Pesquisa e Ensino em Ciências Exatas e da Natureza
4: e1516 (2020)
Research and Teaching in Exact and Natural Sciences

ARTICLE

ISSN 2526-8236 (online edition)
Pesquisa e Ensino em Ciências Exatas e da Natureza

UFCG - Campus de Cajazeiras - PB.

# Investigative study of the potentialities of noble applications for the Brazilian calcium sulfate α-hemihydrate

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Lira M.C.A. & Neiva L.S. (2020) Investigative study of the potentialities of noble applications for the Brazilian calcium sulfate α-hemihydrate. *Pesquisa e Ensino em Ciências Exatas e da Natureza*, 4: e1516. http://dx.doi.org/10.29215/pecen.v4i0.1516

**Academic editor**: Fernando Antônio Portela da Cunha. **Received**: 28 September 2020. **Accepted**: 06 October 2020. **Published**: 07 October 2020.

**Estudo investigativo das potencialidades de aplicações nobres para o hemihidrato de sulfato de cálcio brasileiro do tipo**

**Resumo**: O sulfato de cálcio hemiidratado (comercialmente conhecido como gesso) é obtido através da desidratação parcial do minério gipsita. A depender das condições termodinâmicas de calcinação do minério podem ser obtidas diferentes variações do hemihidrato. O objetivo deste trabalho é realizar um estudo investigativo acerca das possibilidades de aplicações nobres para o hemihidrato tipo α. Para isto, foi analisada uma amostra do gesso α proveniente do estado de Pernambuco, Brasil, por meio de técnicas de caracterizações estruturais e químicas. De acordo com os resultados, a amostra apresentou composição majoritariamente a base de sulfato de cálcio associado à água, com teor de pureza igual a 98.79%. As partículas apresentaram tamanho médio, aproximadamente uniforme, em torno de 60 μm. A concentração insignificante de contaminantes detectados na amostra torna o hemihidrato α adequado para aplicações nobres como, por exemplo, na área da saúde dentre outras aplicações que impliquem em contato literal com organismos vivos.

**Palavras chave**: α-Hemihidrato de sulfato de cálcio, gesso alfa, investigação de potencialidades, caracterizações estruturais.

**Abstract**: Calcium sulfate hemihydrate (commercially known as gypsum or plaster) is obtained through partial dehydration of the gypsum crude ore. Depending on the thermodynamic conditions of calcination of the ore, different variations of the hemihydrate can be obtained. The aim of this work is to carry out an investigative study on the possibilities of noble applications for α-type calcium sulfate hemihydrate. For this, a sample of α plaster from state of Pernambuco, in Brazil, was analyzed using structural and chemical characterization techniques. According to the results, the sample presented a composition mainly based on calcium sulfate associated with water, with a purity content equal to 98.79%. The particles had an average size, approximately uniform, around 60 μm. The insignificant concentration of contaminants detected in the sample indicates that the α plaster sample analyzed can be considered suitable for noble applications such as, in the health area, among other applications that imply literal contact with living organisms.

**Key words**: Calcium Sulfate α-Hemihydrate, alpha plaster, investigation of potentialities, structural characterizations.

## Introduction

Gypsum or plaster is a calcium sulfate hemihydrated that has a chemical formula $CaSO_4.0.5H_2O$. It uses a single raw material for its production, the gypsum crude ore (calcium sulfate dihydrate – $CaSO_4.2H_2O$) (Ferreira *et al.* 2019). For Tritschler (2015) *apud* Gurgul *et al.* (2019), depending on the thermodynamic conditions of the calcination of the gypsum crude ore,

the hemihydrates obtained can be presented in two main variants, namely α and β. Currently, there are several calcination processes for the production of comercial plaster, basically distinguishing into two categories: (1) calcination at atmospheric pressure for the production of β type plaster and (2) calcination at high pressure to produce α type plaster (Soares 2005).

In general, the gypsum crude ore calcination product is a very important inorganic material for the industry, given its wide range of applications that range from civil construction, to medicine, automobile segments, cosmetics and ceramics industries (Lira 2018). Chemically and crystallographically, there are not differences between the α and β types of plaster, however due to the conformation and size of the crystals the specific surface presented by α plaster is smaller in relation to β. The α type plaster acquires a consistency with less mixing water and produces parts with less porosity and superior mechanical resistance to flexion (Abreu 2005).

