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MECHANICAL AND PHYSICAL PERFORMANCE OF LOW ALKALINITY CEMENTITIOUS COMPOSITES REINFORCED WITH RECYCLED CELLULOSIC FIBRES PULP FROM CEMENT KRAFT BAGS

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ABSTRACT

The objective of this work was to study the addition of cellulosic pulp in low alkalinity cement composites as well as its mechanical behavior under bending stresses before and after accelerated ageing cycles. The cellulosic pulp was obtained from recycled Portland cement kraft bags used for packaging. Low alkaline cementitious matrices were tested, reducing from 80 to 85% the content of Portland cement, in order to reduce the use of the conventional raw materials, energy cost and mainly to avoid a possible alkaline degradation of the cellulosic pulps. The cement matrix resulted from the ternary blend Portland cement-gypsum-pozzolan (fly ash or catalytic cracking catalyst residue), with 50% by weight of gypsum and different percentages by weight of pozzolans. These composites were prepared in the laboratory using a slurry vacuum dewatering followed by pressing technique. The four point-bending tests were carried out to evaluate the mechanical behavior of the low alkalinity cementitious composites and composite without pozzolans at 28 days and after soak & dry accelerated ageing tests. The low alkaline cement composites presented average values of modulus of rupture about 10 MPa after the ageing cycles, with the indication that its flexural strength was not significantly affected by the degradation tests. In addition, the average values of specific energy of these composites were also acceptable after 100 soak & dry cycles as compared to the composites with the Portland cement plain matrix. These results suggest that the use of low alkalinity ternary binder system can be an effective contribution in order to avoid the severe damage on cellulosic fibers (which occurred when traditional pure Portland cement matrix is applied).

Keywords: Cement composites; reinforced recycled cellulosic fibres pulp; low alkalinity binder; durability; Portland cement replacement

1. INTRODUCTION

The addition of Kraft cellulosic pulp, as reinforcement in cement based composites is being developed by several research groups. The idea of using discrete, ductile, vegetable fibers to reinforce brittle materials, taking advantage their high tensile strength (between 150-750 MPa), has been explored since a long time despite the degradation effect due to the alkaline effect (Bentur and Mindess, 2007; Coutts, 2005; Akers et al., 2010; Bentur and Akers, 1989). The cellulosic fibers are produced in large quantities, different morphologies and sizes, derived from renewable resources, such as agro-industrial and waste material, and available at reduced cost as compared to synthetic fibers (i.e. polypropylene and polyvinyl alcohol fibers).

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The increase of the agro-industrial activities generates a large quantity of waste in this sector (Savastano et al., 2000). The use of lightweight cellulosic pulp from agro-industrial by-products as reinforcement in low cost building materials is an interesting strategy for managing these by-products, from the point of view of their mechanical properties, environmental and social-economic impacts (Tonoli et al., 2010; Jarabo et al., 2012; Tonoli et al., 2009; Savastano et al.; 2003, Ardanuy et al.; 2012). On the other hand, the generation of waste is one of the most important problems highlighted the construction sector. It has been estimated that the construction sector is responsible for up to half of material resources taken from nature and total waste generation (Formoso et al., 2002; Choate, 2003; Edwars and Bennet, 2003; Aïtcin, 2000). Many types of waste are generated in the construction industry; some of them can be recycled easily as high added value materials (e.g. steel, thermoplastic polymers). However, in most cases, reusing of waste is difficult or, alternately, the reusing way does not produces high valued products (e.g. concrete, cement mortar and ceramics from demolition, gypsum, thermosetting polymers). An interesting waste is the paper packaging used for binders (e.g. Portland cement, gypsum, hydrated lime): the recycling of this paper-based residue is not trivial as it is for other types of paper. It is a consequence of the residual content of cement (or powdered binder, in general) in the bags what can be highly abrasive and, consequently, undesirable for the recycling processing. In many countries (e.g. Brazil) the amount of Portland cement commercialized in kraft bags is significant and the destination of the used bags is considered a key issue for the Association of Cement Producers in these countries. Therefore, the approach of recycling any Portland cement packaging is interesting as reinforcement in fiber-cement and requires simple techniques for processing and consequent application for manufacturing theses composites.

