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The role of bentonite in two-component grout: A comparative study

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A B S T R A C T

Two-component grout is the most used backfilling technology in shielded mechanised tunnelling. Bentonite is an important ingredient in the production of component A, since it plays a key role in the mortar stabilisation. Furthermore, recent studies have highlighted its involvement in the mechanical and elastic performance of the hardened grout after short curing times, while no information is available for long curing times. This study proposes an innovative test campaign, based on a comparison between a standard two-component grout mix design (reproduced with 5 different bentonites) and a special mix design, produced without using the bentonite. This interesting comparison highlights the important role of the bentonite and its activation time in properties related to fresh grout (component A) and hardened grout. Assessments of bleeding, viscosity, unit weight, gel time, surface compression strength (SCS), uniaxial compression strength (UCS) and tensile strength (T_0) have permitted a deep quantitative analysis that, undoubtedly, highlights the importance of bentonite in this widespread backfilling grout technology.

1. Introduction

When a shielded machine is used for tunnelling excavation, the backfilling phase cannot be avoided, since it is intrinsic to the shield technology. In fact, because the installation of pre-cast linings is carried out under the protection of the shield, a difference in diameter exists between the extrados of assembled linings and the extrados of the shield (Thewes and Budach, 2009). This annular space must be constantly filled during the machine advancement (Liu et al., 2021) and this operation is named backfilling. The backfilling can be performed instantaneously during the machine advancement (from nozzles installed in the tail shield) or after a certain time, short enough to prevent displacements of linings and surface settlements. Backfilling is crucial to mechanised tunnelling projects (Sharghi et al., 2018; Di Giulio et al., 2023) since their proper design and realisation depend on the prevention of surface settlements, the locking of segments into the designed position, the avoidance of punctual load on linings (the grout increases the contact area between the medium intended to be excavated and the lining itself) and increasing waterproofness. Two-component grout is the most popular technique for the backfilling phase and is used on construction sites all around the word. This technology is based on two liquid components, called component A and component B, that are turbulently mixed a few centimetres before the injection nozzles, located circumferentially on the tail shield, from which the two-component grout flows out. The time-lapse between the first contact among components and the loss of fluidity of the mixed grout is called 'gel time' and it has an order of magnitude of seconds (Hashimoto et al., 2005). After the gelation, the obtained gout starts its curing by increasing its mechanical (Oggeri et al., 2021;) and elastic performance (Oreste et al., 2021; Todaro and Pace, 2022). Standard component A is composed of bentonite, cement, a retarder fluidifying agent and water. In recent years, innovative mix designs have been tested, including the use of blast furnace slag, fly ash and industrial solid waste, among other materials (André et al., 2022; Schulte-Schrepping and Breitenbücher, 2019; Song et al., 2020, Song et al., 2022). Component B is a solution of sodium silicate and is called 'accelerating agent' or 'accelerator' (Reschke and Noppenberger, 2011). Recently, superabsorbent polymers have also started to be tested as component B (Schulte-Schrepping et al., 2019).

Two-component grout has become the most widely used backfilling technique in mechanised shielded tunnelling applications. Its success and application in many projects is mainly due to its aptitude for controlling and reducing surface settlements (Fargnoli et al., 2013; Grasso et al., 2023); this occurs because of a gelation process that permits an almost instantaneous hardening reaction of both grout components once injected into the annular gap.

Bentonite (a clay mineral) is a standard ingredient in two-component grout; it stabilises the cement particles which would otherwise tend to segregate downward due to the force of gravity. Bentonite also increases the homogeneity and waterproof properties, helping to give a thixotropic consistency to the grout (Peila et al., 2011). However, the use of bentonite has contra-indications. Indeed, an incorrect, excessive dosage could cause a decrease in fluidity due to the absorption of water on its surfaces, which reduces the lubrication of particles (Mesboua et al.,

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2018) and the potential choking of feeding pipes that connect the batching station to the machine, in the construction site.

The development of the bentonite structure (swelled bentonite) is able to bear the cement particles and is strictly correlated with the procedure used for mixing bentonite and water, as well as the time of activation (Todaro et al., 2022). The latter is defined as the duration of the mixing between water and bentonite only. Water is absorbed in this interval and the particles develop their pack-of-cards structure (Garshol, 2003; Van Olphen, 1977).

The goal of this work is to highlight the role played by bentonite on the properties of component A (before the addition of the accelerator) and on the strength of the hardened material after short and long curing times (after gelation). In this work, the identification of short and long curing times is based on the working principle of a shielded machine (where the backfilling phase is carried out). For short curing time is intended a time-lapse after the gelation of 1 and 3 h (and anyway shorter than 1 day), i.e. commonly the time between the casting phase related to a ring and the loading of the two-component grout due to the transport of the linings needed for assembling the next ring. For long curing time is instead intended a time-lapse of 1 and 28 days.

The influence of the activation time on the material properties is also studied. The work is structured with a comparative approach: the standard grout is compared to another grout, expressly prepared without using bentonite. A unique mix design was selected as a reference material (fixed dosages of the ingredients) and used to prepare five variants of standard two-component grout, by using five different bentonites. A standard duration of 7 min was proposed for the bentonite activation by Todaro et al. (2019) but, in order to also highlight the influence of the activation time, a further testing campaign was performed without activating the bentonite.

The outcomes highlight the key role played by bentonite in two-component grout technology. The properties of the fresh component A (bleeding, viscosity, unit weight, gel time), as well as those of the hardened grout (surface compression strength, uniaxial compression strength and tensile strength), are strongly influenced by the presence of the bentonite, whose role is proved to be more than a simple stabiliser of the liquid mortar.

2. Materials and methods

A laboratory testing campaign was planned, with the purpose of highlighting the role of bentonite and the importance of the activation phase. According to previous research (Todaro et al., 2022; Todaro and Pace, 2022), the effects of the bentonite have been evaluated in three different stages. The first stage is related to component A when it is still fluid and not yet gelled; the second stage is expressly related to the gelation process; the last stage is related to the hardened grout in both short and long curing times (a short curing time was intended as a time lapse, up to 24 h). The testing protocol used to quantify the effect of the bentonite on the studied material was established, based on Todaro et al. (2019). With regard to the first testing stage, the assessments of bleeding, unit weight and viscosity were performed. The second testing stage concerned the assessment of the gel time (both liquid components A and B were involved), while tests on hardened grout constituted the third stage, specifically SCS, T_0 (assessed indirectly by the 3-point flexural test) and UCS testing. The mix design considered as a reference is reported in Table 1. Mix designs B1-B5 are related to the five respective bentonites used, while the mix design 'B-less' is related to the case without bentonite. The quantity of water, calculated upon completion (after having established dosages of other ingredients) to reach a volume of the admixture equal to a cubic metre, slightly varies from one case to another due to the different specific weight of the related bentonite.

