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Improvement of two-component grouts by the use of ground granulated blast furnace slag

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Highlights:

- GGBS improves grout stability against bleeding
- Increasing GGBS content extends gel time
- Gel time of the grout can be reduced by decreasing accelerator content
- Durability in water is improved by slag

Abstract

This work investigated the impact of a partial replacement of ordinary Portland cement (OPC) by ground granulated blast-furnace slag (GGBS) in two-component grouts. Maximum GGBS content reached 50%wt of the binder. Fluidity, open time, bleeding behaviour, gel time, compressive strength development and durability with respect to sodium sulfate were assessed. The impact of a variation of the volumetric ratio between the grout base and the accelerator (components A and B) was also considered. The results showed that increasing GGBS content led to multiple benefits of grouts behaviour. At fresh state, the bleeding was limited without decreasing the flowability. At hardened state, it increased the 28-day compressive strength, and improved the durability of the grouts in water/sodium sulfate solutions when used at 16% of the binder mass (mix designs based on jobsite trials).

Keywords: two-component grouts; ground granulated blast-furnace slag; flow time; gel time; compressive strength; sulfate resistance

1 Introduction

During the digging of a tunnel with a Tunnel Boring Machine (TBM), whether in soft or hard soil, an annular void is left between the segment lining and the soil. This empty space can cause significant ground settlement and must be filled rapidly using an appropriate grouting method (Sharghi et al., 2017). The grout must ensure the stability of surrounding strata and the tunnel by distributing the constraints. Most grouts used are cement-based as cement is readily available and inexpensive. This category includes several types of grout, including pure cement-based grouts, clay-cement mixes, grouts with filler, quick-setting grouts with controlled hardening or two-component grouts (AFTES, 1991). Two-component grouts are being increasingly used worldwide due to their numerous advantages and good adaptability to various types of soils (Dal Negro et al., 2019). In particular, they allow rapid hardening, which limits settlement risks (Sharghi et al., 2017). Traditionally, two-component grouts are based on a highly fluid Portland cement grout stabilized with bentonite, and a retarder to offer a long workability time (component A) (Shah et al., 2016). A hardening accelerator (Component B) is added just before the injection of the grout behind the tunnel segment lining, and leads to jellification of the overall mix in a few seconds.

A few scientific papers or tunnelling projects have so far reported the use of secondary cementitious materials (SCMs) in two-component grouts. A conference paper from 1950 presented characteristics of

grouts formulated with slag cement, bentonite and silicate (Florentin and Heriteau, 1950). Sculte-Schrepping and Breintenbücher (2019) demonstrated the interest of using ground granulated blast furnace slag (GGBS), fly ash, or metakaolin to replace some of the ordinary Portland cement (OPC). According to these authors, the higher calcium content of the slag, compared to the other Al-Si materials, allows faster jellification and higher compressive strength. Other recent papers have associated fly ash and GGBS for eco-friendly two-component grout design (Aboulayt et al., 2020; Song et al., 2020). However, grouts formulated in these studies used a lower water-to-binder ratio and a higher accelerator content than the mix-design presented in this work. One of the rare cases of a tunnelling project using SCMs in its two-component grout design is the Warsaw metro. A blended CEM IV/B-P cement, containing between 36 and 55% of natural pozzolan (AFNOR, 2012) was used (Peila et al., 2015).

GGBS is known to behave better than Portland cement in non-accelerated cementitious grouts containing bentonite (Trinh Quang, 2012). It can also help to enhance the durability of construction materials, particularly against sulfate attacks (Gollop and Taylor, 1996; Whittaker and Black, 2015). This last property could be of interest for tunnels passing through mineralogical strata of gypsum, which lead to the presence of sulfates in seepage water. Finally, it is generally recognized that GGBS improves the environmental impact of cement-based materials, due to its lower carbon footprint (Higgins, 2007).

The aim of this paper is to assess the feasibility of using GGBS for two-component grouting, by presenting the results of an experimental campaign that shows the effect of a partial replacement of Ordinary Portland Cement (OPC) by GGBS. It focuses on three main aspects of grout properties that must be managed: fresh state characteristics, compressive strength development and durability in the presence of sulfates.

This study took place in the context of the Lyon-Turin railway connection project, on the Saint-Martin-La-Porte 4 (SMP4) jobsite. It mainly consisted of constructing a 9 km reconnaissance tunnel, excavated with a tunnel-boring machine (Single Shield Hard Rock TBM), from Saint-Martin-La-Porte, France, in the direction of Italy. The request was to design two-component grouts using GGBS and responding to particular specifications determined by the jobsite (Table 1). This exploratory tunnel, bored in the axis of the base tunnel, was foreseen to face geological formations potentially charged with aggressive water. Thus, a sulfate resistant cement or a binder able to safely maintain its properties in chemically aggressive environments (XA2 or XA3 according to European standards (AFNOR, 2014)) had to be implemented in the mix design of component A. Figure 1 presents a picture of a two-component grout injection in the annular void of this tunnel.

Table 1: Specifications for grout properties, considered as acceptable quality parameters on jobsite

Property	Criteria
Components A* only	
Marsh cone flow time (nozzle 4.75mm)	30 to 45 seconds
Open time	No setting at 3 days
Bleeding	< 3 % at 3h < 8 % at 24h
Components A+B**	
Gel time	5 to 15 seconds
Compressive strength	> 0.25 MPa at 1h > 0.45 MPa at 3h > 0.70 MPa at 24h > 1.20 MPa at 28 days

*Component A: cement grout stabilized with bentonite + retarder

** Component B: hardening accelerator

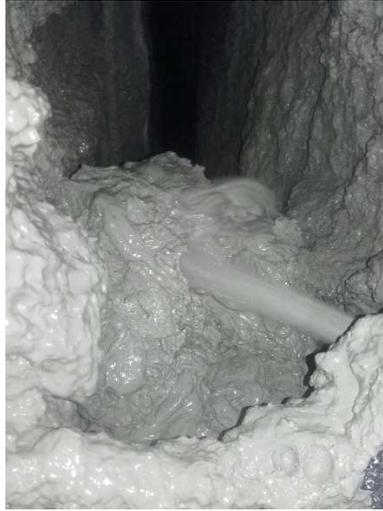


Figure 1: Detail of two-component injection in the annular void at the TBM launching chamber of SMP4 jobsite

2 Materials

2.1 Raw materials

As already stated, two-component grouts are made of:

- Component A, composed of cement and water stabilized with bentonite. The bentonite enhances the grout's stability and homogeneity by providing the necessary thixotropic consistency. It helps to minimize bleeding of the grout that could occur in the pipes when the grout stops flowing. Component A also includes a retarder to increase the open time. Thus, in the frequent cases when the mixing plant is not on the jobsite, the grout can be transported over several kilometres before being injected. The role of the retarder is to inhibit the mix setting and so guarantee a long open time, above 72 hours. It allows more flexibility for the utilization of the grout, particularly at times when the Tunnel Boring Machine (TBM) has standstills of various nature.
- Component B, which is a hardening accelerator added just before the injection to enable the grout to gel.

In this study, Component A was made of a CEM I 52.5 R and a class A GGBS, both complying with European standard requirements (AFNOR, 2006, 2012, 2014). It was completed by sodium bentonite, a commercial retarder (Mapei Mapequick CBS System 1) and water coming from a drilling well on the jobsite. The accelerator used as component B was an alkali-silicate solution (Mapei Mapequick CBS System 3), with a high silica-to-sodium ratio (> 3). The specific gravity of all the materials used is given in Table 2. Chemical and physical properties of CEM I and GGBS are indicated in Table 3.

Table 2: Specific gravity of the raw materials

	CEM I	GGBS	bentonite	retarder	accelerator	water
Specific gravity (kg/m ³)	3.1	2.9	2.5	1.22	1.37	1

Table 3: Chemical and physical properties of the CEM I and GGBS

	Chemical composition (%)											Blaine specific surface (cm ² /g)
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	TiO ₂	SO ₃	Cl ⁻	S ²⁻	Na ₂ O	K ₂ O	
CEM I	20.4	4.9	1.9	63.8	1.67	0.2	2.8	0.05	-	0.34	0.76	4650
GGBS	37.7	10.2	0.6	43.8	6.4	0.7	0.1	0.02	0.7	0.21	0.28	4450

2.2 Mix designs

Formulations of two-component grouts were established on the basis of one cubic metre of total grout (components A+B), considering a typical volumetric ratio B/A of 6.5 %. The reason for using a volumetric ratio was related to the dosing of the components on the jobsite, which was mainly based on flow rates.

Mix designs for component A are given in Table 4, where the composition of each formulation is noted both in volume and in mass per cubic metre. Names of formulations are expressed in mass percent of GGBS (S) relative to the binder (GGBS+CEM I), associated with the B/A ratio (B) once accelerator is added (and when needed). For instance, "S23-B6.5" means that the binder in component A was composed of 23% GGBS and 77% CEM I, by mass, and that the volumetric ratio B/A was equal to 6.5%.