Calcium sulfate hemihydrate is produced in great abundance in the region of Araripe in the state of Pernambuco, northeastern Brazil, and presents degrees of purity that vary between 80 and 95%. The gypsum crude ore can be present in nature under several mineralogical species, the most well-known are: Cocadinha, Estrelinha, Rapadura, Johnson among others, which vary basically between themselves in relation to the purity content (Barbosa *et al*. 2014).

The calcium sulfate α-hemihydrate is widely recognized in the literature as a high value-added cementitious material due to the ease that this material presents for molding processes in addition to presenting high mechanical strength and favorable biocompatibility (Jiang et al. 2016; Singh & Middendorf 2007 *apud* Ma *et al*. 2018). For Kwon *et al*. (2016), plaster is an almost exclusive material for the development of dental models for prosthetic and orthodontic rehabilitation or for the analysis of temple and jaw joints. Following this line of applications in the health area, case studies involving the use of plaster as an inorganic material associated with special therapy techniques aimed at correcting anatomical deformities in the treatment of orthopedic injuries, such as pseudoarthrosis, can also be mentioned, for bone grafts and for making a provisional nasal prosthesis for the treatment of recovery from a geriatric oncological rhinectomy (Rosen *et al*. 2019; Chang *et al*. 2020; Chen *et al*. 2020; Zhao *et al*. 2020).

Research reports describing or ratifying the well-known antipyretic activity of gypsum crude ore, not calcined, when applied as a paste to the skin of a fevered person are not uncommon in the literature and, due to this property, this inorganic material has been widely used with satisfactory performance in the treatment of mild symptoms such as fever in people infected with the new coronavirus (Shahrajabian *et al*. 2020; Wang *et al*. 2020; Zhou *et al*. 2020)

Aligned with this context, the aim of this work is to perform structural and chemical characterization in α type sample of calcium sulfate hemihydrate, belonging to Johnson mineralogical classification, from Plasterer Polo of Araripe, located in state of Pernambuco, in Brazil. The driving motivation for carrying out this work consists of investigating, in a speculative way, the potential of noble applications or distinct from the trivial application in civil construction, for the material object of study of this research from the results of its characterizations.

## Material and Methods

The α-type sample of calcium sulfate hemihydrate, also known commercially as α-type plaster, analyzed in this work comes from the large gypsum crude ore deposits located in the mineral reserves belonging to Chapada do Araripe. Specifically, the sample analyzed in this work comes from the reserves that are located in state of Pernambuco, in Brazil. The sample studied in this work was converted from its crude form of gypsum ore, in mineralogical variety classified as Johnson, into calcium sulfate α-type hemihydrate by means of industrial calcination processing under water vapor pressure in autoclave oven with a temperature at 140°C. The processing of conversion of gypsum crude ore into α-type hemihydrate was carried out in the industrial facilities of one of the companies that make up the industrial complex known as Plasterer Polo of Araripe, which provided a sample of the material ready to be marketed. Henceforth, the sample calcium sulfate α-type hemihydrate, which was received and analyzed in

this work, will be called only α plaster. The plaster sample α was then prepared for characterization by sieving in a 200 # mesh, 44 μm aperture, and the sieving product was then submitted to characterizations analysis. The following is a brief summary of the characterization analyzes to which the α plaster sample was submitted. X-ray diffraction analysis was applied in order to identify the phases present in the sample structure of the material, in order to quantify its purity. This analysis was then performed on a Shimadzu diffractometer, model XRD 7000, which is equipped with the following parameters: copper target; 40 kV voltage and 30 mA current. A characterization analysis via X-ray fluorescence - XRF - was performed in order to identify the qualitative and semi-quantitative chemical composition of the α plaster sample. This XRF analysis was performed on a dispersive energy X-ray fluorescence spectrometer, brand Shimadzu, model EDX 7000/8000. Morphological aspects of the unitary particles and their agglomerates that make up the α plaster sample were observed using the scanning electron microscopy technique - SEM. This analysis was performed using a scanning electron microscope from the manufacturer TESCAN and identification by veg3, operated at 20 kV. The observation of the behavior of the α plaster sample with regard to its mass loss capacity, under heating conditions, was performed using the characterization technique known as thermogravimetry; for this, was used a Shimadzu thermal analyzer, model TGAA-50/50H using the following parameters: dynamic air atmosphere of 50 $mL.min^{-1}$ and heating rate of $15°C.min^{-1}$.