Before proceeding with the description of the used material and methods, it is important to underline the limitations of this research. These aspects could be considered as starting points for future investigations. In this research, the potential effect of the component B dosage on the properties of the two-component grout has not been considered. However, it should be mentioned that the dosage of component B could affect the grout properties due to the interaction with the bentonite. Furthermore, it should be reported that important parameters such as the time for surface reaction and the activation phase of the bentonite (infiltration of the water between clay laminas) have not been deeply investigated from a chemical point of view.

Table 1
Mix designs: the ID number of each mix design (B1 – B5) corresponds to the number of the bentonite (bentonite type) reported in Table 2. As an example, the mix design B1 was prepared by using bentonite 1.

	Two-component grout mix (kg/m^3)					
Element	B1	B2	B3	B4	B5	B-less
Cement	230.0	230.0	230.0	230.0	230.0	230.0
Bentonite	30.0	30.0	30.0	30.0	30.0	/
Retarding/fluidifying agent - Mapequick CBS1	3.5	3.5	3.5	3.5	3.5	3.5
Accelerating agent - Mapequick CBS3	81.0	81.0	81.0	81.0	81.0	81.0
Water	851.8	852.0	852.0	851.8	852.7	863.8

2.1. Used materials

For the production of the two-component grout samples, Portland Cement type CEM I 52.5 R (CEN, 2011) was used. Accelerator and retarding/fluidifying agents were provided by Mapei company. Five bentonites were used for preparing component A; all bentonites are sodic and characterised by different smectite contents and swell indexes (SWI) (ASTM D5890, 2019). Bentonite data were taken from Todaro and Pace (2022) and are reported in Table 2.

2.2. Component A production and sample casting

The two-component grout is a sensitive technology and the procedure used for producing the component A and the hardened grout can affect significantly the properties of the backfill grout. All relevant authors that studied the two-component grout agree on this statement but the procedure used for the sampling production and curing is still not unique.

Oggeri et al. (2021), for example, activate the bentonite naturally, i.e. simply leaving the powdered bentonite in water, for 24 h. After that, the ingredients are mixed together (mechanical and manual mixing have been adopted). The mixing of components A and B is performed mechanically by using a high-speed mixer. The sample curing is performed in water.

André et al. (2022), instead, prefer manual mixing. The two-component grout was prepared by pouring the right amount (according to the mix design) of component A inside the tank with component B, and after no more than 3 transfers from one tank to the other, the homogenous mix was poured into the mould. After 24 h of sealing, samples were demoulded and put in water for curing.

Oreste et al. (2021) and Di Giulio et al. (2023), alternatively, proposed the sample production by using innovative equipment that permits the mixing of the A and B components in a single grout flow, reproducing the effects of a nozzle of a TBM. Both these authors suggested the curing phase of samples in water.

Wan et al. (2021) suggest the mechanical mix between components A and B for a duration of 30″ by using a stirrer and a curing temperature of 20 ± 2 °C. Rahmati et al. (2022) prescribe natural hydration of bentonite. Instead, the mixing phase is not clearly described while a curing phase characterised by a relative humidity higher than 95% and a temperature of 20 °C is suggested.

In this work, Component A was prepared according to the mixing procedure presented in Table 3, by using an impeller. Special care was

Table 2
Properties of the bentonites used.

Bentonite	Unit weight (g/cm^3)	Smectite content (%)	SWI (mL/g)	Photo
Type 1	2.70	92	23.0	
Type 2	2.50	98	12.1	
Type 3	2.50	88	14.0	
Type 4	2.55	94	12.7	
Type 5	2.55	72	19.7	

Table 3
Mixing procedure.

Phases	Impeller rotation speed (rpm)	Duration (min)
Start – only water	800	/
Bentonite mixing phase	2000	According to the activation time (0 or 7)
Cement mixing phase	2000	3
Mix of retarding/fluidifying agent - End	2000	2

taken in order to verify the homogeneity of the produced component A. When more than 1 batching phase was necessary (with the used procedure no more than 3 L can be produced for batch), the assessment of the unit weight permitted to check the homogeneity and the quality of the mortar produced in different batches. It is important to note that the activation phase of the bentonite can be 0 or 7 min if the bentonite is not activated/not used or activated, respectively. When the bentonite is not present the activation phase is skipped, hence the activation time is 0 min. The activation phase is related to the swelling of the bentonite that is accelerated to develop in some minutes by providing kinetic energy, in this study with the impeller as in construction sites by the turbomixer (in standard condition, i.e. no kinetic energy added, the swelling of bentonites can occur in hours, sometimes days). In this research, if the time of activation of the bentonite is 0 min, bentonite and cement can be added at the same time. For mixes with bentonite (B1-B5), the duration of the whole mixing process is 5 or 12 min, respectively, for the activation time of 0 and 7 min. The duration of the mixing phase for the mix design B-less is 5 min.

It has been decided to fix the activation time for all bentonites in order to avoid the introduction of further variables. From previous studies (Todaro et al., 2022), indeed, it is known that the performances of component A can be slightly managed by the activation time of the bentonite. Consequently, it has been chosen to test the different bentonites providing them the same "activation energy" and to assess potential differences in the produced two-component grout.

Fig. 1. Casting phase of a two-component grout sample according to the used procedure. From the left to the right: the component A is poured into the component B, the mixed grout is poured in the empty tank and after that the casting of the sample is carried out.

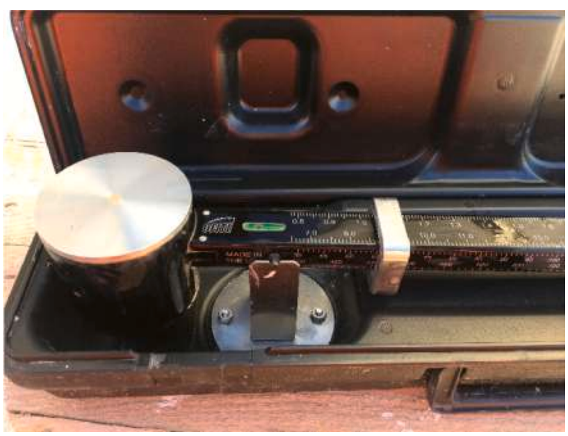

Fig. 2. Mud balance used for the unit weight assessment.

The casting is the most critical phase. Special care has to be taken in order to face the fast gelation process. Due to the lack of a standard procedure expressly suitable for the two-component grout, the experimental procedure described in Todaro et al. (2020) was followed. Briefly, two containers with a volume of at least of 0.5 L are filled, one with component A and the other one with component B (for a correct casting, the total volume should be 0.256 L + 20 %). Component A is poured into component B (if the opposite the right turbulence is not generated), the operation is repeated and finally the mix is poured inside the mould, trying to create a continuous and homogeneous flow (Fig. 1). A sample trimming performed by using a spatula permits to obtain the right shape of the sample.

Standard mortar moulds (40 × 40 × 160 mm) were used, in compliance with CEN (2016).

Also for the hardened grout, special care was taken for the acceptance criteria of the obtained samples. As previously described for component A, each sample was weighed after the demoulding phase and the unit weight was assessed. If a difference higher than 0.03 – 0.04 kg/L with respect to the expected value was recorded, the sample was discarded.

