ABSTRACT:
Building with earth is one of the most ancient and traditional ways of building. Consequently, many historic and new-built earthen buildings can be found all over the world. However, the specific characteristics of earthen materials make the earth constructions very sensitive to external damaging agents. In particular, the weakness and fragility extensively recognized on earthen materials make this type of constructions very vulnerable from a structural perspective. In general, the structural damage on earth constructions occurs in the form of cracks, which debilitate the structural behaviour and compromise the durability. Injection of mud grouts can be seen as a reliable and economic solution for repairing earth constructions. The success of such intervention on a historical construction requires following an appropriated design methodology. However, the knowledge about mud grouts is still limited, and thus research efforts are needed in order to develop such methodology and to surpass many of the difficulties that the restorers of earth constructions are currently facing. Understanding the rheology of the mud grouts is one of the key issues of the all process. Hence, this paper explains an initial proposal of methodology for the design of mud grouts and an experimental campaign that aimed to understand the role of the clay fraction in the rheology of mud grouts.

Keywords: Adobe, rammed earth, mud grout, rheology.

1 INTRODUCTION
Building with earth is one of the most ancient and traditional ways of building. Nowadays, earthen buildings can be found all over the world. However, they show special presence in developing countries, where modern building materials are still limited resources and present unaffordable costs for the majority of the population. Therefore it is understandable that the tradition of building with earth was maintained till the current days. In fact, the one third of the world population that currently lives in earthen houses proves the statement that earth is still a popular building material [1].

Obviously, a great stock of valuable heritage places and buildings resulted from the extensive use of earth as building material. This explains why, in 2007, around 16% of the cultural heritage at the UNESCO world heritage list was partially or entirely built in earth [2]. Despite of the successful use of earth as a building material through the times, several problems are recognized on the constructions made with this material. Once again, this is reflected by UNESCO in the world heritage at risk list, where, currently, 31% of the cultural heritage is partially or entirely built with earth [3].
In fact, the earthen materials are very sensitive when compared with modern materials. In general, they are more vulnerable to external aggressive agents, revealing most of the times a faster degradation rate [4]. Moreover, earthen materials are known for their low strength and brittle behaviour, which make the buildings made with these materials very vulnerable, especially from a structural perspective. In part, these limitations of the earthen materials are responsible also for the seismic vulnerability of the earth constructions. The condition of the structure is another major factor contributing to the seismic vulnerability of earth constructions. In general, the structural damage on this kind of constructions is reflected in the form of cracks, loss of material and partial collapse of the structural elements, resulting from several factors such as foundation problems, structural defects, shrinkage, biological activity and earthquakes [5]. The presence of major cracks on the walls of an earth construction is undesirable, since the parts divided by the cracks are unable to work together. Therefore, repairing these cracks is essential to recover the monolithic behaviour, and thus improving the structural behaviour of the damaged construction.

In general, cracks are repaired by hiding them aesthetically with plaster or by filling them with coarse material (pieces of bricks and stones) and huge quantities of mortar. However, proceeding this way does not promote an effective repair solution, since it does not assure an acceptable continuity to the wall, neither assures the bonding of the repairing materials to the damaged wall, which frequently causes more problems than it solves [6]. According to Keefe [6], the structural repair of cracks on monolithic walls of earth constructions, requires cutting horizontal chases with a determined spacing, in order to create a key pattern around the crack. The removed material is then replaced by new compatible materials, such as mud bricks and earth mortar, which must be well bonded to the original wall material, in order to promote a mechanical connection between both sides of the crack. However, this can be very disturbing and intrusive to the construction, since the wall may became unstable when the chases are cut, due to the reduction of the cross section.

Grout injection can be applied with the same purpose of the repair technique proposed by Keefe [6]. As an advantage, grouting constitutes a solution that is more practical and a less intrusive. Additionally, grouting can also be used to consolidate voids and gaps caused by biological activity, such as the penetration of plant roots or tunnels excavated by animals, or to complement other strengthening/repairing techniques, such as the application of ties. In non-monolithic earthen structures, such as adobe masonry, grouting can also be used for repairing cracks on walls, which can only be acceptably achieved by rebuilding the wall partially.

