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Physico-chemical characterization of paint industry residues for incorporation in cementitious matrix

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ABSTRACT

Before being reused, industrial waste needs to have its composition determined so that in the future, it will not cause harm to the people and to the environment. The present study proposes the characterization and reuse of the residue remaining in the production of leveling masses and textures as a component in the Portland cement mortar specimens in order to analyze the effects on the axial compressive strength. The residue generated was characterized by Infrared (IR) Absorption Spectroscopy, X-ray Diffraction (XRD), Atomic Emission (AE) and Thermogravimetric Analysis (TG/DTG). All analyzes confirmed the predominant presence of dolomite ($CaMg(CO_3)_2$) as the main component and absence of potentially toxic metals. The addition of 10% of the residue on the cement and sand mass or the replacement of 10% of the sand mass in the mortars had small impact on the axial compression strength. The standard deviation values found lead to the conclusion that the compressive strength of the studied mortars can be considered statistically similar.

Keywords: Industrial waste, solid waste characterization, dolomite and Portland cement mortar.

1. INTRODUCTION

Large industrial centers contribute substantially to the increase in the solid waste production. Depending on the characteristics of this waste, its disposal can cause damage to the health of the population and the environment. In this context, the paint industry can be considered as one of the industries that generate produces a high volume of solid waste [1-4]. According to the Brazilian Association of Paint Manufacturers (ABRAFAT), the sector sold 10,026 billion reais, in 2016. This indicator places Brazil among the six largest producers of paints in the world due to the large production and commercialization of paints in recent years [5].

Inks and their derivatives are generally made from resins, pigments, additives and water or organic solvents (Scheme 1) which may exhibit high flammability, strong odor and high toxicity. Studies reported that the improper disposal of some of these wastes can cause some pathologies in humans like nausea, allergic reactions and irritations in various parts of the body. In addition, these residues can also cause damage to the environment [6, 7].

The problematic related to the waste is not only restricted to the amount that is generated, but also by the way it is discarded. Often the disposal of this material is done in open-air areas such as landfills and dumps [7]. In this context, one of the major consequences to the environment is the alteration of the hydrogen potential (pH) of the soil or the water of rivers. This significant change in the ecosystems may result in compromising the natural life of species that live there. Thus, the considerable increase in the amount of industrial solid waste has become an environmental problem [8, 10].

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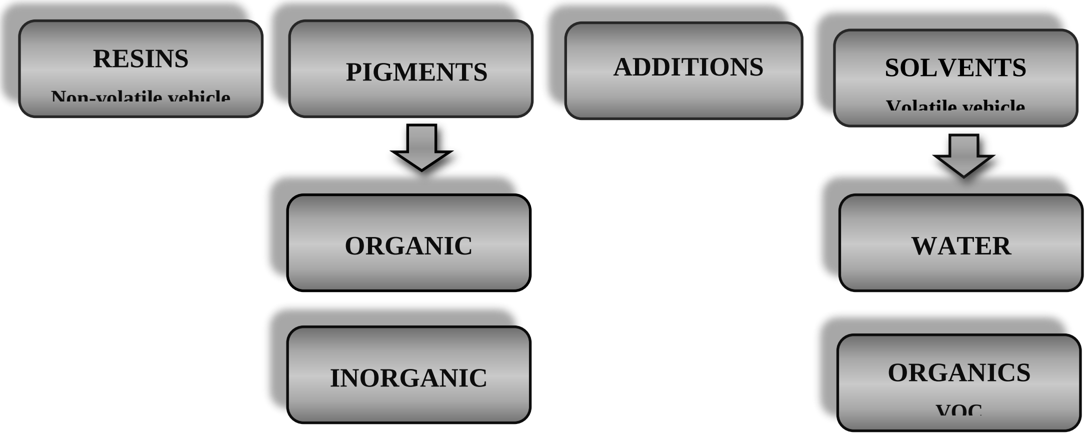

Scheme 1: Flowchart of basic paint constitutions.