Fig. 3. Marsh funnel used for the viscosity assessment and main phases of the procedure. Left: leveling of the funnel. Centre: filling of the funnel (1.5 L) with the component A till the notch. Care must be taken to plug the exit hole. Right: removal of the plug and contemporary starting of the time record till the complete flow of 1 L. Todaro et al. (2019).

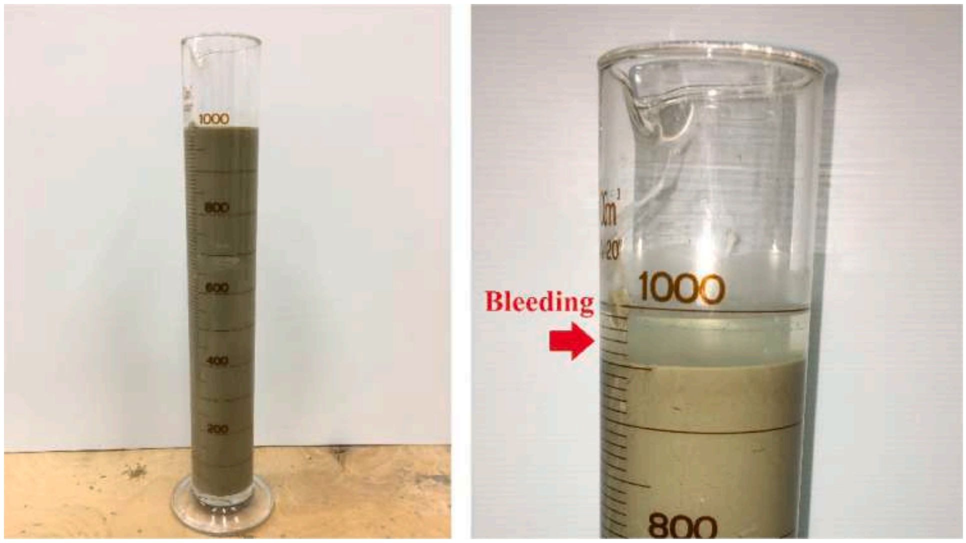

Fig. 4. Bleeding assessment. Left: 1 L of component A (V_t) has been poured into a standardised cylinder. Right: the segregated water (V_w) on the top of the grout indicates the occurrence of the bleeding phenomenon. Todaro et al. (2022).

2.3. Performed tests

All of the tests concerning the mix designs produced by using bentonites (B1-B5) were performed by producing the component A in two different variants, i.e. by activating the bentonite for 7 min and without activating it. The mix B-less, produced without using bentonite, was produced by only following the last two steps of the procedure reported in Table 3.

2.3.1. Gel time

Gel time was assessed according to the experimental procedure proposed by Todaro et al. (2019). Commonly expressed in seconds, it identifies the time-lapse between the mixing phase of component A and B and the loss of fluidity of the obtained grout. At least three experimental determinations have to be assessed, in order to give a reliable result. The accuracy of the measured values, according to the authors' experience, is $\pm$ 2 s, if the operator that performs the test is always the same.

2.3.2. Unit weight

A mud balance was used for the assessment of the unit weight of the component A (Fig. 2). The standard ASTM D4380 (2020) was taken as a reference. The used mud balance had a resolution of $\pm$ 0.01 g/cm^3.

2.3.3. Viscosity

Viscosity tests were performed according to UNI 11152-13 (2005). The time required to drain 1 L of grout out of the Marsh funnel (previously filled with 1.5 L of the grout intended to be tested) was measured. The accuracy of the measured time, according to the authors' experience, is $\pm$1 s if the operator that performs the test is always the same. Fig. 3 depicts the Marsh funnel and main steps of the test.

2.3.4. Bleeding

The bleeding assessment (Fig. 4) permits evaluation of the physical stability of the grout. The bleeding index is defined as a percentage ratio:

$$Bleeding = \frac{V_w}{V_t}(\%) \tag{1}$$

where V_w is the volume of the segregated water and V_t is the total considered volume of the grout. V_w, equal to 0 when the test begins, increases as a function of time, due to the gravity force applied to the cement particles, which tend to settle. Bleeding tests, performed according to UNI 11152-11 (2005), were carried out at different curing times: every 10 min from the ending of the production phase, up to 1 h (6 determinations) and every 30 min for 3 h of curing (3 determinations).

Fig. 5. Penetrometer used for the surface compression strength (SCS) assessment. Left: the used dynamometer. Centre: details of the flat circular bit (15.05 mm of diameter and 5 mm of thickness). Right: phase of the SCS assessment with the reading of F (N). Todaro et al. (2020).

Fig. 6. Left: tensile strength assessment performed according to the 3-point flexural test. The frame for the three-point flexural test is depicted. Right: uniaxial compression strength assessment, performed by using the comprimitor. Both depicted devices are in compliance with the CEN (2016).

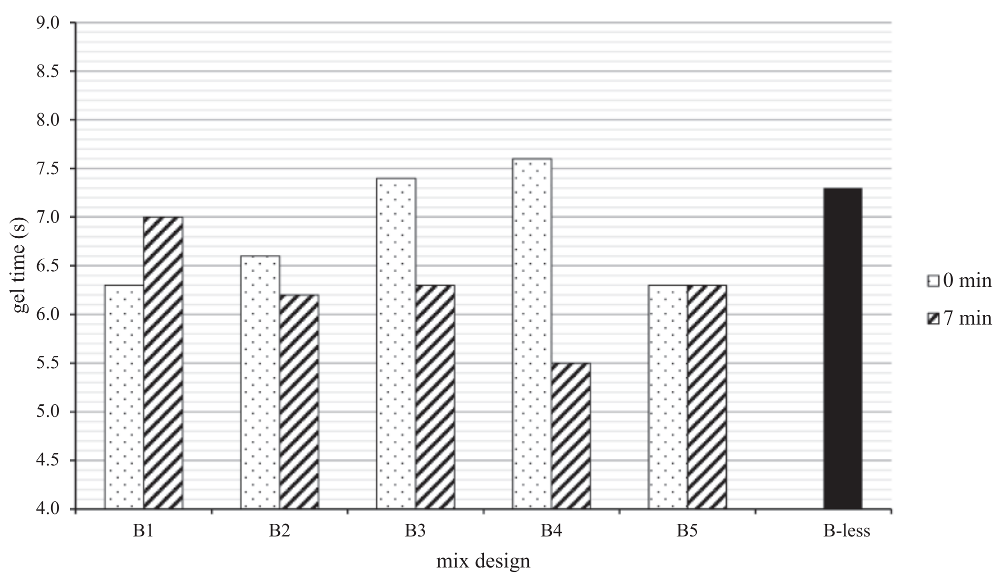

Fig. 7. Gel time of the studied mixes.

