

## ASSESSMENT OF COMPRESSION RESISTANCE OF NATURAL FIBERS – REINFORCED GYPSUM COMPOSITES

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### ABSTRACT

*The objective of the present work was to obtain gypsum reinforced with natural fibers. Gypsum reinforced matrices were prepared from the admixtures of sugarcane bagasse, sugarcane straw and classified pulp of bagasse, at 1, 2, 5, 10 and 20 wt.% concentrations. Water to gypsum ratio was 0.7 for all preparations but 0.4 and 0.7 for pure gypsum. The reinforcement effect by natural fibers to the gypsum was evaluated by compression tests in an Universal Testing Machine, flexural essays in a Dynamic Mechanical Analyzer (DMA) and microstructure assessment by using Scanning Electron Microscope (SEM). Best results were obtained at 1 and 2 wt.% fiber levels. Elastic modulus was more significant for the sample to exhibit a better distribution and compaction between fibers and gypsum.*

**Keywords:** composites; fibers; gypsum; mechanical properties

### 1. INTRODUCTION

In the past few decades, gypsum has been used as finishing material for walls and ceilings in many countries. Its excellent performance, attractive appearance,

easy application, and its healthful contribution to living conditions have made gypsum a most popular finishing material for these applications <sup>(1)</sup>. In addition, the availability in subsoil, relative low cost, easy handling and mechanical characteristics suitable for different applications, makes the gypsum a widely used construction material <sup>(2)</sup>.

However, gypsum presents some undesirable characteristics, as heaviness, brittleness and permeability, impeding exterior application. <sup>(2)</sup>. Heaviness and brittleness may be appreciably reduced by combining gypsum with mineral particles or natural fibers <sup>(2, 3)</sup>.

Nowadays, research for new materials area is very intense. The competition in the industry, seeking materials that meet both socioeconomic expectations and environment preservation drives toward the use of vegetable fibers. The agricultural-industrial residues are generated in a great amount and the proposal of their use, for example, bagasse (that can be considered a by-product), for use in energy generation and sugarcane straw, to reinforce of materials, is of fundamental relevance <sup>(4)</sup>. Bagasse is a complex material that consists of approximately 50% cellulose, 25% polyose and 25% lignin <sup>(5)</sup>.

Gypsum-base composites have more and more extensive application in the development of new-type wall body materials. A lot of companies in other countries have put out new-type building materials that compound gypsum with wood shaving, and have successfully developed several kinds of production equipment. This kind of composite has the following merits: low density, good properties of insulating against heat and sound and high rate of strength/density make it suitable to be used as raw material of light parting wall <sup>(6)</sup>. Thus, investigations of several types of fibers have been aimed to improve the mechanical properties of gypsum <sup>(2, 7)</sup>.

The objective of the present work was the preparation of gypsum reinforced with natural fibers. Fibers / gypsum composites were evaluated mechanically by compression, DMA and SEM techniques.

## **2. MATERIALS AND METHODS**

The gypsum was kindly provided by Imagens Bahia Ltda, located in the municipal district of Ferraz de Vasconcelos, in São Paulo State. Humidity content of gypsum was 1%.

## 2.1 Sugarcane Bagasse and Straw

Sugarcane bagasse and straw were kindly supplied by Usina Ester, from Cosmópolis, SP, Brazil. Chemical composition of fibers is given in Table 1. Straw and sugarcane bagasse were triturated and particles of 5-20mm length and 0.5- 3mm diameter were used as gypsum reinforcement. Pulps of sugarcane bagasse were also used.

