Construction and Building Materials 50 (2014) 352-360

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat



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• Development of a composition that is deemed consistent with the original render.

• Development of lime based grouts for consolidation.

• Rheological behaviour of analysis grouts.

• The rheological behaviour was evaluated with a proper mortar rheometer.

Study of the hardened state properties of grouts.

ARTICLE INFO

Article history: Received 4 March 2013 Received in revised form 4 September 2013 Accepted 6 September 2013

Keywords: Conservation Old renders Lime Mortars Grout Adhesion

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A study was conducted on the development of lime based grouts for consolidation of renders and plasters detached from the support. The aim is to develop grouts that should be compatible with the preexisting materials and allow the restoring of the adherence of coatings to the background. Special attention was paid to the rheological behaviour of the grouts and to other features of the fresh state. The role of components such as binders, aggregates and admixtures used in the formulations was tested through an outlined series of mechanical and physical tests. In terms of hardened state properties, a set of basic requirements (related to strength and capillarity) were evaluated and a test for adhesion loss measurement was developed in order to test the ability of developed grouts to restore adhesion of coating layers.

The rheological behaviour was evaluated through a distinct procedure, which involved the test with a specific speed profile (dwell profile). The dwell profile allows studying the rheological behaviour along measuring time, making possible to observe changes in rheological parameters in mortar suspensions, through the measurement of flow curves along the time test. The dwell profile allowed obtaining the rheological parameters (viscosity and yield stress) according to the Bingham model.

Grouts based on lime, fine sand and metakaolin together with the right amount of water and admixtures were developed and adjusted in order to be used in consolidation works of old renders.

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1. Introduction

In Europe the importance of old buildings restoration is growing in the construction sector. The use of cement mortar for the replacement of old renders does not in many cases respect the features of the original applied materials, as well as their traditional process of application. Therefore, problems such as detachments, cracking and crystallization of soluble salts, among others, appear or are accentuated. It is very important to achieve a composition that is deemed consistent and compatible with the original render

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at both chemical and physical levels. The aim of any action of restoration and maintenance of old renders is to solve the problems accumulated over the years. Equally important, is the safeguard for future generations of a valuable urban and architectural heritage. Indeed, a rehabilitation intervention begins as an operation that aims to conserve the largest part of various elements and materials of the property over which rehabilitation is focused on [1].

A grout is a mortar used to fill or to ensure homogenization, consolidation and the improvement of mechanical properties of systems that present cavities, cracks or loss of adhesion [2].

The loss of adhesion in old renders has a destructive effect, requiring the application of specific techniques such as the use of





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^{0950-0618/\$ -} see front matter © 2013 Published by Elsevier Ltd. http://dx.doi.org/10.1016/j.conbuildmat.2013.09.006

grouts. Grouts based in lime mortars have a good compatibility potential with old supports, but may need several additions with the aim to grant some hydraulic character and to model performance in the fresh state. The development of grouts should involve the study of their rheological behaviour, because this is essential for their penetration inside thin cavities between detached layers and fulfillment of existent voids. The mixing water and admixtures content are factors under consideration [3].

Grout must be compatible with the original coating and one must realize that repair or restoration with grouts is practically an irreversible technique, since it involves works that are difficult to be removed. The injection of the grout in existing holes in the wall aims to fill cavities, whether internal cracks or detachments. This technique becomes more effective if inside the intervening elements there is a communicating network of open porosity. In such delicate cases, the injection should be done by gravity, vertically or with a minimum inclination [3,4]. This technique consists in the introduction of a fluid lime paste into the void area in the detachment occurring between the substrate and the render layers [5,6].

Table 1 summarizes the requirements for the development and performance of grouts.

