

Effect of inserting multi-walled carbon nanotubes on the physical and mechanical properties of the gypsum matrix

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ABSTRACT

The growing demand for gypsum in civil construction has stimulated studies to improve flexural strength and reduce its layer with water. Therefore, the objective was to evaluate the influence of incorporating multi-walled carbon nanotubes (MWCNT) into the gypsum matrix on its physical and mechanical properties. In the production of composites, the gypsum mass was obtained by different levels of functionalized MWCNT: 0%, 0.001%, 0.002%, 0.003% and 0.004%. Apparent density, observation of water and flexural strength and analysis of the nanocomposites were verified. Replacing gypsum with MWCNT can cause water absorption by 8.33% compared to the control. The apparent density of the compounds showed an increase, with the concentration of 0.004 MWCNT showing an increase of 3.21% when compared to the reference. Compounds with contents of 0.001 and 0.003% of MWCNT showed an improvement in flexural strength values, presenting of 6 MPa. There were improvements for the insertion of 0.001 MWCNT (9 MPa) compared to the control (7 MPa). Thus, the results indicate that replacing gypsum with MWCNT is a viable alternative to improving the mechanical and physical properties of gypsum, which can increasingly expand its use in civil construction.

Keywords: Civil construction; Functionalized; Nanocomposites; Mechanical and physical properties.

1. INTRODUCTION

Gypsum is widely used in civil construction due to its excellent thermal and acoustic properties, in addition to its easy molding and quick hardening, among other advantages [1]. However, this material has limitations caused by low mechanical strength, reduced toughness and high affinity with water [2]. These characteristics are mainly explained by the high solubility of calcium sulfate and the formation of large amounts of micropores when crystallization and hardening of the gypsum paste occurs, with the amounts of these micropores around 50 to 60% of its volume, and most have sizes between 0.3 – 1.0 μm , where capillary suction forces are active [3]. Thus, the physical and mechanical properties of gypsum materials are influenced by the structure of the matrix and the introduced additives, which can affect hydration, the size and morphology of gypsum crystals, the state of the interface and the porosity of the material.

Nanotechnology is a line of research under development that presents different applications in the areas of science and technology [1]. Among the areas that show promise, civil construction stands out, which has been consolidating itself in the global sphere as an important development sector, since products containing incorporated nanomaterials have better physical, mechanical, electrical, thermal, among other properties [4, 5].

Carbon nanotubes (CNT) and multi-walled carbon nanotubes (MWCNT) have attracted the attention of the scientific community due to their potential applications, such as reinforcement of polymers and composites, energy storage, electronics and catalysis [6]. These materials can improve the compaction of composites, making the material denser and less porous, filling the voids caused by gypsum hydration products. TOKAREV *et al.* [7] studied the incorporation of CNT into a gypsum matrix with various additives and found that the insertion of nano reinforcement at levels of 0.001 – 0.003% caused an improvement in the mechanical properties of the gypsum. DEREVIANKO *et al.* [8] also worked with the inclusion of CNT as reinforcement in gypsum, with an

improvement in the hydration process of calcium sulfate dihydrate and in the physical and mechanical properties of the matrix being observed, with 0.18% of CNT resulting in an increase in compressive strength by up to 30%.

There are still few records in the literature regarding the effects of incorporating MWCNT on the physical and mechanical properties of gypsum matrices, making it necessary to carry out studies to improve these properties. In this context, we sought to evaluate the physical and mechanical properties with the incorporation of carbon nanotubes in the gypsum matrix.

2. MATERIALS AND METHODS

In the production of the composites, commercial fine gypsum for casting was used as a matrix, which has an apparent specific mass of 781 kg/m³ and a fineness modulus of 0.85. For reinforcement commercial, multi-walled carbon nanotubes (MWCNT) produced via chemical deposition process was incorporated (Figure 1).

2.1. Characterization of carbon nanotubes

MWCNT samples were diluted to a concentration of 0.001% (m/m) and sonicated at 150 Hz for 2 min using an Eco-Sonics/Utronic Ultrasonic Tip sonicator (750 W) [9]. Subsequently, small aliquots were added to the carbon tape adhered to the aluminum sample holder (stubs). After an overnight period in a desiccator with silica gel, the samples were metallized in gold with a spray device (SCD 050). MWCNT morphology was observed in an ultra-high resolution (UHR) field emission scanning electron microscope TESCAN CLARA (Libušina, Czech Republic) using 10 keV, 90 pA and 10 mm working distance. MWCNT diameters and lengths were measured for a minimum of 100 individual structures using ImageJ software [10].