Nevertheless, the repair of earth constructions through grout injection requires the development of specific grouts that assure the success of the intervention, especially in the case of historical constructions. Currently, the trend of the restorers and conservationists of earth constructions is to adopt grouts where earth is used in the composition (mud grouts) [7-10]. This option arises from the necessity to employ grouts compatible with the original earthen materials. However, the properties of mud grouts are still not satisfactorily studied, which hinders the further development of methodologies and guidelines for their design.

The rheology of fresh mud grouts is a key issue in the grouting process, since the prepared mud grout needs to present, between other requirements, an adequate injectability in order to allow a successful intervention. The colloid behaviour of the clay fraction in a mud grout has great importance to its rheology. In fact, the colloid behaviour of clay particles is quite important since it may hinder the flow of the mud grout, which is undesirable for a grouting intervention.

This paper presents and discusses an initial methodology proposal for the design of mud grouts for historical earth constructions. Moreover, the role of the clay fraction in the rheology of mud grouts during the fresh state is also analyzed. This was carried out by measuring the flow time in a Marsh cone of several suspensions containing different proportions of kaolin, limestone powder and clay dispersant (sodium hexamethaphosphate). Additionally, rheological measurements were carried out with a Viskomat mixer-type rheometer in the stiffer suspensions that were unable to flow through the Marsh cone.
2 DESIGN METHODOLOGY FOR MUD GROUTS

Following a methodology for designing mud grouts is fundamental to promote successful grouting interventions on historical earth constructions. The design methodology of grouts for consolidation of historical masonry is a procedure exhaustively discussed during many years, although not yet well established [11]. In terms of discussion, the same cannot be stated for earth constructions. Nevertheless the principles that rule the methodology for historical masonry are common to a possible methodology for designing mud grouts. Therefore, the methodology for historical masonry can work as good starting point. Yet it must be adapted to the specificities of earth constructions and to the properties of mud grouts.

The design of a mud grout is a complex task that assembles several variables depending on each other. In order to design properly a mud grout a “non-linear system” has to be solved. The variables in this system are the properties that define the mud grout in both the fresh and hardened states (see Figure 1): rheology, strength, fresh state stability, bond, chemical stability and microstructure. The definition of each one of these variables accounts for requirements related to the mechanical behaviour and durability of the earth construction. As result of the balance between the properties that best suit the requirements of the construction, the materials that constitute the mud grout and their proportions, can be established.

During a grouting intervention it is aimed to completely fill cracks or voids/gaps with the injected mud grout. Therefore, the mud grout needs the appropriate injectability, i. e., it must be fluid enough to penetrate through the crack or network of voids/gaps and reach every single empty space. This is essential, for example, to assure the continuity of damaged earthen walls, and in this way to re-establish the monolithic behaviour of the earthen structure. Thereby, all the factors influencing the rheology, such as the texture (particles size, distribution and shape), the interaction between particles (dispersion or flocculation), the water/solids ratio, the mixing procedure, the injection process and pressure, and the action of superplasticizers or dispersants, must be carefully accounted for during the design of a mud grout. In fact, from the previous factors, the interaction between clay particles is of great importance to the rheologic behaviour during the fresh state. In normal conditions, clay particles tend to flocculate and to form a gel that opposes to the flow, which does not allow the injection of the mud grout without employing an exaggerated quantity of water in their composition. Moreover, the capacity of the dry earthen materials of absorbing quickly high amounts of water requires that the mud grout presents a great water retention capacity during its injection. This is essential in order to maintain its fluidity and penetrability during all the injection process.

Figure 1. Schematic representation of the methodology for the design of mud grouts [5].
Another important property of a mud grout is its strength. However, the general idea that strong mud grouts provide better structural results is misleading. Instead, the strength developed by the mud grout must be adjusted to the demands of the level of structural damage of the construction. To employ grouts much stronger than the original earthen materials is, thus, to be avoided. Moreover, stiff grouts can cause problems of mechanical incompatibility, since the hardened grout hardly follows the displacements occurring in the earth construction, resulting into damage to the intervention or construction. Thus, the addition of stabilizers, such as cement or lime, has to be carefully accounted, since they increase greatly the stiffness of the earthen materials and in this case of the hardened mud grouts.