In this setting, there is a need for governamental policies in order to regulate and control the n disposal and reuse of these industrial wastes. Among these measures, it can be highlighted the Brazilian Federal Law 12.305/2010 that requires large companies to destinate a viable purpose for its waste, choosing to reduce, recycle or reuse it. Such legal regulation allows the company to choose the best alternative to deal with the type of waste generated [11]. Additionally, Conama Resolution Nº 313/2002, which refers to Industrial Solid Waste (ISW), describes a set of information about generation, characteristics, storage, transportation, treatment, reuse, recycling, recovery and final disposal of ISW [12].

Considering the legal norms, brazilian industries have made a huge effort to try to set a proper waste disposal generated in their productions. A number of this actions is due to the laws created that regulate and establish ways of dealing with the waste generated by industries as well as the principles of green chemistry that aim to propose production mechanisms that do not result in damage to the environment. With these measures, the industries have an accurate information/guidance regarding to the quantity, type and destination of the ISW. Thus, from this information, relevant characteristics can be obtained regarding the reuse of waste generated [13-15].

Studies that address the reuse of industrial waste indicate a wide application in the most varied matrices, mainly in cementitious matrices. [16, 17]. However, few studies emphasize the characterization and reuse of waste from the paint industry. Recently, Alves *et al* (2018) approached the complete characterization of residues from automotive paint industries, looking for a possible reuse [18]. Onyeji (2010), by means of destillation and filtration steps, observed an average value of 71.43% recovery of solvents used in the production of paints, being useful, also, for reuse and recycling [19]. These studies show the need to properly deal with this type of waste, in order to reduce the environmental impacts and to allow their reuse. there are few studies that address the physical-chemical characterization of these residues, mainly those produced from the paint industry.

The levelling masses and textures used in the painting systems are characterized by a pasty product, where a high content of fillers is mixed with an acrylic resin emulsion. The fillers are composed of calcium carbonate (calcite), dolomite or the mixture of these minerals. Therefore, the residue from the production of the masses and textures tends to be composed mainly of finely fragmented carbonate material. The Brazilian standard NBR 16,697 (2018) allows the incorporation of carbonate material in the composition of Portland cement in varying levels, reaching 25% by weight [20]. The carbonate material used in cement is calcite (calcium carbonate), although several studies have proved the viability of using dolomite [21-23].

The presence of finely comminuted carbonate material in the Portland cement composition alters the hydration kinetics of the anhydrous phases [19, 22] and provides the filler effect, in which the spaces between the cement particles are filled with the tiny particles of the added carbonate material, densifying the microstructure [25].

The partial replacement of Portland cement by carbonate material (calcite or dolomite) in levels between 10 and 30%, reduces the compressive strength of the mortar [20, 23]. The present study, intends to characterize the waste from the paint industry and use it in the composition of mortars in two ways: partially replacing the fine aggregate and in addition to the cement and sand masses. Thus, cement consumption is kept practically constant and, consequently, the compressive strength should not be significantly impacted.

2. MATERIAL AND METHODS

2.1 Waste Collection

The solid residues were obtained from a paint industry located in the city of Barreiras in the state of Bahia, Brazil. The waste samples were collected in an appropriate place for their storage, thus avoiding possible contamination. The collection was performed at fourteen points at the waste containment site. Each sample was collected using a spatula and a plastic bag and separated by collection points. The first collection was performed on 07/02/2016, being collected on that day two samples. The collection dates were established according to the period in which new residues from the production line were discarded on the waste containment site. Thus, there were always new residues added to the existing ones, allowing a better representation of the samples that characterized the real content of the residues.

2.2 Infrared Absorption Vibrational Spectroscopy (IR)

Fourier Transform Infrared (FTIR) spectra were obtained on IRAffinity-1S spectrophotometer (SHIMADZU) equipped with a DTGS detector (accessory that provides greater sensitivity to the detector, formed by the deuterated triglycine sulfate salt). Measurements were performed using KBr pellets and readings between 400 cm^{-1} and 4000 cm^{-1} (90 scans and 4 cm^{-1} resolution) at a ratio of 1:100 residue and KBr, respectively. KBr underwent heating to eliminate moisture.