2.3.5. Surface compression strength (SCS)

The mechanical characterisation of the hardened mortar after short curing times was evaluated by performing the SCS test, according to the procedure proposed by Todaro et al. (2020). According to the standard, samples cured for 1 and 3 h were tested. A SAUTER GmbH Ziegelei 1 D-72336 Balingen digital model dynamometer (maximum force of 1000 N and 0.5 N of resolution) was used, equipped with a circular bit of 177.9 mm^2 area (A) (Fig. 5). The test began by pushing the bit on the surface of the sample intended to be tested and ended when the bit penetration reached 5 mm. The maximum value of the applied force epressed in N (F) was recorded. SCS is a pressure (MPa), computed as:

$$SCS = \frac{F}{A} \tag{2}$$

2.3.6. Uniaxial compression strength (UCS) and tensile strength (T_0)

Mechanical characterisation of the hardened mortar, after a long curing time, was evaluated by assessing UCS and T_0 (Fig. 6). Tests were performed according to CEN (2016). Both of these tests were carried out on samples cured after 28 days while, for the curing time of 1 day, the UCS was assessed. After 1 day, in fact, it was impossible to correctly perform the 3-point flexural test due to the weakness of the material, which exhibits behaviour more similar to a clay than a hardened grout. The three supports of the frame for the three-point flexural test punched the sample, rather than applying the correct stress able to break the sample into two halves.

Samples were demoulded 24 h after casting. Samples intended to be tested after 1 day were immediately broken, while those intended to be tested at 28 days of curing were left in water until the time of the test (the curing temperature of the water was 23 ± 2° C).

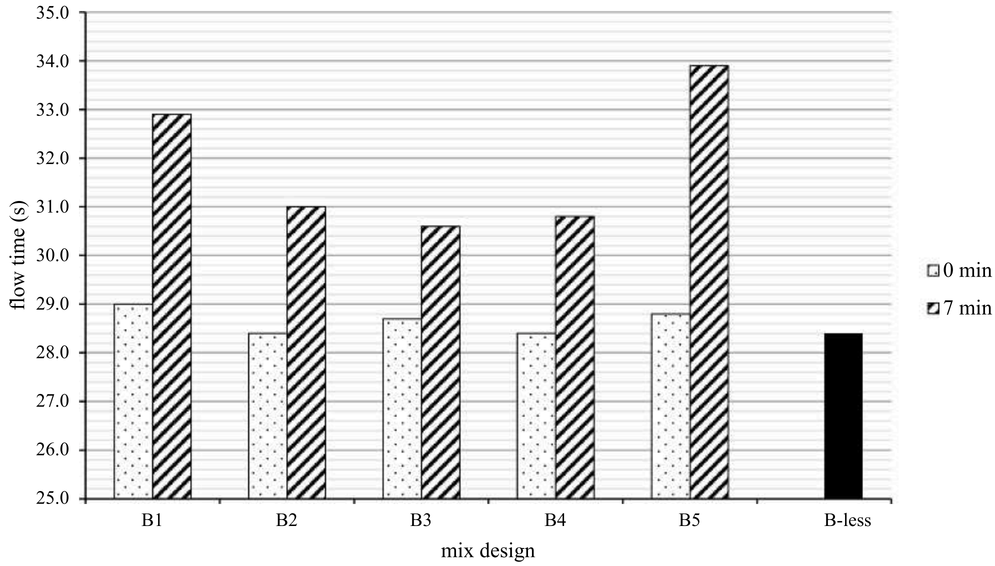

Fig. 8. Flow time of the studied mixes.

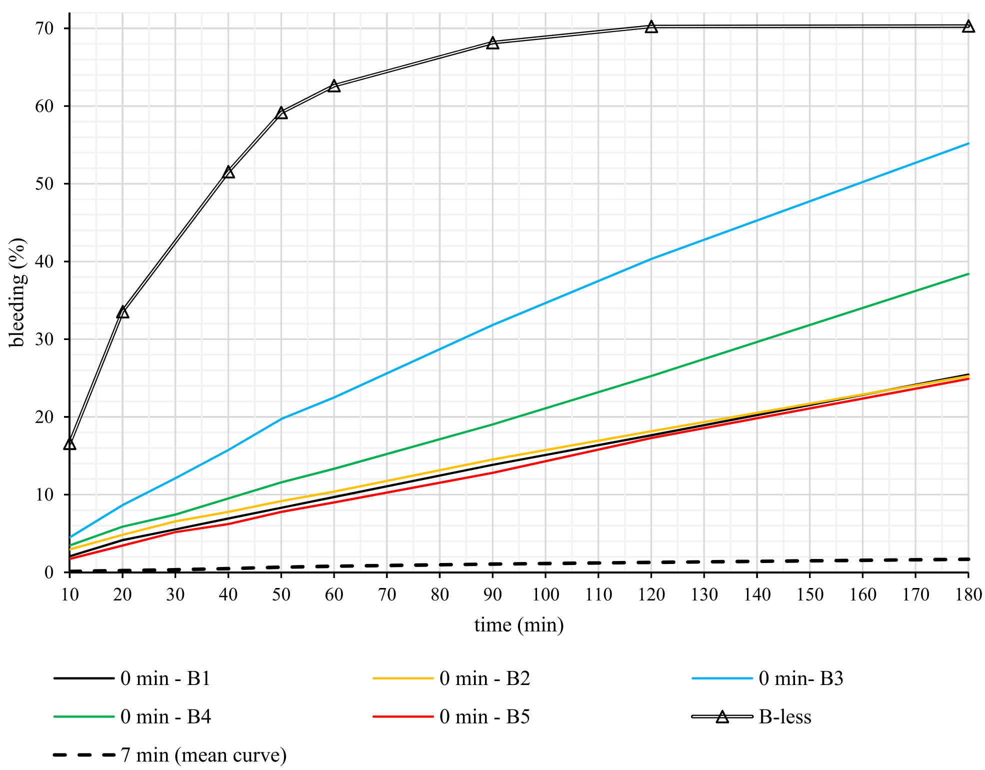

Fig. 9. Bleeding of studied mixes.

3. Results

Concerning component A, the unit weight ranges from 1.18 to 1.19 g/cm^3, irrespective of the activation time and irrespective of the type of bentonite used. Instead, without bentonite the unit weight is a bit lower (1.17 g/cm^3). Concerning the gel time and viscosity, the results are reported in Fig. 7 and Fig. 8, respectively.

With respect to the bleeding, Fig. 9 depicts the relevant outcomes. In order to streamline the chart, only the average function for the activation time of 7 min was reported. This simplification was made possible because all of the bleeding values related to 7 min of activation, irrespective of the bentonites used, and were very similar. By contrast, for the mix produced without activating the bentonite, the bleeding functions were quite different and so they were plotted separately.

