Grouts with Improved Durability for Masonry Consolidation: An Experimental Study with Non-Standard Specimens

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Abstract. Multi-leaf stone masonry walls are among the most vulnerable elements of historic constructions. Grout injection is a common and efficient technique to consolidate such masonries. It consists of introducing a grout into the masonry inner core in order to upgrading the cohesion of the wall by ensuring the transversal bond between the external leaves and improving its monolithic behaviour. Notwithstanding, the recrystallization of salts due to changes in moisture content causes several damages in these masonries, even after the consolidation intervention. This paper aims to assess the potential use of linseed oil in natural hydraulic lime-based grouts to mitigate the water penetration and therefore the damages from salts crystallization. Linseed oil was used in former times as an additive for mortars in order to grant hydrophobicity. In this study several properties of the grouts were evaluated: rheology, mechanical strength, water absorption, adhesion and durability assessed by testing the resistance to sodium chloride. Moreover, this paper also analyses the correlation between non-standard specimens (with reduced size) and standard specimens (40x40x160 mm\textsuperscript{3}). The experimental results revealed that the grouts durability and water transport are significantly improved with added linseed oil. It was also possible to observe a small reduction in mechanical resistance with the presence of linseed oil; however, acceptable strength values to promote an appropriate consolidation were ensured. Furthermore, the reduced size specimens revealed to be a viable alternative to the standard ones.

Introduction

Old stone masonry walls present peculiar mechanical behaviour which is different from that of brick masonry walls or regular stone masonry in new buildings. The main problem concerns the load-carrying capacity of the multi-leaf stone masonry walls, especially when the masonry wall has to carry additional forces (for example a seismic loading). This kind of masonry was built many years ago and frequently designed only to resist static loads. The inhomogeneous inner core of the wall, missing interlocking and the poor bonding between leaves can lead to buckling and deboning of the external leaves and, consequently, the collapse of the whole building. Thus, the consolidation measures for old stone masonry walls have to take into account their specific requirements and, at the same time, not compromise the historical and architectural style of the wall/building. In this way, the grout injection (or grouting) is an effective technique to repair and consolidate stone masonry walls, since it provides a more homogeneous masonry’s inner core and increases the cohesion between masonry elements, with minimal changes in their morphology. Some laboratory studies have proven the promising contribution of linseed oil on improving durability of cementitious material by granting hydrophobic properties [1,2]. Linseed oil contains fatty acids which will be trapped inside the cementitious material and the hydrophobic part of the molecule will create hydrophobic behaviour [2]. The research presented in this paper focuses two main objectives: (i) to study the effect of linseed oil in the fresh and hardened properties of injection grouts and (ii) to analyse the influence of the size of test specimen on mechanical strength, capillarity, and durability towards sodium chloride ageing tests in order to evaluate the possibility.
of obtain a proper correlations between standard specimens and reduced size specimens (non-standard). The potential of using reduced size specimens was evaluated in this work in order to optimise the amount of material used in future experimental campaigns, within the framework of a research program involving nanoparticles.

**Experimental program**

**Materials.** Grouts were manufactured (Table 1) by changing the water/binder ratio (w/b), the high range water reducer (HRWR) content and the presence of linseed oil. Both linseed oil and HRWR was incorporated in weight percentage of total binder. Two grout types were prepared: single grout with natural hydraulic lime (NHL5) and binary grouts with NHL5 + cement (CEM II/B 32.5).

<table>
<thead>
<tr>
<th>Mix</th>
<th>Composition</th>
<th>Linseed oil (wt%)</th>
<th>w/b (-)</th>
<th>HRWR (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>NHL</td>
<td>-</td>
<td>0.50</td>
<td>0.8</td>
</tr>
<tr>
<td>II</td>
<td>NHL</td>
<td>1.5</td>
<td>0.50</td>
<td>0.8</td>
</tr>
<tr>
<td>III</td>
<td>NHL+CEM (60%+40%)</td>
<td>-</td>
<td>0.45</td>
<td>0.6</td>
</tr>
<tr>
<td>IV</td>
<td>NHL+CEM (60%+40%)</td>
<td>1.5</td>
<td>0.45</td>
<td>0.6</td>
</tr>
</tbody>
</table>

Grouts were prepared at room temperature of 20 ± 2 °C and a relative humidity of 60 ± 5 %. For the preparation of grouts a mechanical mixer was used. The mixture procedure adopted was the following: the whole powder was added to 70 % of the total mix water and mixed for 10 min. The remaining water (with diluted HRWR) is added within 30 s (without stopping the mixer). Regarding grouts with linseed oil addition, a bit of dry binder was blended manually with oil (oil/binder ratio of 3/4) in a plastic cup. Then the oiled mixture was added to the mechanical mixer 2 min before the introduction of the HRWR. After all materials had been added, the mixture was maintained for an additional 3 min. For each grout composition two different specimens were performed (see Fig. 1), namely the standard prismatic specimens of 40x40x160 mm$^3$ and the non-standard specimens (reduced size) of 20x20x40 mm$^3$.

