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Portland cement, gypsum and fly ash binder systems characterization for lignocellulosic fiber-cement

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HIGHLIGHTS

- pH and electrical conductivity assessment of Portland cement, gypsum and fly ash binders.
- TG and DTA analysis of ternary blended systems and their components.
- SEM for lignocellulosic fiber durability evaluation under low-alkaline environments.
- Fibers preservation within low-alkaline matrices by specific energy conservation in flexural tests.

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ABSTRACT

The present work aims to obtain an optimal Portland cement, gypsum and fly ash (OPC-G-FA) ternary binder matrix and assess both the addition of paper pulp-by means of mechanical dispersion in aqueous suspension-for cementitious composites reinforcement and the fiber properties over time. To evaluate microfibers preservation from pulp in low-alkaline environments, ternary binder matrices OPC-G-FA are optimized to achieve lower pH values. For that purpose, pH and electrical conductivity over time were analyzed. Only samples with the lowest content in Portland cement (15–20%) offered low alkalinity for short-term. The use of ternary binder systems enhances microfibers conservation compared with control samples (matrices 100% Ordinary Portland Cement) by using FA that, as expected, reduces the presence of $Ca(OH)_2$ in the matrix. Mechanical results prove that obtained matrices yield to a mechanical properties maintenance unlike samples with OPC matrices where toughness is reduced by 95%.

1. Introduction

Recent efforts to produce vegetable fiber reinforced composites for construction purposes have been intensified in order to enable these products from a technical and economical point of view. Lignocellulosic fibers present a potential use as reinforcement due to their optimal mechanical performance, availability and reduced cost if compared with synthetic fibers usually applied in air-cured fiber-cement. However, vegetable fibers durability as reinforcement for fiber-cement is one of the most important shortcomings [1–6]. Although this problem has been widely addressed, up to now this degradation mechanism is still one of the biggest concerns related to a loss of mechanical properties within the cementitious matrices. Between the most recent studies, different hypotheses are exposed to explain this phenomenon:

(a) an ageing process due to fiber mineralization, resulting in a reduction of the tensile strength of the fibers and a decrease of the fiber pull-out ligament after fracture. This mineralization process is a result of migration of hydration products (mainly calcium hydroxide [$Ca(OH)_2$]) to the fiber structure [7];
(b) deterioration due to the alkaline hydrolysis because the low corrosion resistance of lignin and hemicelluloses that exist in the middle lamellae of the fibers and cellulose molecules in high alkalinity environment [8];
(c) secondary ettringite precipitation into pulp fiber within cement matrix [4,9].

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A definitive solution to permit vegetable fibers to be used as reinforcement in the conventional fiber-cement is far from being offered. Also, many publications have broached natural fibers durability in cementitious environments with alternative approaches. A possible solution is fiber treatment to preserve thereof from a chemically aggressive environment [10–12]. Another option to avoid fibers degradation is to limit matrix alkalinity, by reducing pH of the pore solution in cementitious matrices. This alternative has also been tested by different studies achieving remarkable results by means of diverse techniques: cement matrix carbonation [13–15] and using pozzolanic materials [16].

Nevertheless, in all these cases alkalinity reduction is based on calcium hydroxide [$Ca(OH)_2$] reduction which is generated during cement hydration. These techniques require a certain reactivity time [17–19], before decreasing alkaline environment in early stages [14,15]. Therefore, calcium hydroxide reduction from the matrix by using pozzolanic materials may not be considered a fully effective method.

There are binder matrices with lower cement content with possible application in civil construction and acceptable mechanical performance, like cement, gypsum, pozzolan blends. According to Roldán [20], it is possible to use Fly Ash to enhance the compatibility between cement and gypsum and at the same time achieve a satisfactory mechanical performance. Thus it is possible to obtain new economic and environmental low cost materials with a considerable reduction of cement content. These binder materials, besides reducing calcium hydroxide content, might also help to preserve fibers, surrounding them with gypsum particles. In this way, more chemically compatible matrices can be obtained without the prejudice of mechanical performance degradation.

