

EXPERIMENTAL CHARACTERIZATION OF GYPSUM-CORK COMPOSITE MATERIAL REINFORCED WITH TEXTILE FIBERS

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ABSTRACT

The study presented herein focus on the analysis of a series of experimental tests aiming at characterizing the performance of distinct textile fibers acting as a reinforcement of a gypsum-cork composite material. Two groups of textile fibers were selected, namely synthetic fibers (glass, recycled textile, acrylic and basalt) and natural fibers (banana and sisal). The reinforced composite material was assessed taking into account the mode I fracture energy by considering indirect tests on notched beams. Additionally, this material was submitted to distinct types of loading, namely uniaxial compression, from which it was possible to obtain the compressive strength and to calculate the elastic modulus, and flexural loading.

KEYWORDS: composite material, plaster, cork, fiber, fracture behavior, characterization of the material.

1. INTRODUCTION

The shortage of environmental, economical and even social resources demands the increase on research of new alternatives in the civil construction relating to new materials and processes. On this basis, the building industry has been developing new construction processes, as well as the study and application of new materials. The building industry has been playing an important role on the reuse of by-products from other industries. The results obtained in a recent study pointed out that by Vasconcelos et al. (2013) [1] show that it is possible to obtain a composite material resulting from the combination of FGD gypsum (flue gas desulfurization gypsum), regranulated cork and textile fibers resulting from recycling process of tyres that can be applied in blocks for partition walls. All these materials can be considered as by-products coming from distinct industries. It was seen that the textile fibers, composed mainly by polyamide, exhibit a great variation on the length and diameter. This dimension scattering possibly can prevent a better performance of the composite material particularly to flexural and tensile loads. Based on this, it was decided to reinforce the gypsum-cork composite material with textile fibers with a known diameter and length, being selected four synthetic fibers (glass, textile, acrylic and basalt) and two natural fibers (sisal and banana). The natural fibers

of and sisal banana have been used in distinct studies in order to evaluate its performance to work as a reinforcing material. Different matrixes have been used to apply natural fibers, namely cement matrix [2-4] and glue matrix [5-7]. Glass fibers have been also used in the reinforcing of gypsum based composite materials [8].

Thus, the major objective of this work is the assessment of the role of textile fibers in the mechanical performance of the gypsum-cork composite material. Different percentages of these textile fibers were considered being possible to evaluate the improvements in the compressive strength and especially in the tensile fracture energy. For this the reinforced composite material was submitted to distinct types of loading, namely compression tests, from which it was possible to obtain the compressive strength and to calculate the elastic modulus, flexural loading. Additionally, aiming at assessing the mode I fracture energy, indirect tests on notched beams were carried out.

The improvement on the fracture behavior of the composite material with textile fibers should be mainly attributed to the fibers with higher diameter and length, able to undertake tensile stresses that are transferred by composite material after crack initiation and development. The performance of the reinforcing textile

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fibers should be associated to the compatibility and adhesion existing between them and the matrix. It should be stressed that the textile fibers are very important to avoid brittle tensile fractures, which can be important when the mortar is being loaded mainly out of plane. Besides the presentation of procedures and results an analysis and discussion on the results is provided.

2. MATERIALS

2.1. Gypsum and granulated cork

The granulated cork used in this work was a granulated of expanded cork, a byproduct of a Portuguese industry of black agglomerate cork boards as it results from the cork-oak tree branches. It can be classified as a light aggregate with a density rather lower than the traditional aggregate. The granulated cork has similar properties to the expanded polystyrene and expanded clay in terms of thermal insulation and commonly applied without structural role. The granulated cork is constituted by different particles sizes namely 2/4mm, 2/9mm and 4/8mm, with density of respectively 166, 182 and 198kg/m³ and bulk density of 65, 73 and 72kg/m³. Taken into account that the main aim of the application of the composite material composed of gypsum and granulated cork is the production of blocks for partition walls, it was decided to adopt the granulated cork with the lowest granule dimensions (2/4mm), given the relatively small thickness of the external shell of the block [9].

The gypsum is a comercial material commonly designated by plaster. It used often in the finishing of construction elements surface, namely partition walls. This gypsum is thus distinct from the gypsum used in [1], which resulted from the dessulfurization of the effluent gas formed in a termal power station.

