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Effect of Various Treatment and Drying Methods of Blood Meal Additive on Fresh State Properties of Grouting Materials

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Abstract: Grouting is one of the most commonly preferred repair methods used for historic buildings exposed to various deteriorating environmental conditions during their life span. Being that grouting is an irreversible method, it is crucial to determine the original material properties of the historic building to be repaired. In the literature, it is revealed that protein sources have long been used as popular additives to enhance the physical, mechanical, and durability properties of buildings. Specifically, blood additive has been known to have a potential to benefit as a water resisting or air-entraining admixture. Nonetheless, there is a lack of information regarding the use of animal blood in grouting materials, while the impact of blood additive on the properties of lime or cement-based mortars has been investigated recently. In this study, the impact of blood meal incorporation into the lime-based grouting material is discussed in terms of its fresh properties, i.e., fluidity, penetration, and stability. The blood meal samples to be tested were treated with various chemicals to enhance its performance and dried by various drying mechanisms. According to the test results, it was determined that the mixture containing blood meal treated with tri sodium citrate and dried in the drying oven met the requirements of all kinds of fresh state tests and had the potential to be used as a repair material.

Keywords: Civil engineering, Historical building, Grouting, Blood additive

Introduction

Damage in the historic building is an inherent situation due to their exposure to various deteriorating effects, which can amount to hundreds of years since the period they were constructed. Hence, the repair processes have a crucial role in sustaining the cultural identity of the society through historical buildings. There are different methods for repairing the cracks that occurred due to environmental, internal, or man-made sources (Feilden, 2003). The repair process begins with the determination and investigation of the damaged part of the building, and subsequently, the most proper repairing method and mixture for the repairing material are decided. In the repairing process, the original material properties need to be revealed in order for the repairing material to represent the mechanical, physical, microstructural, and durability properties. The repairing method and mixture of the repairing material have a critical role since the repairing is an irreversible process, and once the repairing method or mixture has an inconformity with the original material, it may bring about more severe damages to the building.

Grouting is one of the most commonly used repairing methods for consolidating masonry structures and is generally beneficial for strengthening and/or providing the structural integrity of buildings that have damaged through their structural members (Miltiadou-Fezans & Tassios, 2022; Penelis et al., 1989; Militadou, 1985).

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Besides, it is reported that the grouting method is used for the consolidation of the decorative elements as well (Bicer-Simsir et al., 2009; Bicer-Simsir & Rainer, 2013). The mixture designed as a grouting material consists of binder, water, and, if any, additives. Before the on-site application, the fresh state properties of the mixtures need to meet the requirements of the fluidity, penetrability, and volume stability criteria, which are called "injectability properties" as a whole (Miltiadou-Fezans & Tassios, 2013; Jorne et al., 2015). In the literature, researchers investigate the fresh state properties of the grouting materials with various mixture proportions or effects of introducing various additives (Bras & Henriques, 2011; Miltiadou-Fezans & Tassios, 2012; Miltiadou-Fezans & Tassios, 2013a; Miltiadou-Fezans & Tassios, 2013b; Baltazar et al., 2014; Jorne et al., 2014).

The obligation of compatibility with the original material properties imposes the use of lime as a binder in the grouting materials since cement has quite distinctive mechanical, physical, chemical, and durability properties that are incompatible with the properties of the original material (Baltazar et al., 2019). Nonetheless, both air lime and hydraulic lime have various deficient properties to escalate by benefiting organic or inorganic-based additives and/or admixtures. Previous studies have shown that organic additives were frequently employed during the construction of historic buildings during that time (Li & Zhang, 2018; Pahlavan et al., 2018; Elert et al., 2019; Zhang et al., 2021). Specifically, blood additive is reported as an additive used in historic buildings in ancient times, and the reproductions of the mixtures showed that the blood additive escalates the physical and durability properties of the mixtures (Pegoretti, 1843; Brey, 1844; Zhao et al., 2015).

The prominent properties provided by the blood additive encouraged the research conducted to investigate the impact of blood additive on the physical, mechanical, and durability properties of both cement and lime-based mortars, and it is shown that the blood additive enhances the water resistance and durability to freeze-thaw effect while diminishing the compressive and flexural strength of the mortar (Jasiczak & Zielinski, 2006; Xu et al., 2011; Fang et al., 2015; Dinc-Sengonul et al., 2023; Yuzer et al., 2023). There is, however, a knowledge gap about the characteristics of the lime-based grouting materials with blood additive in both their fresh and hardened states.