![Figure 1 – Illustration of (a) standard specimen and (b) non-standard specimen](image)

**Experimental procedures.** The following tests were carried out to study the efficiency of linseed oil as additive on the fresh and hardened performance of injection grouts:

(i) **Rheological measurements** - to evaluate the flowability of grouts rheological measurements were carried out using a Bohlin Gemini HR$^\text{nano}$ rotational rheometer, equipped with parallel-plate geometry. The diameter of the geometry was 40 mm and the gap was set to 2 mm. To avoid the undesirable influence of any previous shear history, fresh samples were subjected to a pre-shearing at an identical shear rate of 1 s$^{-1}$ for 1 min and left standing for an additional 1 min before measurements took place. The sample was then subjected to a stepped ramp with shear rates from 1 to 100 s$^{-1}$. According to previous studies [3-6] the NHL grout’s rheological behaviour can be modelled fairly well using the Bingham model (Eq. 1).

\[
\tau = \tau_0 + \eta \times \dot{\gamma}.
\]

where: $\tau$ is the shear stress (Pa), $\tau_0$ is the yield stress (Pa), $\eta$ is the plastic viscosity (Pa.s) and $\dot{\gamma}$ is the shear rate (s$^{-1}$).
(ii) **Water absorption by capillarity** – to evaluate the capacity of the grout to absorb water by capillarity a test based on the standard EN 1015-18 [7] was performed.

(iii) **Flexural and compressive strength** – to evaluate the mechanical resistance of the grouts. The mechanical strength tests were done following standard EN 1015-11 [8].

(iv) **Adhesion** – to evaluate the adhesion strength between the grout and two types of support, mortar and ancient brick. The adhesion was analyzed through the flexural tensile test using mixed specimens made with half grout and half support (as illustrated in Fig. 5a).

(v) **Durability** – to evaluate the durability of grouts the specimens were exposed to artificial ageing test. Specimens underwent 15 cycles of dissolution and crystallization of salt, using a solution of sodium chloride (14 % volume concentration) as by the standard EN 12370 [9].

**Results and discussion**

**Rheological measurements.** Rheological properties of injection grouts are decisive parameters since they affect the fresh performance and therefore the success of the consolidation operation. Fig.2 presents the evolution of the yield stress and plastic viscosity of each grout composition. A smaller yield stress and plastic viscosity is better than larger ones. From the rheological parameter obtained, it is clear that the addition of linseed oil increases the yield stress values. The yield stress values of mixtures with oil (mix II and IV) increase to double in comparison to mixtures without linseed oil (mix I and III). The yield stress values enable to understand the energy required for grout to start flowing, since it represents that threshold. Regarding the plastic viscosity, it can be noted that it also increases when linseed oils is added. So, based on these results, it can be concluded that the linseed oil negatively affects the grout’s fluidity which can be seen as a disadvantage.

**Flexural and compressive strength.** From Fig. 3a, it can be observed that the addition of linseed oil leads to a reduction of flexural strength ($R_f$). $R_f$ values decrease about 71% between the mix I and mix II in the standard specimens and about 46% in the non-standard specimens. Between mix III and IV the decrease is about 20% in both specimens types. These results can be compared with those obtained by Justnes [10], where reductions of about 15% in mechanical strength in cement based mortars incorporating linseed oil were observed. Concerning the compressive strength (Fig. 3b), it is clear that for both single and binary grouts there is a decrease of about 40% in compressive strength values when the linseed oil is added, regardless of the type of specimen. Nevertheless, the typical compressive stress in old masonry is in range of 1 to 2.5 MPa [11,12] and all the analysed grout’s compositions have higher compressive strength.
Considering the relationship between the mechanical strength values of standard and non-standard specimens presented in Fig. 4, it is clear that similar trends and good correlation coefficients ($R^2 = 0.99$) were obtained. Given that, it can be concluded that non-standard specimens can be used instead of standard ones.

**Adhesion.** Grout adhesion to two types of support (ancient brick and air lime mortar) was studied through mixed specimens (see Fig. 5a) that were analyzed by flexural tensile test. The flexural strength values obtained at the interface between the grout and the two types of support are presented in Fig. 5b.