Table 4: Formulations of two-component grouts

Name	Unit	Component A						Total A	Component B Accelerator (vol%)
		CEM I	GGBS	Bentonite	Retarder	Water			
S0-Bxx*	kg/m ³	244	-	32	3.8	905	1185	6.5	
	L/m ³	79	-	13	3.1	905	1000		
S16-Bxx*	kg/m ³	201	37	37	3.9	904	1184	6.5	
	L/m ³	65	13	15	3.3	904	1000		
S23-Bxx*	kg/m ³	175	52	35	4.0	908	1174	5.5 ; 6.5	
	L/m ³	56	18	14	3.3	908	1000		
S30-Bxx*	kg/m ³	158	68	35	4.0	908	1173	2.0 ; 3.0 ; 3.5 ; 4.0 ; 4.5 ; 5.0 ; 5.5 ; 6.0 ; 6.5 ; 7.0 ; 7.5 ; 8.0 ; 9.4 ; 10.0	
	L/m ³	51	23	14	3.3	908	1000		
S40-Bxx*	kg/m ³	135	90	35	4.0	908	1172	4.5 ; 5.5 ; 6.5	
	L/m ³	44	31	14	3.3	908	1000		
S50-Bxx*	kg/m ³	112	114	35	4.0	907	1172	4.5 ; 5.5 ; 6.5	
	L/m ³	36	39	14	3.3	907	1000		

*_{xx} corresponds to the volumetric ratio B/A of the two component grout. Ex: S23-B6.5 contains 23wt% of GGBS in component A's binder and has a B/A=6.5 vol%.

3 Methods

3.1 Fresh state properties

To simulate the reality of the jobsite mixing plant, 55 g/L of a previous component A was systematically added to the water (since the jobsite mixer always contains a little of the previous batch when a new one is cast). To be as consistent as possible in comparing all the formulations in this study, the former A-component used was always S0 for formulations containing strictly less than 23% of GGBS in their binder, and S23 for those containing 23% GGBS or more.

Every component A prepared in this study was made respecting the following procedure, using a RAYNERI Turbotest 220 stirrer:

- Addition of 55g/L of A-component from formula S0 or S23 to each new batch
- Addition of water to a pot with a capacity of at least 5 L. Start the stirrer on slow speed and progressively increase it to reach 2500 rpm. A mixing time of at least 20 seconds is required to obtain a homogeneous solution.
- Addition of bentonite and mixing for 120 seconds at 2500 rpm
- Addition of CEM I and mixing for 20 seconds at 3350 rpm
- Addition of GGBS to formulations containing slag and mixing for 20 seconds at 3350 rpm.
- Addition of the retarding agent and mixing for 30 seconds at 3350 rpm.

Bleeding measurements were achieved by pouring 250 mL into a graduated cylinder. The tube was then covered with a Parafilm to avoid evaporation and was not moved for the duration of the tests. Density was also measured at the same time. The percentage of bleeding was determined after 3 and 24 hours, by looking at the volume of water separated from component A, and calculated following equation 1 (separated water was not thrown away between two measurements).

$$\frac{Vol_{separated\ water}}{Vol_{total(=250ml)}} \times 100 \quad (1)$$

The fluidity of component A was evaluated, from the beginning up to 72 h, by means of a standard Marsh cone with a 4.76 mm nozzle. The cone was filled through its sieve until component A reached the grid. Fluidity was measured as the flow time needed for the grout to fulfil a 1 L pot. According to experience on jobsites, common values for fluidity of this type of grout range from 35 to 45 seconds.

Gel time represented the time after which the component A stopped moving when mixed with the component B. It was measured using 200 g of component A and a corresponding quantity of component B, depending on the B/A volumetric ratio chosen. The mass of component B (q_B) was determined following equation (2).

$$q_B = \frac{q_A}{d_A} \times "B/A" \times d_B \quad (2)$$

where d_A corresponds to the density of component A determined while preparing the graduated cylinder for bleeding evaluation, d_B is the density of the accelerator (1.37 kg/L), q_A is the mass of component A used for the test and "B/A" is the chosen volumetric ratio expressed as a percentage. The procedure used to evaluate the gel time was carried out according to the proposition by Todaro et al. (2019). Common values for gel time with B/A=6.5 % are in the range of 6 to 12 seconds, according to experience on jobsites.

The laboratory procedure used to evaluate the gel time is described below:

- Fill a first pot with q_A
- Fill a second pot with q_B
- Pour the component A from the first pot into the one containing component B. Start the chronometer when the two components come into contact.
- Quickly transfer the mix of two components from one pot to another until the mix becomes a gel. Stop the chronometer as soon as the two-component grout ceases to move.

3.2 Hardened state properties

The mixing of components A and B for compressive strength tests followed a protocol similar to the one for gel time measurement. The exception was that the mixtures were transferred a maximum of three times from one pot to another to ensure good homogenization, and then poured into a polystyrene mould designed for 4x4x16 cm³ prisms, before the gelation of the grouts. Samples were prepared one prism at a time, using 350 g of component A. The corresponding quantity of component B was calculated as mentioned above. Directly after the moulds had been filled, they were covered with a plastic film and kept at 20°C for 24 h. After 1 day, samples were demoulded and placed in a water tank at 20°C until tested.

Early compressive strength tests (before 24h) were performed with an IMADA DS2-1000N digital force gauge, using a disc-shaped contact surface (\varnothing 16.3 mm). The penetrometer equipped with its contact tip was manually pressed on the surface of the grout, directly in the polystyrene mould. Strength was then calculated with three compressive trials on a single bar.

At 24 hours of hydration and later, grout bars were cut into three pieces with a hacksaw so that three compressive tests could be carried out per prism. A press with a contact surface of 40x40 mm² was used, at a loading speed of 0.1 kN/s.

3.3 Durability properties

It was decided to assess the durability of grouts in case of external sulfate attacks, since geological formations surrounding the tunnels on the Lyon-Turin jobsite could lead to a significant concentration of SO_3 in ground water. In cement systems, sulfate attack can be attributed to sodium, magnesium and calcium sulfate salts. However, since calcium sulfate has a limited solubility at ambient temperature ($\approx 1.4 \text{ g/L SO}_4^{2-}$) compared to sodium sulfate ($\approx 94 \text{ g/L SO}_4^{2-}$) (Al-Amoudi, 1995), it was decided to use sodium sulfate, as is traditionally done in laboratory studies.

The impact on resistance to external sulfate attack of a partial replacement of CEM I by GGBS in two-component grouts was assessed on samples from two formulations, S0 and S16. This conservative choice was made due to the mix designs tested on real jobsite. A higher GGBS content would probably lead to a higher durability resistance. Three different environments were retained to assess two-component grouts durability. The samples were immersed for one year, in solutions composed of tap water and sodium sulfate solutions (Na_2SO_4) at concentrations of 600 and 3000 mg/L of SO_3 , which were kept at 20°C . Concentration of 600 mg/L was chosen as it represented the mean value of sulfate concentration in surrounding ground water for the tunnel. A higher concentration in SO_3 was also chosen to accelerate the sulfate attack, as it is often seen in accelerated testing. Conservation in water alone was used as a reference to separate the effects of water from those of the sulfates. Samples were stored in 25 L tanks and all solutions (including water) were renewed every 30 ± 2 days.

Three samples were cast for each formulation and each type of conservation to evaluate their dimensional and mass variations with time, following the procedure below:

- The grouts were cast in the moulds, as for compressive strength measurement, and were kept in their mould, covered by a plastic plate, for 24 hours in a room at 20°C . The exception here was that the moulds were made of oiled metal and that stainless-steel bolts were attached to each end of each prism to enable the subsequent dimensional measurements to be made.
- After 24 hours, the samples were demoulded and put into water tanks for 28 days to ensure their good hydration before they underwent the process of accelerated degradation (sulfate solutions).
- After 28 days, samples exposed to sulfate attack were placed in their specified solution tank while some the grout prisms were kept in a tank of tap water.
- At regular intervals, mass was measured with a balance having an accuracy of 0.1 g, while dimensional variations were measured with a retractometer (calibration with an Invar standard bar).

To assess the effect of a partial replacement of CEM I by GGBS on the durability of two-component grout formulations in the environments mentioned above, $4 \times 4 \times 16 \text{ cm}^3$ samples were kept to follow the evolution of their compressive strength.

4 Results

4.1 Fresh state properties

4.1.1 Fluidity and open time

Influence of GGBS content

Figure 2 presents the results of the initial fluidity ($t_0+5\text{min}$) followed by the Marsh cone for all the grouts composed of components A only (i.e. without the accelerator – component B). Mixtures S23 to S50 were also monitored at 24, 48 and 72 h.

It can be seen that the initial fluidity of the grouts was systematically around 40 s and was not influenced by the GGBS content, up to 50% of replacement of CEM I. The same observation could be made for later age monitoring, with no effect of the GGBS content between 23 and 50% of replacement, although the overall flow time decreased to around 36 s after the initial measurements. This lower flow time, sign of an

improvement of the fluidity, remained constant between 24 h and 72 h, and was probably due to the effect of the retarder used to prolong the open time of component A of the grout (Collier et al., 2016). The retarder type and content have an important impact on this phenomenon (rather than the choice of binder), and this might explain the contradictory results found by Antunes (2012), who measured the flow time of a component A grout at 1, 3 and 8 hours after mixing and observed an increase in viscosity over time.