Finally, Fourier transform infrared (FT-IR) spectra obtained in the range between 450 and 4000 cm-1, in the transmittance mode, were plotted using KBr pellets as a reference in a Perkin Elmer Spectrum Two spectrophotometer. This analysis aims to ratify, corroborate or detail information collected in previous analyzes of structural characterizations such as x-ray diffraction and x-ray fluorescence.

## Results and Discussion

### X-ray Diffraction

**Figure 1** depicts the α plaster analyzed sample XRD patterns. This analysis was carried out in order to identify and quantify the constituent phases of the sample structure studied in this work. From the data collected in this analysis, the phases identification was performed using Malvern Panalytical's X'Pert HighScore Plus Software and the ICDD database (International Centre for Diffraction Data).

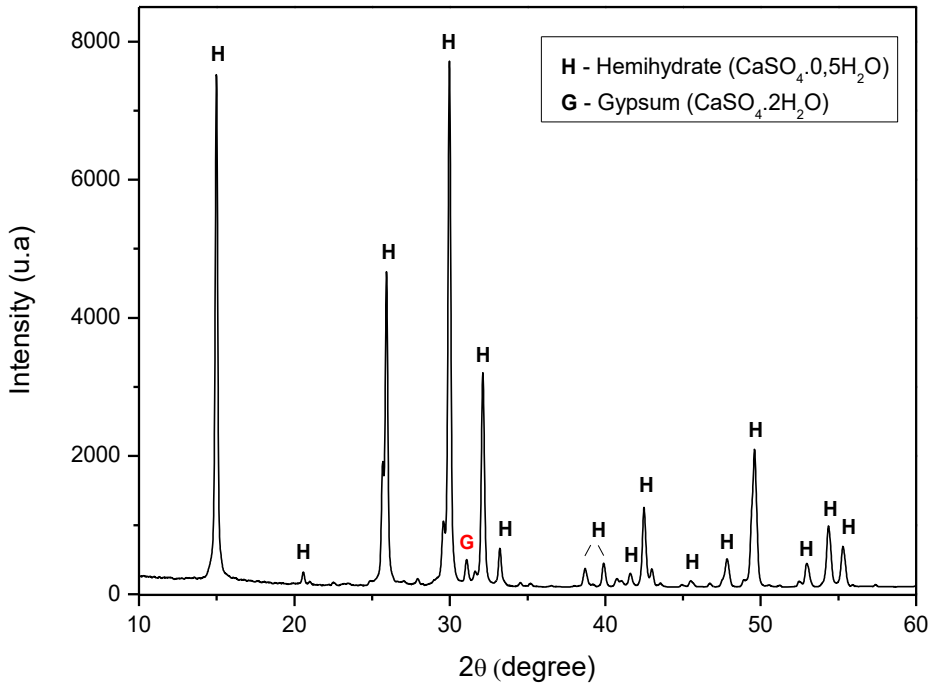


**Figure 1**. X-ray diffraction patterns for α plaster analyzed sample.

When observing at the x-ray patterns, depicts by **Figure 1**, the pronounced degree of purity of the α plaster sample is evident; i.e., the graphical profile of the x-ray patterns shows that the sample in question is essentially composed on Calcium Sulfate Hemihydrate (Card ICDD 00-033-0310), because only the high intensity peaks characteristic of that (H) phase were identified ($CaSO_4.0.5H_2O$). The high intensity peaks characteristic of the phase already mentioned, were detected at 2θ = 14.75°, 25.65° and 29.69° associated respectively to (1 0 1), (3 0 1) and (4 0 0) crystallographic planes. It is also possible to observe the brief occurrence of a low intensity peak at 2θ = 31.121°, referring to the gypsum phase (G) (Card ICDD 00-021-0816), this being associated with the (2 0 0) crystallographic plane. According to Oliveira & Torres (2015), the appearance of the characteristic peak of gypsum can be associated with two factors: (i) deficiency in calcination or (ii) hydration in storage.