In this research, the fresh state properties, i.e., fluidity, penetrability, and volume stability of the blood meal containing lime-based grouting materials, are investigated to provide an insight into the fresh state applicability of the high-performance grouts in terms of their hardened state. The blood meal additives are treated with various chemicals to escalate the performance of the natural hydraulic lime based grouting materials and dried by various drying mechanisms.

Material and Methodology

The binder of the grouting materials in the scope of the experimental program is natural hydraulic lime (NHL 5), which is reported as the most appropriate binder for the repairing works. Animal blood that remains as waste during the slaughtering process is the raw material for blood meal, a commercially available product that is used as a feed for animals. The blood meal obtained after the heat treatment, drying, and grinding processes is utilized as an additive in the grouting material. The chemicals employed to treat the blood meal are stearic acid, citric acid, and tri sodium citrate. Besides, a surface-active organic-based sorbitan monooleate is used to adjust the foaming capacity of the mixtures.

The treatment process begins with preparing the treatment solutions, the proportions of which were determined by considering the preliminary test results and given in Table 1. The blood meal with a particle size of 100 μ m is added to the solution, and the mixture is subjected to one of the three different drying methods: drying oven, freeze dryer, or spray dryer. After the drying process, treated blood meal samples are ground and used as an additive in the grouting materials.

Table 1. Proportio	ons of treatme	ent solutions	
Treatment colution	Chamical	Water	

Treatment solution	Chemical	Water
Tri sodium citrate	0.1	1
Citric acid	0.5	1
Stearic acid	0.03	1

The grouting material mixing procedure is referenced in previously conducted research and consists of the following steps: (i) natural hydraulic lime and water are mixed for 10 minutes at 800 rpm; (ii) blood meal samples and, if any, sorbitan monooleate are added while the mixing continues; and (iii) the ultimate mixture is

mixed for another 3.5 minutes. The denotation of the samples and mixture proportions is given in Table 2. The freshly mixed grouting materials were subjected to injectability tests, i.e., fluidity, penetrability, and volume stability. The experimental research was designated as the mixtures were subjected to the Marsh cone test, sand column test, and bleeding test immediately after the mixing procedure was completed, respectively.

Mixture	Mixture Proportions				Blood Meal Treatment Solution and Drying Method	
	NHL 5	Water	Blood Meal	Sorbitan Monooleate	Treatment	Drying
T-O	1	0.8	0.01	0	Tri sodium citrate	Drying oven
C-0	1	0.8	0.01	0	Citric acid	Drying oven
S-O	1	0.8	0.01	0	Stearic acid	Drying oven
T-O-S	1	0.8	0.01	0.01	Tri sodium citrate	Drying oven
C-O-S	1	0.8	0.01	0.01	Citric acid	Drying oven
S-O-S	1	0.8	0.01	0.01	Stearic acid	Drying oven
T-F-S	1	0.8	0.01	0.01	Tri sodium citrate	Freeze dryer
C-F-S	1	0.8	0.01	0.01	Citric acid	Freeze dryer
S-F-S	1	0.8	0.01	0.01	Stearic acid	Freeze dryer
T-D-S	1	0.8	0.01	0.01	Tri sodium citrate	Spray dryer
C-D-S	1	0.8	0.01	0.01	Citric acid	Spray dryer
S-D-S	1	0.8	0.01	0.01	Stearic acid	Spray dryer

Table 2	Mixture	proportions
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Fluidity

One of the basic characteristics of a grouting material is having adequate fluidity to reach deeply into the cracks by flowing along the sinuous path. To provide this characteristic, the water/binder ratio and the amount of chemical additive used to adjust the fluidity of the grouting material, as well as the mixing duration and speed, are important parameters (Miltiadou-Fezans & Tassios, 2022).

In order to evaluate the fluidity of the grouting materials, Marsh funnel test was conducted according to the procedure given in ASTM D6910 (ASTM D6910, 2010). Since any requirement for the evaluation of fluidity performance is not given in the relevant standard, the criteria given in the referenced research are considered. According to the literature, 500 mL of the grouting material should flow in 45 seconds to have adequate fluidity (Kalagri et al., 2010; Miltiadou-Fezans & Tassios, 2012).

Penetration

The main principle of the grouting process is to provide structural integrity and strengthen the structure by filling the discontinuities, such as voids and cracks, through the structure, and it is crucial for the grouting material to pass through the narrowest discontinuities. Accordingly, the penetrability performance of the grouting material is measured according to the sand column test conducted according to BS EN 1771 by generating a medium with maximum and minimum grain sizes determined considering the crack widths to be grouted (BS EN 1771, 2004). In this experimental research, the granulometry of the grains used to model the cracks and voids was determined to be 2.0-4.0 mm. The elapsed time until the grouting material reaches the upper surface of the sand column is denoted as T_{36} , and this duration should be less than 50 s for an effective grouting application (Kalagri et al., 2010; Miltiadou-Fezans & Tassios, 2012).