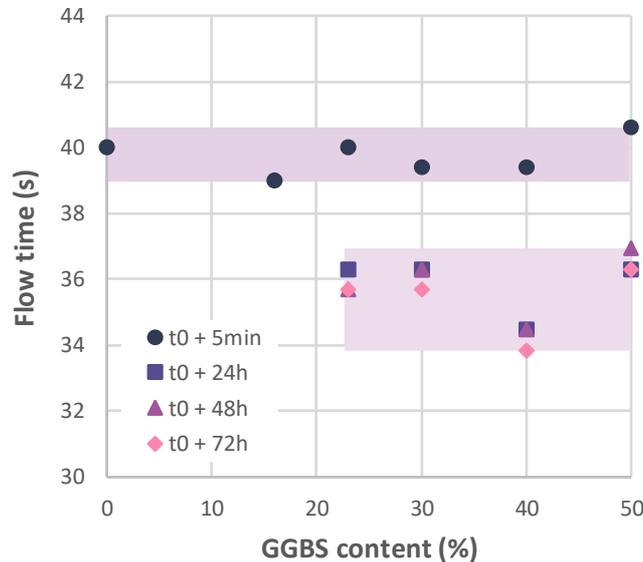


Figure 2: Flow time up to 72 h, measured by Marsh cone, of grouts composed of component A, for increasing GGBS contents (S0 to S50)

Influence of retarder content

Since GGBS is known for its relatively slow hydration rate in a cementitious system, it was decided to investigate the effect of a reduction of retarder content on the evolution of the flow time of the grouts.

Table 5 presents the results of Marsh cone flow time for components A with 30%wt GGBS and various quantities of retarder (from 0 to 4.0 kg/m³), the highest content being the applied retarder dosage used on site.

Decreasing the retarder content from 4.0 to 3.2 kg/m³ had no effect on the fluidity, which remained at a similar level for up to 72 h. The effect was much more pronounced for retarder contents of 1.6 kg/m³ and less, as the grouts had already set at 24 h, meaning that they could not be kept fluid for long enough to be used on the jobsite. It was also noted that the total absence of retarder led to an increase in the initial flow time (46 s vs 36-39 s), probably due to the presence of a plasticizer in the commercial retarder (e.g. lignosulfonate), which helped to improve the fluidity of the grout. From these results, it was concluded that a slight optimization of the retarder content was possible, to reach values equal to or lower than those used in pure CEM I grouts (3.8 kg/m³, as seen in Table 4).

Table 5: Effect of the retarder content in grout composed of component A, based on the measurements of flow time using Marsh cone– Mix design S30 with 30%wt GGBS in the binder – R0 to R4.0 indicates the retarder content in kg/m³, R4.0 being the reference.

Marsh cone fluidity	A-component			
	S30-R0	S30-R1.6	S30-R3.2	S30(-R4.0)
t ₀ + 5min (s)	46	36	39	39
t ₀ + 24h (s)	set	set	36	36
t ₀ + 48h (s)	set	set	36	36
t ₀ + 72h (s)	set	set	36	36

4.1.2 Bleeding behaviour

Figure 3 shows the bleeding behaviour of components A up to 50% of GGBS, monitored during 72 h. All grouts exhibited less than 3% bleeding after 3 hours, which was lower than the limit specified (Table 1). At 24 h, only the grout S0 without GGBS had a bleeding value (9%) slightly above the recommended 8%.

Effect of GGBS content

The general tendency was a decrease of the bleeding when GGBS replaced CEM I, especially when the extreme compositions (S0 without GGBS vs S50 with 50%wt GGBS) were compared: 2% bleeding vs 0% at 3h, and 9% vs 5% at 24h, for G0 and G50, respectively. It was thus obvious from these results that high GGBS contents improved the stability of the grout against sedimentation. Intermediate GGBS contents (16 to 40%) also enabled an improvement in the bleeding behaviour, but to a lesser extent and without the difference between the various mixtures being clearly distinguishable.

Effect of the absence of former grout

As explained in 3.1, almost all grouts made of components A were mixed with 55 g/L of a previous grout batch, to simulate the reality of the jobsite mixing plant. To assess the impact of this pollution, a formulation containing 23% of GGBS without a fraction of the previous grout (S23_{np} – empty markers on Figure 3) was cast and bleeding was compared with that of the S23 mixture. It can be seen that the presence of the previous grout in the new mixtures, even in a small quantity (approx. 5.5%), had a significant impact on the capacity of the grout to resist bleeding. At 3h, no bleeding was seen for S23_{np}, compared to 1% for S23. For later ages, the volume of separated water was 43-50% lower for S23_{np} than S23. This behaviour might be related to an increase of the pH and, more generally, to a higher ion content in the initial pore solution, due to the addition of the former grout to the mixing water. To support this hypothesis, pH and electric conductivity of mixing water were measured. Clean water had a pH equals to 8.1 and a conductivity of 690 $\mu\text{S}/\text{cm}$ while water polluted by a former grout had a pH of 11.7 and an electric conductivity of 2400 $\mu\text{S}/\text{cm}$. It thus means that a mixing water containing a former grout contains more ions in solution. Abu-Jdayil (2011) has shown that sodium bentonite-water dispersions exhibited a lower apparent viscosity when salts were added. Moreover, salts with divalent cations (such as Ca^{2+} , strongly present in cement grouts) had a greater impact on the viscosity drop than those with monovalent cations. The presence of electrolytes disrupts the network structure of bentonite particles (Kelessidis et al., 2007), which could lead to greater bleeding in our case.

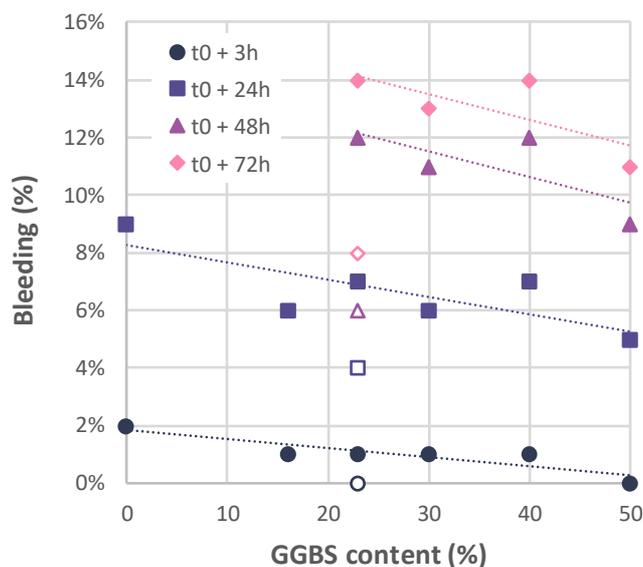


Figure 3: Bleeding behaviour of grouts composed of component A for increasing GGBS content (S0 to S50) – full symbols indicate that the grout was premixed with a former grout while empty symbols indicate that it was not.

4.1.3 Gel time

When the grout made of component A is ready to be injected behind the tunnel segment lining (between 0 and 72 h after initial mixing), component B is added to suddenly accelerate the hardening by a jellification of the overall mix in a few seconds. The mechanism of jellification is related to a quick reaction of the rapidly available silicon dissolved from the activator (e.g. sodium silicate), with the calcium in solution released from the dissolution of cement compounds (C_3A , C_3S , calcium sulfates), to form a gel of calcium silicates like C(-A)-S-H (Song et al., 2020).

Influence of the B/A ratio

For a given accelerator (e.g. commercial admixture based on sodium silicate with fixed dry extract and Si/Na ratio), the modification of the volumetric ratio between component B and component A enables a jellification time to be targeted, here between 5 and 15 seconds (Table 1).

Figure 4a presents the gel time of S30 mixtures for B/A ratios ranging between 2% and 10%. In that range, a fair linear relationship with a positive slope was obtained, meaning that an augmentation of the accelerator content led to an increase in the jellification time. At first sight, this result might seem unexpected, as the accelerator was used precisely to cause jellification of the grout. However, the same trend can be found in other works presented in the literature (Pellegrini and Perruzza, 2009; Todaro et al., 2019). The faster jellification associated with the reduction of the accelerator content in two-component grouts was probably due to an increase of Si/Ca ratio in the solution.

According to the specification given earlier (Table 1), an accelerator content between 3 and 7% should lead to an acceptable gel time for the jobsite here. However, it was noted that, for B/A lower than 4%, the jellification of the grout was only partial, meaning that there was not enough accelerator to mix the two components homogeneously, and the final mixtures stayed partly liquid with lumps of gel. Considering this, a B/A ratio between 4 and 7% is recommended to satisfy the gel time specification on a formula with 30% of GGBS replacing CEM I.

Influence of the GGBS content

Figure 4b reports the gel time of two-component grouts containing up to 50% of GGBS, for B/A of 4.5, 5.5 and 6.5%. On the one hand, it can be seen that all mixtures between 0 and 40% of GGBS met the required specifications for a gel time of less than 15 seconds. Only grout S50 was beyond the limit for high B/A ratios, but a lower accelerator content (4.5%) enabled this mixture to respect the desired target. On the other hand, there was a clear tendency for an increase of the GGBS content to lead to longer gel times, following a non-linear evolution. This meant that (a) for a given GGBS content, less component B was necessary to decrease the gel time (as already shown in Figure 4a); (b) for a targeted gel time, less component B was needed as the GGBS content increased. The increase of the GGBS content can thus bring economic and environmental advantages, as the accelerator based on sodium silicate is an expensive product, which also has a significant carbon footprint (Fawer et al., 1999).