From the results presented it is clear that the adhesion between the grout-brick is higher that the adhesion between grout-mortar. The grout produced with linseed oil (mix II) presents a higher adhesion to both supports types. Moreover, it should be noted that the type of failure observed for
the grout without oil (mix I) was an adhesive failure (see Fig. 6a), in which it is evident the clear separation between the two materials and that only a small permanence of the grout on the brick surface was found. On the other hand, the grout with linseed oil (mix II) a cohesive failure within the grout occurred, showing a much better adhesion between these two materials (see Fig. 6b).

Figure 6 – Illustration of type of failure (a) mix I - adhesive failure, (b) mix II - cohesive failure

**Water absorption by capillarity and drying.** Water is often present in historic buildings and creates problems to the consolidation grouts. Water permits salts to deposit, biological growth, frost action, erosion and softening of the grouts. So it is desirable to have grouts with low water absorption. Based on the water capillarity results it was possible to get the capillarity absorption curve, which relates the time (in the abscissa axis) and the mass of water absorption (in the ordinate axis). Through this curve it can be obtained the capillary coefficient (CC) that represents the initial speed of capillarity absorption and is determined by the slope of the initial section of the water absorption curve. Analyzing the CC values shown in Fig. 7a it can be noted that the CC significantly decreases (reaching a reduction of 90%) when the linseed oil is added. The results obtained can be compared with those of Čechová [1], who reported similar reduction of CC of hydraulic lime mortars with linseed oil.

![Capillarity absorption curve](image)

Figure 7 – Grout behaviour against water (a) capillary coefficient, (b) drying index

The drying of grouts was also analysed since it reflects how the removal of water vapour through the grout occurs. From the drying test it can be determined the drying index value (based on the drying curve) which reflects the global drying behaviour of the grout i.e. a reduced drying index means an easier drying behaviour. As expected the specimens without linseed oil have a higher drying index because they also absorbed higher water content. It can also be noticed that the binary grouts have a lower drying index because the presence of cement. Fig. 8 shows that the correlation coefficients obtained between standard and non-standard specimens are very good ($R^2>0.9$) (as happened with the mechanical strength results), validating once again the use of small size test specimens for this kind of test.

![Drying index](image)
Figure 8 – Correlation between standard and non-standard specimens for (a) water absorption by capillarity and (b) drying index

**Durability - resistance to artificial ageing test toward sodium chloride.** The change of mass of each grout specimen as functions of ageing cycles (i.e. wetting-drying cycles) is shown in Fig. 9.

![Graph showing mass change](image)

Analysing the results it can be noted that at the beginning of the test there is an increase of mass of specimens for mixtures without linseed oil (mix I and III). This mass gain is explained with the salt solution filling the pore of grout’s microstructure that makes the grout specimens seem denser. As the wet-dry cycles are imposed the salt crystallizations leads to cracking due to internal stress which, therefore, makes the specimens integrity diminish. Considering for example the mix I a significant decrease of mass can be observed as soon as cycle 7 is reached; wherein in cycle 12 this mixture has already lost 50% of its initial mass. For the specimens of mix III the mass reduction became more expressive only from the cycle 11. On the other hand, in the mixtures containing linseed oil (mix II and IV) a significant mass decrease during the cycles was not found. From Fig. 10 is can be observed the reduced action of the salts in the specimens with addition of linseed oil. Based on the results obtained, it can be stated that the addition of linseed oil improves the grout behaviour relative to water. This phenomenon can be explained by the presence of fatty acid (component of linseed oil) that is entrapment in the grout, thus causing hydrophobic behaviour.

![Images of specimens](image)
Conclusions

This study assessed the efficacy of linseed oil as a hydrophobic additive on fresh and hardened performance of injection grouts for old stone masonry consolidation. The rheological results have shown that addition of linseed oil increases both the yield stress and plastic viscosity, which is not desirable.

It was found that the mechanical strength values decrease with the presence of linseed oil; however, the mechanical strength values are acceptable for the purpose of consolidating old masonry. Moreover, it was possible to verify that the linseed oil improves the adhesion between the grout and the support in particular to the brick type. Taking into account that grout’s flexural and compressive strength are less determinant that its adhesion to get a successful consolidation process, it can be said that the linseed oil can not be seen as harmful to the strengthening function of injection grouts.

Water absorption by capillarity proved that the linseed oil is an efficient water-repellent since the capillary coefficient was reduced up to 90% in the presence of linseed oil. This way, the harmful consequences of water can be minimized as shown by the ageing test with sodium chloride.

Considering all the results obtained it can be stated that non-standard specimens is a valid alternative to standard ones, allowing to save around 65% of material. It is clear, however, that additional characterisation of the contribution of linseed oil in grout injection are needed, such as the analysis of the performance of those composition during injection test as the ones used in Jorne et al. [13].

Acknowledgments

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