. It reduces the bleeding of grout mixtures as well. Furthermore, its usage up to a boundary percentage has no negative effect on the strength of grouts. Therefore, for cases where strength is important, it is a preferred additive for reducing the bleeding value in comparison with bentonite. In this research, 3% sodium carbonate has been obtained as efficient for bleeding control.

TEA is a gas-producing material by which very small air bulbs are generated in the grout mixture. It reduces the surface tension of the grout mixture to be efficiently injected. Moreover, it reduces the bleeding value and prevents any shrinkage after the grout is set. Almost all the producers of this product make recommendations about its adequate percentage for generating sufficient air bulbs in the grout mixtures. In this study, 0.05%–0.1% of this additive was demonstrated as sufficient.

Finally, it is important to state that all the above-mentioned propositions are recommended for grout mixtures in which all the four characteristics of a grout (bleeding, setting time, strength, and viscosity) are important to be considered and optimization of the additive amounts was required for all the necessities to be satisfied. Therefore, any excessive decrease or increase for the amounts of these additives was prohibited. However, there are many projects in which not all the four characteristics are important for the purpose of grouting and this point is a real help in producing grout mixtures with wider availability of each characteristic.

Conflict of interest

The authors wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

Appendix

Table A1 shows all the results for the 200 grout samples in this study.

Table A1
The produced grout samples with their characteristics.

Sample No.	T_1 (°C)	T_2 (°C)	Content (%)				W/C ratio	Viscosity (s)	Density (g/cm^3)	Bleeding (%)
			Bentonite	Sodium carbonate	Sodium silicate	TEA				
1	20	21	2	3	2	0.1	1	40.23	1.48	0
2	20	21	2	3	3	0.1	1	40.27	1.48	0
3	20	21	2	3	4	0.1	1	40.33	1.48	0
4	20	21	2	3	5	0.1	1	40.42	1.49	0
5	20	22	2	4	2	0.1	1	40.41	1.49	0
6	20	22	2	4	3	0.1	1	40.52	1.49	0
7	20	22	2	4	4	0.1	1	40.89	1.49	0
8	20	22	2	4	5	0.1	1	41.25	1.49	0
9	20	23	2	5	2	0.1	1	41.05	1.49	<1

(continued on next page)

Table A1 (*continued*)

