DISEÑO Y PROPIEDADES DE MORTEROS DE CEMENTO-FILLITA

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DESIGN AND PROPERTIES OF PHYLLITE CLAY-CEMENT MORTARS

Abstract
In this paper, new mortars based on phyllite clay-cement were studied and tested to determine their properties in fresh and hardened state. In this paper, different mixtures were prepared by substituting cement with phyllite clay (0, 20, 50 and 80wt.%). Water demand was determined by fixing the water/conglomerate ratio at a good workability. A comparative study of the most significant mortar properties is conducted, such as workability, occluded air, water retention, water absorption and mechanical strength was carried out on mortars prepared with different contents of phyllite. In addition, a puzzolanic effect experiment in phyllite clay-cement mixes was carried out in order to ascertain the hydraulicity of the mortars. The results showed that increasing the phyllite quantity decreases the occluded air, bulk density and mechanical properties of the mortar while increasing water retention and water absorption. Pozzolanic effect was found in mortars with very high phyllite content. The results indicate that technically viable mortars may be obtained for use in construction and building in a sustainable way due to savings on cement as a raw material to contribute to the improvement of the environment.

Resumen
En este trabajo se presenta un estudio basado en el diseño y caracterización de nuevos morteros de cemento-filita, a través de la determinación de sus propiedades en estado fresco y endurecido. En este trabajo, han preparado diferentes mezclas con distintas sustituciones de cemento por filita (0,20,50 y 80%). La demanda de agua se determinó fijando la relación agua/conglomerante para obtener una buena trabajabilidad. Se han determinado las propiedades de trabajabilidad, aire ocluido, retención de agua, absorción de agua y resistencias mecánicas de los morteros con diferentes contenidos de filita. Además se determinó el efecto puzolánico en pastas de cemento con distinto contenido de filita con el objetivo de conocer su hidraulicidad. Los resultados mostraron que el incremento progresivo de filita reduce el aire ocluido, la densidad aparente y las propiedades mecánicas, a la par que se aumenta la absorción de agua y la retención de agua de los morteros. Se encontró efecto puzolánico en dosificaciones con alto contenido de filita. Los resultados indicaron que los morteros con filita son técnicamente viables para su empleo en construcción y contribuyen a la sostenibilidad ya que ahorran cemento en su fabricación, contribuyendo así a la mejora del medio ambiente.

Key words: Masonry Mortar; Phyllites; Pozzolanic effect; Air Void Content

Palabras clave: Mortero de Albañilería; Filitas, Puzonalicidad, Aire Ocluido
1. INTRODUCTION

Portland cement is widely used in mortar and concrete. It is expensive and has significant embodied energy and carbon emissions therefore a high environmental impact. Consequently, there is a need to find cheaper, more sustainable alternatives. In the last decades, many authors have studied alternative materials that can be used to reduce cement content in mortars and concretes. These have included crushed rock as well as pozzolanic and supplementary cementitious materials such as waste ashes and slags. In many instances, the partial replacement of Portland cement with pozzolanic or supplementary cementitious materials not only leads to an increase in sustainability but also to the improvement of the properties and durability of the resultant material.

In this study, cement is partially replaced with crushed phyllite. The rationale behind this research was that replacing cement with phyllite would increase the sustainability of the material and, furthermore, the (natural) thermal treatment of the clay in the phyllite could potentially trigger pozzolanic reaction with the possibility of improving the physical properties of the material.

Phyllite clays are rocks (metamorphosed to a slight degree) which contain clay minerals (chlorite, illite and mixed-layer illite/smectite), quartz and feldspars [1]. They have been previously studied as a filler in plastics [2] and concrete [3] products. Garzón et al. [4] investigated a new mix for construction of roofs and flexible pavements using phyllite clay and cement at 5, 7 and 9 wt.% which resulted in improved engineering properties over the raw phyllite. Due to their very low permeability, phyllites have been used as a covering, to waterproof roofs and also for urban waste landfill applications. The Spanish patent (PTT2016/005) [5] explains the experimental procedure to obtain a waterproof mortar made with phyllite, white cement and water for civil engineering works such as irrigation reservoirs and canals. However, no published research concerning the use of phyllites in masonry mortars has been found to date.

This paper studies phyllite-based masonry mortars. It outlines an original experimental laboratory study on phyllite clay–cement composites undertaken by the authors to examine the improvement in selected construction properties compared with traditional mortar such as M10 in EN-998 2 [6].