MWCNT were functionalized according to the methodology proposed by FRAGA *et al.* [11] for graphene oxide. Functionalized and non-functionalized MWCNT were subjected to thermogravimetric analysis to verify if there was any change in chemical characteristics. In the thermal analysis, 10 mg of MWCNT were added to the equipment's crucible heated from 25 to 900 °C. The established analysis condition was in an atmosphere of synthetic air (80% N₂ and 20% O₂) flowing at 50 mL/min and heating rate of 10 °C/min [12].

2.2. Production of composites

In the production of composites, the gypsum mass was replaced by different levels of functionalized MWCNT, namely 0%, 0.001%, 0.002%, 0.003% and 0.004% (Table 1). The water/gypsum ratio (w/g) used was 0.6, according to the methodology proposed by GORDINA *et al.* [13] and PERVYSHIN *et al.* [14].

MWCNT were sonicated with water for 30 minutes using an Eco-Sonics/Utronic Ultrasonic Tip sonicator (750 W) at 75% of the device's maximum power. Subsequently, gypsum was added and manually mixed for 3 minutes. Then, 6 specimens were made for each treatment, using molds with dimensions of 40 × 40 × 160 mm, according to the specifications of EN 13279-2 [15]. Figure 2 shows the composite production scheme.

After a period of 24 hours, the specimens were removed from the molds and placed in a climate chamber at a temperature of 20 ± 2 °C and relative humidity of 65 ± 3% for 28 days.

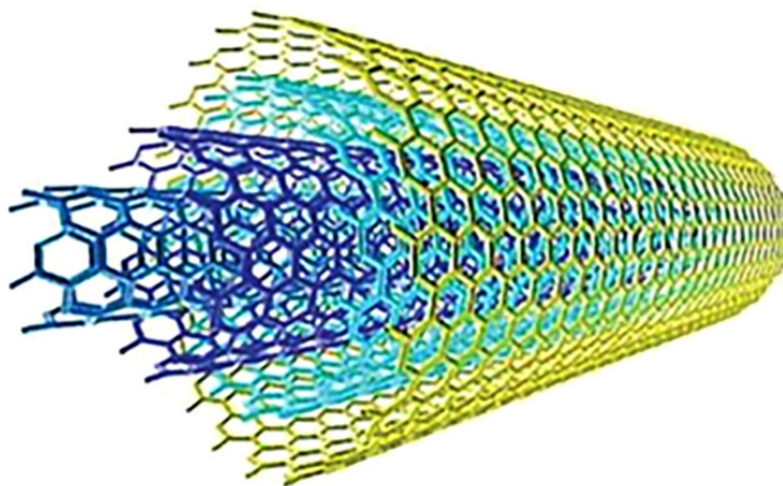
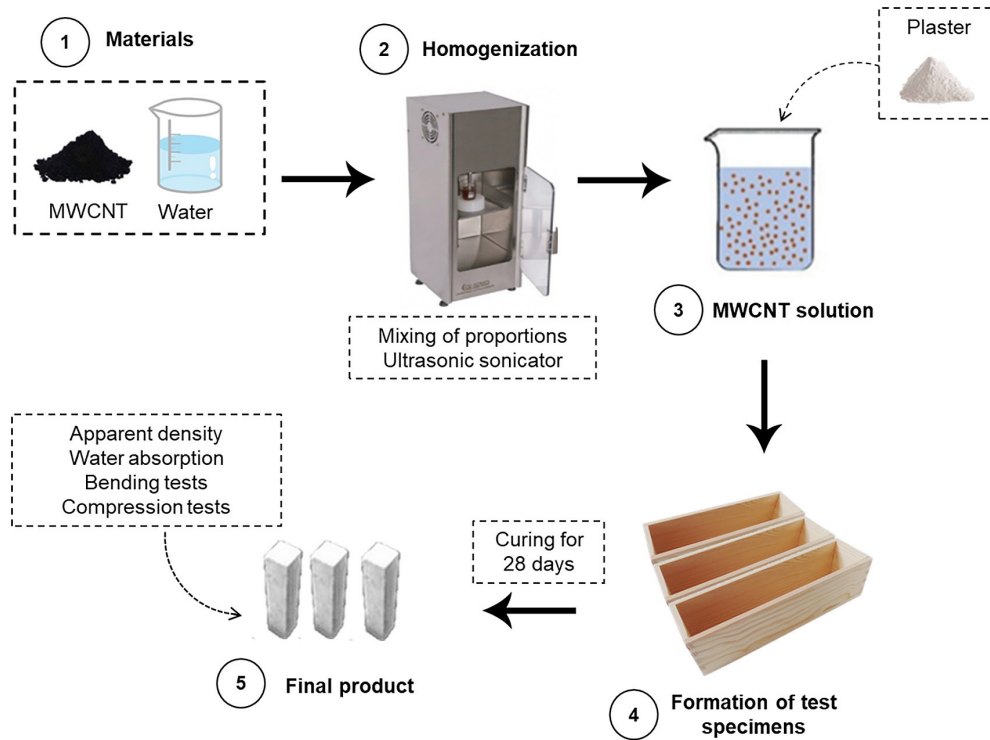


