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Contribution of the use of nanomaterials in cement matrix through additive manufacturing aiming at sustainable construction

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ABSTRACT

Reducing the environmental impact and complying with the social commitments, as well as respecting the environment to promote sustainability in production processes has been debated in various sectors and, in construction processes, technology is an ally, as automation and the insertion of components into materials, there is a decrease in waste and an improvement in the quality of buildings. Therefore, this study aimed to evaluate the constructability and extrudability in civil construction by inserting graphite and graphene oxide in a cementitious matrix. The cementitious filaments produced were evaluated for physical, Chemical, and mechanical parameters. The results indicated that the insertion of both graphite and graphite oxide in the cementitious matrix did not interfere with the extrudability and constructability of the material. Values of 9.34 MPa, 8.99 MPa, and 8.91 MPa were obtained for flexural tension in the samples added with 0.04% and 0.1% of graphite and 0.04% of graphene oxide, respectively. When evaluated for axial compression, the samples containing 0.1% and 0.2% graphene oxide and the reference sample had 127,546.00 MPa, 127,350.05 MPa, and 126,844.12 MPa, respectively. The sample with 0.1% graphite showed a positive behavior in relation to the tensile test in flexion, compression, and less deformation after printing, making it feasible to add nanomaterials in cementitious filaments for additive manufacturing to produce more resistant materials.

KEYWORDS: Civil construction. Cementitious filament. Constructability. Extrusion.

1 INTRODUCTION

In light of many ongoing constructions, the construction industry's impact on ecosystems has become an important issue (ZOLFAGHARIAN et al., 2012). These adverse environmental impacts such as waste, noise, dust, solid and toxic waste, air pollution, water pollution, bad smell, climate change, land use, operation with vegetation, and dangerous emissions, have been significant and irreversible to the environment, in addition to becoming one of the most prominent explorers of renewable and non-renewable natural resources (KAUR, 2012).

According to data from the World Watch Institute (2022), civil construction consumes 40% of raw stones, gravel, and sand and 25% of wood per year. It also consumes 40% of energy and 16% of water annually. The extraction of natural resources is responsible for irreversible changes in the natural environment of the countryside and coastal areas, both from an ecological and landscape point of view. And the consequent transfer of these areas to other geographically dispersed locations culminates in greater energy consumption and a more significant amount of particulate matter dispersed into the atmosphere. The extraction and construction of raw materials also contribute to the accumulation of pollutants in the atmosphere (LIMA *et al*., 2021).

Dust and other emissions which include toxic substances such as nitrogen and sulfur oxides are also constantly released. These emissions occur during the production and transportation of materials and site activities and are responsible for severe environmental threats (KAUR, 2012). Negligence in the construction environment still reflects severe consequences for the biosphere and toxic spills are reaching groundwater systems and reservoirs (LIMA *et al.*, 2021). According to Lima *et al.* (2021), about 30% of the land in the affected area is experiencing degradation, further depleting the environmental quality along with the presence of pollutants. In addition to that, a large volume of solid waste is generated.

For Wu *et al.* (2021), a significant portion of construction waste is unnecessary, and many construction and demolition materials possess a high potential for recycling and reuse. Nevertheless, the recycling of construction waste is a time-consuming activity and the lack of environmental awareness among professionals in the area creates significant barriers. As a result, most of the recyclable material ends up being disposed in landfills. The authors also claim that a waste management plan could reduce site waste by up to 15% and save about 50% in waste handling costs (WU *et al.,* 2021).

Lands that were once cultivated have been transformed by civil construction or lost due to the extraction and mining of raw materials. Forests have been cleared up in order to provide wood to be used either to supply energy for the manufacture of materials or even directly as a basis for construction. Deforestation and the burning of fossil fuels directly contribute to global warming and air pollution. Furthermore, the construction industry has been regarded as a significant energy consumer, and the use of finite fossil fuel resources significantly contributes to carbon dioxide emissions (MARLON *et al.*, 2019; THOMAS *et al.*, 2020; LIMA *et al.*, 2021).