Considering the mechanical performances at short curing times (tests performed after 1 and 3 h of curing), SCS outcomes are reported in Fig. 10 and Fig. 11. For long curing times (Fig. 12 and Fig. 13), the results obtained were very similar, irrespective of the bentonite type, and, consequently, only the average values are reported. As previously stated, the results of 3-point flexural testing are only available for 28 days of curing since, for shorter curing times, it was impossible to properly perform the test. In the following charts, all of the singly performed tests are reported. The average values are highlighted with a specific symbol, slightly larger than the others. Statistical data (mean,

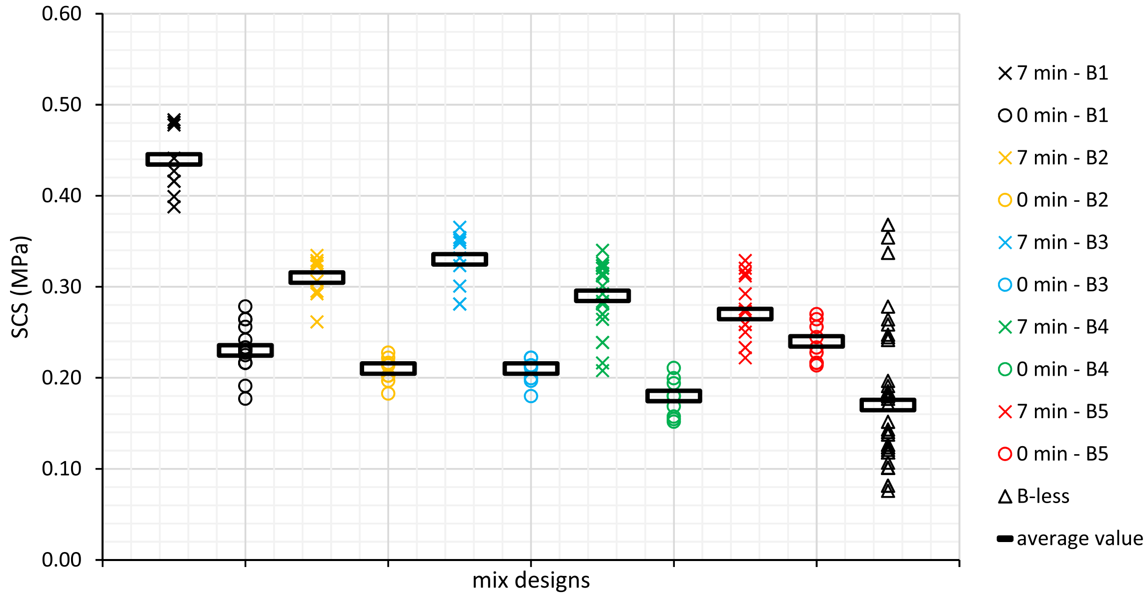

Fig. 10. SCS of studied mixes for a curing time of 1 h.

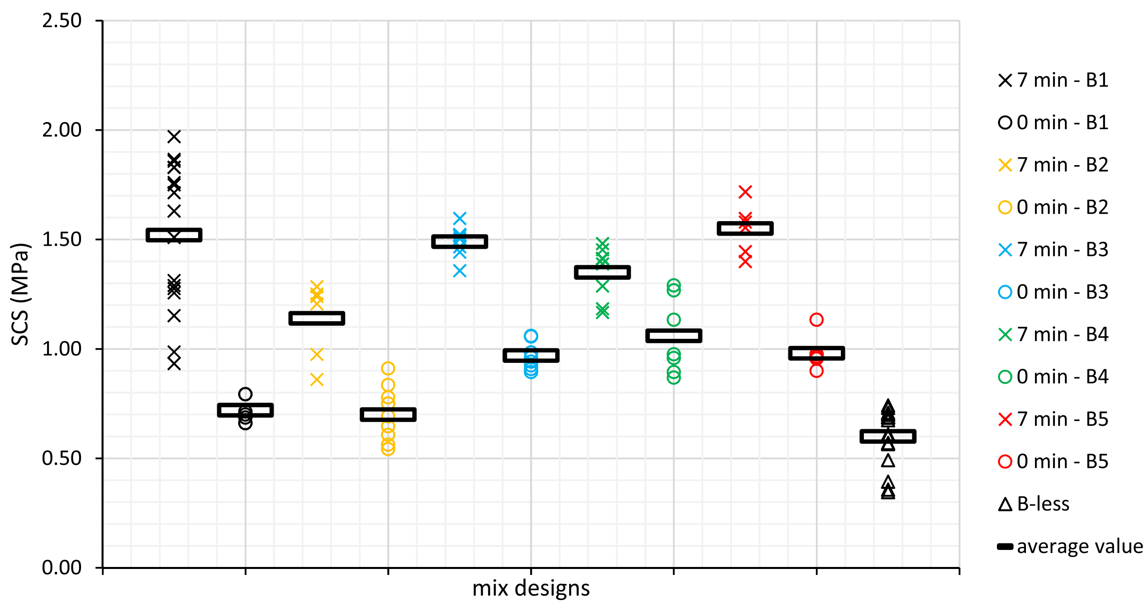

Fig. 11. SCS of studied mixes for a curing time of 3 h.

standard deviation, coefficient of variation and α-level of T-test) are reported in Table 4, Table 5, Table 6 and Table 7. As concerning the α-level, refers to the Student test (T-test), a statistical tool that has been selected in order to analyse the stability and the quality of the results. More in detail, a T-student single-tailed model has been used, since the aim of the analysis was to check if the mean of the considered mix (H_0) differs significantly by the mean of the correspondent mix produced without the bentonite ($H_{B\text{-less}}$). Consequently, 2 hypotheses have been formulated:

1. (H_0) = ($H_{B\text{-less}}$) – the means are not significantly different;
2. (H_0) >< ($H_{B\text{-less}}$) – the means are significantly different.

Results of the statistical analysis are provided in terms of α-level (also called significant level), i.e. the probability of making the wrong decision rejecting hypothesis 1. Each mix design has been studied compared to its relative B-less.

For completeness, Fig. 14 and Fig. 15 depict the failure mode of samples tested at short and long curing times respectively. In Fig. 14, a sample of two-component grout has been tested with the penetrometer. In order to have a "natural" image of the failure mode, the sample have been first removed from the mould and then tested (differently by the standard procedure that prescribes performing tests constraining lateral displacements), continuing the pressure of the bit some millimetres over the standard limit (5 mm), causing voluntarily the splitting of the sample. This procedure has been followed since other attempts to obtain a cross-section by sawing the sample have led to an unclear picture. However, analysing Fig. 14 a failure mechanism similar to the punching failure (the typical trapezoidal acting surface against the punctual load) is clearly visible at the bottom of the picture.