The mechanisms explaining the effect of the GGBS were probably related to its kinetics of reaction. When blended with Portland cement, GGBS participation to heat of hydration starts after 15 hours, suggesting its lower solubility at early age (Steger et al., 2021). Also, GGBS is considered to have a congruent dissolution (Snellings, 2013). Therefore, As GGBS was less soluble and contained less calcium than CEM I, its addition by substitution for cement reduced the Ca^{2+} in solution. Thus, the Si/Ca ratio increased in the two-component grouts, as in the case of an augmentation of the B/A ratio, and it took a longer time to create a jellified network of calcium silicates. This phenomenon has already been observed by Song et al.(2020) as they compared the jellification of a pure cement grout to a GGBS-Fly Ash based grout, both activated by sodium silicate. They also showed that decreasing the Si/Na ratio of the component B could help to reduce the gel time of two-component grouts without Portland cement.

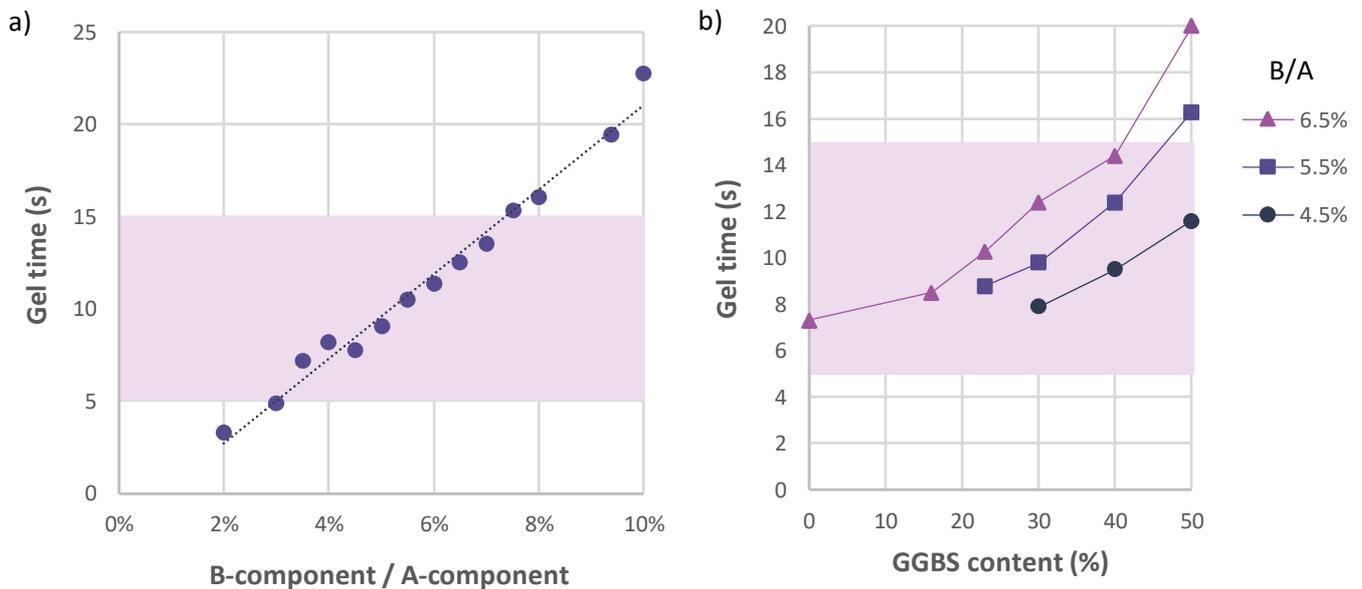


Figure 4: Gel time vs a) B/A ratio – Formulation S30; b) GGBS content

Evolution of gel time with the ageing of component A

Since component A can remain in the pipes in the tunnel for several hours or even days, the impact of its ageing on jellification time was a point of interest. Tests were performed with the S23 component A and 6.5% B/A (Figure 5a). For this study, all materials were carefully stored in a room at 20 ± 0.5 °C before and after mixing. During the 1st hour, the gel time remained approximately constant at 11 s, but it evolved to reach a value around 14.5 seconds, i.e. at the limit of the specification of 15s. This 3.5s increase in jellification time between 0 and 48 hours might have been due to a lower calcium availability. Clays, such as bentonite, are subject to ion exchange (Velde, 1992). Calcium can replace sodium on or in its double layers. Thus, it would increase the Si/Ca ratio, leading to a higher jellification time in the same manner as for B/A augmentation.

Influence of the temperature of component A on gel time

Several tunnelling projects where these grouts are used are deeply buried under the ground at temperatures often warmer than 20°C, as is the case in the Lyon-Turin project, with an average temperature of 28°C. However, the preparation and storing of the grouts, as well as their possible transportation for kilometres before injection, imply that the component A (grout without the accelerator) might be subjected to higher or lower temperatures, depending on the season concerned. Therefore the effect of the temperature at which component a was stored was assessed, by keeping the grout at approximately 10 °C, 20 °C or 30 °C for 24 h after mixing and before the incorporation of the accelerator (component B).

Figure 5b presents the effect of the temperature of S30 components A on jellification time for three B/A ratios. The results show a significant effect with an increase of the gel time with decreasing temperature. On average, the difference of gel time with one component A at 10 °C and another at 20 °C was almost 7 seconds, while the difference was lower (approximately 3.5 seconds) between 20°C and 30°C. The fact that grouts were kept at the desired temperature for 24 h prior to the trial explains the relatively long gel time.

The high values of gel time at lower temperature were probably due to the lower reactivity of GGBS, as mentioned earlier. As the dissolution of GGBS decreases with the temperature, fewer Ca ions are available in the pore solution, thus limiting the formation of calcium silicate compounds when silicon is provided to the system by the accelerator. The opposite happens at higher temperature. For each temperature levels, curves of gel time as a function of accelerator content were approximately linear and parallel. Thus, it would mean that the effect of B/A ratio was independent to the effect of temperature.

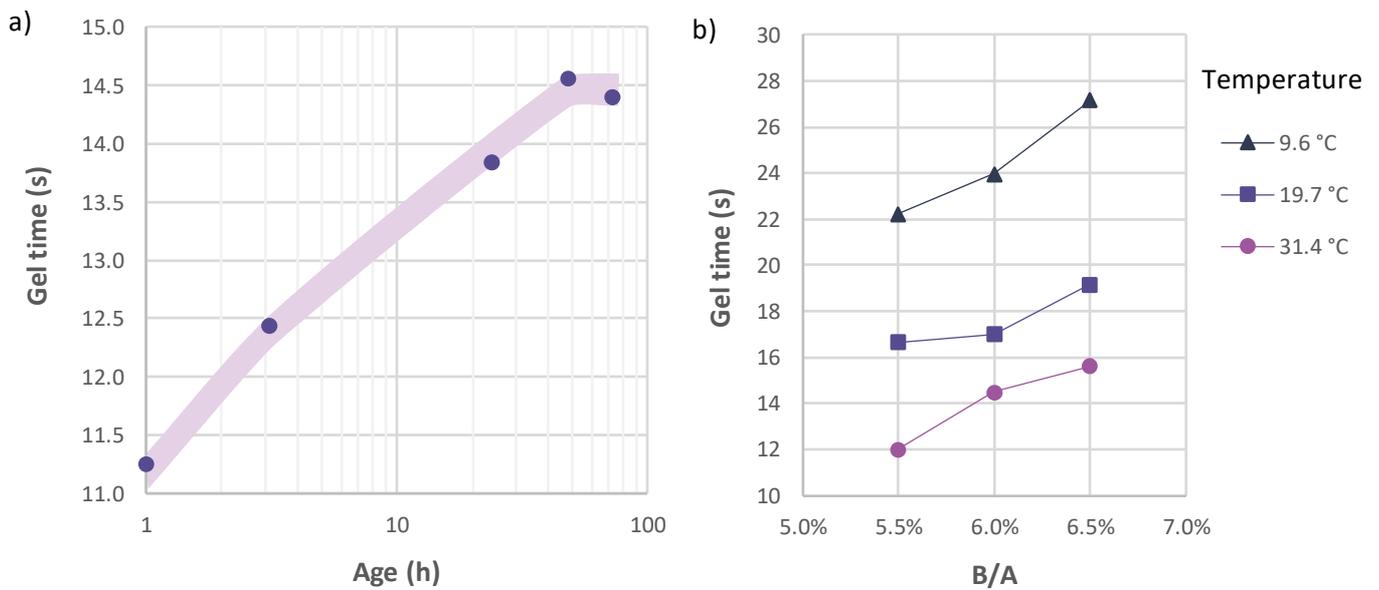


Figure 5: Gel time according to a) the age of component A – Formulation S23-B6.5 - b) the temperature of component A – Formulation S30-B5.5 to S30-B6.5

4.2 Hardened state properties

4.2.1 Early age behaviour

Figure 6a presents the early age compressive strength (between 1 and 24 hours) of formulations S0 and S16. Results at 1 hour are identical for both grouts, with a compressive strength of 0.4 MPa, and are superior to the required specification of 0.25 MPa. At 3 hours and 24 hours, a slight difference of 0.1 MPa appears between formulations but is within the experimental error. The results are higher than the minimum requirements, 0.45 and 0.70 MPa at 3 and 24 hours, respectively, with strengths in the 1.1-1.2 MPa range for both mixtures.