2. METHODOLOGY

2.1. Raw materials and mortar design

The Portland Cement used is CEM-I 42,5-R with a real density of 3150 Kg/m³ (as per manufacturer’s specifications). The chemical composition of the cement, obtained through X-ray fluorescence, confirms that it is basically composed of CaO and SiO₂. The aggregate is a fine, natural siliceous sand. The characterization of the sand was carried out in accordance with EN 13139 [7]. The maximum particle size is 2 mm, as can be seen in Figure 1.
The Phyllite was sourced from Berja, Almería, Spain and it had been characterized in a previous study [8]. The representative bulk phyllite-clay sample used in this investigation was oven dried at (105-110°C) to constant mass and left to cool at temperature of 20°C. It was then crushed and sieved to obtain the fraction passing the 0.08 mm sieve in accordance with EN-933-1 [9].

It was noted that in its natural state, the phyllite had a very low gravimetric water content ranging between 1 and 2% (mean 1.8%), a void ratio (volume of voids to volume of solids) of ~0.39, and a dry density of 2003 Kg/m³.

Garzón et al. [1, 4] reported the chemical composition of this phyllite as typically containing 45-50 wt.% silica and 22-24 wt.% alumina. Minor amounts of other oxides were also found such as CaO (1.7-4.4 wt.%), MgO (2.8-3.4 wt.%), Na₂O (1.8-2.4 wt.%), K₂O (3.3-3.9 wt.%) and iron oxide as Fe₂O₃ (8.3-9.4 wt.%). The phyllite together with the other raw materials appear in Figure 2.

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**Fig. 1: Granulometry of the sand**

**Fig. 2: Raw materials. Phyllite, Sand and CEM I**

Different masonry mortar mixes were prepared. The applications in mind included renders and masonry mortars complying with European standards EN 998-1 [9] and EN 998-2 [6]. Thereby, reference cement mortars made from cement, sand and water, with a cement/sand weight ratio of 1/3 were prepared for the experiments. Mortars were also prepared by substituting cement with phyllite clay (20, 50 and 80wt %) as a binder.
Table 1 shows the nomenclature and the phyllite-clay/cement ratio used for each mixture. The binder is the total amount of cement and phyllite contained. The consistency is defined as the water/binder relation (water/(cement + phyllite)), w/(c + phy), both for the reference specimens and those with different samples of phyllite. It was measured through the slump flow test, with reference to an average spreading diameter of 175 ± 10 mm, which sets the amount of water needed to obtain plastic consistency according to standard EN 1015-3 [10].

The mortars were prepared in a mechanical mixer in accordance with EN196-1 [11]. A total of 3 prismatic specimens were prepared for each mix, with dimensions 160 x 40 x 40 mm to perform the flexural and compression tests and water absorption, in accordance with EN 1015-11 and EN1015-18 respectively [12, 13]. They were then demolded and cured in a moist chamber at 95% relative humidity for 7 and 28 days.

2.2. Experimental procedure

All mortars were characterised by their air void content, bulk density, water demand, water absorption and mechanical strength after 7 and 28 days.

Flexural strength tests were determined with prismatic specimens (160 x 40 x 40) mm³ supported on rollers positioned at 100 mm. The test was performed on a minimum of three specimens. Compressive strength tests were performed on the broken sections of the specimens previously tested to flexural failure (at least six samples). The load was applied to a (40 x 40) mm² contact section.

The operational procedure to determine water absorption by capillarity is specified in standard EN 1015-18 [13] (Figure 3). A specimen of each mortar type was placed in a moist chamber for curing over a 28-day period, at a constant temperature of 20°C and a relative humidity of 95%. After 28 days, they were dried at a temperature of 60°C to achieve a constant mass. Subsequently, the four longitudinal faces were sealed with paraffin and broken under flexion. Each piece was then submerged (with the broken side in contact with the bottom of the tray) in water to a depth of between c.5-10 mm for 90 minutes in order to obtain the absorption coefficient (c) in accordance with equation (1):

\[ c = (M_1 - M_2) \times 0.20 \]  

Where:

- \( M_1 \) is the mass in grams after 10 minutes submerged in water
- \( M_2 \) is the mass in grams after 90 minutes submerged in water

Fig. 3: Water Absorption test
Immediately afterwards, each specimen was divided along its length and the height of water penetration measured in relation to the centre of the specimen. Water retention was measured according to standard EN 83816:1993EX “Test methods. Mortars. Fresh mortars. Determination of water wettiness” [14].