Sample No.	T_1 (°C)	T_2 (°C)	Content (%)				*W*/*C* ratio	Viscosity (s)	Density (g/cm^3)	Bleeding (%)
			Bentonite	Sodium carbonate	Sodium silicate	TEA				
10	20	23	2	5	3	0.1	1	41.35	1.49	<1
11	20	23	2	5	4	0.1	1	41.79	1.49	<1
12	20	23	2	5	5	0.1	1	42.25	1.5	<1
13	20	24	2	6	2	0.1	1	41.41	1.5	1
14	20	24	2	6	3	0.1	1	41.88	1.5	1
15	20	24	2	6	4	0.1	1	42.19	1.5	1
16	20	24	2	6	5	0.1	1	42.78	1.5	1
17	20	25	2	7	2	0.1	1	42.51	1.5	1
18	20	25	2	7	3	0.1	1	42.81	1.5	1
19	20	25	2	7	4	0.1	1	43.02	1.5	1
20	20	25	2	7	5	0.1	1	43.11	1.5	1
21	20	21	2	3	2	0.1	1.5	36.07	1.37	<2
22	20	21	2	3	3	0.1	1.5	36.45	1.37	<2
23	20	21	2	3	4	0.1	1.5	36.65	1.37	<2
24	20	21	2	3	5	0.1	1.5	36.37	1.37	<2
25	20	22	2	4	2	0.1	1.5	36.25	1.37	<2
26	20	22	2	4	3	0.1	1.5	36.44	1.37	<2
27	20	22	2	4	4	0.1	1.5	36.62	1.38	<2
28	20	22	2	4	5	0.1	1.5	36.73	1.38	<2
29	20	23	2	5	2	0.1	1.5	36.29	1.38	<2
30	20	23	2	5	3	0.1	1.5	36.32	1.38	<2
31	20	23	2	5	4	0.1	1.5	35.45	1.38	<2
32	20	23	2	5	5	0.1	1.5	35.64	1.38	<2
33	20	24	2	6	2	0.1	1.5	35.37	1.38	2
34	20	24	2	6	3	0.1	1.5	35.57	1.38	2
35	20	24	2	6	4	0.1	1.5	35.72	1.39	2
36	20	24	2	6	5	0.1	1.5	36.33	1.39	2
37	20	25	2	7	2	0.1	1.5	37.39	1.39	2
38	20	25	2	7	3	0.1	1.5	38.25	1.39	2
39	20	25	2	7	4	0.1	1.5	38.62	1.39	2
40	20	25	2	7	5	0.1	1.5	39.12	1.39	2
41	20	21	2	3	2	0.1	2	34.18	1.28	<3
42	20	21	2	3	3	0.1	2	34.37	1.28	<3
43	20	21	2	3	4	0.1	2	34.42	1.28	<3
44	20	21	2	3	5	0.1	2	34.56	1.28	<3
45	20	21	2	4	2	0.1	2	34.22	1.28	<3
46	20	21	2	4	3	0.1	2	34.35	1.29	<3
47	20	21	2	4	4	0.1	2	34.39	1.29	<3
48	20	21	2	4	5	0.1	2	34.45	1.29	<3
49	20	21	2	5	2	0.1	2	34.37	1.29	3
50	20	22	2	5	3	0.1	2	35.04	1.29	3
51	20	22	2	5	4	0.1	2	35.42	1.29	3
52	20	22	2	5	5	0.1	2	35.71	1.29	3
53	20	22	2	6	2	0.1	2	34.67	1.29	3
54	20	22	2	6	3	0.1	2	34.82	1.29	3
55	20	22	2	6	4	0.1	2	35.02	1.3	3
56	20	22	2	6	5	0.1	2	35.37	1.3	3
57	20	23	2	7	2	0.1	2	35.32	1.3	<4
58	20	23	2	7	3	0.1	2	36.12	1.3	<4
59	20	23	2	7	4	0.1	2	36.89	1.3	<4
60	20	23	2	7	5	0.1	2	37.28	1.3	<4
61	20	21	2	3	2	0.1	2.5	32.02	1.23	4
62	20	21	2	3	3	0.1	2.5	32.16	1.23	4
63	20	21	2	3	4	0.1	2.5	32.37	1.23	4
64	20	21	2	3	5	0.1	2.5	33.3	1.23	4
65	20	21	2	4	2	0.1	2.5	33.14	1.23	4
66	20	21	2	4	3	0.1	2.5	33.32	1.23	4
67	20	21	2	4	4	0.1	2.5	33.54	1.23	4
68	20	21	2	4	5	0.1	2.5	33.72	1.23	4
69	20	21	2	5	2	0.1	2.5	33.25	1.24	<5
70	20	22	2	5	3	0.1	2.5	33.36	1.24	<5
71	20	22	2	5	4	0.1	2.5	33.55	1.24	<5
72	20	22	2	5	5	0.1	2.5	33.79	1.24	<5
73	20	22	2	6	2	0.1	2.5	33.41	1.24	5
74	20	22	2	6	3	0.1	2.5	33.51	1.24	5
75	20	22	2	6	4	0.1	2.5	33.62	1.24	5
76	20	22	2	6	5	0.1	2.5	33.73	1.24	5
77	20	23	2	7	2	0.1	2.5	34.11	1.24	5
78	20	23	2	7	3	0.1	2.5	34.25	1.24	5
79	20	23	2	7	4	0.1	2.5	34.36	1.24	5
80	20	23	2	7	5	0.1	2.5	34.4	1.24	5
81	20	21	2	3	2	0.1	3	31.18	1.17	6
82	20	21	2	3	3	0.1	3	31.23	1.18	6
83	20	21	2	3	4	0.1	3	31.28	1.18	6

Table A1 (*continued*)