2.3 X-ray Diffraction (XRD)

X-ray diffractograms were obtained on a Rigaku D / MAX-2A / C diffractometer with CuKα radiation at 40 kV and 20 mA. Speed of 2°. min^{-1} and angle of 2θ ranging from 2° to 60°. The identification of the peaks was performed with the help of the software X'Pert HighScore, using ICSD (Inorganic Crystal Structure Database) database.

2.4 Atomic Emission

Measurements were made using an Agilent 4200 Microwave Atomic Emission Spectrometer (MP AES) with nitrogen used by an Agilent 4107 nitrogen generator. The sample introduction system consisted of a Micro-Mist nebulizer and cyclonic spray. Glass with double pass chamber. Instrument operating conditions are showed in **Table 1**.

Table 1: Atomic Emission Spectrometer Measurements.

INSTRUMENTAL PARAMETERS	VALUES
Integration Time (s)	3
Peristaltic Pump Speed (rpm)	15
Number of Replicates	3
Estabilization Time	20
Background Correction	Auto
CCD Temperature (º C)	-0.3

2.5 Thermogravimetric analysis

Analysis performed using a thermogravimetric balance, DTG - 60 (Simultaneous DTA-TG Apparatus, SHIMADZU). The equipment has controlled temperature programming and registers mass variation as a function of time and temperature. Controlled variation from (approximately 25 °C) to 900 °C. Adopted heating rate of 10 °C/min under N_2 atmosphere (50 mL/min.). The sample mass was approximately 10 mg stored in platinum crucible.

2.6 Mixture of Portland cement mortars

The mortar mixed procedure was performed as recommended by NBR 7215:1996 - Portland Cement - Determination of compressive strength. A mechanical mixer was used containing a stainless-steel bowl with a capacity of approximately 5 L and a metal shovel that rotates the entire interior of the bowl in a planetary motion. In a bowl containing water and under stirring (low speed) Portland cement (type CP II F-40 – Brazilian standard), sand and industrial residue were added. The latter received a dispersion treatment in order to its particle size showed a standard uniformity. The residue was added in two formulations out of three, left out of the reference mixture. The residue was ground and sieved, and a residue chosen volume of 100% passing material in the sieve opening 4.75 mm (grit). Some agglomerates composed of resins and not friable were removed manually. Each component was added to the container containing water within 30 seconds. After mixing all the components, the equipment was turned on at high speed for 1 minute and 30 seconds until the machine scraping stopped. Then, the mixture was stirred for another 1 minute at high speed.

2.7 Incorporation of waste into Portland cement mortars

Table 2 shows the proportions of the mixture of Portland cement mortars with and without the addition of residues collected in the paint industry. The first proportion refers to reference mortar, without residue..

Table 2: Reference **f**ormulation, without residue**.**

MATERIAL	MASS (Kg)
Cement	1.00
Sand	1.63
Water	0.60
Water/cement ratio	0.60

In the second mix proportion, 10 % of the sand contained in the reference formulation (0.163 kg) was removed, and 10 % residue was added, according **Table 3**.

Table 3: Formulation with 10 % substitution of sand for residue.

MATERIAL	MASS (Kg)
Cement	1.00
Sand	1.46
Residue	0.16
Water	0.60
Water/cement ratio	0.60

The third proportion of the mixture was prepared from the mass of the reference value added plus 10% of residue, as shown in Table 4.The third mix proportion used the reference plus 10 % calculated residue on the sand plus cement mass, **Table 4**.

Table 4: Reference formulation with 10 % residue addition to cement plus sand mass.

MATERIAL	MASS (Kg)
Cement	1.00
Sand	1.63
Residue	0.26
Water	0.60
Water/cement ratio	0.60

2.8 Molding and curing of mortars specimens

Four specimens were molded for each of the three proposed formulations. The specimens were molded according to NBR 7215/1996 - Portland cement - Determination of the compressive strength. The cylindrical molds with dimensions of Ø 5 x 10 cm, after oil lubrication, were filled by the mortar in four layers. Each layer was compacted with standard socket, applying 30 strokes per layer. The mortars were demolded after 24h and kept in a laboratory environment for 25 days, totaling 26 days. At this age, they were submerged in water for saturation for 48 hours (2 days) to perform the compressive strength test at 28 days of age.