Rheology can be defined as the science that studies the flow of materials, evaluating the relationship between the applied shear stress and deformation in a given period of time [7,8]. Banfill [8] demonstrated that mortars have a rheological behaviour that generally follows the Bingham model, expressed as:

$$\tau = \tau_0 + \eta \gamma \tag{1}$$

In this model, there is a linear relationship between stress (τ) and the deformation (γ) from a certain value of shear stress (Bingham stress or yield stress, τ_0). In other words, these materials resist without flowing to shear stresses below the yield stress, but above that, they have a linear relationship between the shear stress and deformation, and this relation is characterized by the plastic viscosity (η) [9,10]. It is very important to evaluate the rheological behaviour of the grouts due to its relevance to their injection and dispersion capacity through the voids.

Slump in a flow table is a technique commonly used for the assessment of mortars workability, and it is a valuable help for fresh state basic characterization.

However, the use of rheometers allows determining, separately, two important rheological parameters, yield stress and plastic viscosity (τ_0 , η) and, by doing so, to evaluate the role of different components in a formulation separately on each parameter. The main objective of this work is assessing and optimising the viability of grouts for consolidation of old renders through rheometry and other relevant characteristics.

2. Experimental

2.1. Materials

All grouts formulations included air lime as binder and fine sand as aggregate in a binder/aggregate of 1:6.65(w/w). The binder is a CL90 air lime (Calcidrata SA, Portugal) the aggregate is a fine sand (SS-160 from Sifucel SA, Portugal). The aggregate used was a very fine silica sand (<200 μ m) and its particle size distribution is shown in Fig. 1.

The grouts development began with the study of the rheological behaviour of a base grout mortar, consisting of air lime, sand and water. Admixtures were then added to control the rheological behaviour and, in a second phase, a pozzo-lanic additive (metakaolin), Table 2, was also introduced to improve lime based grouts hardened state characteristics. The manufacturer of metakaolin (Argical 1200) is AGS Mineraux and its particle size distribution is in Fig. 2. This pozzo-lanic addition was made in amounts of 10%, 20% and 30% of the total mass of the binder.

The admixtures used were a water retaining agent (cellulose ether), a plasticizer (sodium gluconate) and an adhesive agent (Acril33), with contents varying between 0.5% and 1% of the total mass of solids. This adhesive agent is an acrylic resin in aqueous dispersion characterized by excellent resistance to atmospheric agents and chemical stability, good freeze-thaw stability and binding power and a high resistance to yellowing. The manufacturer is CTS Spain (Products and equipment for restoration, S.L.).

Developed grouts were also compared in terms of rheological behaviour with two commercial grouts (PMLA and PMLI). The PMLA grout is an injection grout, based on air lime, which is used to consolidate wall painting and old renders of historical interest. The PMLI grout is an injection grout based on hydraulic lime, which is used to consolidate old renders and plasters of historical interest. Tables 3 and 4 presents the characterization of these materials developed by Tavares and Monte [11,12].

2.2. Methods

2.2.1. Rheological characterization

Fresh mortar can be considered as a fluid material, where the yield stress represents the initial resistance to the flow, caused by contact between grains, while the plastic viscosity control the behaviour once the required torque was achieved to initiate the movement [13].

For the characterization of the rheological behaviour a specific rheometer for mortars (Viskomat NT – Schleibinger Gerate, Germany) was used. In this equipment, as the cylindrical sample container rotates (Fig. 3), the mortar flows through the blades of the impeller and exerts a torque measured by a transducer [9,10]. This rheometer measures the torque along the test duration time as a function of rotation speed for different speed-time profiles. The characteristic Bingham fluid relation of torque (*T*) with rotation speed (*N*) is T = g + h N, where *g* (N mm) and *h* (N mm min) are coefficients proportional to yield stress and plastic viscosity, respectively [10,14].

The rheological behaviour was evaluated through a distinct speed-time profile at constant rotation speed. The dwell profile allows the study of the rheological behaviour over time, making it possible to measure flow curves (*T* vs. *N*) along the test time. The dwell profile allowed a precise evolution of the rheological parameters, determined according to the Bingham model, e.g., the yield stress and the plastic viscosity related coefficients, *g* and *h*, respectively [9,15,16].

