THE INFLUENCE OF THE ADDITION OF NATURAL GYPSUM ON THE MICROSTRUCTURE OF THE SET PLASTER: STUDY OF THE MECHANICAL AND MICROSTRUCTURE PROPERTIES

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Abstract. *Plaster is a brittle material, which possesses a good compression resistance and low flexural and impact resistance. It is known that the mechanical characteristics of set plaster strongly depend on several factors, such as the nature of initial materials (α- and β-hemihydrate) and the preparation process: consistency (water/dry material mass ratio), and the presence of additives. To improve the mechanical properties of set plaster, particulates, fibers and additives are added. In this work, evaluations of the mechanical properties were conducted on set plaster, prepared from α- and β-hemihydrates with the addition of different amounts of gypsum powder. A total of 300 samples of set plaster blocks were prepared adding powdered gypsum up to 70% in mass. The consistency varied from 0.4 to 0.75. The compression and flexural tests were conducted and correlated with density measurements. As a result, the compressive strength varied from 1 MPa to 12 MPa. The flexural strengh varied from 0.5 MPa to 6 MPa. The density values varied from 0.84 g/cm3 to 1.25 g/cm3. Microstructural investigations were also performed by infrared spectroscopy and electron scanning microscopy.*

Keywords*: Plaster, gypsum particulate, composite, mechanical properties, consistency*

1. Introduction

Gypsum, main raw material of plaster, is characterized by being a fragile material with good compression resistance, but low flexural and impact resistance. The dehydration of gypsum can yield four products, the two forms of hemihydrates (α and β) and the two forms of anidrite (insoluble and soluble) , except only two forms of hemihydrates or plaster find large commercial acceptance (Angeleri at al., 1982).

For the type α plaster, the calcinations occur in autoclave with vapour injection or through gypsum dehydration in aqueous solution under a high temperature. The type β plaster, in turn, is generally produced by the gypsum calcinations at 100°C under atmospheric pressure (Sindugesso, 1983), depending on which purpose the use of the final product will be, the process of type β plaster manufacturing can suffer variations in the calcination temperature or make use of necessary additives.

In crystallographic terms, the two forms of hemihydrates are similar, although they present different morphological characteristics. Beta plaster is formed by irregular and porous crystals, while α plaster presents crystal with regular and prismatic form, permitting thus a greater densification of the crystals. These characteristics of the morphology of the crystals exercise a great influence in the physical properties of the final product, as; the larger the presence of vacuity between the crystals, larger will be the absorption of water during the process of hydration and the formation of set plaster (Angeleri at al., 1983; Satava 1980).

Studies already done, demonstrate that the excess of water in the set plaster leaves the final product with less mechanical resistance due to the greater quantity of pores formed after the evaporation of the water (Singh and Garg 1996; Coquard at al. 1994).

In a general form the mechanical properties of plaster products depend on factors like the nature of the initial material (hemihydrates α or β), of the process of the paste preparation, moulding conditions, the presence of additives and mainly the consistence (relation water/plaster) . (Daluit at al, 1996; Jeulin at al. 2001; Gmough at al. 2000).

The conditions of the hydration process; like temperature, time of mixture, consistency, presence of impurities and of the duration of the set plaster hardening bringing about changes in the crystals format, which in turn, influence the mechanic properties (Antunes, 1999).

The search for lighter and more resistant materials, allied to the great employment of plaster in building construction, influenced research with the use of fillers and fibres that sought to improve the mechanic resistance and reduce the set plaster density (Joshi at al. 1992; Gmoug at al. 2001; Sing and Garg 1993). Light materials, like cork, confer lightness to composite, yet, reduce the mechanical resistance, this deficiency that can be corrected with the addition of glass fibres (Eve at al. 2002). Nevertheless, when introducing additives or elements that seek the structural reinforcement of the plaster, composites of difficult recycling can be produced.

There are studies that seek to improve the mechanic properties through the addition of small grains of gypsum to the pre-moulded, trying to increase the density with the reduction of macro-porosity (Gmouh at al. 2000).

The present work, has as an objective, to study the evolution of the mechanical properties of the several set plaster samples obtained from the pastes formed of plaster α and β, with the addition of different quantities of powdered natural gypsum. Besides the compression and flexural mechanic analysis tests in three areas, a microstructural study using the infrared and electron scanning microscopy was done.