In addition to the non-detection of spurious phases in the crystalline structure of the α plaster sample the high purity of this sample was ratified by the absence of the anhydrite phase, or anhydrous calcium sulfate – $CaSO_4$, since this phase is the most common contaminant found in calcination products of the gypsum crude ore to obtain plaster, according to Baltar et al. (2008).

## X-Ray Fluorescence

The result of the analysis of the qualitative and semi-quantitative chemical composition, performed using XRF technique, for the α plaster sample, is describes below in **Table 1**.

According to describes data in **Table 1**, it appears that the composition of the α plaster sample is constituted by expressive levels of calcium oxide (CaO) and sulfuric anhydrous ($SO_3$), totaling 98.79% of the total composition. Despite the detection of the presence of contaminating oxides in the sample, it is possible to observe that, in general, the concentration of these contaminants is very low, limited to a total of 1.2%. The insignificant content of the concentration of each contaminating oxide, individually, may justify the fact that themselves were not detected in XRD analysis.

**Table 1**. Chemical composition obtained by X-ray fluorescence for the α plaster sample.

| Analyte | CaO | $SO_3$ | $Al_2O_3$ | $SiO_2$ | SrO | $Fe_2O_3$ | $TiO_2$ |
|---|---|---|---|---|---|---|---|
| Content (wt %) | 54.109 | 44.687 | 0.473 | 0.458 | 0.175 | 0.084 | 0.014 |

The most classic or trivial application for plaster is in the construction industry, where a material with an expressive purity content is not strictly required as discussed in the research work carried out by Marvila *et al.* (2020), where the feasibility of using a plaster coating was analyzed, in historic buildings in a city of the state of Rio de Janeiro, containing residues of differents rock materials as a way of reducing the accumulation of solid urban waste. Another example of successful application in civil construction, which also does not require composition with an expressive degree of purity, is the case of the work developed by Buggakupta *et al.* (2020) which employed waste from automotive glass shards and plaster mold hulls used in steel industries as inputs for the manufacture of plaster-based mortars. The result of this research was very positive from an environmental point of view as well as technical and economic feasibility. However, the high purity content of the α plaster sample, object of study of this work, adds a lot of value to this material and makes it very attractive for noble applications such as in the health area, among other applications that directly imply a literal contact with living organisms, for which non-toxic materials are usually required. Therefore, it is worth mentioning that the expressive purity content of the α plaster sample analyzed in this work proved to be superior when compared with the α-type hemihydrate sample successfully synthesized by a group of Vietnamese researchers from of calcium sulfate dihydrate crude ore calcined under high pressure. In this work taken as reference, the α-type hemihydrated sample obtained proved with 98.62% purity degree and in the research in question, the same sample was submitted to

biocompatibility tests for application in bone graft. As conclusion, the referenced research found that the analyzed α-type hemihydrate sample can be considered a promising substitute for the synthetic materials currently used for bone grafts in orthopedic and maxillofacial surgeries (Ngoc Le *et al.* 2020).

## Scanning Electron Microscopy (SEM)

The micrographs obtained as result of the morphological analysis, performed by scanning electron microscopy (SEM) technique, in the analyzed α plaster sample, in powder form, are illustrated in **Figure 2** with 500x and 2000x magnification.

From the observation of the micrographs, it is possible to see that the α plaster sample is formed by a cluster of unitary particles joined most likely by low intensity bonding forces, probably of the Van der Walls type, given the easily of de-agglomeration of this material, analyzed in powder form. The morphological aspect of the unitary particles and their agglomerates that make up the sample, shown in the micrographs, are mostly presented with an elongated, fine physical shape and with approximately uniform lengths, around 60 μm (**Figure 2A**). **Figure 2B**, in which the α plaster sample image is displayed with greater magnification, it is possible to notice that the unitary particles as well as their agglomerates present with irregular and inhomogeneous surface aspects.