However, the main drawback presented by the addition of cellulosic pulp on Portland cement matrices is the durability of the fiber subjected to this alkaline environment with pH > 12 (Tolêdo et al., 2000). Thereby, some progressive degradation mechanisms may take place, such as the destruction of macromolecular chains during the partial alkaline hydrolysis of the cellulose, which causes their rupture and the consequent decrease in the degree of polymerization. Besides, the degradation of the fibers occurs by the easy movement of the pore water towards the surface of the fibers. The inner cores of the vegetable fibers may gradually fill up with the hydration products leading to the embrittlement of the fibers, reducing their mechanical performance (Pavasars et al., 2003; Scrivener and Young, 1997).

Pozzolans are materials with an amorphous siliceous and aluminous content that react with portlandite ($Ca(OH)_2$), which is a by-product of the hydration of the anhydrous calcium silicates and is primarily responsible for the high pH of the Portland cement-based system. It is well known the ability of the fly ash (FA) to promote the pozzolanic reactions when combined with the portlandite (Monzó et al., 2002). Additionally, it has been demonstrated the efficiency of the fluid catalytic cracking catalyst residue (FC3R) when incorporated to Portland cement at short times, which has a similar chemical composition and high pozzolanic activity of metakaolin (Payá et al., 2003). The addition of gypsum in the cement composite can contribute to reduce the content of portlandite in the matrix, with no significant expansion by formation of ettringite, and with the increase of the flexural mechanical performance (Çolak, 2000). In order to mitigate the degradation mechanisms of pulp fiber into cement matrix the objective of this work was to evaluate the gypsum–Portland cement– pozzolan (FA and

FC3R) blends with 50% by weight of gypsum relative to the total weight of binder, reinforced with recycled pulp originated from Portland cement kraft bags.

2. EXPERIMENTAL

2.1 *Materials*

The recycled Portland cement packaging used in this work was constituted of unbleached and unrefined softwood pulp (*Pinus*). In order to prepare the cellulosic pulp, the recycled Portland cement packaging was initially cut in small pieces by hand and dispersed separately by mechanical stirring at 1000 rpm during 1 h, in the proportion of 400 g within 1.5 L of water. After that, the cellulosic pulp was dispersed in water by mechanical stirring (3000 rpm for 5 min). The excess water was removed by filtration. The produced pulp was then kept under refrigeration in sealed plastic bags. The morphological characteristics of fiber length and diameter and other selected properties of the cement bag pulp were analyzed using the equipment TM Pulptec MFA-500 Fibre Morphology and Shive Analyser - MorFiTrac.Characteristics of the pulp are shown in Table 1.

Table 1-Characteristics of the unbleached and unrefined softwood pulp (*Pinus*).

Table 2 shows the chemical composition of the binders. The Brazilian cement (ordinary portland cement, OPC), type CPV-ARI, was used in this work. The use of this cement is justified because is commercially available, without pozzolanic or slag of blast furnace additions and has a lower content of limestone filler. Besides, this cement has a higher content of C_3S and C_2S (C = CaO and S = SiO_2), which is favorable to generate an aggressive environment for the study of alkaline degradation of the cellulosic fibers. Particle size distribution of OPC shown a D_{50} of about 11.0 µm. The FC3R was supplied by the company Omya Clariana (Esplugues de Llobregat-Barcelona, Spain). It is basically an aluminosilicate material with an average particle diameter value of 9.3 µm, $D_{50} = 5.3$ µm, $D_{90} = 21.3$ µm and a specific gravity of 2.73 g/cm^3. The fly ash used is class F and was supplied by the thermoelectric power plant of Andorra-Teruel (Spain). It has a specific gravity of 2.84 g/cm^3. In order to increase its pozzolan reactivity, the fly ash was ground using a 1 L high impact energy mill (Gabbrielli Mill-2) (Payá et al, 1997, 2000). The ground fly ash (GFA) reached an average particle diameter value of 10.2 µm, $D_{50} = 7.8$ µm and $D_{90} = 22.1$ µm.