2. Materials and Methods

The materials were supplied by three different manufacturers from the Polo Gesseiro de Peranmbuco, designated by the abbreviations E1, E2, and E3. With these materials proof samples from the mixture of plaster β + gypsum and plaster α + gypsum were made. The E1 company supplied the gypsum and the α and β plasters destined for the application in ceramic moulds. The E2 company supplied the β plaster used for revetment and the E3 company supplied α plaster destined for odontological application.

The raw materials were submitted to the trial of hardening time and the spreading viscosity. The first trial of viscosity used a tube placed on a glass plaque, where concentric circles were marked. The tube (volume of $63cm^3$) was filled completely with the prepared paste and then lifted, permitting the paste to spread onto the glass plaque. The measure of spreading on the glass plaque is an indicator of viscosity. In this work the consistencies water / plaster (a/g) between 0.75 and 1 for the mixtures of the β plaster and of 0.5 and 0.7 for the α plaster were used. These consistencies were adjusted in order to maintain a good workability, as indicated in Table 1.

Table 1 – Time of Preparation of Plaster/Gypsum Paste

Fractions (%) of gypsum / plaster	Plaster type β – Gipsum					Plaster Type α – Gypsum				
	Cons. (a/g) E1	Cons. (a/g) E2	T. Pow. (s)	T. Res. (s)	T. Agit. (s)	Cons. (a/g) E1	Cons. (a/g) E3	T. Pow. (s)	T. Res. (s)	T. Agit. (s)
0-100	0.75	0.75	60	120	60	0,5	0.5	60	60	60
10-90	1	0.85	15	45	30	0,6	0.5	60	60	60
20-80	1	0.9	15	30	15	0,6	0.5	60	30	30
30-70	1	0.9	15	30	15	0,7	0.6	15	15	15
40-60	1	0.9	15	30	15	0,7	0.6	15	15	15
50-50	1	0.9	15	30	15	0,7	0.7	15	15	30
60-40	*	*	*	*	*	0,7	*	15	15	30
70-30	*	*	*	*	*	0,6	*	150	15	30

Note: Cons. = Consistency; T. Pow. = Powdering Time; T. Rep. = Resting Time, T. Agit. = Agitation Time. * the bulk trial in green did not have mechanic resistance

Twenty-five plaster pastes were produced, starting from a mass considered as a reference (100% plaster α orβ), increasing gradually the gypsum paste added in fractions of 10%. The maximum quantity of gypsum that permitted a good mass workability was of 50% in paste for a mixture with β and 70% for those of α plaster. This procedure was adopted for both types of plaster α and of plaster β.

Based on references NBR 12128 (ABNT 1991a) the mass of plaster β were prepared, which consists in the homogenisation of the powder components; powdering upon the water during 1 minute; rest of the suspension for 2 minutes and mixing of the paste during one minute (through a mechanic mixer). However, for the other mixtures alterations were necessary relating powdering, resting, and mixing duration, in relation to what is indicated by the norm, owing to the gypsum acting as a hardening accelerator. The time spent in preparing the pastes with different fractions of plaster/gypsum are indicated in Table 1.

For the confection of the test bulk, used in the compression and flexural trials, moulds of acrylic were used whose manufacturing was done at the Mechanics Engineering Department of the Federal University of Pernambuco, Recife — PE. The dimensions of 40 x 40 x 40mm^{3}were used for the test corpus destined for the trials of compression and of 40 x 40 x 160mm^3 for the test corpus destined for the trials of flexion.

Immediately after the mixture of the pastes, the moulds were filled in two layers to avoid imprisoning air in the paste during the operation. The compactness of the layers and removal of possible imprisoned air bubbles was effectuated manually with spatula blows inside the still fluid paste. After the beginning of the hardening, the mould was levelled with the help of a spatula. For each fraction of plaster / gypsum mixture, six corpus tests were done for the compression trial and six for the flexion trial.

At the end of the exothermic reaction of the plaster paste, whose conclusion was observed approximately 15 minutes after its preparation, the demoulding was done. This duration was sufficient for the bulk test to acquire reasonable resistance to be manipulated. With the intention of simulating the conditions in which the pre-moulded would be subject to, after the demoulding at the factory, the bulk tests were left exposed to the sun, wind and the variations of humidity and the temperature of the environment, as according to the drying process of the pre-moulded in the Araripe Region — PE.