In this study, the rotation speed profile was defined for 60 min at 0 rpm with speed rising to 160 rpm every 15 min followed by a drop back to 0 rpm again (Fig. 4). The change of speed from 0 to 160 rpm and from 160 to 0 rpm is 30 s. In the speed variation areas, flow curves of torque (T) vs. rotation speed (N) can be constructed [6].

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Summary of requirements for grouts [4].

Rheological requirements	Enough fluidity and uniform penetration capacity, to a better filling of voids – injectability Lack of segregation (avoid heterogeneity) Minimum exudation, reducing the presence of voids
Chemical requirements	Chemical characteristics stable over time Resistance to sulphate salts, preventing efflorescence and expansive products Minimum alkali content
Physical requirements	The setting time should be appropriate for injection Insolubility in water and volumetric stability in presence of humidity Low shrinkage
Mechanical requirements	Similar characteristics to the original material Good adherence to the substrate, for good efficacy in consolidation
Thermal requirements	Low heat of hydration to prevent the development of thermal gradients that can affect the adhesion to the substrate



Fig. 1. Particle size distribution of silica powder.

Table 2Composition of metakaolin.

SiO ₂	Al_2O_3	$K_2O + Na_2O$	Fe ₂ O ₃	TiO ₂	CaO + MgO
55%	39%	1.0%	1.8%	1.5%	0.6%



Fig. 2. Particle size distribution of metakaolin (Argical 1200).

Table 3

Characterization of the grout PMLA.



Fig. 3. The Viskomat NT rheometer measuring system.

2.2.2. Hardened state characterization

The mechanical compatibility of the mortar means that the new material must not transmit tensions to the old ones (old renders), at a level that can contribute seriously to their cracking, delamination or rupture in any way. But, the transmission of tensions occur along a period of time and depends on the dynamic development of several processes, such as the hardening of the mortar, by hydration, carbonation or other reactions [17].

For determination of their characteristics in the hardened state, grout samples of each formulation were produced, with prismatic shape and standard dimensions of $40 \times 40 \times 160$ mm, according to NP EN 1015-11 [18], and curred. The curing process followed standard rules for lime based mortars and involved placing the samples in a chamber at 20 °C and 65% RH for 28 and 60 days. These samples were used for the determination of mechanical strength (EN 1015-11: 1999) [18] and of capillarity (EN 1015-18: 2002) [20].

Another important test in the hardened state involved the simulation of consolidation of detachments. This test starts with the preparation of the supports (ceramic bricks), where two layers of lime render mortar were placed [18]. In the intermediate region, a void is simulated and then is filled with the grout mortar

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Composition		Binder (50%): air lime Load (46%): micro fine clays, powders selected and quartzite's Additives: super-plasticizer, agents retainers water, as cellulose ether
Mineral composition by X-ray diffraction (DRX)	Powder mix Specimens after 360 days	Presence of calcite (C), portlandite (P), calcium silicate [(larnite (C)] and quartz (Q) Presence of calcite (C) and quartz (Q)
Bulk density (kg/dm ³) Differential thermal analysis (ATD) Setting time (hardness) Compression resistance (kg/m ²)		1.1 In powder mixture, one organic content (0.6%) 24–48 h 12.7

Table 4

Characterization of the grout PMLI.

0		
Composition		Binder (50%): hydraulic lime, with low salt content Load (46%): microfine clays, powders selected and quartzite's Additives: super-plasticizer, agents retainers water, as cellulose ether
Mineral composition by X-ray diffraction (DRX)	Powder mix Specimens after 360 days	Presence of calcite (C), portlandite (P) and quartz (Q) Presence of calcite (C) and quartz (Q)
Bulk density (kg/dm ³)		1.2
Differential thermal analysis (ATD)		In powder mixture, one organic content (0.6%)
Setting time (hardness)		24–48 h
Compression resistance (kg/m ²)		25.9



Fig. 4. Speed variation for a base grout base (GB-grout base, lime:aggregate: 1:6.6, 65 (w;w)).