2.9 Axial compressive strength test

The axial compressive strength test was performed for mortars samples at the age of 28 days, immediately after they were removed from the water, in a PC 200C EMIC hydraulic equipment, according to the guidelines from NBR 7215/1996 - Portland cement - Determination of compressive strength for mortar specimens. Steel plates with shore 70 hard neoprene discs were used to reduce the imperfections of the flat faces of the mortar specimens. For each formulation four specimens were tested. The average of the results of the four tests represents the axial compressive strength of mortars samples.

3. RESULTS AND DISCUSSION

3.1 Waste Collection

The paint industry produces 600 Kg residue /month. All samples collected were in solid state. They had a whitish color and some residues showed traces of the presence of pigments from the production of paints and apparently did not appear thermally unstable, as they were stored outdoors. It was possible, in some samples, to detect the formation of low friable agglomerates due to the presence of resin found in few parts of the residue.

3.2 Infrared (IR) Absorption Vibrational Spectroscopy (IV)

A sample was collected from the raw material used in the manufacturing of the leveling masses and textures in collaborating paints factory (as a reference) and analyzed by infrared spectroscopy. Another sample was collected in the waste after homogeneous mixture of all residue samples collected along the time. This sample was also analyzed by infrared spectroscopy. **Figure 1** shows an overlap of waste spectra collected with the dolomite, raw material used in the collaborative paints industry.

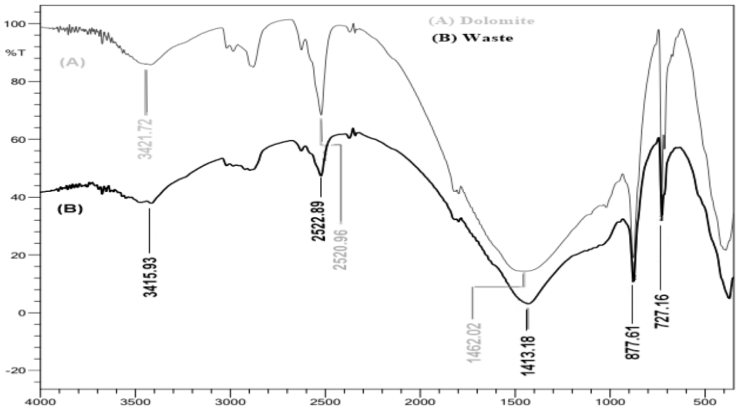

Figure 1: Infrared spectra overlap of the residues and dolomite (values in wavenumber cm ^{1}).

It is noted that the spectral profile of the residue is quite similar to the spectral profile of dolomite, which suggests that the residue have this constituent as being predominant. An absorption band referring to the stretching of the O-H bond appears at 3421.72 cm^{-1} with low intensity in both spectra. The low intensity of the band in both spectra can be justified by the high of energy value of the network and by the difficulty of hydration that this compound has (dolomite), after all, the values of the solubility product (Kps) for this compound left in the order of 10^{-10} and 10^{-19} [26, 27].

Additionally, a broadband appears at 1462.02 cm^{-1} (dolomite) and 1431.18 cm^{-1} (residue) that is characteristic of the CO_3^{2-} vibratory antisymmetric mode. It is also possible to observe vibratory modes in the regions of 877.61 cm^{-1} and 727.16 cm^{-1}, which are related to the folding out-of-plane bend and in plane of carbonate group, respectively [28]. Bands in 1100 cm^{-1} and 450 cm^{-1} in both spectra can be associated with the stretching of the Si-O and O-Si-O bonds respectively, which is in line with the XRD analyzes that are discussed later [29].

In the residue spectra, the absorption bands characteristic of other raw materials such as titanium dioxide (TiO_2), and aluminum sulfate ($Al_2(SO_4)_3$) were not observed. The absence of bands related to these substances is supposed to be due to the smaller amount they are used in the manufacture of paints and derivatives. Another particular factor involving the titanium dioxide in this case is that it is used only in the manufacture of paints, in the manufacture of a liquid product that is almost entirely potted (lossless), not contributing significantly to the formation of waste.