2.2. Textile fibers

The fibers used for the reinforcement of the gypsum-cork composite material are natural fibers from sisal and banana and synthetic fibers such glass, textile, acrylic and basalt (Figure 1). The physical characterization of the natural fibers was made through the use of an optical microscope (Fig. 2 (a)) in the textile department of University of Minho. For the characterization 20 specimens of each natural fiber were considered, being each specimen composed of only one filament. The measurement of the diameter was made by considering 5 cross sections to calculate the average diameter (Fig. 2 (b)). With optical microscope it was possible to obtain amplified images of the filament with the corresponding measurements of the diameter and length, as shown in Fig. 2 (c), where the filament of banana fibers can be seen. The average values of the diameter and length of the natural fibers and textile and acrylic fibers are presented in Table 1.

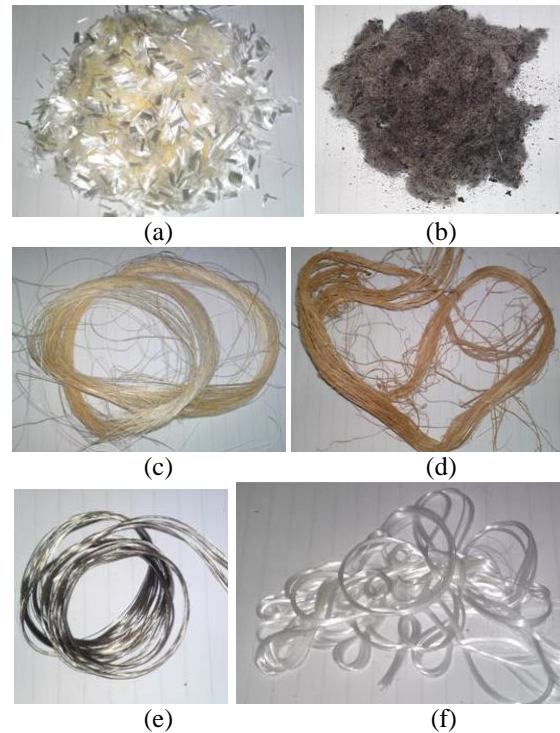
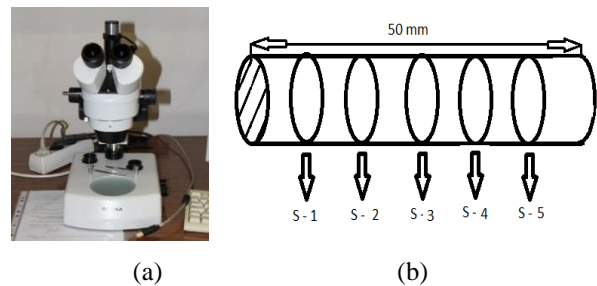
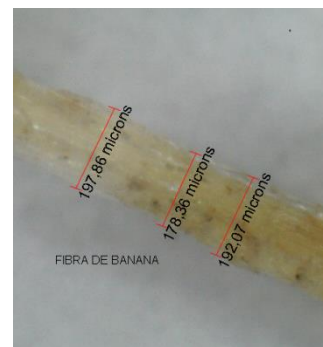


Figure 1. Example of fibers used in the study: (a) acrylic; (b) textile; (c) sisal; (d) banana; (e) basalt; (f) glass



(a) (b)



(c)

Figure 2. Physical characterization of the natural fibers; (a) optical microscope; (b) scheme of the cross section considered to the measurement of the diameter; (c) view of the measurement of the diameter in case of banana natural fiber.

The results obtained for recycled textile fibers revealed great variability on the length, ranging from 108.2 μm to 12469.1 μm. The diameter of the fibers ranges from 7.2 μm and 34.1 μm with an average of 20.7 μm. Based on the results of the Fourier transform infrared

spectroscopy (FTIR) tests it was seen that the recycled textile fibers are mostly composed of polymeric wires and strings, being the main constitutive element the Polyamide 6 [10].

In the same table the average values of the synthetic fibers is also presented according to the information given in the deliver catalogues, as it was not possible to characterize in details the diameter of these fibers. This characteristic and the surface type of the fibers should have a central role on the gypsum-cork composite mortars.