The evaluation criteria given in the relevant standard label the grouting material as (i) "applicable" if the tested material passes through the column filled with sand and 20 mL of excessive material is collected; (ii) "feasible" if the sand column is passed through by grouting material but no excessive material is collected; and (iii) "difficult" if the sand column fails to be passed through by the grouting material (BS EN 1771, 2004).

Volume Stability

Being that the grouting material is a suspension with a high water/binder ratio to meet the previously explained fresh state requirements, segregation and bleeding are always the points in question. Limiting the bleeding of the

grouting material in the first 24 hours is critical to providing the flow of the grouting material through the voids and cracks without blocking them by precipitating or having a severe layering tendency.

To determine the volume stability of the grouting material, the procedure given in ASTM C940 is followed, and the bleeding is calculated by the formula given, in which the terms VW and Vg represent the volume of the water accumulated on top of the grouting material at the end of 24 hours and the total volume of the grouting material to be subjected to the bleeding test, respectively (ASTM C940, 2010). The bleeding value of the grouting material after 24 hours should be less than 5% for an effective grouting process (Bras & Henriques, 2012).

$$Bleeding(\%) = \frac{V_W}{V_g} \times 100 \tag{1}$$

Results and Discussion

The results of the fluidity, penetrability, and stability tests, which are evaluated as components of the injectability properties of the fresh-state grouting materials, are given in Table 3.

MixtureFluidityPenetrabilityVolume StabilityT-O134Not applicable-Difficult0ApplicableC-O150Not applicable-Difficult2.1ApplicableS-O80Not applicable-Difficult0ApplicableT-O-S34Applicable6Easy0ApplicableC-O-S39Applicable6Easy7.8Not applicableS-O-S50Not applicable-Difficult0ApplicableS-O-S50Not applicable-Difficult0ApplicableS-O-S50Not applicable-Difficult0ApplicableS-O-S50Not applicable-Difficult0ApplicableT-F-S62Not applicable-Difficult0ApplicableS-F-S60Not applicable-Difficult0ApplicableT-D-S49Not applicable-Difficult0ApplicableC-D-S40Applicable6Feasible5.4Not applicableS-D-S59Not applicable-Difficult0Applicable			5	7			
MixtureFlow time (s)Evaluation T_{36} (s)EvaluationBleeding (%)EvaluationT-O134Not applicable-Difficult0ApplicableC-O150Not applicable-Difficult2.1ApplicableS-O80Not applicable-Difficult0ApplicableT-O-S34Applicable6Easy0ApplicableC-O-S39Applicable6Easy7.8Not applicableS-O-S50Not applicable-Difficult0ApplicableS-O-S50Not applicable-Difficult0ApplicableS-O-S62Not applicable-Difficult0ApplicableC-F-S40Applicable7Easy6.3Not applicableS-F-S60Not applicable-Difficult0ApplicableT-D-S49Not applicable-Difficult0ApplicableC-D-S40Applicable6Feasible5.4Not applicableS-D-S59Not applicable-Difficult0Applicable	Mixture	Fluidity	Penetrability		Volume Stability		
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T-O-S34Applicable6Easy0ApplicableC-O-S39Applicable6Easy7.8Not applicableS-O-S50Not applicable-Difficult0ApplicableT-F-S62Not applicable-Difficult0ApplicableC-F-S40Applicable7Easy6.3Not applicableS-F-S60Not applicable-Difficult0ApplicableT-D-S49Not applicable-Difficult0ApplicableC-D-S40Applicable6Feasible5.4Not applicableS-D-S59Not applicable-Difficult0Applicable	S-O	80	Not applicable	-	Difficult	0	Applicable
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S-O-S50Not applicable-Difficult0ApplicableT-F-S62Not applicable-Difficult0ApplicableC-F-S40Applicable7Easy6.3Not applicableS-F-S60Not applicable-Difficult0ApplicableT-D-S49Not applicable-Difficult0ApplicableC-D-S40Applicable6Feasible5.4Not applicableS-D-S59Not applicable-Difficult0Applicable	C-O-S	39	Applicable	6	Easy	7.8	Not applicable
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S-F-S60Not applicable-Difficult0ApplicableT-D-S49Not applicable-Difficult0ApplicableC-D-S40Applicable6Feasible5.4Not applicableS-D-S59Not applicable-Difficult0Applicable	C-F-S	40	Applicable	7	Easy	6.3	Not applicable
T-D-S49Not applicable-Difficult0ApplicableC-D-S40Applicable6Feasible5.4Not applicableS-D-S59Not applicable-Difficult0Applicable	S-F-S	60	Not applicable	-	Difficult	0	Applicable
C-D-S40Applicable6Feasible5.4Not applicableS-D-S59Not applicable-Difficult0Applicable	T-D-S	49	Not applicable	-	Difficult	0	Applicable
S-D-S 59 Not applicable - Difficult 0 Applicable	C-D-S	40	Applicable	6	Feasible	5.4	Not applicable
	S-D-S	59	Not applicable	-	Difficult	0	Applicable