3.3 X-ray Diffraction

Figure 2 shows the diffractogram of the residues collected from the paint industry. The peaks at 30.9º 2θ, 41.2º 2θ and 51.1º 2θ found in the diffractogram confirmed that dolomite is the component in greater quantity.

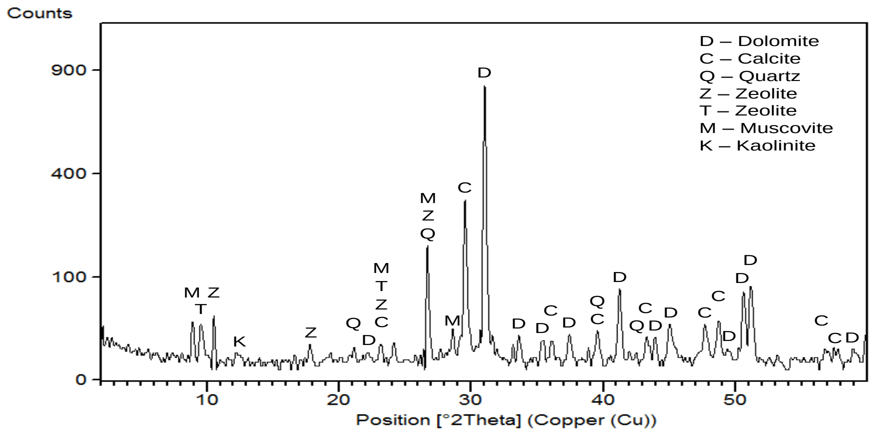

Figure 2: Diffractogram of collected waste.

These findings are in agreement with the data observed in the infrared analysis previously reported. However, it was noted that, in addition to dolomite, the residues still have an amount of calcite ($CaCO_3$) and quartz (SiO_2) due to the presence of peaks of 29.4º 2θ, 47.6º 2θ and 48.6º 2θ (calcite) and 26.6º 2θ (quartz). This shows that, in the reservoir site where the dolomite is located, it is possible to have points containing calcite and quartz. This occurrence is quite comprehensive, since studies documented in the literature show that the most common impurity found in dolomite is precisely the quartz [24]. The minority constituents of the residue were zeolite (SiO_2), muscovite and kaolinite.

The phases identified in the residue of dolomitic base, are identical to those presented in the characterization study of dolomitic limestone carried out by Souza & Bragança [30], except for the zeolite presence. The authors attributed the peaks located in the region close to 10º 2θ to a mineral clay, considering it as an impurity of the limestone rock. The Rietveld refinement applied to the residue diffractometric profile resulted in the quantification of the phases shown in Table 5. The refinement Goodness-of-fit (GOF) was 1.85, with values below 5.00 representing an optimized refinement [31].

Table 5: Quantification of the phases.

PHASE	CHEMICAL FORMULA	QUANTITY (%)
Dolomite	$CaMg(CO_3)_2$	69.3
Calcite	$CaCO_3$	23.7
Quartz low	SiO_2	3.9
Zeolite - Anorthic	SiO_2	1.4
Zeolite – Hexagonal	SiO_2	0.4
Muscovite	$K(Al_{1.91}Fe_{0.09})(AlSi_3O_{10})(OH)_2$	0.7
Kaolinite	$Al_2(Si_2O_5)(OH)_4$	0.6

The phases identified and quantified in the diffractometric profile classify the residue as a dolomitic limestone, with a magnesium oxide (MgO) content in order of 15.1 % and a total calcium oxide (CaO) content of 34.4 %, of which 13.3 % is combined in the form of calcite. The theoretical content of carbon dioxide (CO_2) combined in the form of carbonates, determined from the number of phases present in the residue, is 43.6 %. The CO_2 content is an indicator of thermogravimetric analysis of waste, which volatilizes a small water content consisting of muscovite and kaolinite. Therefore, it is estimated a residual mass of approximately 56 % after calcination of the residue up to 900 ºC in an inert atmosphere.