Table 1. Average diameter of the textile fibers

Fiber	Average length (mm)	Average diameter (microns)
Textile	2.1	20.7
Acrylic	6.0	22.4
Banana	10.0	325.9
Sisal	10.0	269.4
Glass	10.0	13
Basalt	10.0	10

The mechanical characterization of the natural fibers was made based on direct tensile tests carried out at the Department of Textile Engineering of University of Minho. For this characterization, 20 specimens with only one filament were prepared for each natural fiber sisal and banana in order to have reasonable representativeness. The direct tensile tests were carried out by using a machine Hounsfield H100KS, with a load cell of 5kN. The tests were carried out at a displacement rate of 25mm/min. In this characterization, the length and the weight of the fibers were measured. From this data it was possible to calculate the SI unity that characterizes physically the density of the textile fibers, through the eq. 1:

$$Tex = \frac{m}{l} \times 1000 \quad (1)$$

where *Tex* is the unit of measure for the linear mass density of fibers, *m* is the mass of the filament (g) and *l* the length of the fibers (m).

From the knowledge of the linear mass density and the maximum tensile load obtained in the direct tensile tests it is possible to calculate the tenacity, which is a measure of the strength of a fiber or yarn. It is defined usually as the ultimate tensile breaking force of the fiber/yarns (in gram-force units) divided the linear mass density, through eq. 2:

$$Tenacidade = \frac{f_{max}}{Tex} = N/Tex \quad (2)$$

In Table 2, the average values of the linear mass density (tex) and the tenacity for the distinct types of fibers are shown. In case of natural fibers the coefficient of variation is also indicated inside brackets in %. The values of the natural fibers were calculated according to

the procedure defined previously. The tenacity of the synthetic fibers was taken from the deliver catalogues. The comparison of the tenacity among the fibers allows to conclude that the tenacity banana is approximately the double of the sisal but they are considerably lower than the tenacity corresponding to synthetic fibers

Table 2. Average diameter of the textile fibers

Fiber	TEX (g/m)	Tenacity (N/Tex)
Textile	-	-
Acrylic	-	-
Banana	29.98 (28.3)	0.35 (26.7)
Sisal	39.67 (20.6)	0.11 (41.6)
Glass	600	0.61
Basalt	600	0.66

3. DESIGN OF THE COMPOSITE MATERIAL

The raw materials used to produce the composite material are commercial gypsum, working as the binder and the regranulated cork as the aggregate fixed in 5% of the gypsum mass. Additionally, it was used a setting time material in order to obtain a workable material to be molded in a mold to produce blocks for partition walls [9]. Based on previous work performed by Vasconcelos et al. (2013) [1] it was decided to use 0.1% of the gypsum mass of citric acid. The water was calculated taking into account the suitable workability of the material according to European standard EN 13279-2 (2004) [11], corresponding to a flow table of approximately 160 mmm. The percentage of the different fibers was fixed in 1% of the gypsum mass. This means that for all the mixtures the water/binder ratio (*w/b*). Notice that according to several authors, the *w/b* ratio takes a major role on the mechanical properties of cement based mortars [12], but is decided to keep the same percentage of fibers in terms of weigh of gypsum. After preliminary tests, it was decided to consider for all the textile fibers a length of 10mm, as for higher values of the length the mixture was difficult and the workability was very poor. However, in case the acrylic and textile fibers, the length was littler due the inexistence of the considered length. In case of recycled textile fibers, the length has considerably scattered as previously mentioned. Notice that a great advantage of using the textile fibers over the recycled textile fibers is the possibility of controlling the variation of the length and diameter of the fibers. Difficulties in obtaining a workable material were observed when higher lengths of the textile fibers were used, see Fig. 3. So, it was decided to adopt a common length and varying the percentage of the water for each mixture in order to obtained a suitable workability.

In Table 3, a summary of the composite mortar mixes is made with the indication of the percentage of each type of fiber considered taking into account the constant values of regranulated cork (5% of the gypsum mass), *w/b* ratio (variable) and the same percentage of citric

acid (0.1% of the gypsum mass) and the same percentage of fibers (1% of the gypsum mass).



Figure 3. Results of the flow table tests to characterize the workability of the composite materials

As the fibers (synthetic and natural fibers) are very different in terms of surface a physical properties, it was not possible to achieve similar workable properties for the same w/b ratio and percentage of fibers, being necessary to vary the w/b ratio. Other approach is variation of the percentage of fibers and the maintenance of the w/b as mentioned by Vasconcelos et al. (2013) [13]. Additionally, it was developed a standard mortar mix to work as the reference. It should be noticed that the standard mixture and the mixture with recycled textile fibers need to the lowest w/b ratio for the same workability. The mixture with sisal fibers present is slightly higher w/b ratio and other mixes present higher values of w/b ratio ranging from 0.52 (glass) to 0.54 (Banana).