## 2.2 Chemical Analysis of Sugarcane Bagasse, Straw and Pulp

The modified Klason method was utilized to obtain a chemical composition <sup>(8)</sup>. Samples of 1 g of sugarcane bagasse, straw and pulp were treated with 5mL H<sub>2</sub>SO<sub>4</sub>, 72%. After 7 min stirring at 45°C, 25 mL of water were added to the mixture, which was posthydrolyzed under 1.05 bars for 30 min. The product was filtered and the insoluble portion (Klason lignin) quantified by weighing. The hydrolysate was acidified to 1 – 3 pH range, filtered in a Sep-Pak C<sub>18</sub> cartridge and analyzed by high-performance liquid chromatography in a Shimadzu LC10 chromatograph, using an Aminex HPX-87H column at 45°C. Mobile phase was H<sub>2</sub>SO<sub>4</sub> 0.005 mol.L<sup>-1</sup> at 0.6mL.min<sup>-1</sup>. Products were determined by refractive index and quantified by using calibration curves <sup>(9)</sup>. Soluble lignin was determined using the absorption at 280 nm of alkaline solutions obtained from the hydrolysate <sup>(10)</sup>.

## 2.3 Soda/AQ Pulping

The pulps of bagasse were obtained through the process of pulping alkaline soda/anthraquinone. Pulping conditions were NaOH 20.64 (w/v) or (16% of Na<sub>2</sub>O), and liquor: bagasse ratio of 6:1 (v/w) and liquor: straw 7.5:1 (v/m) and 0.15% AQ. Soda cooking was performed in a 10-L stainless steel reactor at 170°C. The time of heating up to 170°C previously determined was of 1.5 h, resulting in a total process time of 3.5 h. Obtained pulps were washed up to 7.0 pH and filtered up to total liquor removal. Pulps were air-dried <sup>(4, 11)</sup>.

#### 2.4 Classification of the Bagasse Sugarcane Pulps

The pulp samples were disaggregated (consistency of approximately 0.3%) in MA - 1032 equipment. The pulp suspension was added in a classifier of the type Somerville with rift of 0.15 mm. The classified pulps were filtered using a Buchner funnel and separated in samples forming disks to facilitate the drying at room temperature.

#### 2.5 Preparation of Reinforced Gypsum Composites

Reinforced gypsum composites were prepared by mixing fiber and gypsum at 1, 2, 10 and 20 wt.% of fiber for 99, 98, 90 and 80 wt.% of gypsum concentrations. Mixtures were accomplished in becker of 1000 mL. Water to gypsum ratio was 0.7 for all mixtures, except for pure gypsum: 0.4 and 0.7 ratios.

#### 2.6 Molds for the Compression Tests

In order to obtain molds, PVC tubes with 28.5 mm diameter and 49.5 mm height dimensions were used <sup>(12)</sup>. Each tube was cut in longitudinal direction in order to facilitate samples removal after drying them at 25°C, for 24h.

#### 2.7 Mechanical Characterization

Samples were submitted to mechanical essays, compression type, by using "Instron" Universal-Testing Machine mod. 4400R, installed in the CCTM laboratory. It was used a 10 ton load cell and the test was run at a 2 mm/min speed. Five specimens for each sample were tested, as per defined dimensions mentioned in section 2.6.

#### 2.8 Dynamic Mechanical Analysis (DMA)

Rectangular shape specimens exhibiting 60 x 13 x 3 mm<sup>3</sup> volume were used for dynamic mechanical experiments. Dynamic mechanical thermal analyzer from DMA

242 Netzsch was used for evaluating the dynamic modulus. Properties were measured at a temperature of 25°C. Tests were carried out at 1Hz frequency.

### 2.9 Scanning Electron Microscopy (SEM)

The microstructures of the pure and reinforced gypsum with 1 wt.% of fibers were investigated by using Scanning Electron Microscope, SEM (Phips XL Serie) in CCTM laboratory. The specimens were previously coated with gold. The magnitude used was 4000X.

## 3. RESULTS AND DISCUSSION

### 3.1 Chemical analysis

Chemical analyses of sugarcane bagasse, straw and pulp are useful for the determination of components. Quantification procedures are based on the hydrolysis of samples in accordance with Klason standard methods: the quantification of lignin followed by the quantification of sugars and derivatives <sup>(9)</sup>.