4.2.2 Effect of the GGBS content on compressive strength

Figure 6b shows the compressive strengths at 1 and 28 days of grouts containing from 0 to 50% GGBS in their binder and a B/A ratio fixed at 6.5%. At 1 day, it appeared that increasing the GGBS content slightly decreased the strength, by a mean value of 21%. However, the strength values were stable for grouts containing between 23 and 50% GGBS and were above the minimum requirement at this age (0.7 MPa). The beneficial effect of GGBS was mainly visible at 28 days, and significantly increased with the GGBS content. Compared to the reference grout containing only Portland cement (S0-B6.5), the S50 mix was almost twice as strong at this age.

Globally, it appeared that the replacement of OPC by GGBS slightly decreased the early strength, but led to a significant improvement in long-term strength. This may suggest a better durability for two-component grouts containing GGBS.

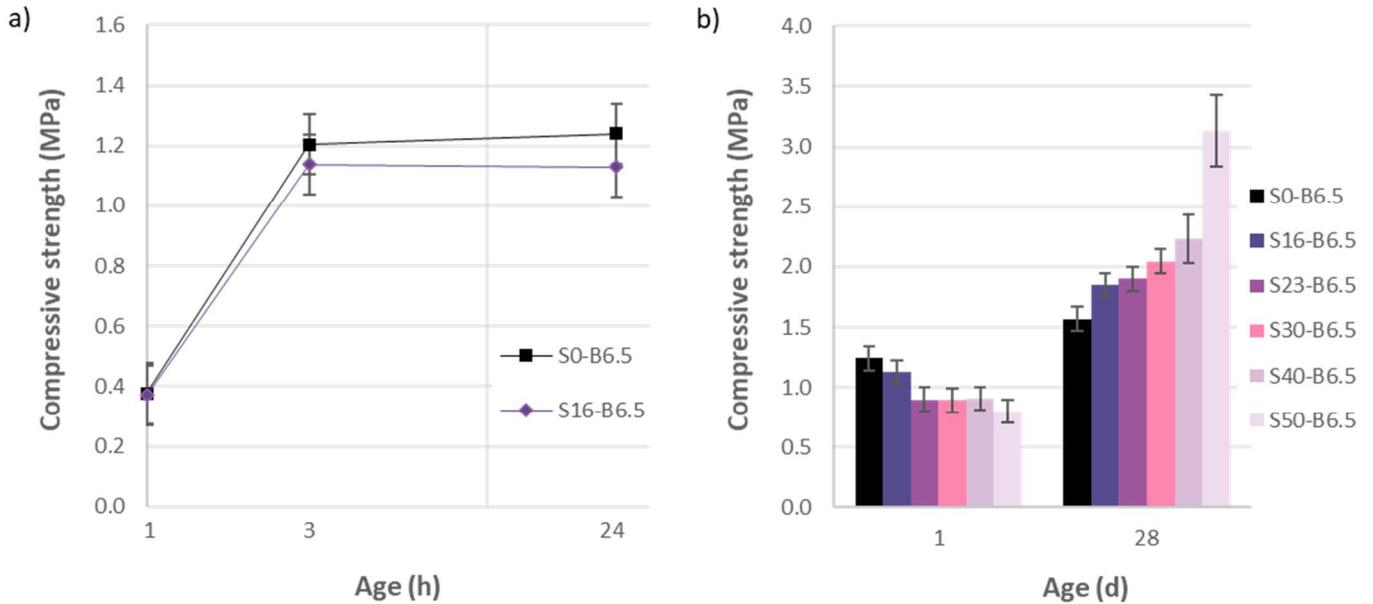


Figure 6: Compressive strength: a) between 1 and 24 hours of mix designs S0-B6.5 and S16-B6.5 - b) at 1 and 28 days of two-component grouts S0-B6.5 to S50-B6.5 containing from 0 to 50% GGBS in their binder – 4x4x16 cm³ bars kept in a water tank at 20°C after demoulding at 24h

4.2.3 Influence of B/A ratio

Figure 7 presents the influence of the volumetric ratio between components A and B (called B/A, varying between 5.0% and 7.5% here) on compressive strength development of two-component grout S30 (containing 30% by mass of GGBS in its binder).

Higher strengths were systematically obtained for a higher B/A ratio (i.e. for a higher accelerator content), whatever the age of the grout bars. The increase in B/A ratio led to a higher alkalinity of the solution, which improved the GGBS reaction. However, adding too much accelerator can also decrease compressive strength because of an increase in water content. (Song et al., 2020).

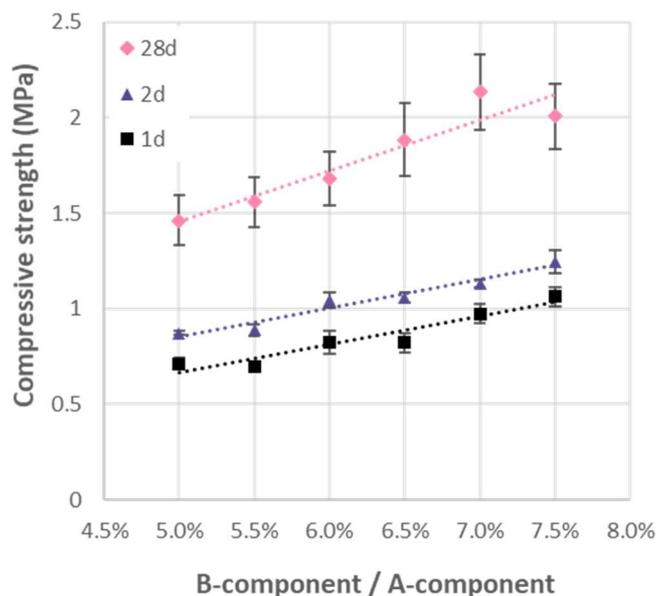


Figure 7: Compressive strength at 1, 2 and 28 days of two-component grouts depending on their B/A ratio – Mix design S30-B5.0 to S30-B7.5 – 4x4x16 cm³ bars kept in a water tank at 20°C after demoulding at 24h

4.3 Durability with respect to water and sulfate attack

The Lyon-Turin tunnelling project crosses geological zones potentially affected by circulation of aggressive sulfatic waters, so it was decided to assess the impact of a partial replacement of OPC in two-component grouts by GGBS, which is particularly efficient against sulfate attack (Gollop and Taylor, 1996). Due to industrial constraints, only two formulations of grouts were studied in this part: S0 (without any slag) as a reference and S16 (with 16%wt of GGBS in the binder). For both formulations, the B/A ratio was kept equal to 6.5%.

4.3.1 Behaviour of two-component grouts in water

Two-component grouts have been described as sensitive to groundwater corrosion (Wang et al., 2019; Song et al., 2020). In order to separate the effects of the immersion in water from the sulfate reactions, a first series of samples were immersed in a tank of tap water at 20°C to follow the possible consequences of this immersion on mass and dimensional variations, as well as on compressive strength evolution, up to 1 year.

Swelling and mass evolutions

Figure 8a and 8b present the swelling and mass gain, respectively, of the two-component grouts S0-B6.5 and S16-6.5, kept for 1 year in tap water at 20°C. On the one hand, it can be seen from Figure 8a that the expansion increased sharply during the first few weeks due to water intake, and then slowed down to reach a value around 0.26% at 1 year. No difference was observed between the two formulations and a replacement of 16% OPC by GGBS did not in any way affect the length deformation of samples kept in water.

On the other hand, the mass of water taken up by the grout samples differed between formulations S0-B6.5 and S16-B6.5. Reference grout S0-B6.5 showed a faster mass gain during the early days and the gap between S0-B6.5 and S16-B6.5 persisted for several weeks. After 60 days, the mass had stabilized for S16-B6.5 and even slightly decreased for S0-B6.5. At longer term, both grouts reached equivalent masses.