Figure 1: Multi-walled carbon nanotubes (MWCNT) [3].

Table 1: Sample composition proportions.

TREATMENT	COMPOSITIONS	
	GYPSUM (%)	CARBON NANOTUBES (%)
MWCNT ₀	100	0
MWCNT _{0.001}	99.999	0.001
MWCNT _{0.002}	99.998	0.002
MWCNT _{0.003}	99.997	0.003
MWCNT _{0.004}	99.996	0.004

**Figure 2:** Scheme of production stages of test specimens.

2.3. Determination of the physical and mechanical properties of composites

To obtain the apparent density, the NM 23:2000 [16] was followed, while the water absorption after 2 hours was determined according to the EN 520:2004 [17]. Flexural strength and compression tests were carried out in a universal testing machine in accordance with the EN 13279-1 [18]. In the flexural test, specimens with dimensions of 40 × 40 × 160 mm were used, and in the compression test, specimens of 40 × 40 × 40 mm.

2.4. Microstructural analysis of composites

For microstructural analysis, fractured specimens of approximately 1 × 1 × 1 cm were made, with proportions of 0 and 0.002% of MWCNT, which were dried in an oven at 70 °C for 10 hours. Then they were positioned on aluminum sample holders (stubs) and metallized in gold with a spray device (SCD 050). The surface of the composites was analyzed using a TESCAN CLARA ultra-high resolution field emission (UHR) scanning electron microscope.

The scanning electron microscope (SEM) was duly calibrated for the analysis of elements in energy dispersive X-ray spectroscopy (EDS). The observation in SEM with EDS analysis was performed with magnification of 200,000x for relative quantification of the elements, obtaining energy spectra and studying the distribution of elements (mapping function), keeping the working distance (WD) close to 9 mm, Kcps value greater than 30 units, voltage at 20 keV and Bean Current around 1 nA. Images made using a secondary electron detector were obtained with magnifications of 2,000, 20,000 and 200,000x.

2.5. Analysis of results

The results of the physical and mechanical analyzes were evaluated using a completely randomized design, and submitted to regression analysis at 5% significance.

3. RESULTS AND DISCUSSIONS

3.1. Characterization of carbon nanotubes

The morphology of carbon nanotubes (MWCNT) with network formation is shown in Figure 3. Length values ranged from 0.5 to 15 μm , with an average of approximately 4.5 μm , while in diameter, values ranged from 8 to 45 nm and an average of 20 nm.

Functionalized MWCNTs showed greater thermal stability (Figure 4). The maximum mass loss rate for non-functionalized MWCNTs occurred at a temperature of 548 $^{\circ}\text{C}$ and for functionalized MWCNTs at a temperature of 595 $^{\circ}\text{C}$. According to HATEL *et al.* [19] and RAHAMAN *et al.* [20], the greater resistance to thermal degradation observed for functionalized MWCNTs corresponds to the presence of magnetite, which confers greater thermal stability to the material.