Table 2- Chemical composition of the binders

The gypsum was supplied by Brazilian company (State of Pernambuco, Araripe gypsum District). Calcium sulfate hemihydrate, with the chemical formula $CaSO_4.0.5H_2O$, are the main component of gypsum. The chemical and physical analysis can be seen in the Table 3.

Table 3. Chemical and physical characteristics of the gypsum*

The composite with 100% Portland cement was used as reference (OPC). The low alkalinity cementitious composites were developed using the following mix designs shown in Table 4. It was used 91% by weight of the gypsum- Portland cement - pozzolan blend, with 50% by weight of gypsum and different percentages by weight of cement and pozzolan (fly ash (GFA) or catalytic cracking catalyst residue (FC3R)). 9% by weight of pulp respect to the binder (cement+pozzolan+gypsum) was used.

Table 4- Mix designs of the low alkalinity cementitious matrices

2.2. Preparation of the composites

The pads of the composites of 200 x 200 mm and around 6 mm thick were prepared in laboratory scale using a slurry vacuum dewatering followed by pressing technique described in detail by Savastano Jr. et al. (2000). Composites were based on matrices listed in Table 3 and reference matrix with 100% of Portland cement. In order to prepare the cellulose pulp, the recycled Portland cement packaging was initially cut in small pieces by hand and dispersed separately by mechanical stirring at 1000 rpm during 1 h, in the proportion of 400 g within 1.5 L of water. After that, previously, the cellulose pulp was dispersed in water by mechanical stirring (3000 rpm for 5 min). The mixture - matrix and cellulose pulp - formed with approximately 20% of solids was stirred at 1000 rpm for 4 min. The slurry was transferred to the evacuable casting box and the vacuum was applied (~70 kPa gauge) until a solid surface formed. Three pads were pressed simultaneously at 3.2 MPa for 5 min. Pads were then sealed wet in a plastic bag to cure at room temperature for 28 days. Pads were cut wet into four 165 mm x 40 mm flexural test specimens using a diamond saw cooled with water. Specimen thickness was approximately 6 mm. On completion of the cure, specimens were tested at 28 days after production. For the mechanical tests five specimens were prepared for each mix design and for the physical characterization (water absorption, apparent porosity and bulk density) three specimens were used.

2.3. Soak and dry accelerated ageing cycles

The soak and dry accelerated ageing cycles intended to partially simulate the thermal and mechanical stresses caused by natural ageing conditions such as rainfall and solar radiations, accelerated hydration products reactions and alkaline attack from pore water on the cellulose fibers. This test involved comparative analysis of the physical characteristics and mechanical performance of the composites before and after this test. Specimens were successively immersed in water at 20 ± 5°C during 170 min, followed by the interval of 10 min, and then exposed to the temperature of 60 ± 5°C for 170 min in a ventilated oven and with the final interval of 10 min. Each soak and dry set represents one cycle and was repeated for 100 times. This procedure was based on recommendations of the EN 494 Standards.

2.4. Mechanical and physical characterization

Mechanical tests were performed in a universal testing machine Emic DL-30,000 equipped with 1 kN load cell. A four-point bending configuration was applied for the determination of the values of modulus of rupture (MOR), limit of proportionality (LOP), and specific energy (SE), according to Eqs. 1 to 3. The LOP was described as the stress corresponding to the upper point of the linear portion of the load versus deflection curve. The SE was defined as the energy absorbed during the flexural test and divided by the specimen cross-sectional area. The absorbed energy was obtained by integration of the area below the load versus deflection curve at the point corresponding to a reduction in the load carrying capacity to 95% of the maximum reached. The modulus of elasticity (MOE) was determined by Eq. 4 and m, which is equal to tangent of the slope angle of the load versus deflection curve during the elastic deformation. A major span of 135 mm and a displacement rate of 1.5 mm/min were adopted. The deflection during the bending test was registered by the deflectometer positioned in the middle span, on the down side of the specimen.