Sample No.	T_1 (°C)	T_2 (°C)	Content (%)				W/C ratio	Viscosity (s)	Density (g/cm^3)	Bleeding (%)
			Bentonite	Sodium carbonate	Sodium silicate	TEA				
84	20	21	2	3	5	0.1	3	31.33	1.18	6
85	20	21	2	4	2	0.1	3	32.12	1.18	<7
86	20	21	2	4	3	0.1	3	32.32	1.18	<7
87	20	21	2	4	4	0.1	3	32.41	1.18	<7
88	20	21	2	4	5	0.1	3	32.53	1.18	<7
89	20	21	2	5	2	0.1	3	32.27	1.18	8
90	20	22	2	5	3	0.1	3	32.33	1.18	8
91	20	22	2	5	4	0.1	3	32.45	1.19	8
92	20	22	2	5	5	0.1	3	32.55	1.19	8
93	20	22	2	6	2	0.1	3	32.33	1.19	8
94	20	22	2	6	3	0.1	3	32.37	1.19	8
95	20	22	2	6	4	0.1	3	32.47	1.19	8
96	20	22	2	6	5	0.1	3	32.65	1.19	8
97	20	22	2	7	2	0.1	3	32.45	1.19	8
98	20	22	2	7	3	0.1	3	32.69	1.19	8
99	20	22	2	7	4	0.1	3	33.18	1.19	8
100	20	22	2	7	5	0.1	3	33.45	1.2	8
101	20	21	3	3	2	0.1	1	41.14	1.48	0
102	20	21	3	3	3	0.1	1	41.25	1.48	0
103	20	21	3	3	4	0.1	1	41.35	1.48	0
104	20	21	3	3	5	0.1	1	41.47	1.48	0
105	20	22	3	4	2	0.1	1	41.23	1.48	0
106	20	22	3	4	3	0.1	1	41.36	1.48	0
107	20	22	3	4	4	0.1	1	41.39	1.48	0
108	20	22	3	4	5	0.1	1	41.42	1.49	0
109	20	23	3	5	2	0.1	1	41.55	1.49	0
110	20	23	3	5	3	0.1	1	41.58	1.49	0
111	20	23	3	5	4	0.1	1	41.63	1.49	0
112	20	23	3	5	5	0.1	1	41.69	1.49	0
113	20	24	3	6	2	0.1	1	41.63	1.49	<1
114	20	24	3	6	3	0.1	1	41.70	1.49	<1
115	20	24	3	6	4	0.1	1	41.82	1.49	<1
116	20	24	3	6	5	0.1	1	41.89	1.5	<1
117	20	25	3	7	2	0.1	1	41.72	1.5	<1
118	20	25	3	7	3	0.1	1	41.95	1.5	<1
119	20	25	3	7	4	0.1	1	42.19	1.5	<1
120	20	25	3	7	5	0.1	1	42.64	1.5	<1
121	20	21	3	3	2	0.1	1.5	36.78	1.37	<1
122	20	21	3	3	3	0.1	1.5	36.89	1.37	<1
123	20	21	3	3	4	0.1	1.5	36.95	1.37	<1
124	20	21	3	3	5	0.1	1.5	36.99	1.37	<1
125	20	22	3	4	2	0.1	1.5	36.88	1.37	<1
126	20	22	3	4	3	0.1	1.5	36.98	1.37	<1
127	20	22	3	4	4	0.1	1.5	37.12	1.37	<1
128	20	22	3	4	5	0.1	1.5	37.31	1.37	<1
129	20	23	3	5	2	0.1	1.5	36.99	1.37	<1
130	20	23	3	5	3	0.1	1.5	37.25	1.38	<1
131	20	23	3	5	4	0.1	1.5	37.4	1.38	<1
132	20	23	3	5	5	0.1	1.5	37.56	1.38	<1
133	20	24	3	6	2	0.1	1.5	37.13	1.38	<1
134	20	24	3	6	3	0.1	1.5	37.31	1.38	<1
135	20	24	3	6	4	0.1	1.5	37.52	1.38	<1
136	20	24	3	6	5	0.1	1.5	37.73	1.38	<1
137	20	25	3	7	2	0.1	1.5	37.22	1.38	<1
138	20	25	3	7	3	0.1	1.5	37.35	1.38	<1
139	20	25	3	7	4	0.1	1.5	37.54	1.38	<1
140	20	25	3	7	5	0.1	1.5	37.98	1.39	<1
141	20	21	3	3	2	0.1	2	34.54	1.28	<1
142	20	21	3	3	3	0.1	2	34.76	1.28	<1
143	20	21	3	3	4	0.1	2	34.92	1.28	<1
144	20	21	3	3	5	0.1	2	35.23	1.28	<1
145	20	21	3	4	2	0.1	2	34.85	1.28	<1
146	20	21	3	4	3	0.1	2	34.99	1.28	<1
147	20	21	3	4	4	0.1	2	35.23	1.28	<1
148	20	21	3	4	5	0.1	2	35.46	1.29	<1
149	20	21	3	5	2	0.1	2	35.11	1.29	<1
150	20	22	3	5	3	0.1	2	35.16	1.29	<1
151	20	22	3	5	4	0.1	2	35.22	1.29	<1
152	20	22	3	5	5	0.1	2	35.37	1.29	<1
153	20	22	3	6	2	0.1	2	35.34	1.29	1
154	20	22	3	6	3	0.1	2	35.56	1.3	1
155	20	22	3	6	4	0.1	2	35.67	1.3	1
156	20	22	3	6	5	0.1	2	35.81	1.3	1