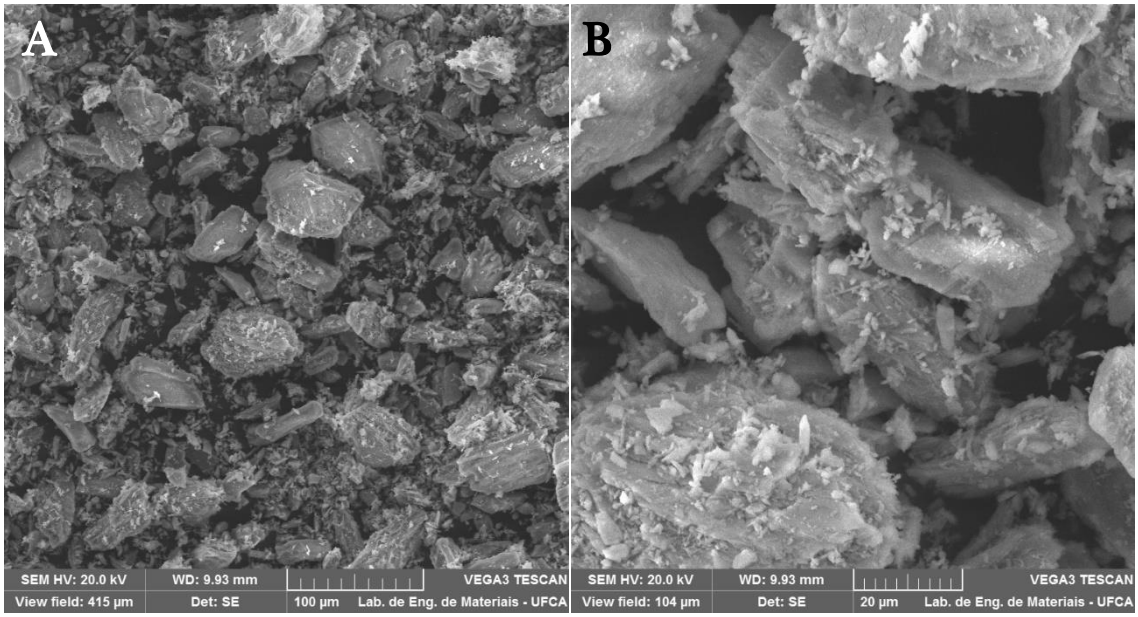


**Figure 2**. SEM micrographs of the morphology of analyzed α plaster sample in powder form: **A**. 500x magnification; **B**. 2000x magnification.

Interestingly, in a research conducted by Guan *et al.* (2020) that investigated the mechanical properties of gypsum-based composite materials concluded that when particles or gypsum crystals have physical characteristics with elongated and thin shapes or even stick shapes, this morphological characteristic tends to promote the formation of shear stresses inside the material structure, thus increasing its mechanical resistance, when compared to other particle morphologies also addressed in the same work. Thus, this type of particle or plaster crystal morphology can be very interesting for applications that require this type of improvement.

## Thermogravimetric Analysis (TGA)

The graphic profiles representative of the curves for mass loss, obtained via thermogravimetric analysis (TGA) as well as its first derivative (DTGA) for the α plaster sample are presented in **Figure 3**.