Considering instead Fig. 15, on the left a sample broken after the tensile test is depicted. Due to the nature of the test and according to the authors' experience, the breaking point on the bottom of the sample can slightly shift from the centre of the sample, due to the presence of weak

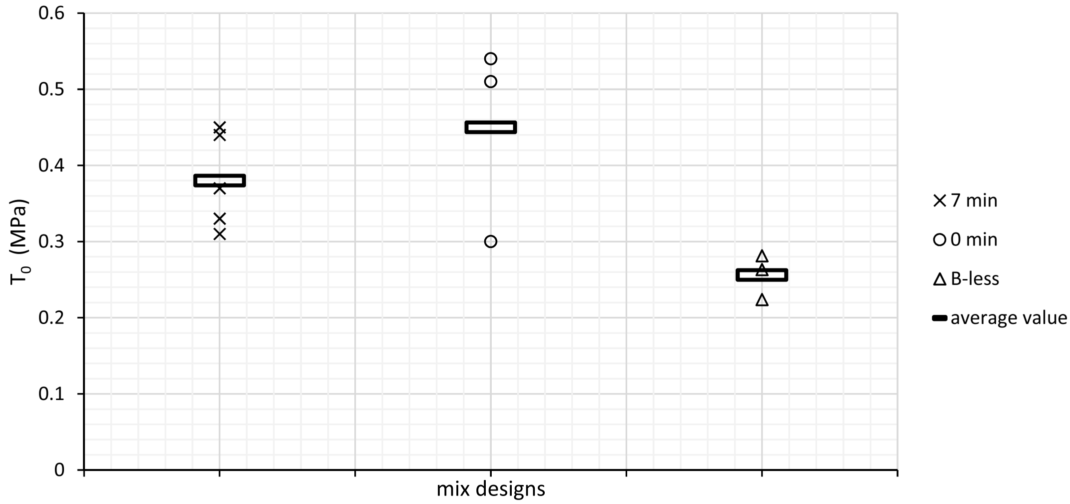

Fig. 12. T_0 of studied mixes for a curing time of 28 days.

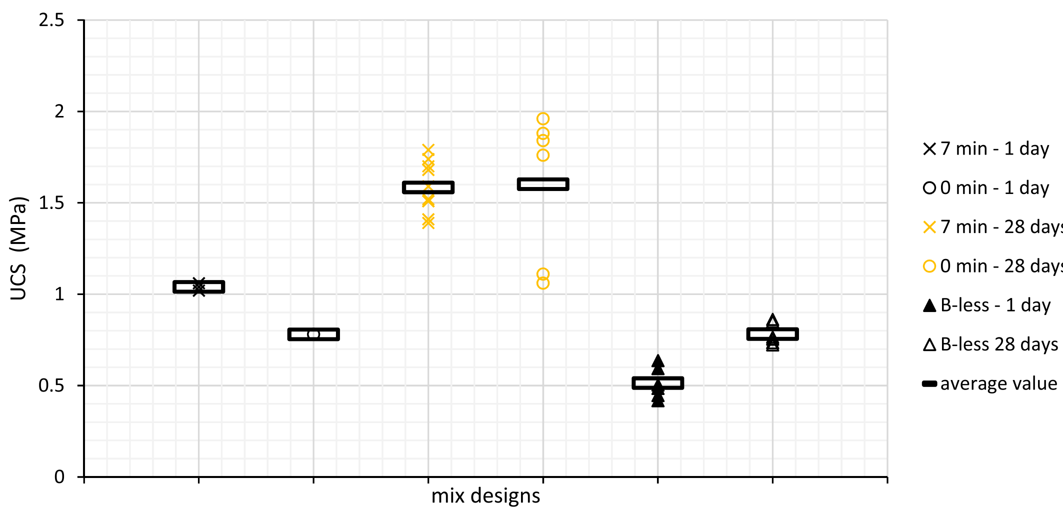

Fig. 13. UCS of studied mixes for a curing time of 1 day and 28 days.

Table 4
Statistics of SCS assessments for a curing time of 1 h.

Mixes	7 min B1	0 min B1	7 min B2	0 min B2	7 min B3	0 min B3	7 min B4	0 min B4	7 min B5	0 min B5	B-less
Mean	0.44	0.23	0.31	0.21	0.33	0.21	0.29	0.18	0.28	0.24	0.16
Standard deviation	0.04	0.03	0.02	0.01	0.03	0.01	0.04	0.02	0.03	0.02	0.06
Coefficient of variation	0.08	0.11	0.08	0.07	0.10	0.07	0.13	0.12	0.13	0.09	0.37
α-level of T-test	0.5 ‰	0.5 ‰	0.5 ‰	1%	0.5 ‰	1%	0.5 ‰	hypothesis H_0 cannot be rejected	0.5 ‰	0.5 ‰	/

Table 5
Statistics for SCS assessments for a curing time of 3 h.

Mixes	7 min B1	0 min B1	7 min B2	0 min B2	7 min B3	0 min B3	7 min B4	0 min B4	7 min B5	0 min B5	B-less
Mean	1.52	0.72	1.14	0.70	1.49	0.97	1.35	1.06	1.55	0.98	0.60
Standard deviation	0.32	0.06	0.17	0.13	0.07	0.06	0.12	0.17	0.11	0.08	0.13
Coefficient of variation	0.21	0.08	0.15	0.18	0.05	0.07	0.09	0.17	0.07	0.08	0.22
α-level of T-test	0.5 ‰	2.50%	0.5 ‰	5%	0.5 ‰	0.5 ‰	0.5 ‰	0.5 ‰	0.5 ‰	0.5 ‰	/

points. On the right of Fig. 15, instead, two different samples after a UCS test are depicted. The two different breaking shapes reported are the two extreme situations: a compressed sample, without a particular shape (Fig. 15 – A) and the typical hourglass shape (Fig. 15 – B). All samples have a failure shape that can be recognised between these 2 models. At present, it is not clear which parameter (or parameters) of the grout, of

Table 6
Statistics for T_0 assessments for a curing time of 28 days.

Mixes	7 min	0 min	B-less
Mean	0.38	0.45	0.26
Standard deviation	0.06	0.13	0.03
Coefficient of variation	0.17	0.29	0.11
α-level of T-test	2%	between 5% and 10%	/

Table 7
Statistics for UCS assessments for a curing time of 28 days.

	7 min 1 day	0 min 1 day	7 min 28 days	0 min 28 days	B-less 1 day	B-less 28 days
Mean	1.04	0.78	1.58	1.60	0.51	0.78
Standard deviation	0.02	#N/A	0.14	0.41	0.09	0.06
Coefficient of variation	0.02	#N/A	0.09	0.25	0.17	0.08
α-level of T-test	0.5 ‰	#N/A	0.5 ‰	1 ‰	/	/

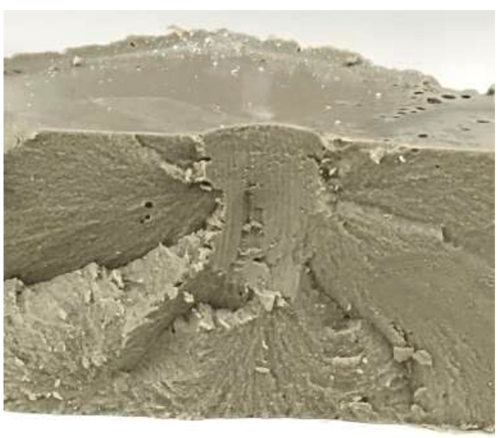

Fig. 14. Failure mode of the sample tested with the penetrometer for the SCS assessment. The trapezoidal surface acting against the applied punctual load is visible at the bottom of the picture.

the sample casting, or of the batching phase mainly controls the failure shape.