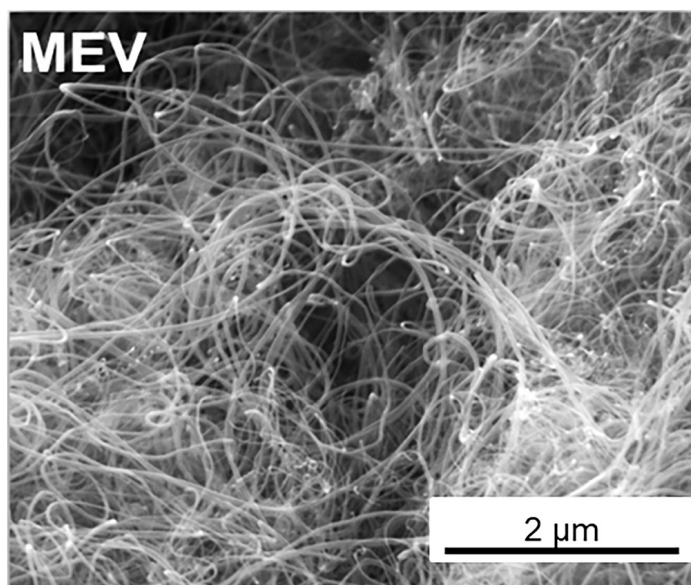


Figure 3: Morphology of carbon nanotubes in a scanning electron microscope.

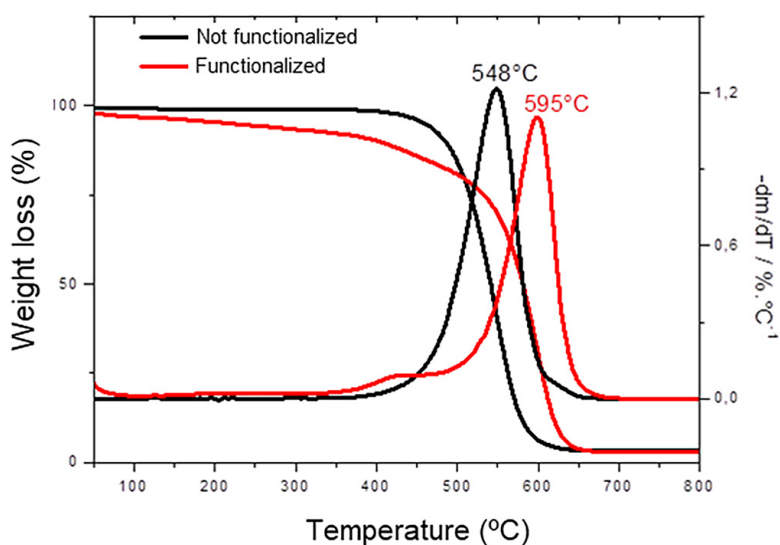


Figure 4: Thermogravimetric analysis of carbon nanotubes before and after functionalization.

Functionalization can improve solubility and processability by facilitating the material's interactions with other polymers [21]. In addition, functionalization allows modifying the intrinsic characteristics of the material, making it more reactive and expanding its area of application [22].

3.2. Physical properties of composites

Apparent density, the regression adjustment was significant ($F_c = 3.31$), indicating a statistical difference between treatments, in which the coefficient of determination increased as higher concentrations of MWCNT were added (Figure 5). According to the trend line, the maximum apparent density values found were for the concentration of 0.004 MWCNT, demonstrating an increase of 3.21% when compared to the control. This increase in apparent density can be considered advantageous, since the gypsum is very porous, which makes the gypsum fragile and highly susceptible to water absorption. MWCNTs possibly filled the pores of the matrix causing densification of the material.

CHEN and AKONO [23] report that MWCNTs act in filling the voids of composite matrices and, consequently, in reducing the capillary porosity of cementitious pastes. Capillary voids have a great influence on the strength and permeability characteristics of cement pastes, so the same behavior is observed in gypsum pastes. VIDALES-BARRIGUETE *et al.* [24] found a lower porosity index in gypsum composites using waste plastic cables, and found a reduction in their water absorption.

Linear regression was also significant for water absorption ($F_c = 6.19$), with a reduction in absorption with increasing concentrations of MWCNT replacing gypsum (Figure 6). Through the adjustment, the minimum point of the curve was at 0.004% of MWCNT incorporation, with a value of 27.55% of water absorption. Comparing with the control, there was a reduction of 8.33% for this property.