In the context of sustainable development, it is essential for industries to strive for increased efficiency, utilizing fewer natural resources and minimizing the pollution they cause. Several natural resources are used during the execution of a construction project, resources that involve energy, land, materials, and water (MARLON *et al.*, 2019). The operation of construction equipment also contributes to the consumption of natural resources such as electricity and diesel oil, which, when not properly managed, can further contribute to pollution.

However, as pointed out by Daniyal *et al*. (2018), it is possible to optimize natural resources and promote environmentally-friendly practices characterized by sustainable planning, construction, and occupation that consider the well-being of ecosystems. The construction industry itself can play a significant role in the management of solid waste through practices such as reuse and recycling. This demonstrates that while the civil construction industry may use and degrade a substantial portion of the environment, it also has the potential to implement measures that mitigate these impacts.

Due to the growing demand for more sustainable practices, significant investments in research have taken place, mainly due to the fact that these more efficient materials can optimize the properties of the final product and contribute to the preservation of natural resources. Additionally, innovative processes can enhance efficiency and performance (DANIYAL *et al*., 2018). Among these processes, additive manufacturing, also known as 3D printing, has emerged as an efficient method for creating objects using digital models that enable the sustainable production of new products. As it is a recent and little-explored technology, one of the biggest challenges of 3D printing in the construction sector is the capability to extrude a cement matrix with the inclusion of nanomaterials for printing parts, structures, and even the building as a whole (BHARDWAJ *et al*. al., 2019).

The application of nanomaterials in these cementitious matrices has been thriving since it enables the production of more resistant materials with excellent durability. It enables the adsorption of CO2 from the atmosphere. Additionally, it contributes to the development of more durable products, reducing the need for interventions in buildings and, consequently, reducing the generation of waste that is released into the environment (DANIYAL *et al.*, 2018). Furthermore, the production of sustainable materials and the implementation of environmental management systems and corporate social responsibility are actions that are also committed to the 2030 Agenda and to the fulfillment of the United Nations Sustainable Development Goals.

These actions are capable of reducing the environmental impacts (DUBRAVSKÁ *et al.*, 2020; WU et al., 2021).

In the coating mortar, the incorporation of nanomaterials aims to achieve improved mechanical results to reduce pathologies in civil construction (DANIYAL *et al.*, 2018). Moreover, it enables the production of structures with enhanced quality and cost savings due to material reduction. This is what happens with the use of graphene oxide, which can improve the structure of the cementitious matrix by increasing its permeability to resist chemical attacks (POKHREL *et al.*, 2018). In addition, the inclusion of graphite oxide and graphite to cement mortar improves compressive strength, tensile strength, and flexural strength (TUFAIL *et al.*, 2022).

Considering the additive manufacturing triad, nanomaterials, and environmental preservation, the purpose of this study was to develop a cementitious filament with graphite and graphene oxide insertion through additive manufacturing.

2 METHODOLOGY

2.1 Raw materials

To conduct this research, Portland cement CPII Z32 (Votoran) was used, with a specific mass ranging from 900 to 1200 Kg/cm3 at 20°C, along with medium sand as fine aggregate. The sand was obtained from a local supplier and sieved using a 4.75 mm mesh, following the guidelines of the Brazilian Association of Technical Standards (ABNT) NBR NM - ISO 3310-1 (2000). The specific mass of the sand, as per ABNT NBR 16916 (2001) was measured at 2631 Kg/cm3.

The powdered graphene oxide was purchased from the brand Amazonas Grafeno. The high-purity graphite powder used was from the Sigma-Aldrich brand. A 0.04% polycarboxylate-based superplasticizer was used, commercially available as Master Glenium 51®, and purchased locally.