Table 3. Design of mortars mixes (values in percentage of the gypsum mass)

Mortar mix	Type of Fibers	cork	Ratio w/b	Citric acid (%)	Fibers
Si	Sisal	5%	0.475	0.05	1%
Ban	Banana	5%	0.54	0.05	1%
Gl	Glass	5%	0.515	0.05	1%
Bas	Basalt	5%	0.533	0.05	1%
Acr	Acrylic	5%	0.53	0.05	1%
Tex	Textile	5%	0.455	0.05	1%
Standard	-	5%	0.45	0.05	-

4. EXPERIMENTAL CAMPAIGN

The experimental program was composed of uniaxial compressive and bending tests, from which mechanical properties such as compressive strength, f_c , elastic modulus, E , and mode I fracture energy, G_f , were obtained.

4.1. Experimental procedures

The uniaxial compressive tests were carried out on cylindrical specimens with 100mm diameter and 200mm height, leading to a height to diameter ratio of 2.0, considering a total of 3 specimens for each mixture. The specimens were kept in a oven at 40°C during seven days, following the recommendations of EN 13279-2

(2004) [11]. The elastic modulus was obtained after two uniaxial compressive tests were carried out to estimate the compressive strength, based on the cyclic load procedure indicated in Fig. 4a, carried out in force control at 0,1kN/s (EN 393 (1993)) [14]. The steady load is estimated to be 30% of the average compressive strength obtained in the two previous uniaxial compressive tests. The mean displacement, from which the strain was calculated, was obtained by averaging the uniaxial displacement recorded by three LVDTs (linear field of ± 2.5 mm and a precision of 0.01% placed in two steel rings distanced of 50mm and placed 120° apart around the specimen, see Fig. 4b). After the determination of the elastic modulus, the uniaxial compressive tests were carried out under displacement control at a rate of 0.005mm/s ensuring that the maximum load is attained in the range between 2 and 15mm after the onset of the test EN 13279-2 (2004) [11] and the record of post-peak behaviour was possible. The deformation was obtained by averaging the displacements recorded by three LVDTs placed 120° apart between the top and the base of the specimens, see Fig 4b. Additionally, tests of compression and flexion were carried according with EN 1052-1 (1999) [15].

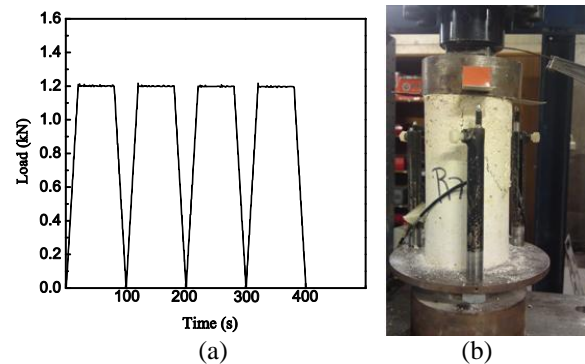


Figure 4. Procedure for uniaxial compressive tests; (a) loading configuration for modulus of elasticity; (b) Instrumentation for compressive tests;

The mode I fracture energy of the distinct composite material mixtures was obtained based on bending tests adopting the three point load configuration, following the testing procedure provided in standard FMC1 (1993) [16] for the obtainment of the mode I fracture energy of concrete and mortar, see Fig. 5. The dimensions of specimens (840mm length, 100mm depth and 100mm width, and effective span of 80mm) were defined according to the maximum dimension of the granulated cork, which was approximately 4.0mm. In each beam, there is a notch with 30mm of depth and 3mm of thickness. In total 3 specimens of each mixture were tested to obtain fracture energy.

The fracture energy tests were carried out in a stiff steel frame equipped with a closed-loop servo control enabling to carry out stable tests. The fracture tests were carried out under displacement control at a rate of 0.002mm/s. The load was applied by means of an

actuator of capacity of 25kN by using a steel roller, see Fig. 5.