For the purpose of using the waste in conjunction with Portland cement, the mineralogical composition is, at first, compatible, since it consists predominantly of limestone filler (93.0 %) and quartz filler (3.9 %), which are considered inert materials.

3.4 Atomic Emission

Atomic emission analyzes were used to evaluate the presence of toxic metals. Arsenic (As), mercury (Hg), cadmium (Cd), chromium (Cr) and lead (Pb) were detected, according to **Table 6**.

Table 6: Presence of heavy metals in the residue.

DETERMINATION	RESULTS (ppm)	EQUIPMENT QUANTIFICATION LIMIT
As	0.36	0.1
Hg	0.008	0.01
Cd	0.27	0.1
Cr	0.12	0.1
Pb	4.62	0.1

The results indicated that the metal that was present in the highest concentration was Pb, 4.62 ppm, however, this value is below the maximum allowed value to be found in paints, which is 0.06 %, in this case, 600 ppm [32, 33]. For Hg, the value found in the sample, 0.008 ppm, value below the equipment limit of quantitation (LoQ). Finally, the other metals (As, Cd and Cr) presented concentrations below the values permit by the CONAMA/ 157 resolution. These findings characterize the residue as low toxicity [34].

The low concentration of potentially toxic metals in the waste, mainly mercury, cadmium and lead, is an important factor for reuse, especially when considering the recognized toxicological effects of these metals on the environment and living organisms.

3.5 Thermogravimetric Analysis

The TG **(a)** and DTG **(b)** curves of the residue can be visualized in **Figure 3**. It is possible to identify a single well-defined decomposition stage, at a temperature of 735 ºC, which corresponds to the production of carbon dioxide (CO_2) (44.85 % by mass). This event identifies the decomposition of the dolomite and the calcite minerals, as exemplified in those shown in **Scheme 2**.

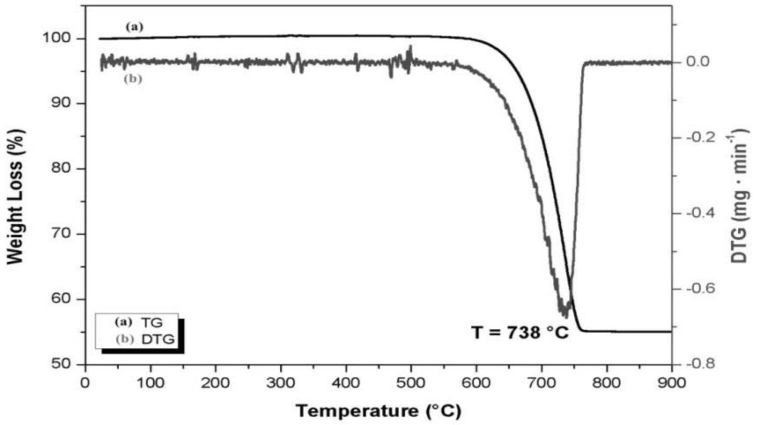

Figure 3: Thermogravimetric analysis (TGA) of collected waste

The thermographic test did not allow distinguishing a decomposition of magnesium carbonate from dolomite, followed by the decomposition of the calcium carbonate from dolomite and calcite. Therefore, from the result of the thermogravimetric analysis, there is no way to estimate the amount of each of these carbonate compounds.

$$CaMg(CO_3)_{2(s)} \overset{\Delta}{\rightarrow} CaO_{(s)} + MgO_{(s)} + 2CO_{2(g)}$$
$$CaCO_{3(s)} \overset{\Delta}{\rightarrow} CaO_{(s)} + CO_{2(g)}$$

Scheme 2: Decomposition reaction of the dolomite mineral and calcite respectively.

As the dolomite content (69.3 %) and calcite (23.7 %), quantified by the Rietveld method applied to diffratometric residue profile, it is possible to estimate the respective levels based on thermogravimetric analysis, since it is considered that the relationship dolomite (69.3 %) / calcite (23.7 %) remains constant, with a value of 2.924. Thus, dolomite and calcite content estimated from the mass volatilized carbon dioxide (CO_2) are 71.3% and 24.4%, respectively. These values are similar to those quantified by Rietveld / DRX. At the end of the analysis, 55.15 % of non-volatile material remained, which in turn is composed of calcium oxide (35.4 %), magnesium oxide (15.5 %) and other minority constituents. The calculated mass percentage for the resulting calcium oxide (CaO) and magnesium oxide (MgO) is 50.90 %.