2.2 Sample preparation

The unitary ratio employed for both the cementitious filaments and the reference sample was 1:1.33 (cement to sand). A polycarboxylate-based superplasticizer, available as Master Glenium 51®, was used in a proportion of 0.04% by weight of the cement. The water-to-cement ratio was set at 0.45g.

A total of six cement filaments were prepared, as outlined in Table 1.

Table 1 - Composition of the reference sample and modified samples

Samples	Cement (Kg)	Sand (Kg)	Superplasticizer (g)	Water (L)	Graphene oxide (g)	Graphite (g)
REF	45	60	45	13.260	0	0
A	45	60	45	13.260	45	0
B	45	60	45	13.260	90	0
C	45	60	45	13.260	15	0
D	45	60	45	13.260	0	45
E	45	60	45	13.260	0	90
F	45	60	45	13.260	0	15

Source: The authors, 2022.

The sample preparation followed the guidelines of ABNT NBR 13281 (2001). The incorporation of graphene oxide and graphite into the cementitious filament was performed by manually dispersing them directly into the dry mixture of sand and cement. Initially, the dry materials (cement, sand, graphene oxide, and graphite) were separated and weighed using a Max Dst-30 scale by Triunfo - Brazil (Table 1). Subsequently, they were inserted into a 100L capacity mixer (MIX 90 from Betomaq - Brazil) and the equipment was operated for five minutes to ensure homogenization of cement, sand, graphene oxide, and graphite. After mixing, two-thirds of the water was added and mixed for five minutes, followed by the addition of the remaining water along with the superplasticizer additive. The mixer was then operated for additional five minutes, resulting in a total of 27 minutes to achieve complete homogenization of the mixture. Once the filaments were prepared, they were transferred to the injection pump subsystem and transported to the printer nozzle outlet through hoses, initiating the extrusion process, as described by Silva (2022).

2.3 Printing of samples

The printing process was carried out at a flow rate of 5 kg/min and at a speed of 50 mm/s, following the parameters of 200 mm width and 250 mm length for each sample. The constructability analysis assessed the height that each layer could support, and it was determined that the maximum printing height supported by the equipment was 165 mm.

After preparing the samples and subjecting them to the pumping and extrusion process, they were characterized in their fresh state through consistency index tests (ABNT NBR 13276/2016), extrudability, and constructability. The samples were collected from the extruder nozzle and placed in a metallic container. Tests were conducted on both the fresh state (consistency index) and the hardened state (flexural tensile strength and axial compression).

Mechanical parameter tests (flexural tensile strength and axial compression) were performed on the samples after 28 days of curing using DL 30000-EMIC equipment, in accordance with ABNT NBR 1327/20058 and 13279/2005 standards.

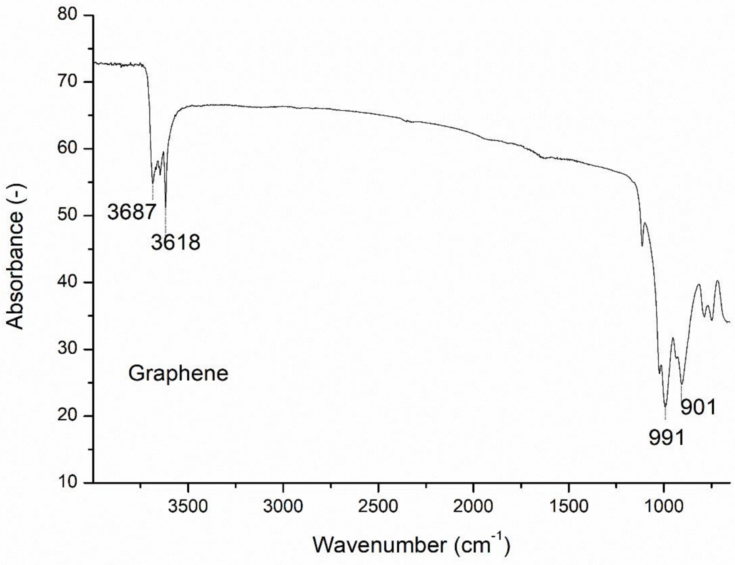
Statistical analyses were conducted using the desirability function, proposed by Derringer and Suich in 1980 (COSTA *et al.*, 2011). Tukey’s test was employed at a 5% significance level for statistical analyses of tensile strength, compression, deformation, and consistency index. This test was used to determine if the samples differed significantly from each other or maintained the same average as the reference sample.