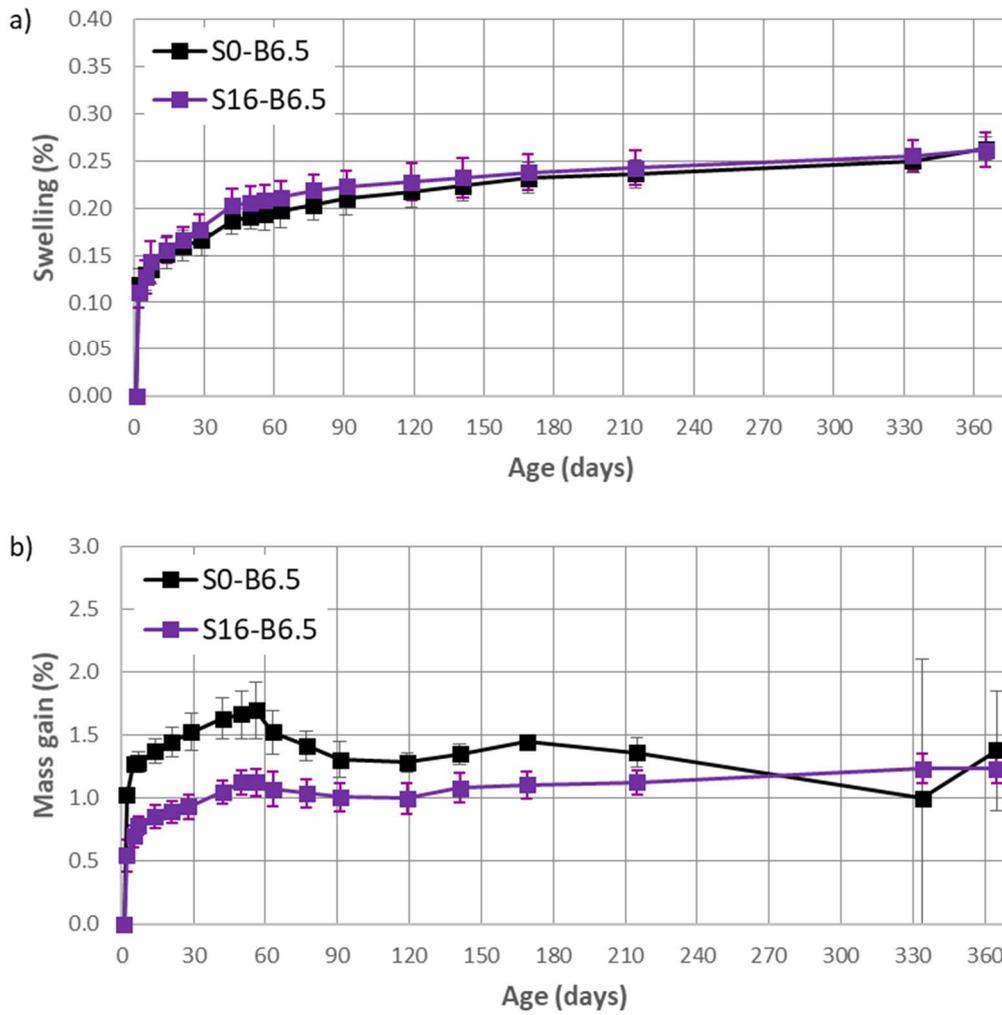


Figure 8: a) Swelling and b) mass variations of two-component grouts S0-B6.5 and S16-B6.5 – 4x4x16 cm³ bars kept in 25L of tap water at 20°C.

These phenomena of relative gain/loss of mass were probably linked to leaching of elements and changes of the water every 30 days. Alkalis, coming mainly from the accelerator, tended to leach out of the grout due to osmotic pressures. Other elements (Ca, Si, Al, etc.) were probably also leached, but less rapidly, as alkalis are known to be highly mobile ions as reported by Lloyd and al. (2010). When leaving, they could be replaced by water molecules. Thus, there was an opposition between the mass of elements lost by the leached grout and the water mass intake. In addition, the effect of changing the water every 30 days also affected the kinetics of leaching. The solubility of the elements of the grout was obviously dependent on the concentration of these same elements in the surrounding water: just after the water was renewed, the leaching proceeded faster.

Figure 9 presents a photograph of S0-B6.5 and S16-B6.5 grout samples after they had been kept in water at 20°C for 10 months. A soft, viscous gel appeared on the surface of the reference grout bars (S0-B6.5) but not on the GGBS samples S16-B6.5. This gel contained a large quantity of water and could be easily detached from the rest of the bar. It indicated that formulation S0-B6.5 was more sensitive to leaching phenomena and could explain the greater mass variability.

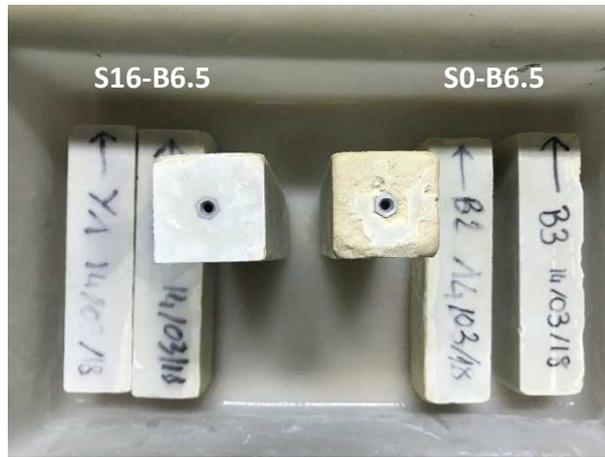


Figure 9: Picture of grout samples after 10 months in 20°C tap water

Compressive strength evolution

Figure 10 presents the compressive strength evolution of two-component grouts S0-B6.5 and S16-B6.5 kept in a 25 L tank of tap water at 20° for 1 year. Both formulations firstly showed an improvement of their strength before exhibiting a loss at later ages. Two-component grout S0-B6.5, without GGBS, gained strength up to 14 days, reaching a maximum of 1.6 MPa. Its compressive strength was then stable until day 90. Then, it began to lose strength, reaching 0.9 MPa at 180 days and 0.5 MPa after 1 year.

Grout S16-B6.5, which contained 16% of GGBS, developed its compressive strength for up to 90 days, reaching a maximum of 2.1 MPa ($\approx + 30\%$ compared to S0-B6.5). Then a loss of strength was observed, as 1.8 MPa and 1.1 MPa were measured at 180 days and 1 year, respectively. The use of GGBS allowed the grout to maintain its strength for a longer time when immersed in water. This was probably due to an improved hydration of the GGBS in this condition, and maybe to a better resistance to leaching, as a study performed on concrete by Müllauer et al. (2015) would suggest.

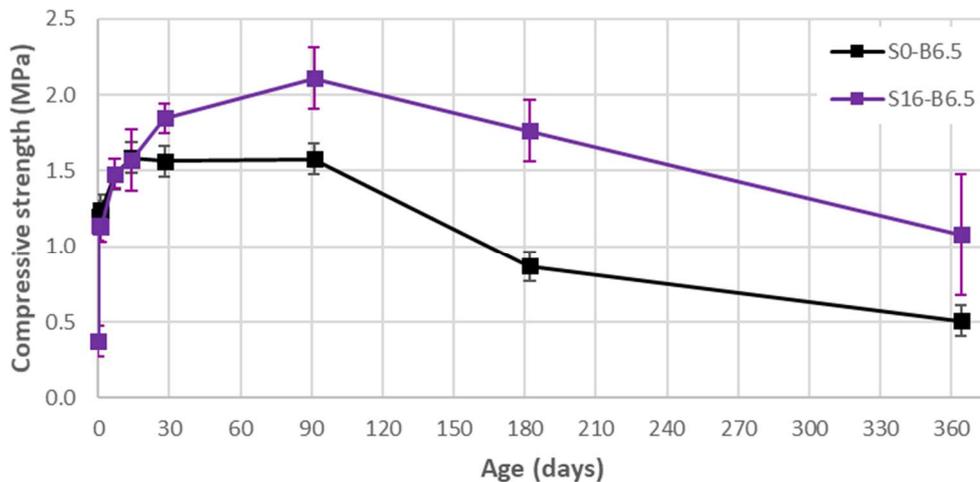


Figure 10: Compressive strength variation of two-component grouts S0-B6.5 and S16-B6.5 – 4x4x16 cm³ bars kept in 25 L of tap water at 20°C.

4.3.2 Behaviour of two-component grouts in Na₂SO₄ solutions

Two concentrations were selected for grout immersion in this study: 600 mg/L and 3000 mg/L of SO₃. In each case, 4x4x16 cm³ grout bars of formulations S0-B6.5 and S16-B6.5 were cured for 28 days in a 25 L water tank at 20°C, then kept in 25 L of a solution with the required concentration, also at 20°C. Solutions were renewed every 30 days. Swelling and mass gain, as well as compressive strength, were followed for up to 1 year.

Swelling and mass evolutions

Figure 11a and Figure 11b show the swelling and mass gain caused by sulfate, for two-component grouts S0-B6.5 and S16-B6.5 kept for up to 1 year in a sulfate solution at 20°C. Two cases are presented: for immersion in a solution at a concentration of 600 mg/L (plain lines and triangles) or 3000 mg/L of SO₃ (dashed lines and crosses). In each case, the corresponding effect of water has been subtracted to consider only the impact of sodium sulfate. Confidence intervals drawn correspond to the sum of those obtained independently for immersion in water or in sulfate solutions.

During the first 28 days, all curves remain close to the abscissa line. This implies that S0-B6.5 and S16-B6.5 grout bars experienced the same behaviour as their corresponding references immersed in water, which is reasonable since all samples were kept in the same condition (water at 20°C) during this period.

Then, regarding the behaviour of grouts kept in a solution at 600 mg/L of SO₃, observations indicate that all formulations experienced a slight swelling and a new mass intake during the first two weeks of immersion in sulfate solution. Afterwards, no more length or mass evolutions seem to be linked to the effect of sulfates.