Another objective of the grouting is to grant the continuity of the earthen structure, both for structural and durability reasons. The hardened grout is supposed to establish the bridge between the parts of a wall divided, for example, by a crack. Thus, both the strength developed by the hardened mud grout and the bond established between the original earthen materials and the hardened mud grout shall be enough for supporting this bridge. Only in this way it can be expected to achieve a structural state similar to that existing before the damage occurred. On the other hand, re-establishing the continuity broken by a crack hinders the propagation of damage on an earthen wall by, for example, enabling that the rain water penetrates through the crack to a major extension, which would decrease dramatically the strength of the wall. The bond established is highly dependent on the shrinkage of mud grout. The shrinkage of the mud grout during its drying can lead to cracking and consequently the required bond cannot be established, which is a problem common to other reparation works using earthen materials [6]. Necessarily, the shrinkage of mud grouts cannot be fully avoided, although it can be minimized, and for this several recommendations can be proposed. The first consists in adopting selected clays with low shrinkage ratio in the composition of the grout, such as kaolinite clays. The texture of the mud grout is another parameter which can be intervened by, for example, decreasing the clay fraction of the mud grout and by correcting the particles size distribution with addition of “unshrinkable” fine material (fly ash, silica fume, calcium carbonate powder, quartz powder and etc.). Another possibility consists in decreasing the water content. However, this has direct consequences in the rheology, which can only be overcome by adding dispersants. Using stabilizers is also an alternative to solve the shrinkage problem, despite that, other problems can arise from this decision. The addition of cementeous materials to the mud grout implies that part of the water used for preparing the grout will be incorporated in the hardened grout, which helps reducing the shrinkage.

A mud grout also needs to present chemical stability over time, in order to not compromise the durability of the earth construction. The salt content has to be limited in order to avoid efflorescence and crypto efflorescence problems. Moreover, the grout must present resistance to aggressive compounds present in the original materials of the construction. For example if a possible mud grout contains Portland cement, there is a possibility of formation of expansive products since the presence of sulphates is very common on earthen materials. In this case the addition of lime or hydraulic lime would be a better alternative.

A mud grout must present stability during the fresh state. Therefore, it shall present limited bleeding, no segregation and adequate water retention. These are essential in order to assure that the mud grout maintains its fluidity and penetrability during the injection and remains homogeneous after hardening. Therefore, using an earth with large particles in the composition of the mud grout for overcoming the shrinkage problem can constitute a major drawback since they easily tend to settle.

Obtaining a hardened mud grout with a microstructure compatible to that of the original earthen materials is another essential condition for fulfilling the durability requirements of the earth construction. The water vapour that penetrates in the earthen materials has to be able to do it freely. If a grout with low porosity is injected, it can constitute a barrier, making the water vapour to condensate. This is harmful for the intervention or for the construction, depending on the size of the intervention. The condensed water can leech the material around the grout disturbing the bond created, and therefore, damaging the intervention. In the cases where a large grout barrier needs to be created, the condensed water can lead to the weakening of the earthen materials that at long-term can be responsible for an eventual collapse. Therefore, the incorporation of materials such as cement has to be carefully evaluated, since it reduces the porosity of the mud grout. The thermal properties of
the hardened grout must also be closer to the ones of the original earth materials. This is even more important in monolithic earth constructions (for example rammed earth), where the grout has to be able to follow the thermal displacements of the earthen materials.

3 THE ROLE OF THE CLAY FRACTION ON THE RHEOLOGY OF A MUD GROUT

The clay fraction in a mud grout can be designed to have an active or a passive function. In the first case, the clay fraction has the role of a binder in the grout. In the other case, the role of the binder is left to another component such as hydrated lime, hydraulic lime or (Portland) cement, which is responsible for developing the grout strength. In fact, the mud grout strength can be substantially increased by adding binders to their composition, although, this is most of the times not necessary. If the design of the mud grout is correctly done the clay fraction is sufficient to provide the required levels of strength. The addition of the aforementioned binders can have, however, a different objective in the mud grout, such as to limit the shrinkage or to improve the water resistance.