3 RESULTS

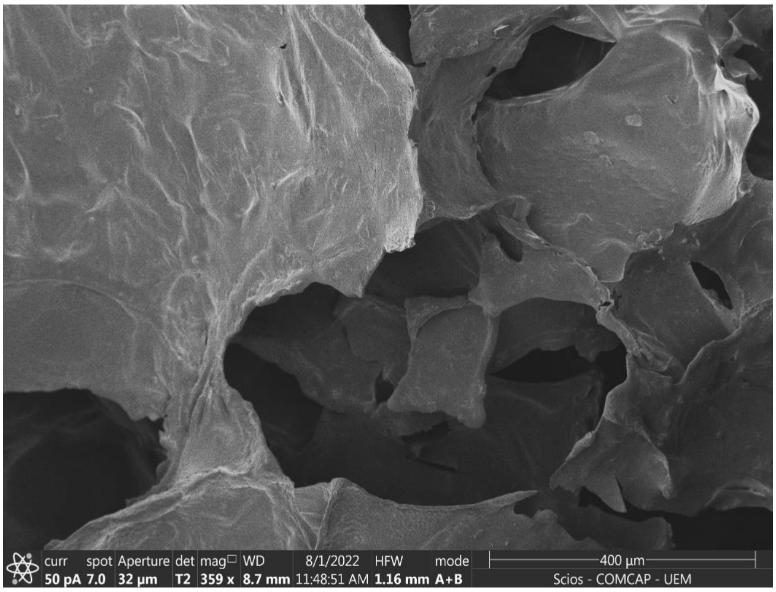
The FTIR analysis revealed a distinctive characteristic, with two bands observed above the 3600 cm-1 regions, at 3618 cm-1 and 3687 cm-1 (Figure 1 (a)).

Gong *et al.* (2015) compared graphite oxide (GO), reduced graphene oxide (rGO), and nitrogen-doped graphene (NG). In their study, they verified: a broad band at 3338 cm-1 in GO, which corresponds to the intense stretching of the OH group; absorption peak at 1635 cm-1 due to C ¼ C elongation mode; found bands at 1716, 1154, and 1033 cm-1, which correspond to the stretching methods of C ¼ O, C–OH and C–O, respectively. As for the rGO molecules, no absorption was observed in the region of 3338 cm-1, which reveals the absence of the OH group after reduction. Furthermore, a band at 1586 cm-1 was attributed to C ¼ C; they found no change in absorption at 1164 cm-1 attributed to C–OH.

Figure 1 - (a) Graphene oxide FTIR analysis (b) Graphene oxide morphology



(a)



(b)

Source: The authors, 2022.