$$MOR = \frac{P_{max} \cdot L_v}{b \cdot h^2} \qquad \textbf{(Eq. 1)}$$

$$LOP = \frac{P_{LOP} \cdot L_v}{b \cdot h^2} \qquad \textbf{(Eq. 2)}$$

$$\mathrm{SE} = \frac{\text{Energy absorbed}}{\mathrm{b} \cdot \mathrm{h}} \qquad \textbf{(Eq. 3)}$$

$$MOE = \frac{276 \cdot L_v^3}{1296 \cdot b \cdot h^3} \cdot m \qquad \textbf{(Eq. 4)}$$

where P_{max} is the maximum load, P_{LOP} is the maximum load reached on the linear portion of the load versus deflection curve, L_v is the major span between the supports, b and h are the specimen width and depth respectively.

Water absorption (WA), apparent porosity (AP) and bulk density (BD) were obtained by following the instructions of ASTM C 948-81 Standards. The water content for the determination of the water/binder ratio was calculated by the difference of weight between the amount of dry raw materials (binder + fibers) and the prepared pad.

3. **RESULTS AND DISCUSSION**

3.1. Mechanical behavior

Table 5 lists the average values and their respective standard deviations for the mechanical behavior of the composites with blend matrix and without pozzolan, as well as the Figures 1-5 reveal some aspects of the mechanical behavior of these materials. Figure 1 shows typical stress versus specific deflection curves of the cement based composites. Figure 2 shows the corresponding curves for aged specimens after 100 soak and dry cycles.

Figure 1- Typical stress versus specific deflection curves for composites reinforced with cellulose pulp from recycled cement bags at 28 days.

Figure 2- Typical stress versus specific deflection curves for composites reinforced with cellulose pulp from recycled cement bags after 100 soak and dry cycles (aac = accelerated ageing cycles).

Table 5- Mechanical properties of the cement based composites (aac = accelerated ageing cycles)

Figure 3- Average values and standard deviations of the modulus of rupture (MOR) of the composites (mix designs) before and after the soak and dry accelerated ageing test (aac = accelerated ageing cycles).

The cement based composites with Portland cement matrix (OPC) at 28 days presented the highest average MOR values, approximately 11.8 MPa (Fig. 3). The composites F1 and F2 (low alkalinity cement based composites with ground fly ash) at 28 days presented 10.3 and 11.7 MPa respectively. However, the composites F3 and F4 (low alkalinity cement based composites with FC3R) reached about 9.4 and 8.0 MPa respectively. These differences in the mechanical behavior between these materials can also be observed in the average values of MOE. The composites 100% C, F1 and F2 presented average MOE values greater than 12 GPa, while composites F3 and F4 about 9 GPa (Table 5). This behavior could be attributed to the water/binder (w/b) ratio of composites: OPC and GFA composites had w/b values (see section 3.2) in the range 0.25-0.29, whereas composites containing FC3R had higher w/b ratio (range 0.32-0.36).

After 100 cycles of accelerated ageing, the average MOR value of the composite without any kind of pozzolanic addition (OPC) decreased about 46% if compared to the corresponding value of MOR at 28 days (Figure 3 and Table 5). Therefore, the observed loss on the bending strength after exposition to accelerated ageing test may be due to the degradation of the cellulose fibers or changes on the fiber-matrix interface (Scrivener and Young, 1997; Payá et al., 1999; Peris Mora et al., 1993; Payá et al., 1996). The LOP/MOR ratio can indicate the conditions of the fiber-matrix interface and how much fibers work after first crack to start growing until the composite to reach the maximum resistance in the four point bending test. After 100 accelerated ageing cycles, LOP/MOR ratio for composites with pozzolanic additions is significantly lower (between 0.57-0.64) than that without pozzolans, where the LOP/MOR ratio is nearly equal to 1 (0.97). Thus, after accelerated ageing the fibers did not reinforce the composites with Portland cement matrix (OPC), as it can be seen in Figure 2. This LOP/MOR ratio was lower for mixtures containing 15% of Portland cement (F1 and F3), suggesting that the reduction in the content of this binder drives to better performance of the fibers within the matrix.