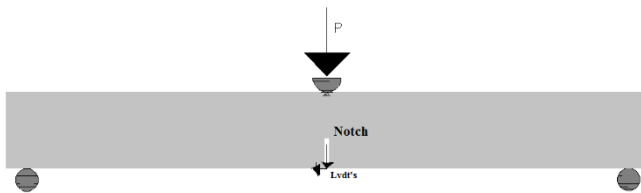


Figure 5. Notched beam for bending tests to obtain mode I fracture energy

The deformation of the beam was measured through an LVDT aligned with the applied load. The calculation of the fracture energy was made based on the expression proposed by standard FMC1 (1993) [16], see eq. 3:

$$G_f = \frac{(W_0 + mg\delta_0)}{A_{lig}} \quad (3)$$

where W_0 is the area under the force-displacement diagram, m is the weight of the specimen and of the load devices, δ_0 is the deformation of the beam at final failure and A_{lig} is the projection of the fracture zone on a plane perpendicular to the beam axis.

5. ANALYSES AND DISCUSSION OF RESULTS

5.1. Compressive behaviour – global behaviour and mechanical properties

The typical mechanical behaviour of the composite material under uniaxial compression reinforced with the distinct textile fibers can be observed through stress-strain diagrams displayed in Fig. 6. In Table 4 are presented the average values of the mechanical properties obtained in cylinders (compressive strength, $f_{c,c}$), and in prims (compressive strength, $f_{c,p}$, and f_f). For all composite mixtures, the pre-peak regime is characterized by two phases, namely an initial linear branch and a nonlinear branch with variable extension, which seems to depend on the ductility exhibited by the specimen. Indeed, the more ductile specimens (smoother post-peak regime) present a more remarkable pre-peak nonlinear range. Low to medium scatter characterizes the linear regime of the composite material. The post-peak behaviour is characterized by a very smooth descending branch, whose smoothness is also variable, resulting in a higher scatter. For all mortar mixes it is seen that low degradation of strength was observed, even if the low strength degradation is accompanied by larger vertical displacements. This is the result of the higher ductility of this gypsum-cork composite material in compression, which appears to be improved by the addition of the synthetic and natural textile fibers. The high ductility of the gypsum-cork composite material in compression is associated to the nature of the aggregate (regranulated cork), which enables that higher deformations develop without collapse, see Fig. 7,

where the failure patterns of some specimens are presented.

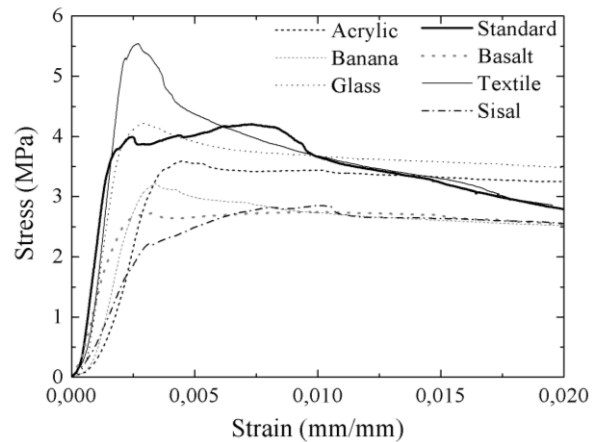


Figure 6. Stress-strain diagrams obtained in compression in cylinders: for all of type of the fibers

It is seen that there is a trend for high lateral deformation are associated to the crushing of material, which corresponds essentially to the collapse of the resisting structure of the composite material attributed to the gypsum. The opening of thick cracks, inclined or sub vertical can be associated also to the relative sliding between both lips of the crack. Besides the elastic modulus, E , obtained in the compressive tests carried out in cylinders, the average values of the compressive strength obtained in cylinder, $f_{c,c}$, and according to EN 1052-1 (1999) [15], $f_{c,p}$, are also given.

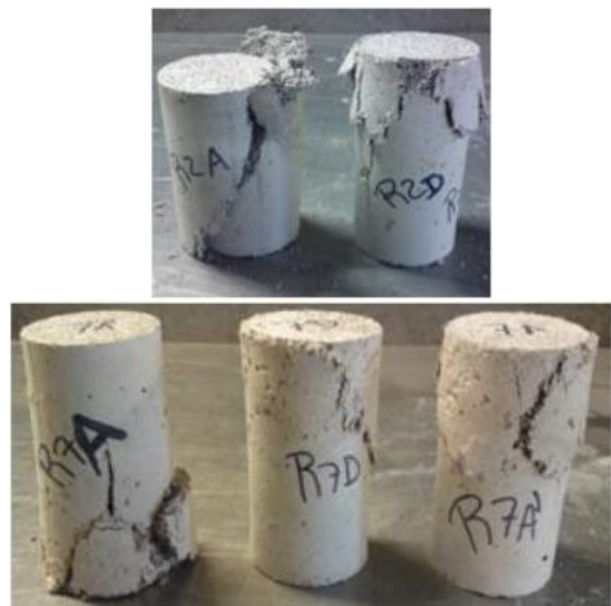


Figure 7. Crack patterns under compression

It is seen that there is a considerable difference between the average values of the compressive strength obtained in cylinders and the one obtained according to the procedure indicated in EN 1052-1 (1999) [15], being the latter reasonably higher. These results should be