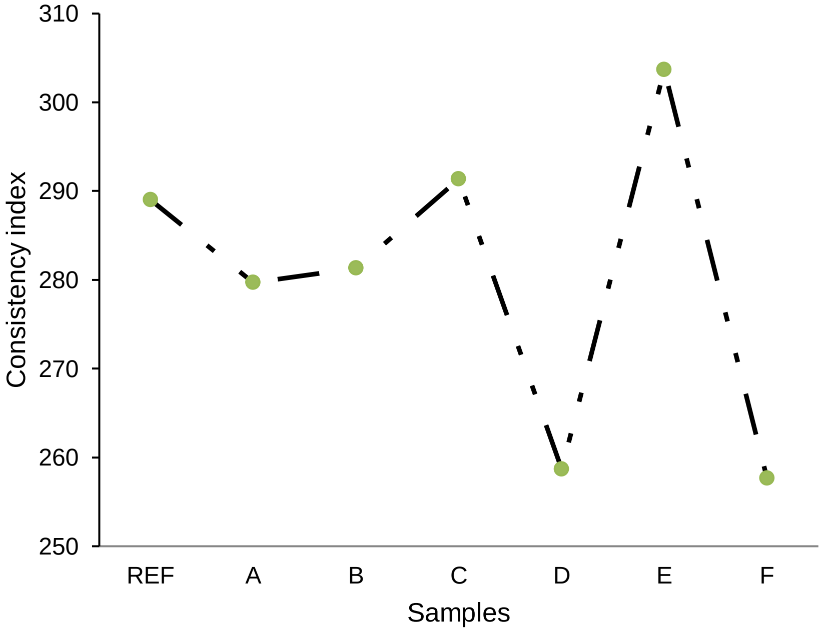
The presence of GO leaves can be observed (Figure 1 (b)). There is a roughness resulting from the increase in the spacing between the sheets, indicating an oxidation process of the material.

The specific mass of the Portland cement used, as determined by the manufacturer, ranges from 900 to 1200 kg/cm^3 at 20°C. The physical characteristics obtained for the fine aggregate align with the recommended values for use in a cementitious filament. The granulometric composition presents the percentage of fine aggregate within a granulometry range of 9.5 mm to 0.15 mm, which is ideal for the composition used in coating mortar, facilitating the pumping and extrusion of the cement filament.

The consistency index test was carried out to evaluate the fluidity of the cement paste and the pumping capacity. This well-known test was performed on the reference sample (REF), and the modified samples incorporated with graphene oxide [A (0.1), B (0.2), C (0.04)] and graphite [D (0.1), E (0.2), F (0.04), according to Table 1].

In Figure 2, it is evident that the samples incorporated with graphene oxide in the proportions 0.1 (A) and 0.2 (B) experienced a reduction in the fluidity of the cement paste but did not significantly differ in the consistency index. Similarly, the samples incorporated with graphite in proportions 0.1 (D) and 0.04 (F) also did not show significant differences. According to Song and Li (2021), graphene oxide tends to reduce fluidity due to the formation of agglomerates of calcium cations by chemical crosslinking, which traps free water and consequently reduces fluidity. Additionally, depending on the size of the nanomaterial, it may interfere with the progression of the hydration reaction and, consequently, with the extrusion of the filament in the printer and the curing of the mass (ZHOU *et al*., 2020). As for graphite, its size and shape can affect its incorporation into the sample, potentially reducing the workability of the sample (MEDINA *et al.*, 2018).

Figure 2 - Variation of the consistency index for the samples



Source: The authors, 2022.

Sample REF; Sample with Graphene Oxide 0.1% (A); Sample with Graphene Oxide 0.2% (B); Sample with Graphene Oxide 0.04%(C); Sample with Graphite 0.1% (D); Sample with Graphite 0.2% (E); Sample with Graphite 0.04% (F).

The consistency index for the sample incorporated with 0.2 graphite (E) was determined to be 303.67 mm ± 1.20 mm (Figure 2), showing an increase of approximately 17% in the consistency index. It was observed by Tufail *et al*. (2022) which showed an increase in the fluidity of the paste due to the lubricating effect of nanomaterials on the solid particles of the cement paste, thus improving fluidity and reducing the use of superplasticizers.