Table 3. Injectability test results of the mixtures

According to the flow durations of the mixtures without sorbitan monooleate, it is obvious that, regardless of the treatment type, the fluidity requirements are not met. Accordingly, it is an obligation to use an admixture that contributes to the fluidity performance of the grouting material. The comparison of the fluidity performances of each of the T-O, C-O, and S-O mixtures with and without sorbitan monooleate revealed that the sorbitan monooleate shortened the flow time. Hence, it is deduced that sorbitan monooleate is an effective admixture that provides the required fluidity to the mixtures.

According to the visual inspection, the foaming capacity of the blood meal varies for each of the treatment methods. Besides, it is seen that the mixtures without sorbitan monooleate, especially the C-O mixture, generate a significantly greater amount of foam, which has an impact on the fluidity of the mixture. After sorbitan monooleate is added, the mixtures including stearic acid-treated blood meal show noticeably greater foaming capacity relative to other treatments, regardless of the drying method, while the ones with citric acid show the least foaming capacity, on the contrary of the mixture without sorbitan monooleate.

For all the drying methods, grouting mixtures, including citric acid-treated blood meal, show applicable flow performances, while the shortest flow time is seen in the mixture with tri sodium citrate-treated blood meal dried in a drying oven. The effect of treated blood meal's drying method on the fluidity of grouting material shows no trend and varies for each of the blood meal treatment methods, while the overall performance of the mixtures, including blood meal dried in a drying oven, has the shortest flow time relative to the other drying methods when evaluated individually for each of the treatment methods.

The penetrability and fluidity performances of the mixtures show that these two phenomena are strongly related to each other. The mixtures that failed to meet the requirements of the fluidity test are evaluated as difficult to penetrate through the cracks according to the sand column performance, and this argument is valid for the

mixtures without sorbitan monooleate as well. Amongst other drying methods, grouting materials with blood meal dried in a drying oven after treatment shows more desirable performances than the others. It is also concluded that the grouting materials subjected to citric acid treatment show better performances than the other treatment methods.

Lastly, the bleeding performances of the mixtures show that the grouting materials with blood meal treated with either tri sodium citrate or stearic acid completely prevent the bleeding and layering tendency. It is seen that the drying method does not have a strong impact on the bleeding performance of the mixtures, including either tri sodium citrate or stearic acid. Moreover, mixtures including either tri sodium citrate or citric acid-treated blood meal result in a bleeding ratio of 0%, regardless of the sorbitan monooleate inclusion, in spite of the fact that the contribution of the sorbitan monooleate admixture to the fluidity of the mixtures was seen as a result of the Marsh cone test. Citric acid-treated blood meal-added grouting materials, however, show significant bleeding, which exceeds the limit value stated by the relevant standard.

Conclusion

- The fluidity values of the treated blood meal including grouting mixtures do not meet the requirements. Sorbitan monooleate admixture significantly shortened the flow time.
- The fluidity and penetrability characteristics of the mixtures are strongly related to each other.
- The foaming capacity has a strong impact on the fluidity and penetrability performance of the mixtures, and it is affected by the existence of sorbitan monooleate and the relationship between the treatment method of the blood meal and sorbitan monooleate.
- The favorable fluidity and penetrability performances of the C-O-S, C-F-S, and C-D-S mixtures are associated with the limited foaming capacity, while the ineffective bleeding performances are related to the possible weak adhesion of the mixtures.
- Amongst all the mixtures, the T-O-S mixture shows the most successful performance in terms of fluidity, penetrability, and volume stability by satisfying the requirements of the related limit values. Thus, this mixture can be used as a high-performance grouting material.

Recommendations

In light of the above findings, various blood meal ratios and the effect of the blood meal additive on the hardened state properties, i.e., physical, mechanical, and durability properties, should be discussed in further research. Additionally, the mixtures having the most desirable properties in terms of fresh and hardened state should be used in on-site grouting applications.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

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