On the other hand, grouts immersed in a solution at 3000 mg/L of SO₃ swelled significantly upon exposure to sulfates. Mass gain corresponded to this length change. Monitoring of grout samples stopped after 120 days, corresponding to 90 days in the 3000 mg/L SO₃ solution, since one bar of S16-B6.5 was broken at this age. Figure 12 shows a photograph of the broken bar. In this case of immersion in highly concentrated sulfate solution, swelling of samples was certainly due to the formation of expansive ettringite, which led to crack formation (Colleparidi, 2003). Yu et al. (2018) have shown that this phenomenon also concerns two-component grouts based on Portland cement binders. Here, formulation S16-B6.5, with 16% GGBS in its binder, appeared to swell more than S0-B6.5, even though the extended confidence intervals indicate a large variability in behaviour of the different grout bars with the same formulation. This result is not consistent with the literature, where it can be found that GGBS usually enhances the durability of binders against sulfates. However, its effect is often visible for higher GGBS content, over 65% of the binder (Gollop and Taylor, 1996; Whittaker and Black, 2015).

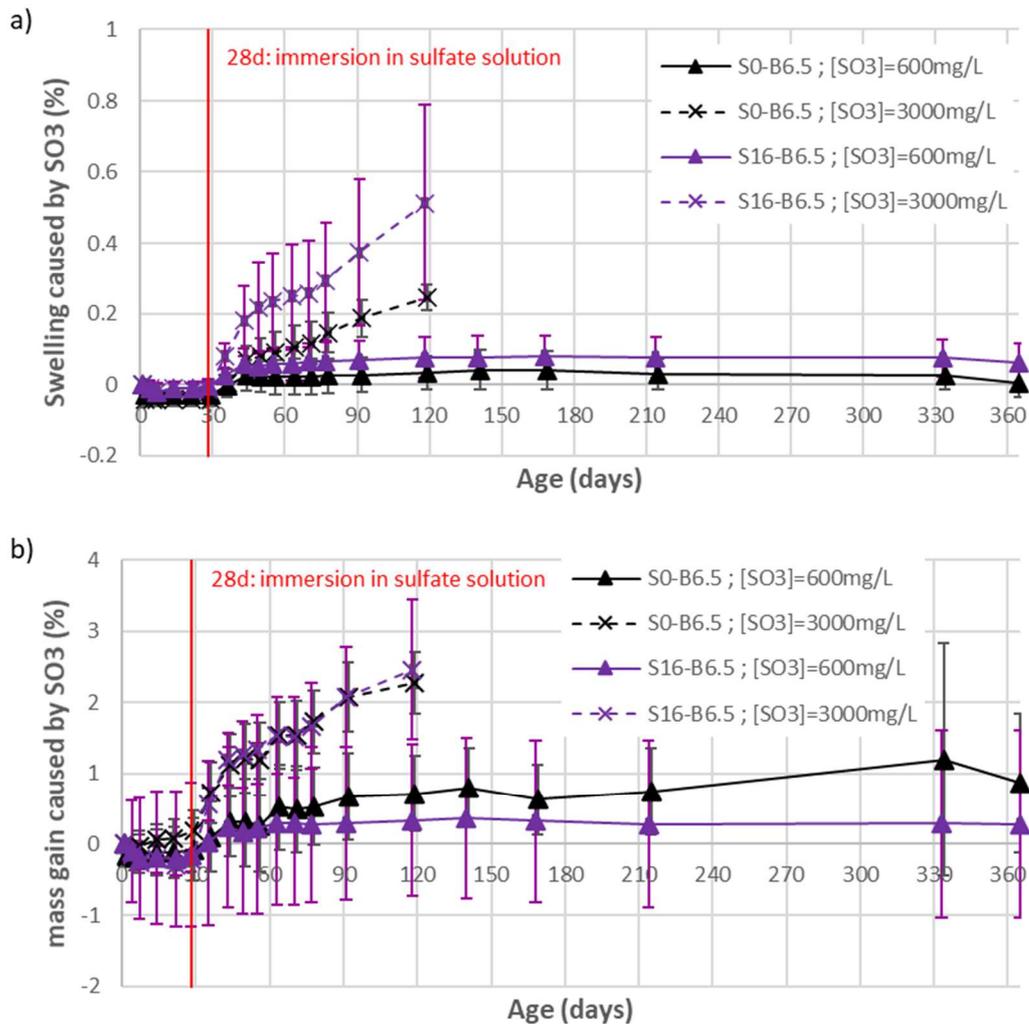


Figure 11: a) Swelling and b) mass gain caused by sulfates in two-component grouts S0-B6.5 and S16-B6.5 – 4x4x16 cm³ bars kept in 25 L of solution at SO₃ concentration of 600 mg/L or 3000 mg/L after the first 28 days in 25 L of tap water.



Figure 12: A grout bar of formulation S16-B6.5, broken after passing 90 days in Na₂SO₄ solution at SO₃ concentration of 3000 mg/L.

Compressive strength evolution

Figure 13a and Figure 13b present the compressive strength evolution of two-component grouts S0-B6.5 and S16-B6.5 immersed in sodium sulfate solutions having SO₃ concentrations of 600 mg/L or 3000mg/L, respectively. The strength of formulations does not start at 0 MPa since only the time spent in the sulfate solution is represented. This means that time “0” corresponds to a grout that has already passed 28 days of curing in water.

Looking at the immersion in the lowest sulfate concentration, and despite slight upward and downward variations for grout S0-B6.5, it can be considered that its compressive strength remained stable after 1 year of immersion. Formulation S16-B6.5 started at a higher value than S0-B6.5 after 28 days curing. Then its strength slowly increased after immersion in the sulfate solution. The stability, or slow evolution, of mechanical strengths of both grouts suggests that there were fewer leaching phenomena in this case than

under tap water. However, even if it was not visible on grout bars destined for the evaluation of dimensional variations, samples containing no GGBS and cast for compressive strength measurements exhibited cracks at their ends after some time. This is visible on the picture in Figure 14. The dark stains appearing on samples from formulations S16-B6.5 were due to GGBS hydration (Chaouche et al., 2017). In the case of an immersion in a solution with an SO_3 concentration of 3000 mg/L, all formulations experienced a loss of strength. After 28 days of exposure to sulfate, the average strength reduction was 0.5 MPa for S0-B6.5 and 0.3 MPa for S16-B6.5. Then, after 90 days of exposure to sulfate attacks, no strength could be measured since all samples destined for compressive strength measurement were found to be totally cracked (Figure 15).

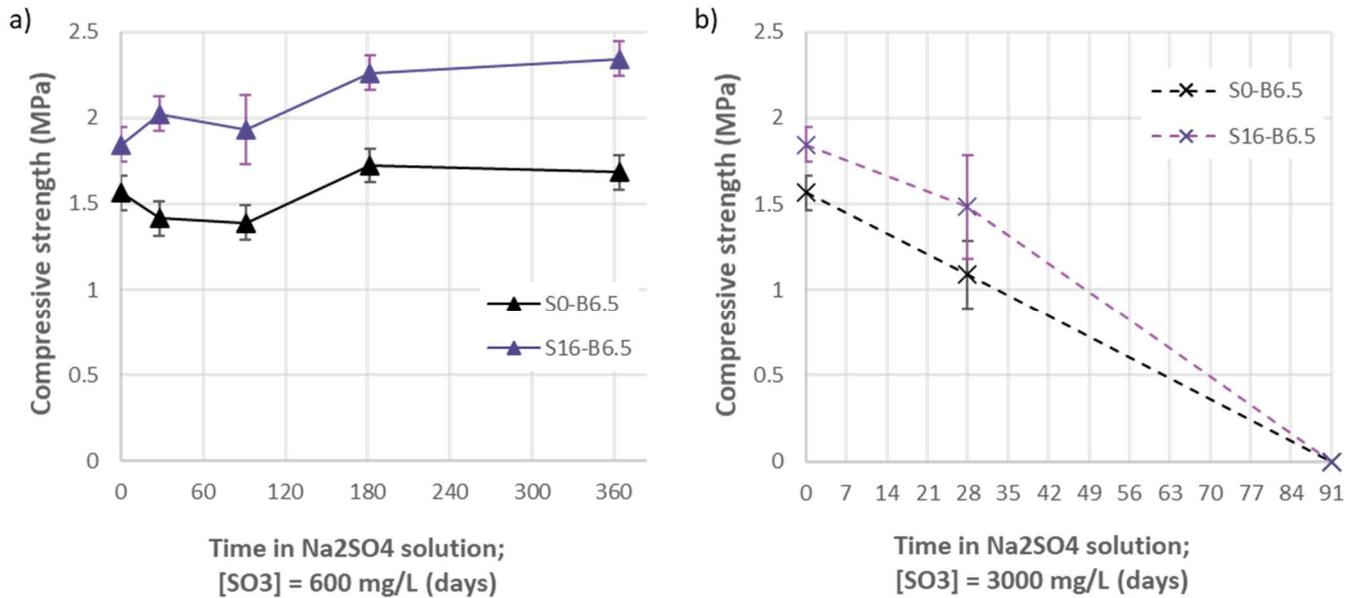


Figure 13: Compressive strength evolution of two-component grouts S0-B6.5 and S16-B6.5 – 4x4x16 cm³ bars kept in 25 L of solution at SO_3 concentration of 600 mg/L or 3000 mg/L after first 28 days in 25 L of tap water.