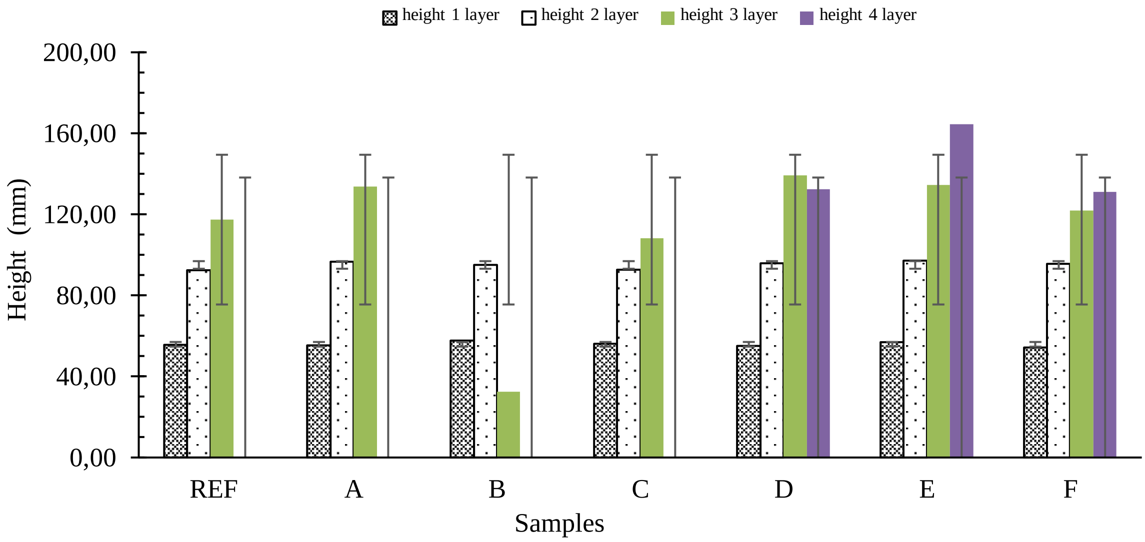
Regarding extrudability, all samples were successfully extruded by the printer, with an average open time of approximately 4 minutes and 40 seconds to construct four layers when it occurred. This open time is an important fact, as it can interfere with the workability of the mixture during subsequent printing (SONG; LI, 2021).

To assess the constructability of the filament, its printing capacity was evaluated. As there is no standardized method for testing constructability in 3D printing, several methodologies were tested and compared in this vertical deformation study, as well as Zou *et al.* (2021) and Ma *et al*. (2018). The results showed that all samples were printed up to the third layer. However, samples modified with graphite, D, E, and F supported up to the fourth layer, with maximum print heights of 157.73, 164.56, and 156.24 mm, respectively, as shown in Figures 3 (a) and 4.

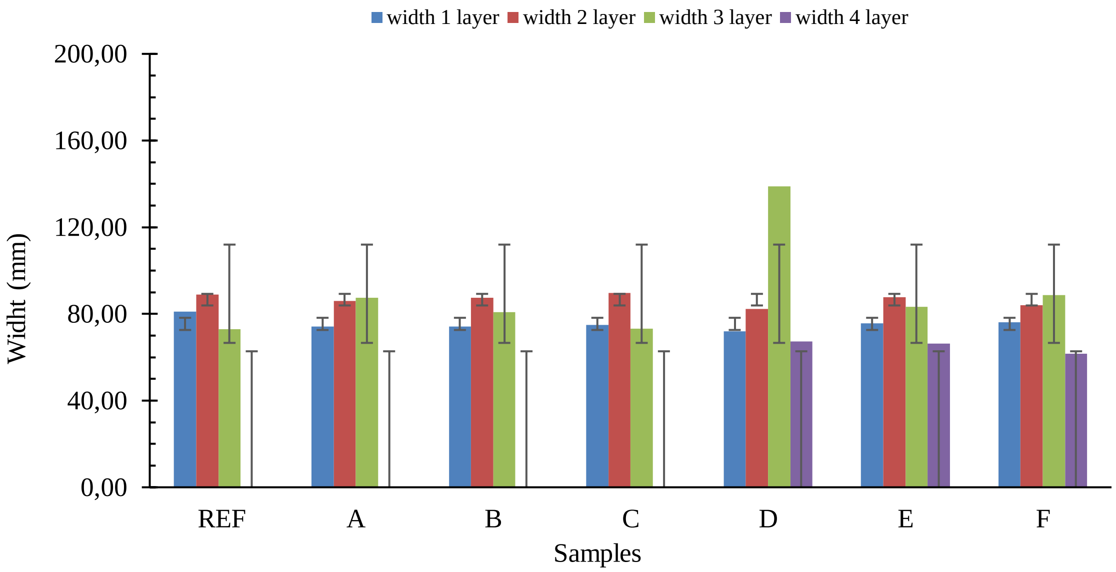
Analyzing the data, it was observed that there were no significant differences in the height and width of the first layer among all filament samples. The same applied to layers 2, 3, and 4 concerning height, except samples REF, A, B, and C did not have any height in the fourth layer. According to Kazemian *et al*. (2017), 3 primary sources are responsible for the deformation applied to a printed layer: the extrusion pressure, the action of its weight, and the importance of the subsequent addition of new layers. In this case, the deformation was due to its weight's activity and other layers' addition.