When 24 and 48 hours of confection were completed, the bulk test were weighed in order to observe the water lose and the stability of the mass. The dimensions of each bulk test were measured before and after the drying using a pachymeter with pressure at 0.05mm. Based on the dimensions of dry bulks test the volumes used were calculated using subsequently the density calculus together with the mass obtained after a dehydration of 48 hours. According to the ISO 3051 norm the flexural resistance trial was done[21] and the compression trial in conformity with the NBR 12129 norm (ABNT 1991b).

Right after the mechanics trials, the analysis of infrared spectroscope and electronic microscopic sweep of the blocks of alpha and beta plaster were done with larger or smaller concentrations of gypsum. For the analysis of the infrared pastilles were made (1mg of the sample and 150mg of KBr), placing the powdered mixture in a press and applying a 5 ton load. The pastilles formed, they were submitted to the Bruker – IFS-66 FTIR spectrophotometer, at a resolution of 2cm^{-1}, fascia 4000 to 400cm^{-1}.

Parallelly analysis of the microstructure was done using a JOEL. Electron scanning microscopy, model JSM 5600 I.V.

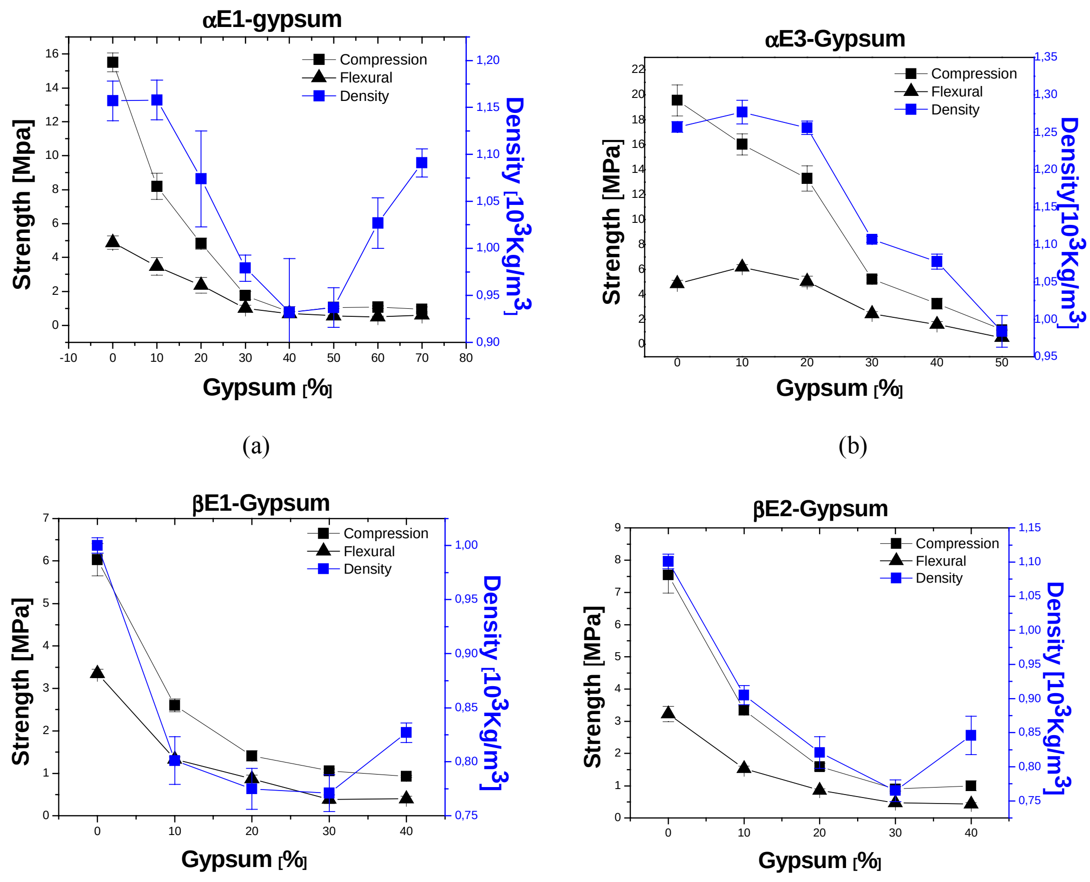
3. Results and Discussions

Figure 1 presents the graphics produced from the results obtained in the compression and flexural testing in three areas for the different α and β set plaster with the addition of gypsum.