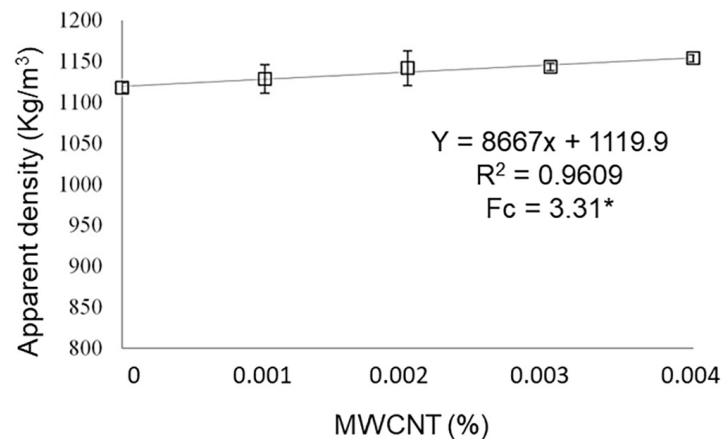


Figure 5: Apparent density of gypsum composites with carbon nanotubes.

*Significant regression analysis at the 5% level.

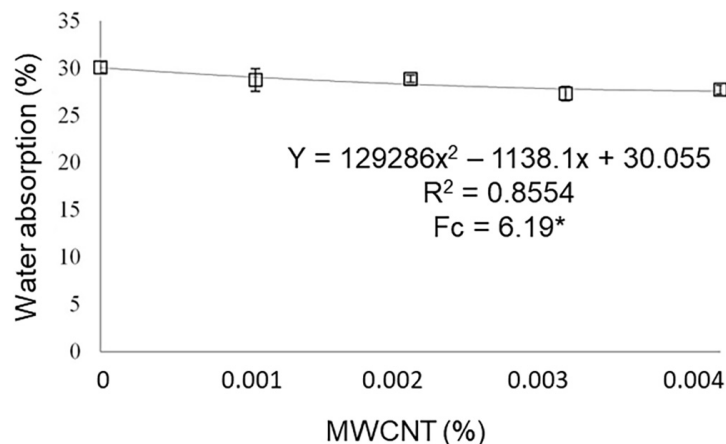


Figure 6: Water absorption after 2 hours of immersion of gypsum composites with MWCNT.

*Significant regression analysis at the 5% level.

MARCONDES *et al.* [25] studied the incorporation of 0.3% MWCNT in the portland cement matrix and concluded that the addition of nanotubes reduced water absorption by 4.4%. TOKAREV *et al.* [7] evaluated several mineral additives together with MWCNT as gypsum binders, finding less water absorption with the incorporation of 0.002% MWCNT together with 3% portland cement, corroborating the present study.

MATVEEVA *et al.* [26] evaluated the effect of carbon nanomodifier (HFNCM) on the structure and properties of gypsum with the addition of carboxylate plasticizer, finding a reduction in water absorption of 27.5%. LI *et al.* [27] studied the influence of nano-silica and silicone paraffin oil emulsion mixed with flue gas desulfurization (FGD) gypsum, and found that nano-silica influenced the affinity of gypsum with water, with a decrease in water absorption from 30 to 13.62%.

One of the biggest difficulties in using gypsum as a building material is its affinity for water, which restricts its use in dry environments [3]. Thus, the incorporation of MWCNT can increase the hydrophobicity of gypsum-based composites, expanding its use in civil construction.

3.3. Mechanical properties of composites

In flexural strength, the most significant values were with 0.001 and 0.003% MWCNT (Figure 7). According to GORDINA *et al.* [13], MWCNTs have high surface energy and crystalline structures, forming an ordered and dense structure, which can result in better mechanical properties of composites. PERVYSHIN *et al.* [14] found an increase in flexural strength and lower water absorption in composites reinforced with MWCNT, which was justified by the dense and ordered structure of MWCNT.

The proportions of 0.002% and 0.004% MWCNT did not improve the flexural strength of the composites, which can be explained by the non-uniform distribution of MWCNT in the gypsum matrix. LUDVIG [28] found that MWCNT levels above 0.005% caused a reduction in flexural strength in mortars, due to despairing in the distribution of MWCNTs in the matrix.

The study by LING *et al.* [29] revealed a significant variation in the absolute flexural strength of phosphogypsum composites with MWCNT. Flexural strength peaked at 8.7 MPa with 1% MWCNT, representing a 61% increase over the control group. However, at 2.0% MWCNT, the absolute dry flexural strength fell below that of the control group. Furthermore, with 1.5% and 2.0% MWCNT, the strength reached 6.7 MPa and 5.7 MPa, respectively, indicating increases of 24% and 6%.

