Lightweight composites based on gypsum with reinforcement of rice husk and polystyrene

Materiales compuestos ligeros basados en yeso con refuerzo de fibra de cascarilla de arroz y poliestireno

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Abstract

Gypsum matrix composites were made with rice husk and polystyrene particles in order to obtain lightweight materials. The composites were mechanically characterized, and morphologically analyzed by Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS). Gypsum and rice husk interfacial transition zone were studied by means of pull-out testing and microstructural analysis. The results showed that the polystyrene allows obtaining lightweight composites with low mechanical properties, which may be considered for non-structural applications. Rice husk fibers reinforcements significantly increased the mechanical properties of the gypsum matrix that due to the good adhesion and the amount of reinforcement used in a major way affecting the toughness.

Keywords: Polystyrene; rice husk fibers; gypsum; toughness; pull-out test.

Resumen

Se obtuvieron compuestos aligerados de matriz de yeso reforzados con cascarilla de arroz y partículas de poliestireno. Las muestras fueron caracterizadas por medio de análisis mecánico y morfológico, mediante microscopia electrónica de barrido (MEB) y espectroscopia de electrones dispersados (EDS). La zona interfacial entre la matriz de yeso y la fibra de cascarilla de arroz fue estudiada por medio de una prueba de arrancamiento y análisis microestructural. Los resultados obtenidos mostraron que las partículas de poliestireno permiten la obtención de materiales compuestos aligerados pero al mismo tiempo se comprometen las propiedades mecánicas de los mismos, lo que hace que el uso de estos materiales compuestos sea de tipo no estructural. Por otro lado, las fibras de cascarilla de arroz reforzaron la matriz de yeso incrementando significativamente las propiedades de flexo-tracción de la

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matriz de yeso y la tenacidad de los compuestos al mismo tiempo que disminuyeron la densidad de los mismos.

Palabras clave: Poliestireno; fibras de cascarilla de arroz; yeso; tenacidad; prueba de arrancamiento.

Introduction

One of the building materials that has been used since ancient times in large quantities is the gypsum material. Archeologists dated the oldest applications of gypsum plaster or gypsum binder to 7000 years B.C. The modern building industry can not only utilize natural gypsum, as commonly done in the past, but also forms of gypsum that appear as waste, secondary-raw material from various sources, such as phospho-gypsum red gypsum, and flue-gas desulfurization gypsum between others (Vimmrova *et al.*, 2011). In many studies the reinforcement of the gypsum matrix for a highly number of applications has been investigated mainly to obtain lightweight materials with insulation properties (Vimmrova *et al.*, 2011; Borreguero *et al.*, 2014; Tonoli *et al.*, 2009; Robayo *et al.*, 2013), likewise it is known that the increase in porosity due to the inclusion of reinforcements decreases the thermal conductivity of the composite (Onésippe *et al.*, 2009). In addition, properties like low density, rapid hardening, insulating behavior and good workability make ''gypsums'' potentially meaningful in the ''must be'' permanent crusade for the search of alternative construction materials free of grey cement contributing in the economics and the reduction of CO_2 liberation in the pursuit of sustainability (Magallanes-Rivera *et al.*, 2012). In this work the possibility of using two types of reinforcements is studied, expanded polystyrene and rice husks for gypsum matrix composites, being the aim to obtain lightweight materials. It was analyzed both the density obtained, as well the mechanical properties for the composites and reinforcements, including the morphological characteristics for the Interfacial transition zone (ITZ) which plays an important role in the final properties of the composites.

Experimental

Materials

The plaster used was commercial gypsum, developed primarily for making stucco. The rice husk (RH) used was provided by a regional rice mill. The RH samples were subjected to a selection and cleaning process. The characteristics of polystyrene used were given by the supplier while the dimensions of rice husk fibers were measured by SEM figure such has been done in other works in the 'Grupo Materiales Compuestos' (Pardo and Salinas, 2013). The spherical particles of polystyrene with a diameter range between 3.3 mm and 4.8 mm are commercial selling. The density and size of these compounds are seen in Table 1.