The experimental campaign presented in this paper is limited exclusively to the case where the clay fraction has an active role in the mud grout. However, it should be noticed that the addition of mineral binders to clay suspensions has great importance in the interaction between the clay particles, and therefore in the rheological behaviour of the suspensions [12]. The experimental work that is here presented and discussed, aimed to evaluate the importance and the constrains that the clay fraction has in a mud grout. This is part of a much more extended research that aims the development of the sate of knowledge about mud grouts.

3.1. Materials

In order to obtain a material that would be easily reproduced with similar properties during the experimental campaign it was decided to prepare an artificial soil, instead of using a natural soil. This artificial soil will be used further for the preparation of mud grouts, and consists on the mixture of kaolin and limestone powder. The kaolin RR40 (*Wienerberger*, Belgium) used in the experimental campaign was provided as a dry powder. The chemical and mineralogical compositions were provided by the company *Wienerberger* and are given in Table 1 and Table 2, respectively. The limestone powder was provided by *Carmeuse* (*calcite 2001 S*). The particle size distribution of both materials was determined according to ASTM D 422 [13] and is presented in Figure 2. Additionally, it was used sodium hexametaphosphate (HMP) as a dispersant in some of the kaolin suspensions.

Table 1. Chemical composition (% per unit mass) of the *kaolin RR40*.

<table>
<thead>
<tr>
<th></th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>TiO₂</th>
<th>CaO</th>
<th>MgO</th>
<th>Na₂O</th>
<th>K₂O</th>
<th>L. I. (1000°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>48</td>
<td>34.5</td>
<td>1.9</td>
<td>1.9</td>
<td>0.44</td>
<td>0.18</td>
<td>0.04</td>
<td>0.04</td>
<td>14.29</td>
</tr>
</tbody>
</table>

Table 2. Mineralogical composition (% per unit mass) of the *kaolin RR40*.

<table>
<thead>
<tr>
<th></th>
<th>Kaolinite</th>
<th>Quartz</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>87</td>
<td>8</td>
<td>5</td>
</tr>
</tbody>
</table>
3.2. Methods

Several suspensions composed by kaolin, limestone powder and HMP were prepared and their flow time was tested according to ASTM C 939 [14], using a Marsh cone with high ratio outlet length over diameter to allow the laminar flow of the suspensions [15]. For this Marsh cone, the average flow time of 1 dm³ of water at an average temperature of 18.3 °C is 34 s. The suspensions were prepared by changing the components, the solid fraction (φ), the quantity of HMP as function of the clay weight ([HMP]) and the weight ratio between the kaolin and limestone powder (K/L), see Table 3.

Table 3. Suspensions prepared during the test campaign.

<table>
<thead>
<tr>
<th>Suspension</th>
<th>φ (%-v.)</th>
<th>[HMP] (kg/t)</th>
<th>K/L (kg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kaolin</td>
<td>4 / 5 / 6 / 7 / 8 / 9 / 10</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Kaolin + hexametaphosphate (HMP)</td>
<td>9 / 12 / 15 / 18</td>
<td>1.4 / 2.9 / 4.3 / 5.8 / 7.2 / 8.7 / 14.5 / 40 / 60</td>
<td>-</td>
</tr>
<tr>
<td>Kaolin (K)+ Limestone (L)</td>
<td>20 / 30</td>
<td>-</td>
<td>0.2 / 0.3 / 0.4 / 0.5 / 0.6</td>
</tr>
</tbody>
</table>

All the suspensions were mixed using a Hobart N50 planetary mixer with a wire whip paddle and by using the same procedure. First the components were mixed manually by adding increasing amounts of tap water. In the case of the blends that had two solid components, both were manually dry mixed in advance. In the case of the suspensions with dispersant, the HMP was firstly dissolved manually in the water of the suspension. After the manual mixing, the suspension were mixed with the mixer at speed 1 during 5 min and then left to rest during 1 min. Subsequently, they were mixed during 5 min at speed 2 and then more 5 min at speed 1 with a resting period of 1 min in between. During the resting periods the bowl was checked and the material stuck into the walls was scrapped. The long mixing procedure aimed to grant that all the suspensions were homogeneous.