(*continued on next page*)

Table A1 (*continued*)

Sample No.	T_1 (°C)	T_2 (°C)	Content (%)				W/C ratio	Viscosity (s)	Density (g/cm^3)	Bleeding (%)
			Bentonite	Sodium carbonate	Sodium silicate	TEA				
157	20	23	3	7	2	0.1	2	35.52	1.3	1
158	20	23	3	7	3	0.1	2	35.89	1.3	1
159	20	23	3	7	4	0.1	2	36.38	1.3	1
160	20	23	3	7	5	0.1	2	37.02	1.3	1
161	20	21	3	3	2	0.1	2.5	32.32	1.23	1
162	20	21	3	3	3	0.1	2.5	32.87	1.23	1
163	20	21	3	3	4	0.1	2.5	33.18	1.23	1
164	20	21	3	3	5	0.1	2.5	33.48	1.23	1
165	20	21	3	4	2	0.1	2.5	33.15	1.23	1
166	20	21	3	4	3	0.1	2.5	33.45	1.23	1
167	20	21	3	4	4	0.1	2.5	33.65	1.23	1
168	20	21	3	4	5	0.1	2.5	33.85	1.23	1
169	20	21	3	5	2	0.1	2.5	33.25	1.24	1
170	20	22	3	5	3	0.1	2.5	33.51	1.24	1
171	20	22	3	5	4	0.1	2.5	33.87	1.24	1
172	20	22	3	5	5	0.1	2.5	34.16	1.24	1
173	20	22	3	6	2	0.1	2.5	33.65	1.24	<2
174	20	22	3	6	3	0.1	2.5	33.97	1.24	<2
175	20	22	3	6	4	0.1	2.5	34.36	1.24	<2
176	20	22	3	6	5	0.1	2.5	34.68	1.24	<2
177	20	23	3	7	2	0.1	2.5	34	1.24	<2
178	20	23	3	7	3	0.1	2.5	34.35	1.24	<2
179	20	23	3	7	4	0.1	2.5	34.74	1.24	<2
180	20	23	3	7	5	0.1	2.5	35.36	1.24	<2
181	20	21	3	3	2	0.1	3	31.68	1.17	<3
182	20	21	3	3	3	0.1	3	31.87	1.18	<3
183	20	21	3	3	4	0.1	3	31.98	1.18	<3
184	20	21	3	3	5	0.1	3	32.09	1.18	<3
185	20	21	3	4	2	0.1	3	32.12	1.18	<3
186	20	21	3	4	3	0.1	3	32.18	1.18	<3
187	20	21	3	4	4	0.1	3	32.26	1.18	<3
188	20	21	3	4	5	0.1	3	32.35	1.18	<3
189	20	21	3	5	2	0.1	3	32.45	1.18	3
190	20	22	3	5	3	0.1	3	32.58	1.18	3
191	20	22	3	5	4	0.1	3	32.69	1.19	3
192	20	22	3	5	5	0.1	3	32.83	1.19	3
193	20	22	3	6	2	0.1	3	32.75	1.19	3
194	20	22	3	6	3	0.1	3	32.89	1.19	3
195	20	22	3	6	4	0.1	3	33.16	1.19	3
196	20	22	3	6	5	0.1	3	33.23	1.19	3
197	20	22	3	7	2	0.1	3	32.98	1.19	3
198	20	22	3	7	3	0.1	3	33.12	1.19	3
199	20	22	3	7	4	0.1	3	33.25	1.2	3
200	20	22	3	7	5	0.1	3	33.78	1.2	3

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