Table 1. Characteristics of materials.

Raw Material	Density Mg/ m^3	Dimensions
Polystyrene particles	0.02	Average Diameter: 4.05 mm
Rice husk fibers	0.65	Average Transversal Area: 0.20 mm^2 Average Lengh: 9.7 mm
Gypsum	2.33	Average size: 22 µm

Source: Authors

Methods

The fiber tension test was carried out following ASTM D3822-07, at the ends of the fibers were adhered pieces of paper to be subjected to the jaws (figure 1, **right**), thus the transversal area and the fiber perimeter were obtained by SEM. The statistical treatment of the data for the tensile strength of the rice husk fiber has appeared in many other work in the 'Grupo de Materiales Compuestos' (Pardo and Salinas, 2013); (Cocuy *et al.*, 2014), in this work being the objective the lightening of gypsum matrix, that part is skipped. Polypropylene molds were used for the pull-out measurements, which have a cubic shape with a cross sectional area of 12 mm x 12 mm, the fiber was immersed in the center of the sample, and in its other edge, a piece of paper was bonded to be clamped with the universal testing machine (Figure 1, **left**).

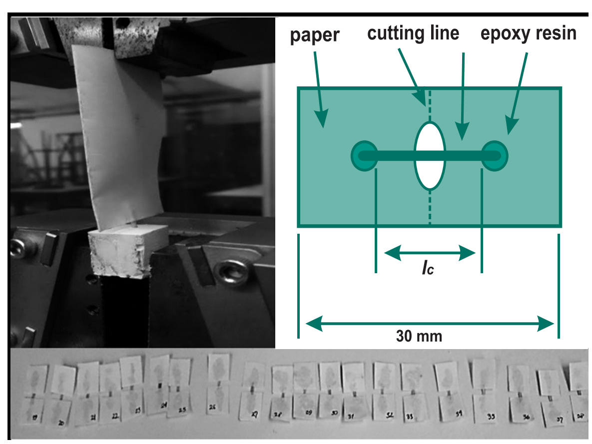

Figure 1. Testing set- up: (Left) pull-out. (right) tension test, (down) Fiber Tension Test Samples.
Source: Authors

Gypsum matrix composites reinforced with 40% (v/v) of polystyrene (PE) and composites reinforced with 26% (v/v) of rice husk (RC) were performed by thermal curing at 40 ºC for one hour and post-cured at room temperature for 7 days according to other studies (Yu *et al.*, 2012; Chinta *et al.*, 2013; Gencel, *et al.*, 2014). The proportion was calculated with a mixtures rule in order to obtain a density lower than 1, however a limiting factor was the workability of the mixture that produced restriction in the addition's selection, thus finally the proportion was mainly obtained from the literature's results (Vimmrova, *et al.*, (2011); Aghazadeh *et al.*, (2011); Borreguero *et al.*, (2014); Madariaga and Lloveras , (2008); Sabrine *et al.*, (2013)). Physical and mechanical properties such as density (displaced volume technique), flexural strength (ASTM C293) and compressive strength (ASTM C 472) were measured. The morphology and composition of the transition zone were analyzed by SEM and EDS techniques. The bond stress was determined using the detachment method or pull- out of the fiber in tension (Valadez, 1999), according to equation 1.

$$\tau = \frac{F_f}{l_e P_e} \qquad (1)$$

Where F_f is the maximum pullout load, L_e represents the embedded length and the P_e the fiber perimeter. The expression of Kelly and Typson is an alternative way to calculate the bond stress (Valadez, 1999) in equation 2.

$$\tau = \left(\frac{\sigma_f}{2}\right)\left(\frac{d_f}{l_c}\right) \qquad (2)$$

The critical length l_c for a fiber can be calculated as in equation 3.

$$l_c = \left(\frac{\sigma_f d_f}{2\tau}\right) \qquad (3)$$

Where:

σ_f: Maximum Tensile strength of the fiber

d_f: Diameter of the fiber

τ: Bond stress

The flexural strength was performed using the universal testing machine Instron 3369 model equipped with 1 KN load cell. Three-point bending configuration was employed to evaluate the modulus of rupture (MOR), following the expression 4.

$$MOR = \frac{3 \times L \times a}{2 \times b \times t^2} \qquad (4)$$

Where L is the Load at failure, a is the beam span between supports, b width of beam and t is the depth of beam. The toughness was obtained by integration of the area below the load vs. deflection curve at four points, to the maximum resistance at first crack, σ_f, and at $3\sigma_f$, $5.5\sigma_f$ and $10.5\ \sigma_f$, values. The toughness index of the specimens (I_1, I_2, I_3), could be obtained using the equations 5, 6 and 7.

$$I_1 = \frac{Area\left(P - 3\delta_f\right)}{Area\left(P_f - \delta_f\right)} \qquad (5)$$

$$I_2 = \frac{Area\left(P - 5.5\delta_f\right)}{Area\left(P_f - \delta_f\right)} \qquad (6)$$

$$I_3 = \frac{Area\,(P - 10.5\,\delta_f)}{Area\,(P_f - \delta_f)} \qquad (7)$$

Results and discussion

The two types of reinforcement induce effects of lightening on the composite material, being more marked the results obtained for expanded polystyrene. In the Table 2 can be appreciated the results for density. The reinforcement of polystyrene produced a decrease in density with respect to the pattern near the 28.6%, giving an important lightness, even having the ability to float on water; these results confirm that is possible decreasing mass by unit surface with the adhesion of polystyrene particles, similar results obtained by Madariaga and Lloveras, (2008).

Table 2. Density results after 7 days of curing.

Gypsum			PE			RH		
Mass (g)	Volume (cm^3)	Density (g/cm^3)	Mass (g)	Volume (cm^3)	Density (g/cm^3)	Mass (g)	Volume (cm^3)	Density (g/cm^3)
22.36	16.00	1.40	18.18	18.00	1.01	26.75	20.00	1.34

Source: Authors

Polystyrene spherical particles in the composite induce a high degree of porosity, functioning as a pore agent, due to the high air contents. In the tables 3 and 4 are shown the results from compression and flexural analysis, where can be noted low mechanical properties for composites with polystyrene compared to the pattern samples, thus their applications may be limited to non-structural elements.

Table 3. Compressive strength Test results.

Mixture	Compressive Strength (MPa)	Specific Compressive Strength (MPa/Mg/m^3)
Gypsum	2.42	1.73
PE	1.47	1.45
RH	2.19	1.63

Source: Authors

Table 4. Flexural Strength after 7 days of curing.

Mixture	Max. Load (N)	MOR (MPa)
Gypsum	31.67	2.19
PE	28.39	1.96
RH	53.51	3.70

Source: Authors

On the other hand, the reinforcement with rice husk produced a significant increase in MOR, although has similar density with respect to the pattern samples. In the Figure 2a can be observed the load vs. deflection curve for the all types of studied composites. A significant effect on the toughness of the composite reinforced with rice husk was observed, as is seen in the Figure 2b. The Table 5 shows the results for the toughness and flexural toughness index, these values clearly reflect the effect of the added fibers on the post-cracking behavior, which it means an efficient response of the fibers on the crack arresting which increases with the grade of the applied load that is transmitted from matrix to the fiber. In Table 5 is shown an increase in toughness to first crack (σf) with rice husk reinforcement of 2.67 times the pattern toughness.

The flexion results in reinforcement with rice husk, suggest besides good adhesion of the fiber to the gypsum matrix also an homogeneous distribution due to the pre-mixed in dry, in contrast to the results obtained by Hamza *et al*., (2013), who obtained a decline in the mechanical properties of the samples reinforced with natural fibers, that is associated with the generation of porosity in addition to the inhomogeneous distribution of the fibers in the matrix.