On the other hand, the average MOR values were maintained for the composites with blended matrices, except for F4 which tended to increase approximately 37% (Fig. 3), after 100 cycles of accelerated ageing test. These results suggest that the cellulose fibers were well preserved and adhered satisfactorily to the blend matrix. In contrast, average MOE values decreases about 30% for F4 and about 60% for other composites with blended cement matrices, but composite with Portland cement matrix presented similar average MOE value before and after the accelerated ageing test. This mechanical property is associated mainly with the stiffness of the matrix. It is known that the main deterioration agent for gypsum is water. Even a small amount of water may produce severe reduction of the matrix stiffness. Thus, the matrices with gypsum and pozzolan tend to present less rigidity, after 100 cycles accelerated ageing test.

The Figure 4 shows that the low alkalinity cement composites (F1 to F4) and composite without pozzolan (OPC) reached higher SE values at 28 days if compared to other composites with approximately the same amount of fibers in similar studies (Tonoli et al., 2009; Roma et al., 2008). Specific energy is directly associated to the behavior of the fibers into matrix of the composites. When a composite is loaded, for example, in bending configuration, before the rise of some micro-cracks, part of the stress of the matrix is transferred to the fibers. After the formation of some micro-cracks, toughening mechanisms appeared as the results of the interaction between fiber and matrix, such as pullout and bridging (Bentur and Mindess, 2007). The cellulose fibers obtained from the Portland cement bags were effective reinforcing elements for the composites analyzed in this work.

Figure 4- Average values and standard deviations of the specific energy (SE) of the composites (mix designs) before and after the soak and dry accelerated ageing test (aac = accelerated ageing cycles).

However, after 100 cycles of soak and dry accelerated ageing test the average SE value of the composites without pozzolan (OPC) decreased drastically. The average SE value was reduced from 2.99 kJ/m^2 at 28 days to 0.06 kJ/m^2 after the accelerated ageing cycles. This result suggests that the toughness reduction is associated to the degradation mechanisms on the fibers by direct contact with calcium hydroxide released during the hydration process of the Portland cement, such as fiber mineralization and alkaline attack (Bentur and Mindess, 2007; Scrivener and Young, 1997). On the other hand, composites with the blended matrices presented less ductility loss as compared to composite without pozzolan, although the composite F2 have suffered a remarkable loss of 63% on toughness. Contrarily, the average SE value of the composite F1was maintained. This behavior could be related with the higher amount of Portland cement in F2 composite compared to F1. For FC3R containing composites, F3 and F4, similar decreasing in SE was observed, probably due to the high pozzolanic reactivity of this pozzolan, which avoided the degradation in large extension of cellulose fibers.

3.2. Physical characteristics

The composite without pozzolan was prepared with lower water/binder ratio about 0.25. The composites F3 and F4 have required the highest water/binder ratio values around 0.32 and 0.33 respectively. Previous work indicated that Portland cement system combined with FC3R pozzolan needs a large amount of water. The FC3R is an inorganic aluminosilicate material with a zeolitic structure, and for this reason it has a high specific surface area (Payá et al., 1997). The composites F1 and F2 have a water/binder ratio around 0.26 and 0.27 respectively. The fly ash acts as a lubricant (Peris Mora et al., 1993, Payá et al., 1996) and counteracting the demand of water by gypsum, which is greater than the Portland cement corresponding demand of water. Besides, there is the influence of the particle-size distribution and packing effect of both pozzolanic materials studied in this work. The excess water initially produces voids in the matrix increasing the apparent porosity of the composites. This effect was observed by physical characterization at 28 days (Table 6). The composites with FC3R (F3 and F4) have a higher average apparent porosity value than those without this pozzolan, probably because the higher water demand for this pozzolan and lower compaction of the matrix. After accelerated ageing test, the composites (F2 and F4) presented an increase of average apparent porosity value as well as they presented a lower bulk density value. The average bulk density values increased for the composite without pozzolans which can be explained by porosity refinement due to the continuation of the hydration process of OPC with curing time.