The analysis of the results obtained in the mechanical testings of the set plaster taken as reference (0% of gypsum), showed that the values of the compression and flexion tensions for plaster β of company E2 are 64 and 20% superior to those achieved for plaster β by company E1. In the case of plaster α, the material supplied by company E3 presented flexural tension values of about 20% larger than those obtained with the material from company E1. For the tensions of compression the values acquired were 21% larger for company E3. These results can be related with the process of the obtainment of plaster or presence of additives.

The Figure 1a of the α / gypsum plaster mixtures of company E1 show the results of the densities, flexion tensions, and compression due to the percentage of gypsum added to the mixture, revealing a reduction of the values for both tensions as the quantity of gypsum is increased. The values of densities also suffer a reduction up to the mixture with 50% of gypsum, presenting immediately afterwards an increase. Imputing this density increase to the fact that gypsum ends up becoming the component in greater proportion. In the alpha plaster graphic of company E3 (Fig. 1b) a similar behaviour can be verified to the one presented to αE1 in terms of reduction of the mechanic properties with the addition of up to 50% of gypsum.

The two samples of beta plaster had their mechanic properties reduced because of the increase of gypsum, considering that their densities started to increase because of the percentage of gypsum added to values from 40% (Fig. 1c and 1d).

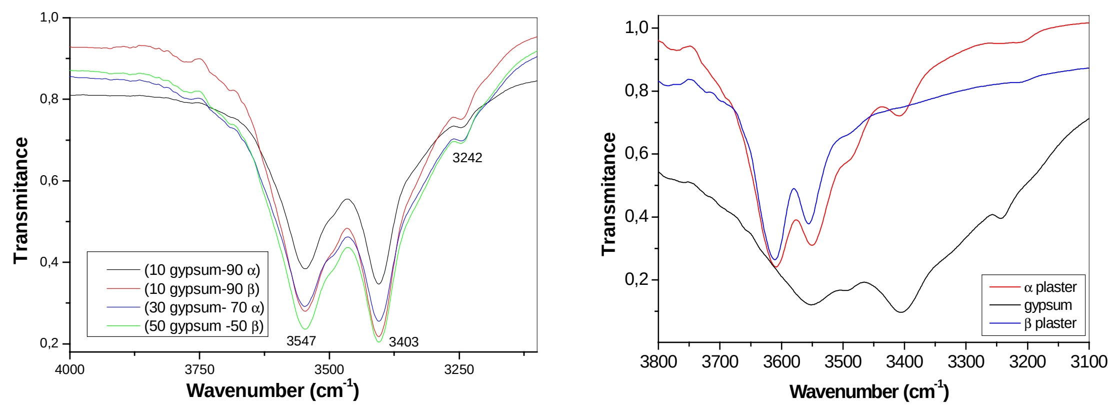


(a) (b)

(c) (d)

Figure 1. The relation between density, flexural strength, and compression strength, due to the addition of gypsum to the plasters αE1, αE3, βE1and βE2

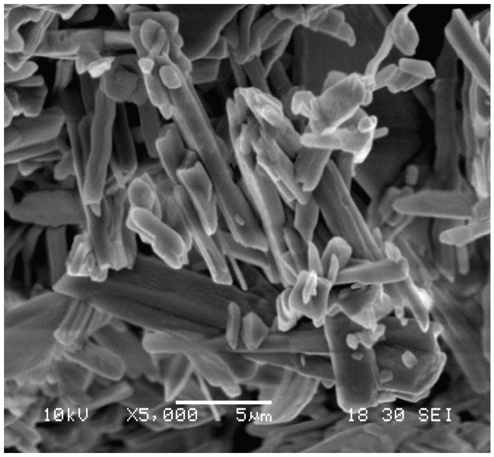
Figure 2a shows the results of the analysis of FTIR in the range of 4000 to 3100cm^{-1}, that is, vibration band from the water. It is observed that the hydrated plaster mixtures α and β showed that the gypsum bands, as already expected, do not present any spectrum variation due to the quantity of gypsum added. In the analysis of the raw materials (Fig. 2b), were observed bands, characteristic of gypsum presented a larger quantity in the spectrum of alpha plaster