For layer 2 widths, significant similarities and differences occurred in the third layer. In the fourth layer, there was also no printing in the width. Comparing the relationship between the layers for each of the samples, what was expected happened, that is, with each new layer, a new height occurred with a significant difference, except layers 3 and 4 of samples E and F due to a significant deviation, as shown in Figure 3 (b) and Figure 4. For the width, sample D showed the same significant average between the 3rd and 4th layers with a large deviation for the 3rd layer. In the width of sample F, only the 1st layer differed from the others. The lowest height occurred for sample A (55.34 mm ± 0.63 mm) and the highest (164.56 mm ± 4.53 mm) for sample E. Smallest width in the first layer for sample E (71, 90 mm ± 0.72 mm) and the largest in layer 3 for sample D (104.06 mm ± 13.71 mm).

Figure 3 - Results of the constructability test: a) height; b) width



(a)



(b)

Source: The authors, 2022.

Sample REF; Sample with Graphene Oxide 0.1% (A); Sample with Graphene Oxide 0.2% (B); Sample with Graphene Oxide 0.04%(C); Sample with Graphite 0.1% (D); Sample with Graphite 0.2% (E); Sample with Graphite 0.04% (F).

The total average vertical deformations for all layers were 37.69%, 36.67%, and 38.62%, respectively, with samples D, E, and F successfully distinguishing themselves from the others and indicating the positive effect of graphite in improving constructability.

To analyze the influence of graphite and graphene oxide on the mechanical properties of the samples, tensile tests were carried out in flexion and immediately after the axial compression test.

From the results presented in Figure 5 (a), in which the significance of the flexion traction test was analyzed, it is possible to verify that there were no significant differences between the samples added with graphite and graphene oxide in the proportions 0.1%, 0.2%, and 0.04%, respectively. Regarding the average, the three best results were: the highest traction (9.34 MPa) was observed in sample F, in sequence sample D (8.99MPa) and sample C (8.91MPa), and the lowest average of traction (7.57 MPa) was in sample A, a difference of about 8% about the effectiveness of graphite (lower percentage). The reference sample showed more significant deviation about specimens 1, 2, and 3 but did not differ significantly from the others.

The increase in flexural tensile strength with the addition of graphite (Figure 5 (a)) is attributed to the synergistic effects of this material with the cementitious paste. In turn, the reduction in bending may be linked to the agglomeration of the plates, as nanomaterials with high specific areas are subject to accretion by secondary interactions, which may cause ineffective dispersion of particles and consequently cause an increase in internal pores and porosity (TUFAIL *et al*. 2022).