Table 6- Physical characteristics of the composites (aac = accelerated ageing cycles).

The average bulk density (BD) values of the composites with blended matrix are clearly lower (between 15 to 28%) than the value corresponding to the composite without pozzolan at 28 days. This behavior is related to the higher porosity in these composites. The low bulk density is a desirable property for some applications in building construction considering the specific strength (mechanical strength/ bulk density, MOR/BD) because for a lower bulk density the specific strength increases. Thus for nonaged specimens, the value MOR/BD for OPC composite was 7.13 MPa/g·cm^{-3} whereas composites F1-F3 were in the range 7.72-8.37 MPa/g·cm^{-3}. Only F4 sample showed a lower value (6.32 MPa/g·cm^{-3}). However, after ageing, the MOR/BD values for F1-4 composites increases significantly and the values were in the range 8.16-9.97 MPa/g·cm^{-3}, in contrast to OPC composite for which the MOR/BD value decreased strongly at 3.37 MPa/g·cm^{-3}. This behavior demonstrated that the good performance of composites F1-F4 respect to the specific strength.

After ageing, the bulk density decreased, probably because the partial dissolution of gypsum. It is well-known that gypsum has a relative high solubility in water if compared to hydrated Portland cement, and there is a leaching of gypsum during the soaking the samples. Contrarily to expected, bulk density value decrease after accelerated ageing test is more pronounced for mix design with more cement (F2 and F4) as well as these composites presented a larger apparent porosity. Finally, water absorption for blended composites is approximately twice than water absorption found for OPC composite, due to the high values of total porosity for F1-F4 composites: porous system is well connected because hydration products from cement, gypsum and pozzolanic reaction between pozzolan and portlandite from cement hydration are not selling completely the pores. For all binder-blended composites F1-F4, despite the low bulk density, high water absorption and high apparent porosities, the mechanical properties after ageing, such as specific energy, MOR and specific strength, are significantly better than those found for OPC composites.

4. CONCLUSIONS

The pulp obtained from recycling Portland cement bags works satisfactorily as reinforcement for composites with cement-based matrix consisting of 100% Portland cement at 28 days of age, the average MOR value was 11.77 MPa and average specific energy was 2.99 kJ/m^2. However, after 100 cycles of accelerated ageing the values of MOR and SE decreased drastically to 6.34 MPa and 0.06 kJ/m^2, respectively. This behavior is indicative of the fibers being degraded very easily.

Changing the type of matrix, reducing the cement content (15-20%) and adding cheaper alternative mineral materials, such as gypsum (as calcium sulphate hemihydrate) and pozzolans (fly ash or fluid catalytic cracking catalyst residue) allowed to prepare composites with good mechanical performance at 28 days of age (MOR values between 7.90-11.72 MPa and SE values between 2.77-5.27 kJ/m^2). The average bulk densities values of the composites with blend matrix are significantly lower than the values corresponding to the composite without pozzolan at 28 days, between 15 and 28% less, due to the larger amount of capillary porosity. These results indicate that these matrices are suitable for such composites because of the higher specific strength (mechanical strength/bulk density), even more after ageing, given that higher specific strength is a desirable quality for materials with a good flexural performance.

The low alkalinity cementitious matrices composed by Portland cement-gypsum-pozzolanic blends, decisively helped to preserve the mechanical behavior of the fibers from recycled Portland cement packaging after 100 cycles of accelerated ageing. Thus, while the composites with 100% cement matrices lose much of their mechanical performance, the composites with blended matrices were able to maintain high values of MOR and SE, 9.9-10.9 MPa and 1.93-3.60 kJ/m^2 respectively. This behavior is quite remarkable considering that the cement-gypsum-pozzolan matrices composites have lower bulk density (between 25-35% less) and higher porosity values (between 226-303% more) than the reference OPC composite.

The good results obtained in this work show the possibility of using this ecomaterial in low-cost housing.

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