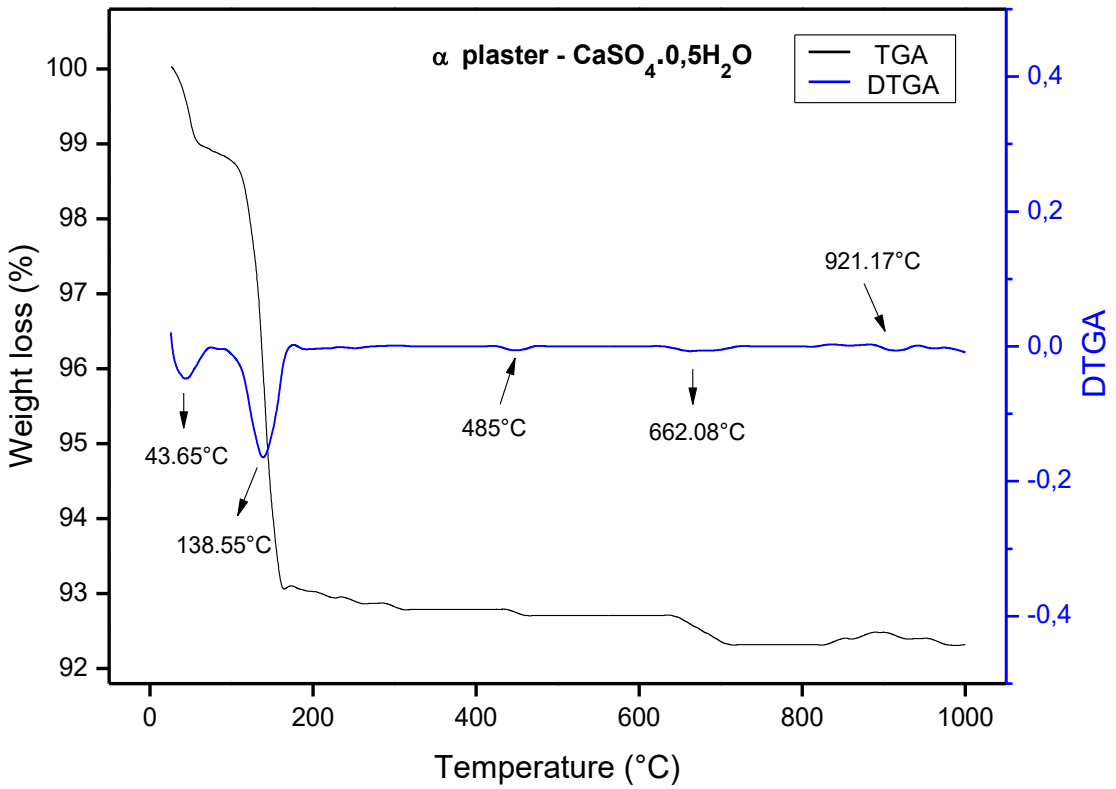


**Figure 3**. Graphic profiles representative of the data from the thermogravimetric analysis (TGA/DTGA) for α plaster sample, in dynamic air atmosphere, 50 mL.min$^{-1}$.

When analyzing the thermogravimetric profile of the α plaster sample it was possible to notice the presence of three main peaks representative of mass loss occurrences. According to the graph, the first occurrence of mass loss occurred under the maximum temperature value of 43.65°C, probably related to a loss of free water still existing in the sample. The second mass loss event, occurred with a maximum of 138.55°C, most likely related to the loss of part of the crystallization water, these values detected in this analysis referring to the temperatures of partial dehydration of the gypsum with its consequent conversion to the form hemihydrate are in accordance with research reports related to the investigation of this type of ore published in the literature (Krause *et al.* 2020; Ritterbach & Becker 2020). Some discrete events were perceived at temperatures above 450°C suggesting the presence of thermally active components, which were not detected by XRD analysis. Comparing this graphic construction based on the data from the TGA/DTGA analysis with the results of the XRF analysis, it is plausible to deduce that these little pronounced events may be related with thermal decompositions of the contaminating oxides detected in the chemical composition which has been described in **Table 1** for the α plaster sample. Engbrecht & Hirschfeld (2016) carried out an investigative research on the thermal behavior of calcium sulfate dihydrate and in their studies it was proven that the gypsum sample investigated underwent thermal decomposition of carbonate components (calcite and dolomite) at temperature close to 700°C, which had not been revealed in the XRD analysis. Another plausible hypothesis to justify the discrete events observed in the graph shown in **Figure 3** may be related to the evaporation of the remaining water in the crystalline structure of the α plaster sample, object of study of this work, with the probable formation of anhydrite. It is not uncommon to find references in the literature that point to the formation of anhydrous calcium sulphate related to the complete dehydration of calcium sulphate hemihydrate (Canut 2003 *apud* Canut 2006; Costa 2013; Marandi *et al.* 2017).

## Fourier Transform Infrared Spectroscopy (FT-IR)

**Figure 4** shows the FTIR spectra for the analyzed α plaster sample in this work. The information presented by result of the FTIR analysis endorses the high purity of the α plaster sample, corroborating the results of the XRD and FRX analysis already discussed.