Fig. 7. Flow curves of base *grout* mortar (GB). The arrows indicate the up and down speed variation (GB-grout base, lime:aggregate: 1:6.6, 65 (w:w)).



Fig. 5. Specimens simulating the detachment between layers, where the grout will be introduced.



Fig. 6. Torque variation for a base grout (GB-grout base, lime:aggregate: 1:6.6, 65 (w:w)).



Fig. 8. Variation of h parameter as a function of testing time for the base and commercial grouts (h GB – viscosity of grout base; h PMLA – viscosity of grout commercial PMLA and h PMLI – viscosity of grout commercial PMLI).



Fig. 9. Variation of g parameter as a function of testing time, for the base and commercial grouts (h GB – viscosity of grout base; h PMLA – viscosity of grout commercial PMLA and h PMLI – viscosity of grout commercial PMLI).

to be tested (Fig. 5). In these specimens adhesion test of the grouts (EN 1015-12: 2000) [19] are made to evaluate the consolidation of the detachment between layers.

The production of brick samples for adhesion test involved: (i) application of a first mortar layer on the brick; (ii) place acrylic plates on the first layer and apply a second layer of mortar; (iii) remove the acrylic plates and this way a detachment on rendering mortar (interlayer, crushed mortar) is produced. The mortar, identified as base mortar, used for the production of brick samples was prepared with binder: aggregate volume ratio 1:3, using air lime as binder and a coarser aggregate [17].

3. Results and discussion

3.1. Rheological characterization

The torque variation with time (Fig. 6) shows that by increasing the rotation speed to 160 rpm, the torque values also increase. Fig. 4 shows the correspondent speed variation with time. On the other hand, when speed is at 0 rpm (at rest), torque tends to increase due to a thixotropic effect leading to the building up of a structure of particles in suspension. As can be noticed, these particle structures are broken down by rotation and agitation because when the vessel speed returns to 0 rpm the torque is also zero again. This happens during the 60 min of the test duration. When the cup is stationary there is no resistance to rotation but the flow curves are taken when the cup is moving every 15 min.

During speed variation flow curves can be built, like the one shown in Fig. 7. The relationship between torque and rotation speed (T = g + hN) confirms this is a Bingham fluid, where g and h are related to yield stress and plastic viscosity, respectively [10,16]. Their values can be extracted from these curves relationship.

Figs. 8 and 9 shows the variation of h and g parameters as functions of testing time for a base grout, prepared with 60% of kneading water. A comparison is also made between this base grout and to two commercial grouts measured in the same



Fig. 10. Shows the effect of increasing of plasticizer agent on h and g respectively (GB_X EC, where X = 0.1%; 0.15%; 0.5%; 0.6% and 0.75% cellulose ether (EC)).

conditions. The base grout (GB) has only lime, silica powder (1:6,65 w/w) and water without any additions. In order to achieve an interesting grout one needs to raise the viscosity value since its original value is quite lower by comparison with the commercial grouts. The next step is then the use of different admixtures that can improve the grout behaviour in the fresh and hardened state.

As an admixture, a water retaining agent allows retaining the water in the mixture but also has a thickening effect that can help to improve viscosity and cohesion of fluid systems. It should improve the workability and mortar adhesion.

In this work a cellulose based admixture (EC) was used and Fig. 10 shows the effect of thickening on h and g, respectively. It

is clear that both *h* and *g* increase with increasing thickening agent content and the values of most formulations are well inside the range of the tested commercial grouts (PMLA and PMLI) values.