Additionally to the flow time tests, the suspensions of kaolin and HMP that were unable to flow were tested using a Viskomat mixer-type rheometer with a mortar paddle of diameter 83 mm [16]. The applied profile had a stepwise increase from 20 to 120 rpm and a stepwise decrease from 120 to 20 rpm. The duration of each step was 30 s and the increment/decrement between steps was of 20 rpm.

Figure 2. Particle size distribution of kaolin and limestone powder.
3.3. Results and discussion

The flow time in function of the solid fraction of the kaolin suspensions is represented in Figure 3a. This figure shows that by increasing the solid fraction, the flow time increases as well, till a critical value is reached, after which the kaolin suspensions are unable to flow through the Marsh cone. This critical solid fraction was found to be between 9% and 10%, which is a very low value and is not suitable to prepare a mud grout.

The addition of HMP to the kaolin suspensions allowed increasing further the critical solid fraction as it can be seen in Figure 3b, where \([HMP]\) is represented in a base 2 logarithmic scale. This time it was possible to obtain flowing suspensions till a solid concentration of 21% for the range of \([HMP]\) tested. This figure also shows that the effectiveness of the HMP depends on the solid fraction of the kaolin suspension, i.e., the lesser is the solid fraction the lesser is the \([HMP]\) required to obtain suspensions capable of flowing through the Marsh cone.

The inability of the kaolin suspensions in flowing through the Marsh cone results from the interaction between the clay particles. In aqueous suspension, the typical plate-like kaolin particles present a surface charge both on the face surfaces (F) and edge surfaces (E). The face surfaces have a negative charge that results from isomorphous substitution that occurs in the surface layers of the crystalline structure, and therefore it constitutes a permanent charge. On the other hand, the charge on the edges is variable and depends on protonation/deprotonation reactions that occur on the silanol and aluminol groups exposed on this surface. Therefore, the charge of the edge surfaces depends on the pH of the medium, and it can be positive or negative if the pH is acidic or alkaline, respectively [17]. The interaction between the clay particles is then dominated by the electrostatic forces resulting from the heterogeneous surface charge and by the van der Waals attractive forces. In mediums with high ionic strength, the electrical double layers formed due to the surface charge tend to collapse and allow the attractive van der Waals forces to dominate over the repulsion forces promoted by the electrical double layers with same charge signal. This promotes the flocculation of the clay particles that can occur in three different modes: face-to-face (FF), edge-to-edge (EE) and edge-to-face (EF). The random flocculation of the clay particles, according to the previous modes, creates a “house-of-cards” structure, which makes the clay suspensions to exhibit a thixotropic behaviour at high shear rates that corresponds to the breaking of this structure [18]. This structure also opposes to the flow of the suspensions, which requires that a minimum stress develops before the flow starts (yield shear stress) that is related directly to the strength of this structure.

The prepared kaolin suspensions were expected to have high ionic strength, since the water used was tap water (and not distilled water) and the kaolin was not purified to eliminate salts that would contribute for increasing the ionic strength of the medium. Measurements of the pH also showed that the water used for preparing the suspensions had variable pH, still it was found to be slightly alkaline.

![Figure 3. Flow time tests: (a) suspensions of kaolin; (b) suspensions of kaolin + HMP.](image-url)
Even if the kaolin particles presented surfaces with the same charge signal, due to the alkaline pH, the ionic strength of the medium was enough to collapse the electrical double layers and make the attractive forces dominate over the repulsion forces. This made the kaolin particles to flocculate and oppose to flow through the Marsh cone. The addition of HMP allows disturbing the “house of cards” structure that the flocculated clay particles form. The HMP is a negatively charged polymer that is mainly adsorbed on the kaolin particles edges, which increases the overall negative surface charge and consequently promotes the repulsion between the kaolin particles. Moreover, the HMP has the ability to complex the dissolved flocculant alkaline earth cations and to replace them by sodium cations, therefore increasing the thickness of the electrical double layer [19].