The pozzolanicity test was carried out in accordance with EN-196-5 [15] to determine the concentration of calcium ions (expressed as calcium oxide) in an aqueous solution, when in contact with hydrated cement after a fixed period of time. This was compared with the amount of calcium ions that saturated a solution of the same alkalinity.

3. RESULTS

3.1. Consistency and water demand

The water demand of the mortars is shown in Table 1. The results show that increasing the amount of phyllite raises the mortar's water demand. Thus, mixtures (MPhy20) showed an increase of 0.5 points as regards the reference mix (without phyllite), mortar MPhy50 the increases by 2.5 and there is a 3-point increase in samples with high phyllite content (MPhy80). This effect may relate to the specific surface area of the binder's particles. Phyllites have a larger specific surface area than cement therefore they have the ability to fix more water. As a result, we recorded a greater need for water to achieve a specific plastic consistency in the mortars including phyllite. This implies an increase in the water / binder ratio of 12% in binders with 20% of phyllite (MPhy20), and a 24% increase in mortar (MPhy80).

3.2 Air content and density

Table 1 shows the entrained air in the different mortar mixes. A higher phyllite content reduces the entrained air in the fresh state. This reduction indicates a potential improvement of the material's porosity leading to a lower permeability coefficient. Mortars mixed with phyllite, (MPhy20), (MPhy50) and (MPhy80), contained less occluded air, at 5.2% and 4.4% and 3.9%, respectively.

Contrary to the water demand which rises with increasing phyllite, the occluded air and the bulk density of the phyllite mortars lowers as the phyllite content increases.

The phyllite reduces mortars' bulk density with a 2% reduction in mortars (MPhy20) and 7% in mortars with a high content of phyllite (MPhy80). One possible explanation is the lower density of the phyllite (2003 kg/m³) in substitution of cement (with a density of 3150 kg/m³). Another possible explanation for this reduction in density is the increase in water content in the phyllite mortars leading to a more open pore structure. This has been the case in other materials [16].

<table>
<thead>
<tr>
<th>Table 1: Mortar composition (reference and phyllite series) and properties including weight ratio, water/binder ratio, bulk density, air content and water retention.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Series no.</td>
</tr>
<tr>
<td>Reference</td>
</tr>
<tr>
<td>Phyllite Series</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>
3.3. Water absorption

Table 2 shows the water absorption by capillarity of the mortars after 90 minutes. Standard EN-998-1 [9] classifies the mortars in three categories in accordance with the value of the absorption coefficient by capillarity (c):

- W1 when c≤0.4 Kg/(m²·min0.5)
- W2 when c≤0.2 Kg/(m²·min0.5)
- W0 when the value 3 is not declared

According to these, the mortars mixed with phyllite, up to 50/50 (cem/phyllite), are classified as W2, therefore, they are expected to show good resistance to filtration against water penetration. An exception is MPhy80, with a coefficient by capillarity of 2.83 Kg/(m²·min0.5) which didn't show good waterproof behaviour and it shouldn't be used in exterior rendering or walls.

<table>
<thead>
<tr>
<th>Series no.</th>
<th>Sample</th>
<th>Absorption coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference</td>
<td>MR</td>
<td>0.19</td>
</tr>
<tr>
<td>Phyllite Series</td>
<td>MPhy20</td>
<td>0.15</td>
</tr>
<tr>
<td></td>
<td>MPhy50</td>
<td>0.18</td>
</tr>
<tr>
<td></td>
<td>MPhy80</td>
<td>2.53</td>
</tr>
</tbody>
</table>

3.4. Mechanical strength

Figure 4 shows the results obtained for flexural and compression strength at 7 and 28 days for the reference and phyllite mortars. Here, it can be seen that both strengths decrease proportionally to the quantity of phyllite present in the mortar. The lower strength is due to the reduced amount of cement in the specimens with increasing phyllite. It can also be partly attributed to the high water demand of the phyllite reducing the amount of water available for the hydration of the cement binder, leading to a reduction in mechanical strength.

As aforementioned, there is a decrease in strength as larger percentages of phyllite are used. Nevertheless, these results evidence that mortars MPhy20 and MPhy50 compare well with traditional mortars such as standard mortar M10 in EN 998-2.