In order to get lower alkalinity matrices this ternary system was assessed in different stages;

- Firstly pH and electrical conductivity of different paste mixes are studied over time;
- Once the mix with a lower pH at early ages is obtained, it is employed in the production of fiber-cement elements to evaluate their flexural performance and their ability to preserve the fibers after being aged.

2. Materials and methods

2.1. Materials and preparations

The cement used for this research is Portland CEM I-52.5R according to BS EN 197-4:2004 [21], with a mean particle size value of 14 μm. The gypsum used, with no hardening regulator additives, meets the BS EN 13279-1 [22] standards. This gypsum has a purity index over 75%, retained fraction on the 200 mesh below 50%, maximum combined water value of 6%, minimum pH value of 6 and flexural strength higher than 2.0 MPa at 28 days.

Fly Ash used is F class, from silica or silicoaluminte rich fly ashes, with a specific gravity of 2.52 g/cm^3. Chemical composition by means of X-rays fluorescence of the Fly Ash is shown in Table 1. The original Fly Ash (FA) has a mean particle size of 29.9 μm and 10.4 μm for milled Fly Ash (FAm). Particle size distribution was determined by laser diffraction spectroscopy.

Table 2
Water characterization.

Chloride	Ca^{2+} and Mg^{2+}	Sulfates	pH	Conductivity
93 mg/l	480 g/l	298 mg/l	7.88	939 μS/cm

Table 3
Fibers characterization.

Characteristics of the unbleached and unrefined softwood pulp (Pinus)	
Ashes (%)	3.36 ± 0.62
Fibers (million/g)	1.32 ± 0.07
Length arithm. (mm)	2.19 ± 0.16
Length weighted in length (mm)	3.325 ± 0.12
Width (μm)	24.17 ± 0.91
Coarseness (mg/m)	134.25 ± 20.76
Kink angle (°)	38.05 ± 0.02
Kinked fibers (%)	11.08 ± 0.21
Curl. (%)	0.82 ± 0.06
Rate in length of macrofibrills (%)	47.23 ± 1.76
Broken ends (%)	36.17 ± 3.53
Fine elements (% by length)	3.51 ± 0.34
Fine elements (% by area)	4.55 ± 0.64

The water used for specimen's preparation and pulp extraction is characterized in Table 2. For paste molding and fiber observation at optical microscopy deionized water is used.

Cellulosic pulp fibers used in this work (pine fibers) are obtained from cement packaging kraft pulp. The process to get this cellulosic pulp is mechanical dispersion in water solution. For this purpose, cement packaging kraft is previously torn in smaller pieces and immersed in water for 24 h. After water immersion the kraft pieces are mechanically dispersed in water suspension for 20 min at 2000 rpm. The excess of water from the suspension is removed by filtration and humid pulp is kept refrigerated at 5 °C until its use. Table 3 presents the main physical properties for this recycled pulp.

By using the pulp from the kraft paper the ratio of fine elements is reduced when compared to other pulp sources as hemp or eucalyptus. Also pine kraft pulps present longer fibers which could difficult their dispersion in the matrix. However, the high arithmetic length, length weighted in length, average width and coarseness values of the pine pulp used in this work offers the possibility of promoting high pull-out bonding with lower porosity. This factor may help to see the efficiency of lower alkalinity matrices to keep high adherence between long pine fibers and matrix after ageing.

2.2. Test methods

The different tasks carried out during this study have been structured according to the next diagram (Fig. 1):

2.2.1. Blended paste system analysis

2.2.1.1. pH and electrical conductivity analysis of the blended paste systems. According to the ternary systems to study, three groups of blends may be observed, as shown in Fig. 2. This pH and electrical conductivity analysis is conducted to observe the alkalinity and

Table 1
Fly Ash composition.

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	SrO	TiO_2	P_2O_5	LOI
% by mass											
38.34	22.98	20.14	13.25	0.95	0.35	1.06	0.25	0.07	0.94	0.35	1.32