Therefore, the findings suggest that the addition of MWCNT can significantly improve flexural strength, but this effect tends to decrease with increasing MWCNT concentration, and excessive concentrations can impair strength [30].

In Figure 8, it is observed that there was no increase in compressive strength with the substitution of 0.004% of gypsum by MWCNT, with the most significant value in the proportion of 0.001% of MWCNT, related to improving hydration and accelerating gypsum curing. DEREVIANKO *et al.* [8] added 0.18% of MWCNT in the gypsum matrix and observed a 30% increase in compressive strength, a value close to that found in this study. This fact occurs due to the ability of MWCNT to improve the hydration of calcium sulfate dihydrate present in the gypsum, which results in increases in the mechanical properties of the composite. MELO [31] working with nanotubes in mortars obtained increments of around 34% in compressive strength. The lowest

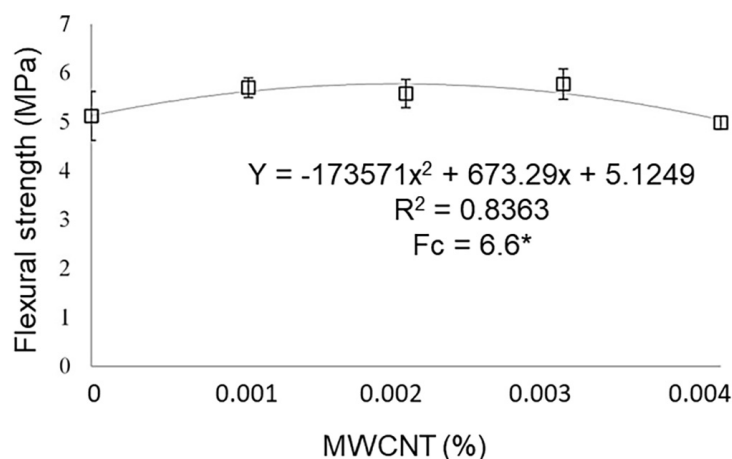


Figure 7: Mean flexural strength values of gypsum composites with MWCNT.

*Significant regression analysis at the 5% level.

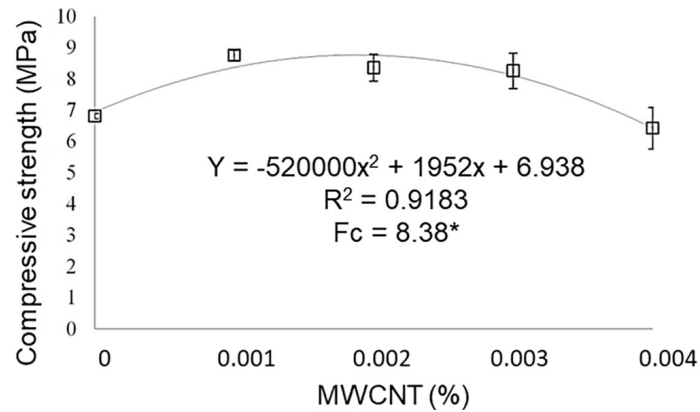


Figure 8: Mean Compressive strength values of gypsum composites with MWCNT.

*Significant regression analysis at the 5% level.

value of compressive strength was with 0.004% of MWCNT, which can be attributed to the presence of microcracks in the composites and to the format and size of the MWCNT, which causes heterogeneity and segregation in areas of the composites, making them more fragile.

According to LING *et al.* [29], the absolute dry compressive strength of phosphogypsum with MWCNT shows a tendency to increase and then reduce as the nano content increases. At 2.0% MWCNT, resistance is below that of the control group. However, at 1% MWCNT, strength peaks at 24.3 MPa, which represents a 47% increase compared to the control. With 2.0% MWCNT, the strength drops to 11.5 MPa, indicating a 30% reduction. Thus, although MWCNT can increase the absolute dry compression strength of phosphogypsum, this effect tends to decrease with increasing MWCNT concentration and may even become unfavorable when the concentration is excessively high.