According to the FTIR graphic spectra, shown in **Figure 4**, the composition of the α plaster sample analyzed in this work is essentially composed by calcium sulfate in humidity presence. Furthermore, no peak or band occurrence was detected, alluding to the presence of compounds other different those already identified in previous structural and chemical characterizations. Analyzing this FTIR spectra, it was found that the peaks that were located between the range of 3067 and 3490 $cm^{-1}$ are according to reports published in the literature, attributed to the bond length of the functional group OH, an indication of the humidity presence in the sample. The peak identified exactly at 1619 $cm^{-1}$ alludes to the flexion vibrations of water molecules present in the sample. Finally, it was found that the peaks located in the wave range between 1100 and 590 $cm^{-1}$ are attributed to the asymmetric vibrations of the bonds of the sulfate group - $SO_4^{2-}$ (Huynh *et al.* 2020; Li & Zhang 2020; Maale *et al.* 2020; Zhang *et al.* 2020).

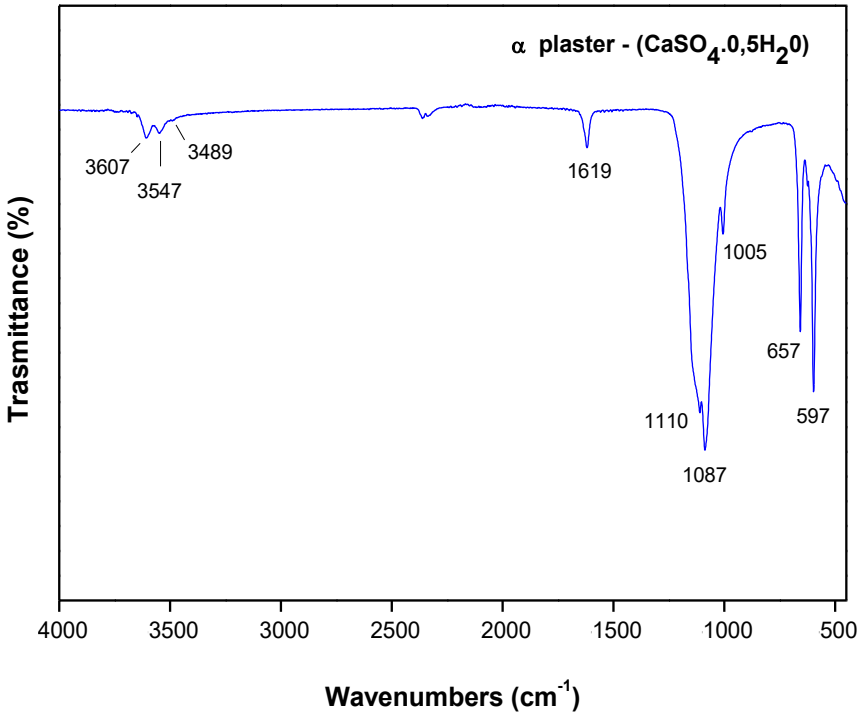


**Figure 4**. Fourier Transform Infrared (FTIR) spectra for the α plaster sample.

## Conclusions

The sample of Brazilian gypsum, from state of Pernambuco, analyzed in this work, belonging to the mineralogical variety Johnson and called α plaster, presented mineralogical composition mostly based on calcium sulfate hemihydrate, α-$CaSO_4.0.5H_2O$, with a content of purity around 98.79%; thus, the sample of the α plaster analyzed was shown to be suitable for applications that require minimal possibilities of toxicity, such as, applications in the health area, among others that imply literal contact with living organisms.

The expressive degree of purity of the α plaster sample analyzed in this work was superior when compared to the degrees of purity of samples of calcium sulfate hemihydrates, also α type, from other countries.

The combination of the results of the structural and chemical characterizations (XRD, XRF, TGA and FT-IR) converged to a uniqueness, endorsing each other, with regard to the presence of contaminating components in concentrations that do not disprove the indication of

α plaster, analyzed in this work, for applications considered more noble and distinct from the classic and trivial applications for civil construction.

The morphological analysis, carried out by the SEM technique, revealed that the unitary constituent particles of the α plaster sample, as well as their agglomerates, presented with elongated and thin shapes with average sizes, approximately uniform, around 60 μm. The morphology presented by the particles presented to be suitable for applications that require the manufacture of parts with appreciable mechanical resistance, thanks to their physical predisposition to form residual shear stresses.

## Acknowledgements

The authors are grateful for the financial support of CNPq and the Federal University of Cariri, which made possible the development of this research. We would also like to thank the company of the Plasterer Polo of Araripe that provided the α plaster sample analyzed in this research.

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