Another type of admixture was also studied in order to reduce the amount of water in the grout needed to guarantee the necessary fluidity. A plasticizer or water reducing agent usually has this effect in mortars of decreasing the amount of water used in mixing. A plasticizer can keep the workability when the water content is decreased. In this study, the effect of the plasticizer (GS) in the rheological behaviour of the base grout (GB) is reflected on the increase of viscosity and yield stress. However, looking for its isolated effect the changes in viscosity are lower but the yield



Fig. 11. Shows the effect of increasing of plasticizer agent on h and g respectively (GB_X EC_YGS, where X = 0.1%; 0.15%; 0.5%; 0.6% and 0.75% cellulose ether (EC) and Y = 0.5%; 0.6%; 1% and 1.5% sodium gluconate (GS)).



Fig. 12. Shows the effect of increasing of metakaolin (10%, 20%, 30%) on h and g, respectively (GB_YMK where Y is 10%, 20% and 30% de Metakaolin).



Fig. 13. Effect on h and g of increasing metakaolin content (10, 20, 30%), in grouts with admixtures (GB_Y MK_admixture – where Y is 10%, 20%, 30% Metakaolin, and admixture = 0.5% cellulose ether, 1% sodium gluconate and 0.5% Acril 33).



Fig. 14. Shows the effect of increasing content of metakaolin in flexural strength.



Fig. 15. Shows the effect of increasing content of metakaolin in compressive strength.



Fig. 16. Shows the effect of increasing content of metakaolin in capillarity coefficient.

stress decreases with the increase in plasticizer (Fig. 11). The overall effect is a more fluid system.

In order to improve the ability of a lime based mortar to develop necessary strength faster, pozzolanic additions can be used.

Originally the term pozzolan was associated with natural volcanic ashes and calcined clays, which react with lime in the presence of water at ambient temperature. Now the term has been extended to cover all siliceous/aluminous materials which, in finely divided form and in the presence of water, will react chemically with calcium hydroxide to form compounds with cementitious properties [13].

Metakaolin was introduced in these grouts formulations with this purpose and so its effect. Therefore its effect in both the hardened state but also in the fresh state had to be assessed. Metakaolin was used as an additive to improve the adhesion of grout to the substrate and consequently, to improve the lime mortar's mechanical resistance. Metakaolin is obtained through clay calcination, resulting in a material of high pozzolanicity that is due to its ability to react with calcium hydroxide, providing products such as the ones developed in cement hydration [11,21,22].

Fig. 12 shows the effect of addition of metakaolin to the base grout in the fresh state behaviour. The metakaolin does not present a significant effect in viscosity but helped to increase the yield stress of the grout.

Grouts for consolidation of older renders also need some gluing effect that can be ensured by polymeric resins. In this work such addition (Acryl33) was also tested in order to verify if the hardened state characteristics improved. This admixture was introduced in



Fig. 17. Adhesion simulation sample test scheme.



Fig. 18. Adhesion on grout and render in the samples of simulation of detachment.

the grouts formulations containing metakaolin as a pozzolanic additive (10%, 20% and 30% of total mass of binder), and the other admixtures tested earlier.

Fig. 13 shows the effect of these additions on the grouts used in Fig. 12. An increase in h and g was observed in comparison to the grouts without admixtures. The behaviour of these grouts in terms of injection improved, due to the good workability that was achieved.

3.2. Hardened state characterization

Figs. 14–16 show the effect of the metakaolin introduction on the developed grouts, namely, on the increase of mechanical strength and average capillary of the grout. The results are within the range of acceptable values in terms of compatibility with the strength values of old renders. The values of flexural strength are between 0.2 and 0.7 MPa and compressive strength is between 0.4 and 2.5 MPa. According to Veiga [11,23], the developed compositions have values compatibles with old renders. The capillary coefficient is better to the grout with 30% metakaolin, and this value is approximately 14 kg m² h^{0.5}.