expected as in the case of cylinders the restriction effect of the boundaries is minimized, leading to lower values.

Table 4. Mechanical properties of the reinforced composite material

Mortar Mix	$f_{c,c}$ (MPa)	$f_{c,p}$ (MPa)	f_f (MPa)	E (MPa)	G_f [N/m (J/m ²)]
Si	3,1	4,9	3,0	2839,8	230,4
Ban	3,3	3,9	2,4	2470,0	179,0
Gl	4,4	4,8	5,1	3257,4	428,1
Bas	3,4	4,7	4,2	2689,2	394,6
Acr	3,7	4,1	3,3	2887,5	240,0
Tex	5,7	5,1	2,8	3745,7	208,3
Standard	4,9	4,1	3,1	3589,4	76,0

The compressive strength tends to be higher in case of the composite material is reinforced with synthetic fibers, namely textile fibers and glass fibers. It is believed that the increase of the compressive strength of the composite materials reinforced with recycled textile fibers in relation to others, namely glass fibers, can be associated to the lower w/b ratio used in the manufacture of the composite material with recycled textile fibers. Notice that as above mentioned, the w/b ratio takes an important role on the strength properties of the mortars, being the strength lower for higher values of this ratio. This can hinder the effect of the reinforcing fibers on the compressive strength. On the other hand, it is observed that the synthetic fibers lead to higher values of the flexural strength in relation to the standard composite material. The increase is of 65% in case of glass fibers and of 36% in case of basalt fibers. On the other hand the effect of natural fibers and recycled, particularly textile fibers does not improve the flexural strength. For this loading configuration, the contribution of the synthetic fibers is effective for the improvement of the flexural strength the gypsum-cork composite material.

5.2. Fracture behavior under tension

The typical force-displacement diagrams found in the fracture energy tests are presented in Fig. 7. The average values of the fracture energy calculated according to eq. 3, are given in Table 4.

Apart from the specimens reinforced with acrylic fibers it appears that the initial stiffness is similar to all mixes. The results obtained for the three specimens are consistent with this trend. The main differences detected among the distinct blends are at the level of the maximum force reached and in the post-peak regime. Mixtures with glass and basalt fibers are the ones that achieve higher maximum strength and a very ductile behavior, indicating the good performance of the respective fibers.

The post-peak behavior characterized by a decrease of resistance with the increase of the displacement values is different, although some groups follow a trend. The standard mixtures present clearly the steepest post-peak

branch, which allow anticipating that the energy released during fracture is lower than the reinforced mixtures. In fact, the area under the force-displacement diagrams is considerably higher for composites reinforced with fibers and consequently higher values of fracture energy are expected. Notice that in the case of mixtures without fibers it is possible to define the deflection corresponding to the zero loading and in other cases the diagrams is more elongated. This can be associated to the complete loading release in case of the mixture without reinforcement as no bridging effect exists between both lips of the crack. When analysing Fig. 8, one can arrive to the division of the composite into 3 groups. The reference mix reflected a mixture showed no ductility compared with the other, and so that is the mixture that has the lowest fracture energy, as expected.