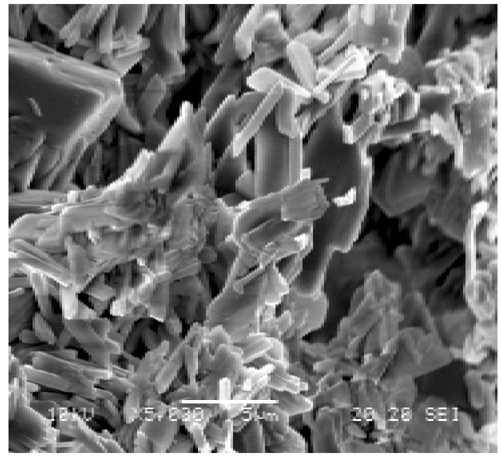
(a) (b)

Figure 2. Infrared spectrums of the alpha/gypsum and beta/gypsum mixtures in different concentrations (a); and of the raw materials (b).

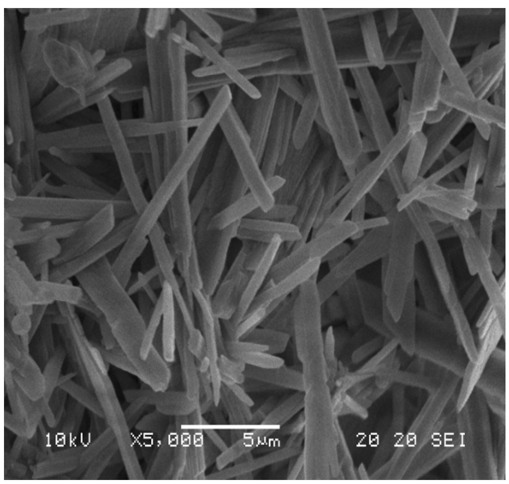
The results obtained through the electron microscope (Fig.3) show that with the increase of gypsum an alteration occurred in the format of the crystals, which became longer and with more irregular formats, which is more evident in the case of plaster β (fig. 3c and 3d). The grains of gypsum act as impurities interfering in the formation of the crystals, in which to obtain structures of irregular formats that do not present good compactness that promotes the reduction of the mechanic properties.



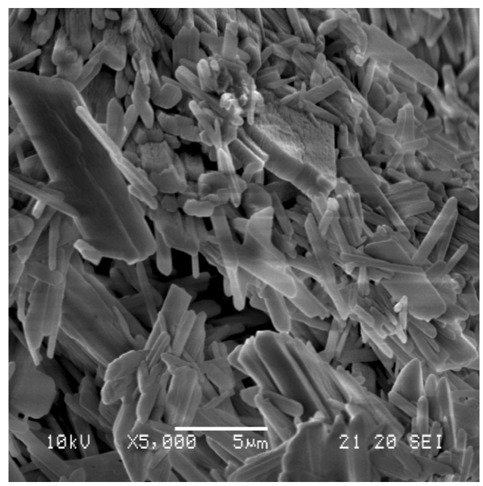
(a)



(b)



(c)



(d)

Figure 3. Microstructures of plaster α with 10% (a) and 70% (b) of gypsum, and of plaster β with 10% (c) and 50% (d) of gypsum added.

4. Conclusions

The addition of gypsum to plaster causes a reduction in the density of the final product accompanied by a reduction of the mechanic properties. The initial addition of 10% of gypsum promotes a larger reduction in the compression and flexural tensions of the beta plaster than in the alpha plaster. From a certain quantity of gypsum added to the plaster paste an increase in the density of the final product occurs, without however being sufficient to elevate the mechanic resistance.

The fact that gypsum acts as an accelerator of hardening it causes besides the reduction of mechanical properties, modifications in the morphology of the crystals, which start to acquire irregular formats that make the compacting difficult demanding in this way, a greater quantity of hydration water.

The analysis with infrared spectroscopy did not show great differences among the paste spectrums with different concentrations of gypsum. Yet the analysis of raw material in the region of the OH group of stretched band showed bands of gypsum present in larger quantity in alpha plaster, indicating an incomplete calcination of the raw material.

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