There is a good correlation between flexural strength and compressive strength (R:0.999) therefore, based on this relationship, phyllite mortars can be designed which comply with the EN-898 2 standard strength requirements such as M2.5, M7.5, M10 by fixing the strength parameter as desired. For example, by extrapolation, to obtain a standard M10 we need to design mixes with a 60% phyllite (MPhy60) these mortars would have a flexural strength of 2.48 MPa.
### Areas of Interest:

<table>
<thead>
<tr>
<th>Series no.</th>
<th>Sample</th>
<th>Compressive strength (MPa)</th>
<th>Flexural strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>7 days</td>
<td>28 days</td>
</tr>
<tr>
<td>Reference</td>
<td>MR</td>
<td>30.0</td>
<td>42.9</td>
</tr>
<tr>
<td>Series Phyllite</td>
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<td>19.0</td>
<td>30.7</td>
</tr>
<tr>
<td></td>
<td>MPhy50</td>
<td>7.42</td>
<td>13.4</td>
</tr>
<tr>
<td></td>
<td>MPhy80</td>
<td>1.15</td>
<td>1.9</td>
</tr>
</tbody>
</table>

3.5. Pozzolanic effect

The [OH]⁻ and [CaO] concentrations obtained for each sample are shown as mmol/l, in the diagram in Figure 5. These represent the saturation concentration of calcium ions in the solution (shown as CaO) based on the concentration of hydroxyl ions at 40 °C. In view of the data in Table 3, it can be noted that mortar (MPhy80) is below the curve in the graph, thus indicating a pozzolanic effect. Contrary to what might be expected, the pozzolanic effect does not contribute to the mechanical resistance of the material.
Fig. 5: Puzzonalic effect of different mixtures.

Table 3: Puzzolanic effect in reference and phyllite mortars

<table>
<thead>
<tr>
<th>Sample</th>
<th>ml HCl</th>
<th>mmol/l [OH]</th>
<th>[OH]</th>
<th>ml EDTA</th>
<th>mmol/l [CaO]</th>
<th>[CaO]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ref.</td>
<td>34.8</td>
<td>69.6</td>
<td>70.5</td>
<td>10.6</td>
<td>6.4</td>
<td>6.5</td>
</tr>
<tr>
<td>MPhy20</td>
<td>35.7</td>
<td>71.4</td>
<td></td>
<td>11.0</td>
<td>6.6</td>
<td></td>
</tr>
<tr>
<td>MPhy50</td>
<td>30.1</td>
<td>60.2</td>
<td>61.2</td>
<td>12.4</td>
<td>7.4</td>
<td>7.6</td>
</tr>
<tr>
<td></td>
<td>31.1</td>
<td>62.2</td>
<td></td>
<td>12.9</td>
<td>7.7</td>
<td></td>
</tr>
<tr>
<td>MPhy80</td>
<td>25.9</td>
<td>51.8</td>
<td>51.5</td>
<td>18.1</td>
<td>10.9</td>
<td>10.8</td>
</tr>
<tr>
<td></td>
<td>25.6</td>
<td>51.2</td>
<td></td>
<td>17.8</td>
<td>10.7</td>
<td></td>
</tr>
<tr>
<td></td>
<td>19.9</td>
<td>39.8</td>
<td>39.2</td>
<td>22.2</td>
<td>13.3</td>
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<tr>
<td></td>
<td>19.3</td>
<td>38.6</td>
<td></td>
<td>21.3</td>
<td>12.8</td>
<td></td>
</tr>
</tbody>
</table>

4. CONCLUSIONS

- It has been confirmed that large phyllite substitutions of cement increase the \( \text{w/cem} + \text{Phy} \) ratio by weight to ensure good workability. In turn, an increasingly lower amount of water is placed in each specimen, which means longer pores, and in consequence, lesser densities.
- The inclusion of larger amounts of phyllite increases the water demand of the material, which isn’t related with the percentage of the mixture’s entrained air. This implies that the increase in water demand is due to the specific surface of phyllite itself.
- The mechanical strength greatly depends on the density of the material and the binder of the mortars. There is a good correlation between compressive and flexural strength, which gives an idea of the optimal proportions in which phyllite may be added to the binder to achieve the minimum strength required by standards.
Areas of Interest:

- Puzzollanic effect exist only at cement replacements of 50% and higher. Large phyllite contents show a slight puzzollanic behaviour which did not improve the mortars' properties.
- The mortar with 50 wt% of phyllite was deemed the most suitable masonry mortar in accordance with the standards, showing a compressive strength of 13.4 MPa, flexural strength of 3.3 MPa, 3.9% of occluded air, 89.1% of water retention and a low water absorption value of 0.18. It can be appropriate for exteriors, both as a pointing and a rendering mortar.

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