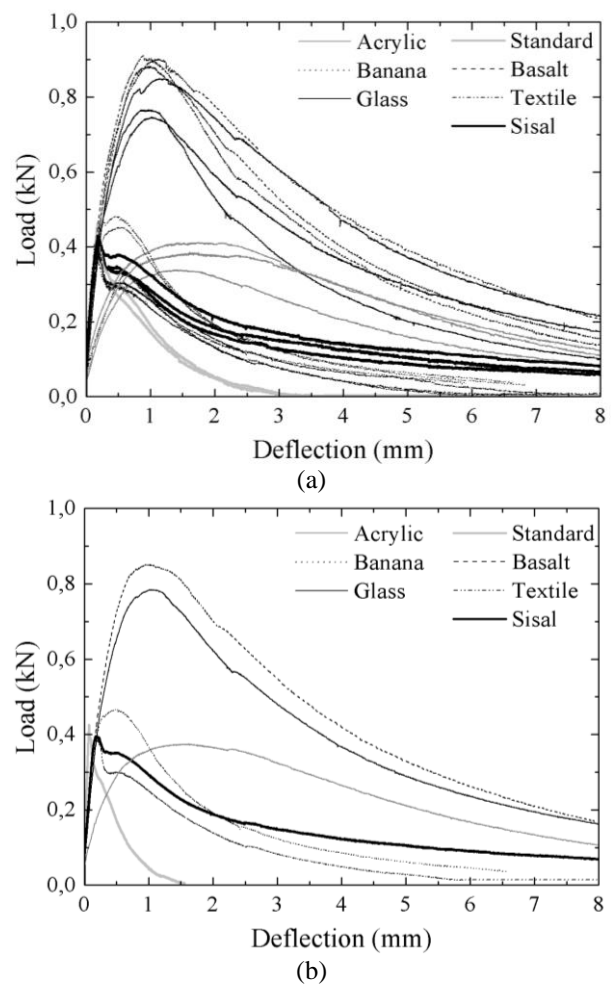


Figure 8. Representative force-displacement diagrams obtained for the mortars; (a) all specimens; (b) average curves

In the second group the mixtures of basalt, glass and acrylic fibers with a very smooth post-peak branch. In spite of the composite material reinforced with acrylic fibers, the post-peak branch is really smooth and approaches the post-peak branch of the composite material reinforced with glass and basalt fibers. The third group is composed by the natural fibers and by the recycled textile fibers. In this group the composite

material with banana fibers behaves better than the other ones. In case of composite material with recycled textile and sisal fibers the post peak branch presents a sharp decrease immediately after the maximum strength, similarly to what happens with the unreinforced mortar. This behavior should be associated to a more difficulty load redistribution of the bearing forces from the mortars to the fibers (Fig. 8). In this group the maximum forces are relatively similar and the post-peak is also very similar, which mean that the fracture energy should be close. However, the fibers confer some benefit when compared with the standard mixture in terms of maximum elongation. Although not exhibit ductility such as the second group is highly visible graphically that they present better performance than the standard mixture

The fracture energy of the specimens with the glass fibers is practically the double of the fracture energy obtained in the specimens with mortars reinforced with banana and sisal fibers. The differences found in the behavior of the synthetic and natural fibers should be associated to the nature of the fibers. The synthetic fibers exhibit a considerable higher tenacity than the natural fibers, which justify the better mechanical performance when added in the mortar. It is interesting to notice that the recycled textile fibers present a similar performance to the natural fibers, in spite of its lower length. The increase on the fracture energy of the natural fibers is about 2.5 in relation to fracture energy of the unreinforced mortar. The presence of fibers allows the progressive redistribution of forces between the matrix and the fibers and the lips of the crack in the fracture zone are connected by the fibers. The lower performance of the natural fibers can be also associated to the surface of the fibers which can promote its sliding from the matrix.



Figure 9. View of the crack after the fracture test in beam with composite material with sisal fibers

6. CONCLUSIONS

In this work a gypsum-cork composite material reinforced with distinct types of textile fibers was studied under uniaxial compressive and three point bending load tests, from which fracture energy was derived. Synthetic textile fibers (glass, basalt, acrylic and textile) and natural fibers (sisal and banana) were

selected in order to evaluate and compare its effectiveness in the mechanical behavior of the gypsum-cork composite material. From the experimental results it was possible to see that:

- (1) the compressive behavior of the gypsum-cork composite materials is similar in terms of the general configuration of the force-displacement diagrams;
- (2) the synthetic fibers result in better mechanical properties, namely, compressive strength and elastic modulus when compared to natural fibers;
- (3) the natural banana fibers performed better in terms of compressive and flexural mechanical properties;
- (4) the synthetic glass, basalt as well as acrylic fibers lead to a more ductile behavior when compared to natural fibers, which is confirmed by the considerable higher values of the Mode I fracture energy;
- (5) the reference mixture showed no ductility and lowest fracture energy;
- (6) the differences in the mechanical performance of the distinct fibers should be associated to the distinct nature and physical and mechanical properties as the synthetic fibers have much higher tenacity. These issues should be addressed in a future work.

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