Table 1. Chemical composition of the fibers

Components	<i>in natura</i> bagasse(%)	Non- classified pulps (%)	Classified pulps (%)	<i>in natura</i> straw (%)
Cellulose	43.8	83.1	88.1	33.3
Polyoses	28.6	4.9	4.0	27.4
Klason lignin	23.4	2.7	2.0	21.3
Soluble lignin (%)	0.1	5.7	4.0	4.8
Ash (%)	2.8	1.1	0.8	2.6
Other*	2.8	2.5	1.1	10.6
Total	97.2	97.5	98.9	89.4

\*Presence of other components not determined by analysis carried out.

Table 1 shows chemical composition differences for *in natura* bagasse, non-classified and classified pulps of bagasse and *in natura* straw. It can be observed that the straw presents a lower cellulose percentage in composition (33.3%). It also shows a reduction of 86% in the polyoses content for obtained pulps compared to the

*in natura* bagasse and an increase of 50% of cellulose content. The difference in the chemical composition of the classified pulp compared to the non-classified was due to the material that was not disaggregated during the pulping process.

### 3.2 Compression Tests

Compression test results for pure gypsum and fibers / gypsum composites are shown in Table 2.

Table 2. Mechanical strength of pure gypsum and fibers/gypsum composites

Pure gypsum and (fibers*/gypsum composites)	W	Compressive strength (MPa)	Stress (MPa)
100% gypsum	0.4	5.60 ± 0,46	5.75 ± 0.04
100% gypsum	0.7	2.79 ± 0.48	2.97 ± 0.55
1% sugarcane bagasse	0.7	7.60 ± 0.17	8.00 ± 0.19
1% classified pulp	0.7	8.84 ± 0.59	4.35 ± 0.75
1% straw	0.7	4.20 ± 0.40	6.68 ± 0.07
2% sugarcane bagasse	0.7	6.35 ± 0.33	6.73 ± 0.07
2% classified	0.7	7.89 ± 0.29	8.24 ± 0.36
2% straw	0.7	5.90 ± 0.25	4.52 ± 0.08
10% sugarcane bagasse	0.7	3.55 ± 0.25	3.70 ± 0.23
10% classified pulp	0.7	7.74 ± 0.34	6.79 ± 0.02
10% straw	0.7	2.79 ± 0.23	1.17 ± 0.07
20% bagasse	0.7	1.74 ± 0.06	1.18 ± 0.07
20% classified pulp	0.7	0.38 ± 0.03	0.42 ± 0.01
20% straw	0.7	2.47 ± 0.27	2.67 ± 0.30

(\*) wt% of reinforcement  
W=water / dry materials ratio

Table 2 indicates that a 43% raise of water content in the pure gypsum sample resulted in a decrease of 50% for the compressive strength. Other authors verified that gypsum characteristics were superior when water / dry materials ratio was of  $w=0.4$ , when compared to a  $w=0.8$  ratio. Mechanical properties were dramatically affected by water <sup>(13)</sup>.

Figure 1 presents results for compression modulus of fibers / gypsum composites as a function of fibers percentage.

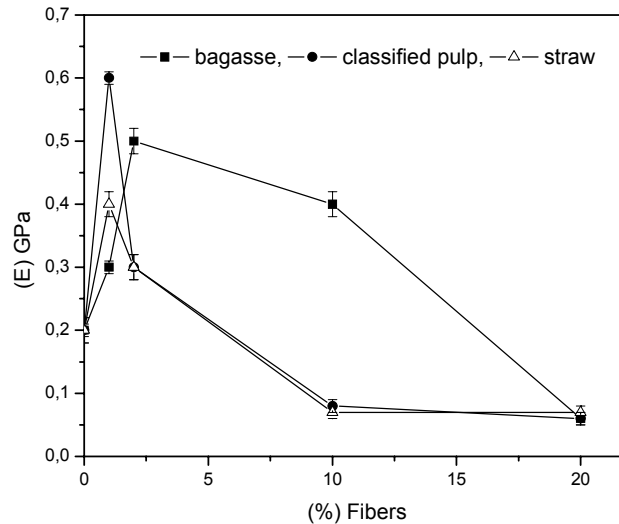


Figure 1. Results of compression modulus of fibers/ gypsum composites as a function of the fiber percentage.