These analyzes make it possible to determine with precision that the waste can be classified, according to NBR 10005/2002, in category II, as non-toxic. Thus, it can be concluded that the waste could be reused, for example, in civil construction, in the process of inserting cement production bases [12].The joint consideration of the analyzes allows to us determine with precision that the waste in question can be classified in NBR 10005/2002 in category II, as non-toxic. Thus, it can be concluded that the waste could be reused, for example, in construction, for insertion of production bases cement [12].

3.9 Resistance to axial compressive strength of specimens

The reference sample consists basically of cement, sand and water. Samples 1 and 2 have their formulation based on the reference sample, with the replacement of 10 % of the sand by the residue (sample 1) and by the addition of 10 % of the residue to the cement and sand mass (sample 2) respectively.

Figure 4 shows the compressive strength of the reference mortar, sample 1 and sample 2, referring to specimens aged 28 days.

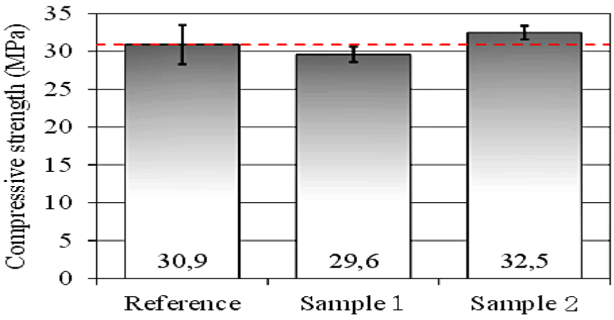

Figure 4: Compressive strength of mortar specimens.

Sample 1: replacement of 10 % of sand by residue. Sample 2: added 10 % of the residue on mass of cement and sand

The sample using the reference formulation has, in average, a compressive strength higher than the sample where 10 % of the sand was replaced by the residue (sample 1). Comparing the same reference sample with that where 10 % of the residue was added to the total sand mass plus cement (sample 2), the latter obtained, in average, a compressive strength gain when compared to the reference sample.

In general, the use of the residue in the content of 10 % by mass, both for the partial replacement of sand and for the addition of solid materials (cement + sand) over the mass, had little impact on the compressive strength of mortars. The analysis of the standard deviations of mortars shows that the compressive strengths can be considered statistically similar.

Dolomite is widely used in the manufacture of paints and complements because it is also an inert product. However, when the dolomite is removed from its deposit, it passes through a mill, where it has a particle size of 15 to 18 microns. This small particle size coupled with its low reactivity will have a good entry into the mortar mix so that it fills the voids. This causes the mortar to gain more density and then increase its compressive strength.

4. CONCLUSIONS

All analyzes performed on the waste samples confirmed dolomite (CaMg (CO3) 2) as the main component. This finding can be supported, mainly, by the infrared analyzes that showed the carbonate absorption bands (antisymmetric, in the plane and outside the plane side), Si-O and O-Si-O and the XRD analyzes by the identification and quantification of the phases. Other materials such as calcite, quartz, muscovite and zeolite were determined in lesser quantities and were classified as impurities.

The infrared and thermal analyzes also confirmed that the water content even after processing, the dolomite is low, corroborated by the appearance of a weak band at 3400 cm^{-1} in the infrared spectra and by the lack of decomposition stages in the temperature range from 100 to 250 ° C. In addition, atomic absorption analyzes indicated potentially toxic metal levels below the limit permit legislation.

Specimens (mortar) containing residues in their formulations showed a similar axial compressive strength when compared to the reference sample. The sample, which had 10% of the mass of cement and sand in its composition, obtained, on average, a small increase in strength. For a sample in which 10% of the sand was replaced by the residue, the resistance value obtained was within the margin of error equal to the reference.

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