Figs. 14 and 15 also show the increase of flexural and compressive strength on curing from 28 to 60 days. The grout composition with 10% of metakaolin was the one with largest increase.

Fig. 17 show the adhesion simulation sample test scheme. Brick samples were also prepared to simulate the situation *in loco*. These samples were then injected with the grouts.

Fig. 19 show the effect of increasing content of metakaolin in the adhesion values. The values of adhesion are low, but it is normal in grouts, because the quantity of water in the grout is high (60% water).

Fig. 19 show that after 28 days of curing, there is a significant evolution of pull-out strength in the consolidation zone when it proceeds to increasing the percentage of metakaolin from 10% to 30%. The maximum adherence stress at 28 days, occurs for the grout with 30% metakaolin in the binder, and is about 0.015 MPa.

At 60 days of curing, there is an inversion of pull-out stress, quite visible in grouts containing 10-20% of metakaolin. The maximum adhesion stress for the 60 days of curing specimen is in the



Fig. 19. Show the effect of increasing content of metakaolin in adhesion values.

grout with 10% metakaolin content. Increasing from 10% to 30% metakaolin, a gradual decrease of the respective yield strength occurs.

This phenomenon was predictable, since metakaolin presents a late effect in terms of its pozzolanic activity, the higher its content within the formulation. Lime/MK grouts compositions displayed different reaction kinetics during curing time, being pozzolanic products content directly proportional to the substitution rate of lime by MK [24].

The evaluation of the hardened state properties revealed that the addition of metakaolin played its pozzolanic effect increasing mechanical strength even in a situation of diminishing the lime content by replacement. Grouts with a good mechanical strength and compatibility with lime based renders could be achieved, as shown from the consolidation simulation test performed with the grouts, see Fig. 18.

It was also noted that the increase of the metakaolin content improved the adhesion of the grout. This property was also favored if grouts used metakaolin together with the admixtures (0.5% cellulose ether, 1% of sodium gluconate and 0.55% of Acryl33).

It is important to point out, that although the recommended values for pull-out stress in substitution mortar are ≥ 0.1 MPa, [6,16]. But, the values obtained are not comparable due to the

difference of used supports. Tested consolidation grouts, with metakaolin in the binder, still present low stress values compared with the desired ones.

The fact that weak rupture forces (0.2 MPa) are enough to cause the pull-off of the grouts are due to the fact that they were not totally carbonated as confirmed afterwards. This is justified by restricted diffusion of CO_2 through the porosity of the render, needed for the hardening process of the lime based grout.

4. Conclusions

It was useful to use rheometers to evaluate the rheological parameters of grouts in order to better assess the requirements for their development. The introduction and role of certain components as admixtures or other additions (such as pozzolans) could be assessed through rheology. It is shown that the studied grouts present a behaviour related to the Bingham model, exhibiting plastic viscosity and yield stress coefficients that can be independently determined.

The developed grouts show rheological characteristics that can be appropriate for injection applications to consolidate old renders in conservation actions.

Water retaining agent presents a thickening effect, increasing the values of g and h, and has ensured a good workability. Looking at the isolated effect of the used plasticizer, viscosity (h) slightly decreases while the yield stress (g) decreases with increasing content. Its effect was not so predominant. The adhesive agent ensured a gluing effect to the grout and in the used amounts no significant impact was observed in the rheological parameters.

The metakaolin used as an additive in the base grout did not affect too much the viscosity (h) values, but it increases the yield stress as its content increases, allowing a good workability to be achieved.

The success of a good consolidation treatment depends not only on the chosen product, but also on its application and the intrinsic characteristics of the conservation state of the material to treat. In this context, the study of traditional air-lime mortars for rehabilitation of buildings resulted well and they constitute a suitable solution in interventions for rehabilitation of old building renders.

Acknowledgement

The authors wish to thank the Foundation for Science and Technology through the project Limecontech – PTDC/ECM/100234/ 2008.

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