Figure 1 results showed that 1 wt.% of bagasse and straw contents contributed for improvement of the mechanical modulus in the composites. It was also observed that the addition of 10 and 20 wt.% fibers to the matrix contributed for decreasing the modulus.

### 3.3 Dynamic Mechanical Analysis (DMA)

The reinforcement effect imparted by natural fibers to the gypsum was also evaluated by DMA. Figure 2 presents results of elastic modulus of fibers/ gypsum composites as a function of fiber percentage. The elastic modulus values found for reinforced gypsum with 1 wt.% of bagasse and straw were 6.6 GPa and 7.1 GPa, respectively. For pure gypsum and gypsum reinforced with 1% of classified pulps of bagasse, the result obtained was 5.1 GPa.

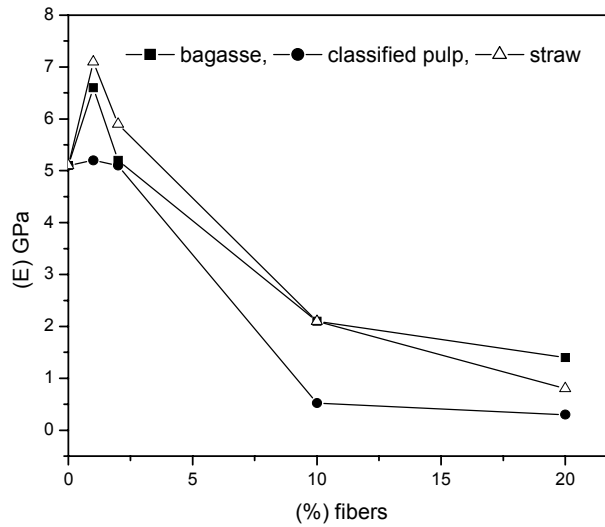
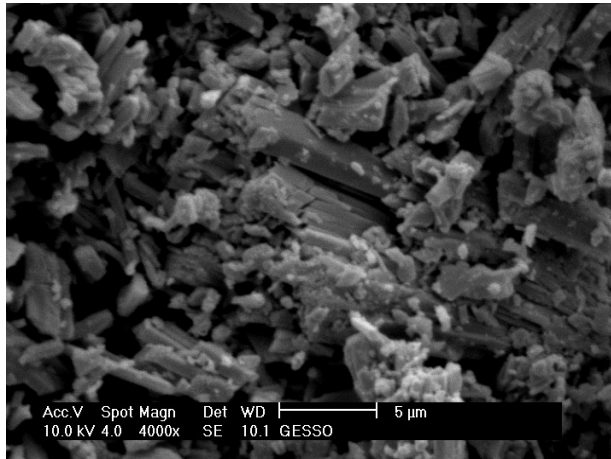


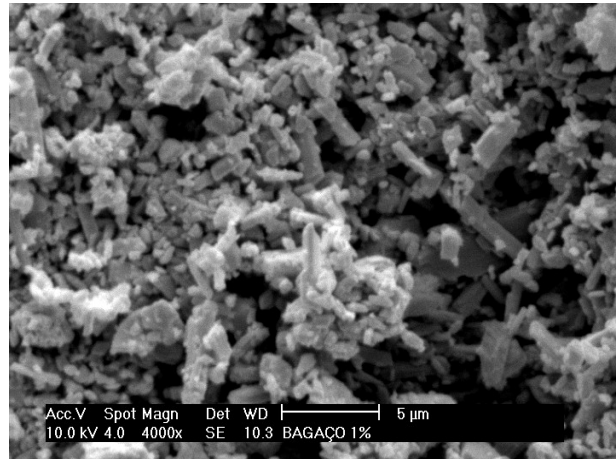
Figure 2. Elastic modulus results for fibers/ gypsum composites as a function of fiber percentage.