3.4. Microstructural analysis

In the reference samples (Figure 9A), elongated, prismatic and needle-shaped gypsum crystals are seen, and a greater amount of empty spaces in relation to the composite with 0.002% MWCNT (Figure 9C). Thus, the

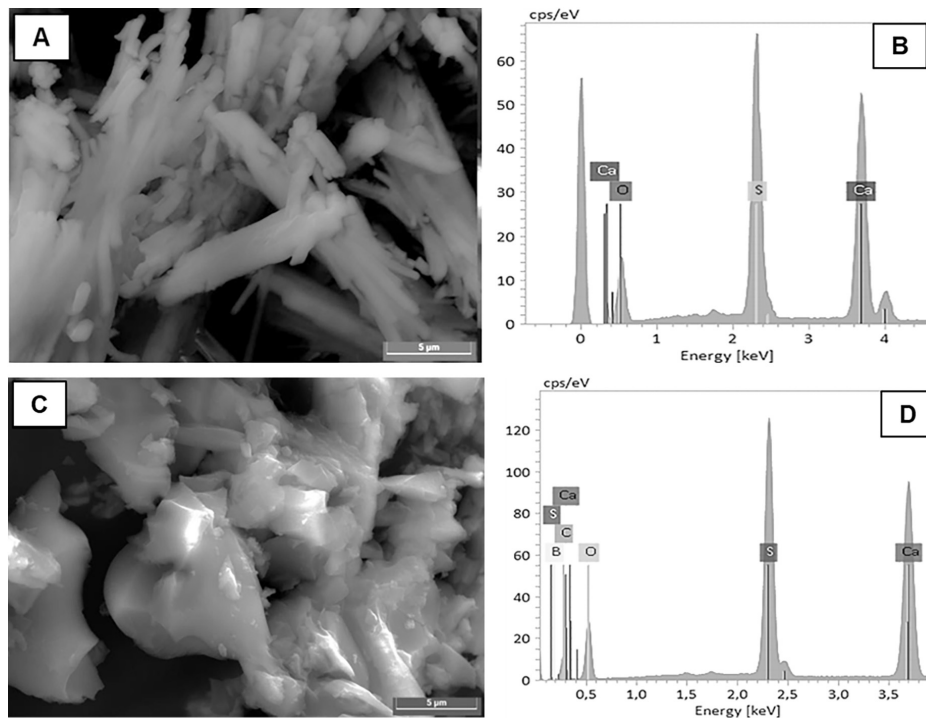


Figure 9: Micrographs: A) SEM of the composite with 0% MWCNT; B) EDS of the composite with 0% MWCNT; C) SEM of the composite with 0.002% MWCNT; D) EDS of the composite with 0.002% MWCNT.

addition of MWCNT modified the morphology of the gypsum crystals, resulting in changes in the microstructure and a decrease in the porosity of the composites, leaving the material denser, with less water absorption and greater compressive strength.

In the EDS analysis of the composite with 0% MWCNT (Figure 9B) the presence of constituent elements of the crystalline phases corresponding to gypsum β was verified, presenting characteristic peaks for the elements calcium, oxygen and sulfur. For the composite with 0.002% MWCNT (Figure 9D), peaks can be seen that demonstrate the presence of carbon, the main constituent of MWCNT, and also the presence of boron, which, according to YUDASAKA *et al.* [32] and ALI *et al.* [33], can be attributed to the existence of impurities resulting from the catalysts used during the MWCNT synthesis process.

4. CONCLUSIONS

The analysis of the effects of incorporating multi-walled carbon nanotubes (MWCNT) into the gypsum matrix revealed significant results that point to the predictions of these replacements in civil construction. Research has shown that water absorption was reduced by 8.33%, showing a specific improvement in the impermeability properties of the gypsum. With a concentration of 0.004% of MWCNT, there was an increase of 3.21% in density, resulting in a more robust material. The composites containing 0.001% and 0.003% MWCNT showed flexural strength of 6 MPa, while the compressive strength increased to 9 MPa with the insertion of 0.001% MWCNT, compared to 7 MPa in the control.

These results suggest that the addition of MWCNT not only improves the physical and mechanical properties of gypsum, but can also expand its application in civil construction, making it a more efficient and versatile material. Therefore, research in this field is promising and can significantly contribute to innovation in construction.

The production and functionalization of carbon nanotubes can be expensive, which can increase the final cost of the composite. However, the composite can be used in coatings, panels and structures that require greater resistance to flexion and compression, as well as less water absorption. The strength and lightness of composites make them suitable for prefabricated elements, reducing weight and improving durability. Therefore, homogenization of the mixture can be challenging, especially at higher MWCNT concentrations, which can affect the distribution and effectiveness of the material.

5. ACKNOWLEDGMENTS

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