The flow curves obtained from the measurements with the viskomat rheometer for the suspensions of kaolin + HMP with solid concentration 15% are presented in Figure 4. The hysteretic loop of the flow curves show an anti-thixotropic behaviour of the suspensions, which means that the rotation speed was not enough to break down the “house of cards” structure formed in these suspensions. Instead, the shear flow applied was helping to build up this structure. To the descending branch of each one of these curves a rheological Bingham model was fit, which can be applied in terms of torque ($T$) and rotation speed ($\Omega$) according to equation (1) [20]:

$$T = g + h \times \Omega$$

(1)

Where $g$ and $h$ are characteristic constants of the suspension that are related to the Bingham yield stress and plastic viscosity of the Bingham model, respectively. The disturbing power of the HMP is reflected essentially in the decrease of $g$ with the increasing addition of HMP, see Table 4. This means that the strength of the “house of cards” structure decreases with the addition of HMP, and consequently the resistance of the suspension to initiate the flow.

<table>
<thead>
<tr>
<th>[HMP] (kg/t)</th>
<th>$g$ (N.mm)</th>
<th>$h$ (N.mm.min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.4</td>
<td>43.81</td>
<td>0.0642</td>
</tr>
<tr>
<td>2.9</td>
<td>36.58</td>
<td>0.0412</td>
</tr>
<tr>
<td>4.3</td>
<td>30.85</td>
<td>0.0326</td>
</tr>
<tr>
<td>5.8</td>
<td>22.52</td>
<td>0.0402</td>
</tr>
</tbody>
</table>

Table 4. Rheological parameters of the kaolin suspensions with solid fraction 15%.

Figure 4. Flow curves of the kaolin suspensions with solid fraction 15%
Despite the addition HMP allowed increasing the solid fraction in the kaolin suspensions, this is still inadequate to produce a mud grout. Higher values of solid fraction are required to limit the shrinkage of the dried mud grout to acceptable values. The addition of fillers can be a solution to overcome this problem. Filler powders have, normally, particle sizes above the colloid dimension, whereby the particles interaction do not have the same remarked importance as for clay particles. To show this, suspensions of kaolin and limestone powder were prepared for which the flow time was, subsequently, tested for two solid fractions (20% and 30%). The results are presented in Figure 5. The content of kaolin is shown, once again, to be limiting the flow time of the suspensions. The higher is $K/L$ the higher is the flow time of the suspensions, which stop flowing for $K/L$ higher than 0.5, for the suspensions with solid fraction 20%, and for $K/L$ higher than 0.2 for the suspension with solid fraction 30%. Suspensions with solid fraction above 30% did not flow through the Marsh cone. This is still a very low value for preparing a grout without the occurrence of great shrinkage.

![Figure 5. Flow time tests of the suspensions of kaolin + limestone powder.](image)

\[ Flow time (s) \]

\[ 0 \quad 20 \quad 40 \quad 60 \quad 80 \quad 100 \]

\[ 0.0 \quad 0.2 \quad 0.4 \quad 0.6 \quad 0.8 \quad 1.0 \]

$\phi = 20\%$

$\phi = 30\%$

4 CONCLUSIONS

Grout injection of mud grouts has many advantages as a repair technique for earth constructions, when compared with traditional techniques for structural repair of cracks. Nevertheless, the knowledge about mud grouts is still very limited. The complexity of the mud grout design demands following a design methodology that grants safety and long-term durability. Therefore, an initial methodology was presented in this paper, based on the grout design methodology for historical masonry. However, the continuous development of this methodology can only be achieved if the knowledge about mud grouts is also developed through research.

In this paper an experimental campaign was presented that aims at understanding the importance of the clay fraction in the rheology of a mud grout. It was shown that the interaction between the kaolin particles is a drawback in the formulation of mud grouts since it develops a yield stress that hinders the flow. The addition of a dispersant (HMP) allows increasing the critical solid fraction of the kaolin suspensions. Still, its low value is not compatible yet with the formulation of a mud grout. The addition of filler (limestone powder) seems to be a promising solution to further increase this critical solid fraction, but again not enough to formulate a mud grout without shrinkage problems. Further experiments will test the addition of HMP to suspension of kaolin and limestone powder, in order to check to what extent the solid fraction can be further increased.
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