4. Discussion

Regarding the gel time, Fig. 7 shows that this parameter seems to be independent of the type of bentonite used, as well as their activation. Above all, gel time seems to be independent of the presence of the bentonite. This last remark is very important since it is in contrast to some scientific contributions (Pelizza et al., 2010, 2011) which highlight the involvement of the bentonite in the gelation process. Without activation, the gel time results in an average close to 7 s but, with activation, the gelation occurs in about 6 s, on average. According to the authors' experience in the field of two-component grout, a precision of ± 2 s is intrinsic of the performed test since the measures are affected by the involved operators (Todaro et al., 2019). Consequently, the gel time can be considered to be unaffected by the bentonite activation. Finally, the gel time of approximately 7.5 s assessed for mix B-less can also be considered as being aligned to the others.

Pertaining to viscosity, observable increments occur due to the activation time, according to Todaro et al. (2022). It can be observed that, irrespective of the bentonite used, an increasing percentage of flow time ranged between 6.6 and 17.7% (mix B3 exhibited the smaller increment of the reported range, while mix B5 the larger one) has been obtained thanks to the activation process. It should be noted that mix B-less is well aligned to the trending of the tests performed without activating the bentonite (about 28 s). Consequently, it can be stated that, concerning the viscosity, the presence of the bentonite in the mix design is irrelevant if the bentonite is not activated. However, Marsh funnel tests were performed immediately after the end of the batching phase and it cannot be excluded that, even without activation, the bentonite can swell with time, leading to higher flow times after 1 or 2 days after batching. The reason for limiting flow time assessments to fresh component A is the stability; without activating the bentonite, the obtained component A is highly unstable (see the paragraph related to the bleeding assessments). Instead, considering the results for the activated bentonite, it is notable that B1 and B5 exhibited higher flow times, about 33 and 34 s, respectively, compared to 31 s for other mixes. By analysing Table 2, it is notable that bentonite 1 and 5 are characterised by the higher value of SWI.

Bleeding is strictly correlated with the time of activation of the bentonite, where a higher activation time creates a better structure and bonds between bentonite particles, providing a higher capacity to stabilise the admixture. The type of bentonite also influences this behaviour: some bentonites are characterised by a better stabilising capacity than others. In the case of the batching procedure with bentonite activation, the average bleeding of the five mix designs is lower than 1%, after 1 h, and lower than 2% after 3 h. However, by analysing Fig. 9, it is clear that the presence of the bentonite, even if not activated, has an effect on the mortar stability. For example, mix B3 (the worst mix, in terms of bleeding for not-activated bentonite) reduces the bleeding by 64% and 22%, compared to the B-less mix design, for 1 and 3 h, respectively. After 1 h, this effect is more evident since the B-less bleeding curves reach 90% of its maximum value. Taking into account the bentonite properties (Table 2), there is no evidence of correlation between the SWI and/or the smectite content and the bleeding.

As concerning the SCS, it should be strongly remarked that, according to the authors' experience, the casting phase of samples is very

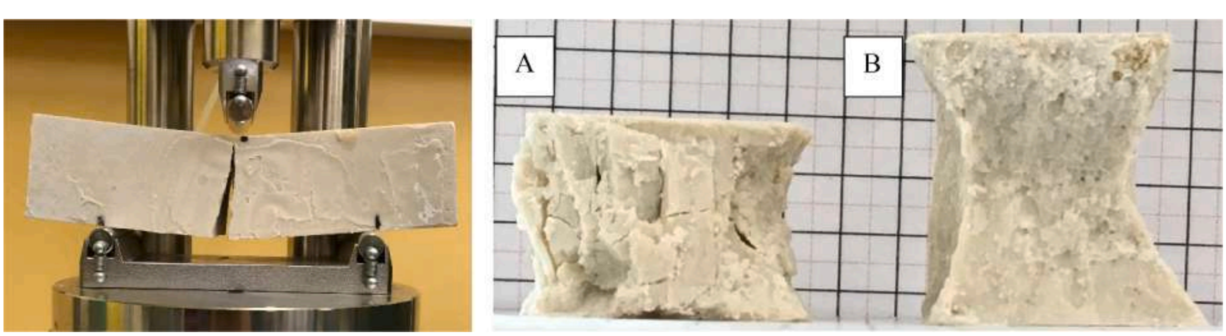

Fig. 15. Example of failure mode of samples tested at long curing time. On the left: failure of s sample during a tensile strength assessment. On the right: two different failure modes of samples after a UCS test: an irregular shape (A) and an hourglass shape (B).

important and that a wrong casting, embedding for example too much air in the sample can lead to strongly scattered data. However, in this research high level of care was taken for the sample production phase, according to Todaro et al. (2022). Analysing the results, it can be stated that the bentonite plays an important role. Furthermore, it should be always considered that the grout cured in a construction site is subject to a mechanical stress evolution due to the machine advancement (the application of the machine load) and that this effect has not been considered in this research.

Considering 1 h of curing, it should be reported that the tests performed exhibited a standard deviation (SD) ranging between 0.01 and 0.04 MPa for mixes B1-B5, while B-less was characterised by a slightly higher value of SD, equal to 0.06 MPa. It can be speculated that, for the analysed curing time, the absence of bentonite in component A leads to more scattered results, in terms of SCS. It can be hypothesized that a two-component grout material produced without bentonite is less homogeneous, reason for the higher dispersion. From analysing the data, SCS is higher when the bentonite is activated, compared to cases of no activation. Taking into account 1 h of curing, an average SCS of 0.21 MPa was found, in the case of no activation, while SCS reaches an average of 0.33 MPa when the activation is performed. It is also important to note that the mix B-less exhibits an SCS comparable with values of mixes produced without activate the bentonite. Consequently, in terms of strength at short curing times, it can be stated that if the bentonite is used but not activated its involvement in strength is negligible. On the other hand, all mixes with activated bentonite (7 min) exhibit SCS higher than the corresponding without activation (0 min) and higher than the mix B-less. Taking into account the coefficient of variation (CV), the value is always close to 0.1 and however never over 0.2. This value, according to the authors' experience with SCS test is acceptable. It is however important to underline the value of CV for B-less is higher than 0.35. This uncommon value could be due, as already written, to the absence of bentonite.

Wider data scattering was observable compared to the testing performed after 3 h of curing; SD ranged between 0.06 and 0.17 MPa. The only exception is related to the mix B1, where a SD close to 0.3 MPa was computed. This higher dispersion of data could be justified by considering the different temperatures of ingredients used for the batching and curing of these samples (tests performed in open spaces during summer and winter). Neglecting the high dispersion of mix B1, also for 3 h of curing, the influence of the activation process on the strength is appreciable. The effect of bentonite activation on the strength is more marked in samples with a curing time of 3 h: SCS has an average value of 0.88 MPa in the case of no activation, while it reaches an average of 1.41 MPa when activation is performed. The mix design B-less exhibits the lowest SCS (0.6 MPa). It can be speculated that, for a curing time longer than, or equal to, 3 h, the presence of bentonite (even if not activated) guarantees better mechanical performance, with respect to a mix prepared without using this ingredient. Considering CV, values slightly higher compared to tests performed at 1 h have been obtained. However, CV was for all mixes close to or lower than 0.2. B-less exhibited however the higher value of the set.