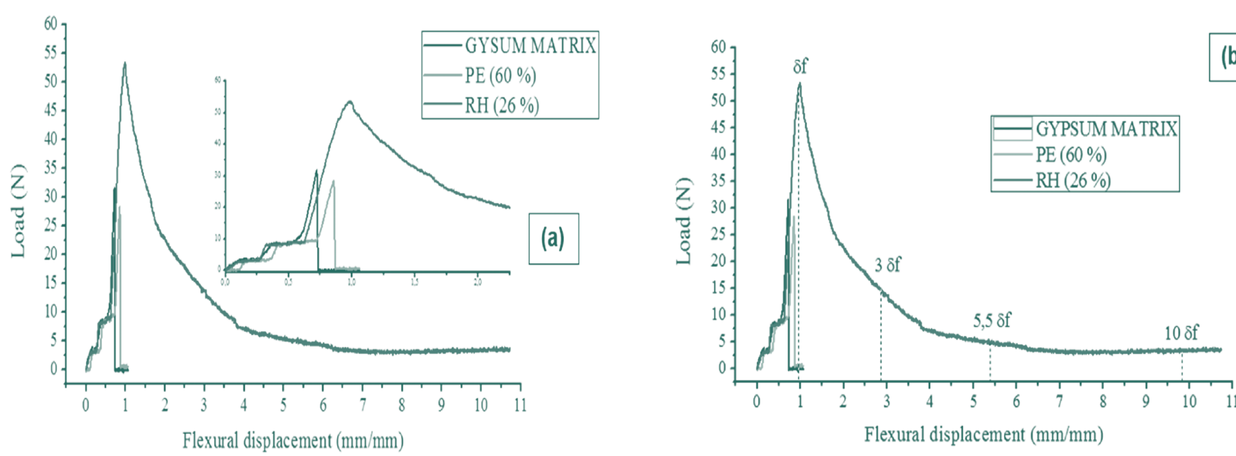

Figure 2. Results from flexural strength analysis: (a) load vs. deflection curve (b) flexural toughness index.
Source: Authors

Table 5. Toughness and flexural toughness index after 7 days of curing.

Mixture	Load $_f$ (N)	δ_f (mm)	Toughness (N.mm)				Toughness Index		
			)	3 δ_f	5,5 δ_f	10,5 δ_f	δ_f I_1	I_3	I_5
Gypsum	31.66	0.73	5.88	-	-	-	-	-	-
PE	28.39	0.86	6.21	-	-	-	-	-	-
RH	53.51	0.98	15.70	67.82	86.62	101.79	4.32	5.52	6.48

Source: Authors

The results of the pull-out test (Figure 3 and Table 6) and the obtained values for MOR suggest a significant adherence of the rice husk fiber on the gypsum matrix, related to the hydrophilic characteristic of the fiber and good wetting of the matrix, Also the morphological characteristics of rice husk with high roughness allows a significant contribution of mechanical anchoring (Pardo y Salinas, 2013).

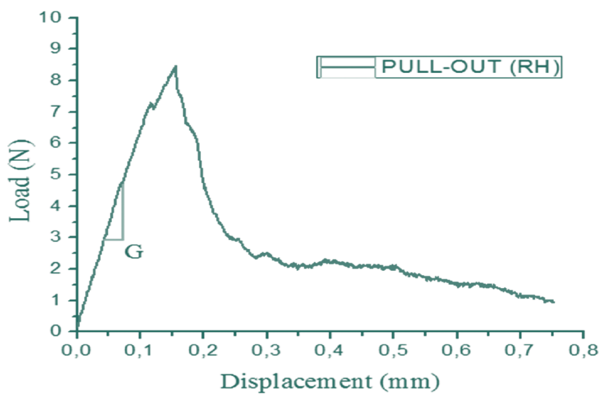

Fig. 3. Pull-out curve.
Source: Authors

Table 6. Test results Pull Out.