### 3.4 Scanning electron microscopy (SEM)

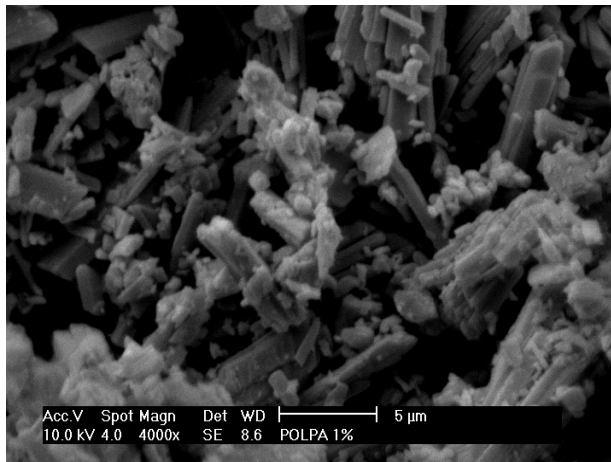
The scanning electron microscopy micrographs (Figure 3) show the pure and reinforced gypsum samples. It can be observed that the fibers are randomly dispersed and not strongly bonded to gypsum.



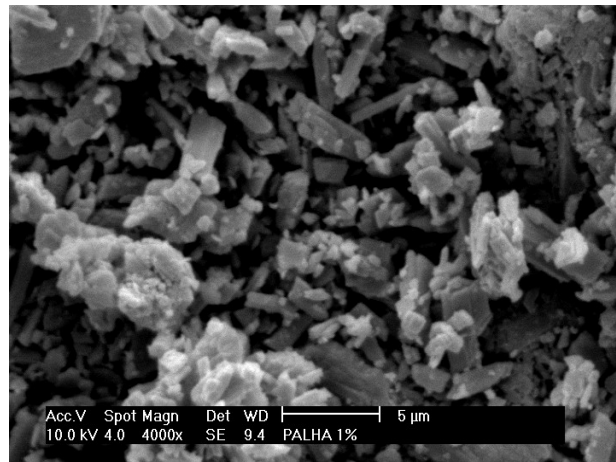
(a)



(b)



(b)



(c)

Figure 3. SEM photographs of (a) pure gypsum, (b) mixture (1 wt.% of bagasse with 99 wt.% of gypsum), (c) mixture (1 wt.% of classified pulp bagasse and 99 wt.% of gypsum) and (d) mixture (20 wt.% bagasse and 80 wt.% of gypsum).

Figures 3 (b) and (d) show a better compacting of the samples with 1 wt.% of bagasse and straw fibers with the gypsum, than the pure gypsum and classified pulp samples of the Figures 3 (a) the and (c). Those more compacted samples showed a larger elastic modulus. The mechanical properties can be improved with the matrix and fibers mixture in a compact and well distributed way <sup>(6)</sup>. Interfaces play an important role in physical and mechanical properties of composites <sup>(13)</sup>. It will be required further fiber modifications, in order to produce a higher and more effective fiber adhesion to the matrix <sup>(14)</sup>.

#### 4. CONCLUSIONS

Fibers/gypsum composites were evaluated by compression test, DMA and compared to SEM. Best results were obtained when 1 and 2 wt.% of fibers were used as reinforcement. The modulus was larger for the sample where the fibers and gypsum were more compacted. Mechanical properties can be affected by the water amount used in the gypsum preparation. A study of the interfacial mechanism of adhesion fiber/gypsum will allow a better understanding of composites properties. The application of a resin can improve the required performance of reinforced gypsum composites.

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