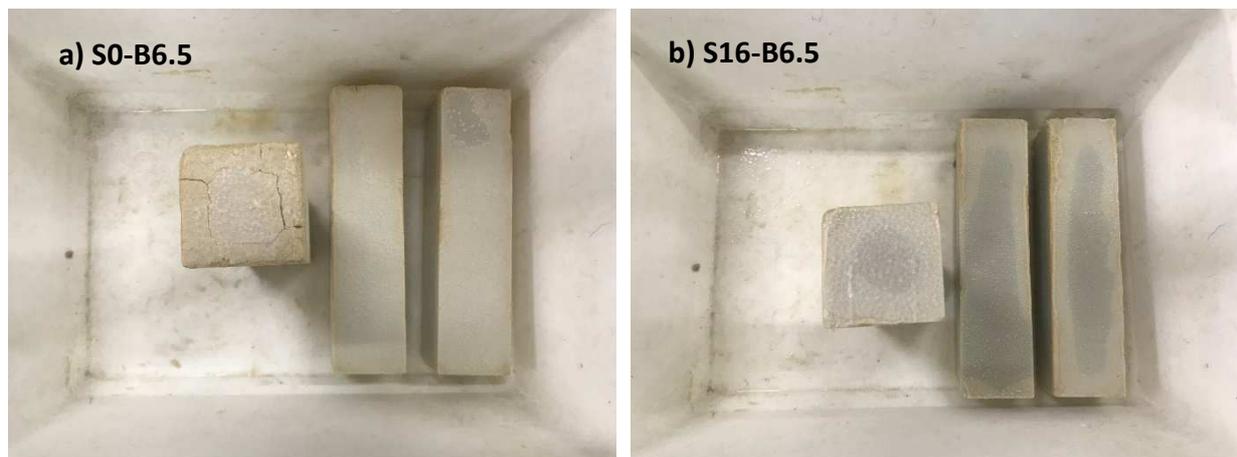


Figure 14: Two-component grout bars exposed to a sulfate solution at 600 mg/L of SO_3 for 90 days (after 28 days of curing under tap water) – a) formulation S0-B6.5, without GGBS– b) formulation S16-B6.5, containing 16% GGBS in its binder

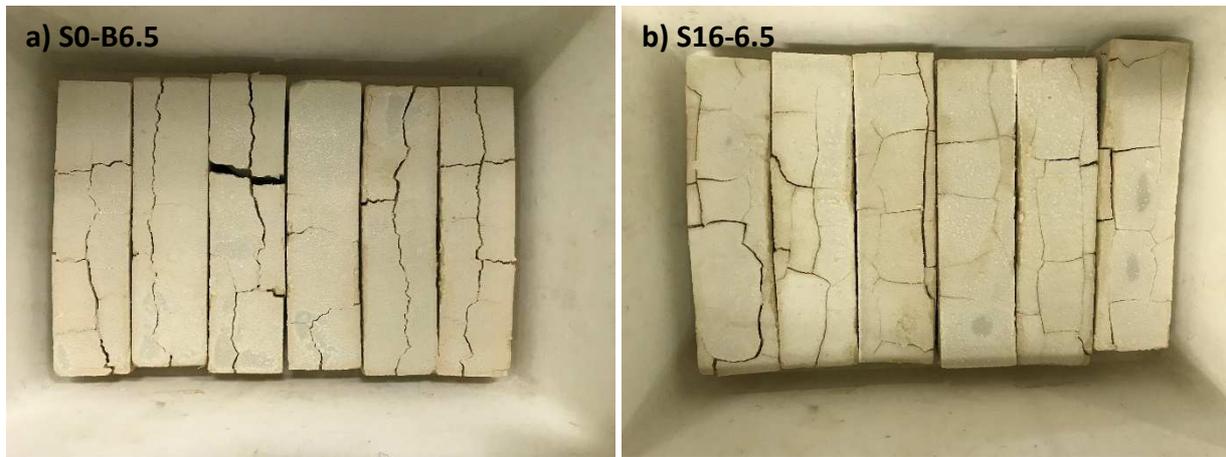


Figure 15: Two-component grout bars exposed to a sulfate solution at 3000 mg/L of SO_3 for 90 days (after 28 days of curing under tap water) – a) formulation S0-B6.5, without GGBS – (b) formulation S16-B6.5, containing 16% GGBS in its binder

5 Discussion

During the digging of a tunnel using a TBM, the grout (component A) mixer operates continuously and is not cleaned between each batch. Consequently, part of the previous grout remains in the mixer when making a new one. Laboratory trials realized during this study have shown that adding 55g/L of a former grout to the mixing water increased its pH and electrical conductivity. Thereby, because of a high ions content in the mixing water, bentonite was not able to fully develop its grout stabilizing role (Abu-Jdayil, 2011; Kelessidis et al., 2007) and component A tended to develop more bleeding. Thus, the addition of a former grout to the mixing water during laboratory trials on two-component grouts is crucial to be representative of real application. Theoretical conclusions drawn without respecting this stage of the mixing protocol could lead to failure in terms of grout rheology during jobsite implementation.

On the contrary, the partial substitution of OPC by GGBS in component A formulations reduced the bleeding phenomenon. The calcium reduction brought by increasing GGBS content allowed bentonite to better develop its network, resulting in a better stability of the grout. Also, this substitution did not change drastically the fluidity nor open time of the grout. It did however have an impact on the jellification process of the two-component grout. Increasing the proportion of GGBS lengthened the gel time. As for bleeding phenomenon, the most probable explanation came from the lower availability of calcium with higher GGBS content. As component B, the accelerator was an alkali-silicate solution with high Si/Na ratio, which reacted with the calcium in solution to produce a calcium silicate gel (Song et al., 2020). This reaction was faster for high Ca/Si ratio in solution. The fewer the calcium, the longer it took for the two-component grout to transform into a gel. This was also confirmed by the observation made on the effect of component B to component A ratio. An augmentation of the accelerator content increased the gel time as Ca/Si ratio was reduced. Thus, to include larger GGBS content in component A binder and respect jobsite specification, one key would be to work on B/A ratio to reduce the accelerator content.

At hardened state, the addition of GGBS tended to lower the 1-day strength of two-component grouts. As previously explained, it was probable that the lower calcium availability generated by OPC substitution led to less initial C-S-H formation responsible for the strength development (Song et al., 2020). However, it was possible to reach more than 0.70 MPa at 24 hours, even with 50%wt slag. In the long term (28 days), the addition of GGBS greatly enhanced the compressive strength of two-component grouts. GGBS is known to be a compatible material for alkali activation (Shi et al., 2003). As GGBS contained more alumina compared to OPC, its activation by an alkali-silicate solution led to the formation of C-A-S-H gel. Some authors have shown that the increase in C-A-S-H phase, rather than C-S-H, improved strength development of alkali-activated materials based on GGBS (Puertas et al., 2011; Yang et al., 2018).

Regarding durability to external sulfate attack, it would probably require more than 16%wt of GGBS in component A binder to prevent grouts from swelling in a strong sulfate solution (Gollop and Taylor, 1996;

Whittaker and Black, 2015). However, even a small addition of GGBS had a beneficial impact on water resistance. It prevented the formation of a soft, easily detached gel on the surface of the grout. According to Song et al. (2020), the better behaviour with respect to water corrosion of two-component grout containing GGBS could be due to the modification of C-(A)-S-H gel. The increase in silicon and alumina brought by GGBS could form a less soluble gel.

6 Conclusion

This paper has shown that a partial replacement of CEM I by GGBS in two-component grouts is possible. A grout containing 30%wt GGBS in its binder and activated with a B/A ratio of 6% respects all the required specifications. Enhancing GGBS content to 40%wt could also fulfil the needs of the jobsite. In addition, this study helps several points concerning two-component grout behaviour to be understood. The major conclusions drawn are listed below:

- (1) An increase B/A ratio (Hardening accelerator/Retarded grout stabilized with bentonite) leads to an augmentation of the gel time, but also to higher strength of the two-component grouts.
- (2) At fresh state, an augmentation of the GGBS content in component A formulations does not modify its open time and fluidity. However, it limits the bleeding phenomenon. Also, it tends to enhance the gel time.
- (3) At hardened state, increasing the GGBS content tends to lower the early strength (1 day) but to improve the 28-day strength.
- (4) Regarding durability, the substitution of 16% OPC by GGBS does not permit to prevent swelling and cracks formation in strong sulfate solution. It would probably require more GGBS. However, even this slight substitution already increases water corrosion resistance. It prevents the formation of a soft and easily detached gel on the surface of the grout.

In parallel to this study, a two-component grout containing first 35 kg/m³ of GGBS and then 50 kg/m³ (respectively 16%wt and 23%wt in the binder) has been used on the jobsite for almost 2 years. Comments received on this subject mention the formulation stability provided by using slag, thus demonstrating the reliability and robustness of the solution.

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