Fiber	Embedded area (mm^2)	Extraction load (N)	τ (MPa)
RH	24.74	8.47	0.34

Source: Authors

Using the equation (3) with the values obtained for the adherence tension is calculated the critical length for the rice husk fibers in a gypsum matrix, the results are showed in the Table 7, can be observed that the average critical length value is superior to the fiber length, indicating that pull out phenomena is more prone to occur. The increase in toughness is due to the dispersed energy for the fibers in the displacement process where the pull-out is dominant on the interface zone due the length of the fiber (Singh *et al.*, 1994; Savastano *et al.*, 2003) that reported from Figure 3, a module G of 62.58 N/mm.

Table 7. Test Critical Length.

Fiber	Fiber tensile resistance (MPa)	Fiber Length (mm)	Fiber Transversal Area (mm^2)	Average Critical Fiber Length (mm)
RH	54.53	11.98	0.21	41.46

Source: Authors

The morphologies of the interface for the expanded polystyrene as well as for rice husk are seen in the SEM micrographs presented in figure 4. It can be seen an interface with high porosity due to the wall effect in the transition zone, the polystyrene (fig 4c, 4d) presents a very poor adherence, which explain the results of the mechanical tests for the two cases (Martias *et al.*, 2013). The observed morphology suggests a high fragility of the transition zone, similar to the findings of other authors (Tonoli *et al.*, 2009; Pardo and Salinas, 2013).

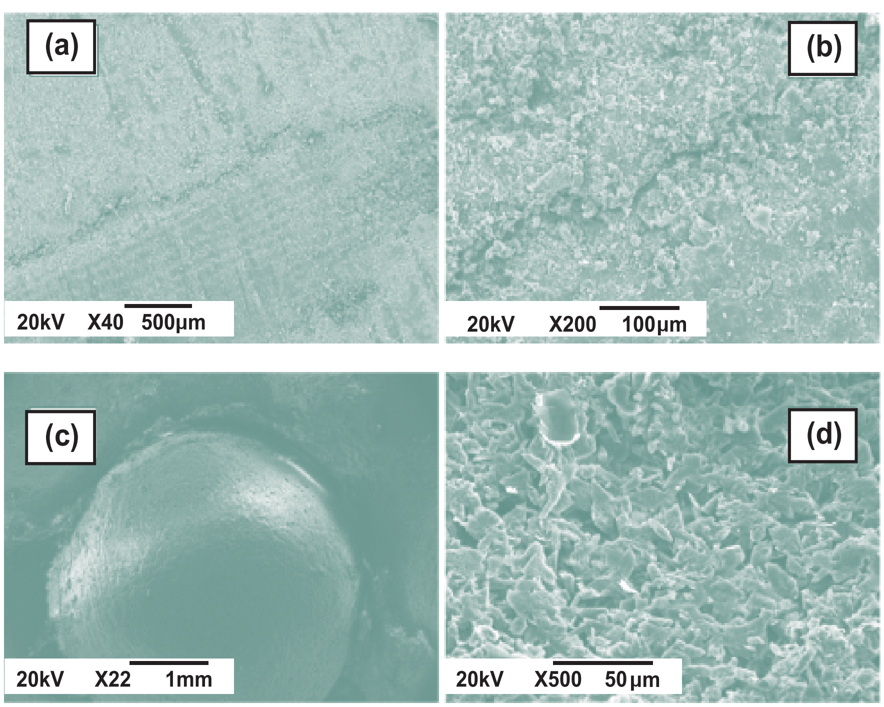

Figure 4. SEM of ITZ for rice husk: (a), (b) and ITZ for the PE (c), (d).
Source: Authors

Conclusions

Based on the experimental results is concluded:

• The reinforcement with polystyrene particles produces a significant lightening effect in the matrix of gypsum. However, there is a decrease in the mechanical properties due not only to the induced porosity also to the poor adhesion of the polystyrene in the gypsum matrix.

• The reinforcement with rice husk fibers improves the mechanical strength and the toughness. Test results of pull-out and the length critical showed that during fissuration the main cause of the increase of the absorbed energy is the detachment phenomenon and pull out of the fibers.

• It is possible to obtain lightweight gypsum base composites with improved mechanical properties and resistance to cracking, by their reinforcement with rice husk fibers.

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