As for the previous tests, it is not possible to correlate the obtained SCS results specifically to the considered bentonite properties (Table 2).

As a final remark, outcomes relating to longer curing times, i.e. 1 and 28 days, have to be analysed. All of the tests achieved an SD lower than 0.14 MPa. The only exception was for the test set related to 'no activation bentonite – 28 days of curing' which yielded an SD of 0.41 MPa. This high dispersion was caused by two tests that exhibited values much smaller than the others, possibly because of hidden surface defects. Considering CV, the dispersion of data is confirmed with a value of 0.25. However, it has been decided to not remove the 2 lower data since it is not so rare, by working with the two-component grout, to have sometimes samples with mechanical performances slightly different from what is going to be expected. This behaviour can be also found by analysing two-component grout injected by the machine. The results from the 3-point flexural strength test and the UCS test show a higher development of the strength for the grout made with bentonite, compared to the one without it. The presence of the bentonite produces an increase in UCS of 53% and 104% at 1 day of curing, for 0 and 7 min of activation of the bentonite, respectively. Alternatively, at 28 days of curing time the activation of the bentonite seems to have no influence on the UCS, since the value is, in both cases, close to 1.6 MPa. An increment higher than 100% of strength is obtained with respect to the mix B-less, also at 28 days. Concerning the tensile strength, the use of the bentonite leads to an increment of 46% of T_0 in the case of 7 min of activation while, for the mix without activation, the increase is greater than 70%. This last result appears in contrast to globally obtained outcomes. It should be noted that, according to the authors' experience, T_0 can be strongly affected by the presence of bubbles trapped inside the grout (that lead to crack propagation aligned to the trajectory of less resistance dictated by the eventual bubbles) and this possibility is considered the reason for a CV close to 0.3 for the mix without bentonite activation.

Generally speaking, as concerning uniaxial compression test carried out on two-component grout performed at long curing time (more or equal to 1 day), CV values lower than 0.4 are acceptable, being common also to other authors. Briefly discussing the statistical analysis performed by using the T-test, the results obtained confirm that hypothesis 1 can be always rejected and this evidence confirms that the obtained averages are really different and that it is not a question of causality. The only case that shows the impossibility to reject hypothesis 1 is the case of "0 min - B4". However, this is not a critical issue if it is considered that according to the authors' experience and according to the evidence obtained in this work, B-less exhibits a SCS comparable with values of mixes produced without activating the bentonite.

5. Conclusions

This research continues the previous studies by Todaro et al. (2022) and Todaro and Pace (2022), and was conceived in order to study and provide answers pertaining to the influence of bentonite on two-component grout technology. The proposed study was designed with a comparative approach, i.e. a fixed mix design, realised by testing different bentonites (B1-B5) and compared to a special mix, made up without using any type of bentonite (B-less). This work can be considered well-structured since all of the tested mixes are comparable, considering the w/c ratio: the difference in water between mixes B1-B5 and B-less is largely inside the tolerance commonly acceptable in the batching station of construction sites (EFNARC, 2005).

According to the obtained results, density and gel time are not influenced by the bentonite activation time or by its presence in the grout, while the bleeding is strongly influenced by the use of bentonite in the mix design since the segregated water can be strongly reduced even if the bentonite is not activated. Concerning the strength, the presence of the bentonite, irrespective of the activation, strongly affects the SCS after 3 h of curing. SCS at 1 h of curing is clearly improved if the bentonite in the grout is used, but the activation is needed if increased SCS is required. At longer curing times (1 and 28 days), evidence related to shorter curing times are confirmed. Indeed, both the T_0 and UCS of grout with bentonite are higher than the same parameters related to a grout which is B-less. Furthermore, at 28 days of curing time, the activation phase seems not to play an active role pertaining to mechanical performance.

It can be speculated that when the bentonite is used and activated, since the grout completes the gelation, particles of cement are more homogeneously distributed in the sample. This better and more homogeneous distribution could be the reason for the better mechanical performances obtained. A component A prepared with bentonite has a homogeneous distribution of cement particles and this 'balanced distribution' is preserved after the gelation. Consequently, it can be assumed that a hardened sample of two-component grout, obtained from a component A produced with bentonite, is characterised by less potential 'weak-points' and this peculiarity leads to higher mechanical performances. However, this evidence is strongly appreciable at short curing times while, at long curing times (1 and 28 days), the presence of bentonite, also without activation, seems to guarantee better performances compared to the mixes prepared without bentonite. This work is absolutely the first that investigates this issue and further testing campaigns are necessary, in order to confirm what has been reported previously.

Concerning the type of bentonite, to date, it must be reported that it is not possible to unequivocally predict the behaviour of a two-component grout as a function of a specific bentonite, in terms of bleeding, viscosity or strength. If the gel time and the unit weight are clearly not dependent on the type of bentonite, the viscosity seems to be affected by the type of bentonite if an activation process is performed, as well as the bleeding (strong differences without performing the bentonite activation, differences of a smaller order of magnitude if the activation is carried out). Pertaining to the strength, if for long curing times appreciable differences between bentonites have not been recognised, the dependence of the SCS from the type of bentonite cannot be excluded. Unfortunately, the SWI and the smectite content, selected as bentonite-distinguishing parameters in this study, seem to not be effective 'prediction parameters'. It should be noted that special chemical additives are often used as ingredient in the commercial bentonite products, in order to confer certain properties to the grout. The presence of these additives could be recognised as the main cause of criticism of bentonite characterisation, since no information is provided on the datasheets of products. However, the effects of these additives on the characterisation test results are not negligible, consequently different bentonites can lead to different grout performances. Unfortunately, the potential presence of these additives is still undetectable and specific expansive tests (e.g. XRD) should be performed, even if the result in terms of identification is not ensured. Consequently, the cost of the chemical analysis could exceed that of the simple experimental approach which remains the method suggested by authors for the best bentonite selection in two-component grout applications.

Concluding, it is due to underline that even if the evidence introduced in this work constitutes important innovation on the key role of bentonite in the two-component grout technology, some chemical aspects linked to the dosage of component B on the bentonite and the infiltration of water between clay laminas have not been considered. Future investigations can improve the limited knowledge on the role of bentonite deepening also the chemical point of view.

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CRediT authorship contribution statement

Carmine Todaro: Conceptualization, Methodology, Validation, Formal analysis, Visualization, Investigation, Writing – review & editing. **Davide Zanti:** Investigation, Data curation, Writing – original draft, Writing – review & editing. **Andrea Carigi:** Investigation, Validation, Writing – review & editing. **Daniele Peila:** Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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