For the analysis of resistance to axial compression, the samples of graphite and graphene oxide, as well as the reference, did not show significant differences (Figure 5 (b)); this can be because the samples reached the limit of the force of the machine of the test and do not break. Sample E showed the lowest compression and the highest deviation of the other samples (9.69 MPa ± 1.58 MPa).

Figure 4 - Images of the constructability test

ARG REF A B C

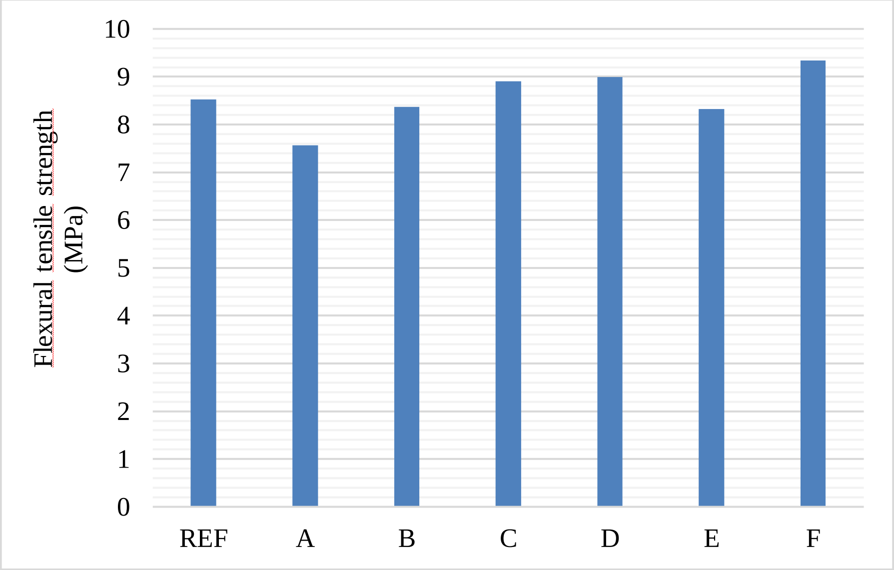
D E F

Source: The authors, 2022.

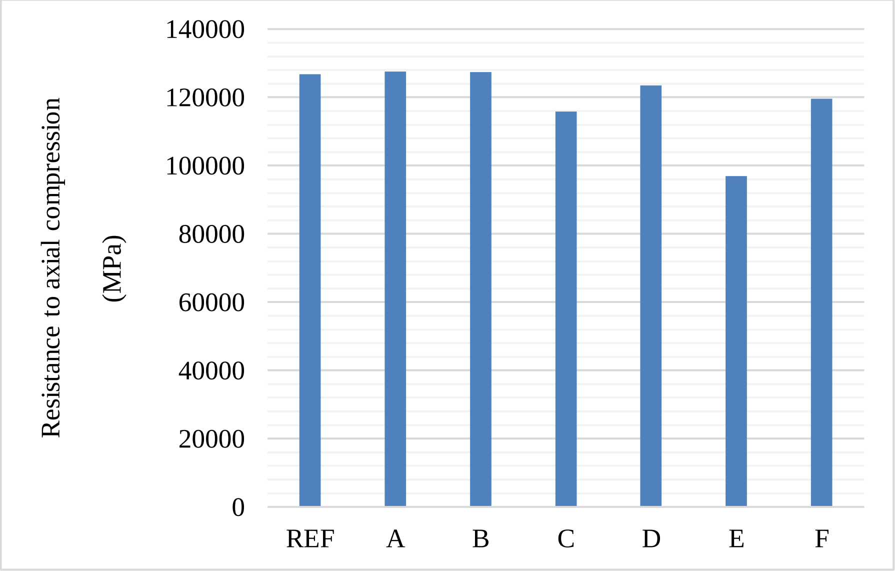
Arg. REF Arg. Graphene Oxide 0.1 (A) Arg. Graphene Oxide 0.2 (B) Arg. Graphene Oxide 0.04 (C) Arg. Graphite 0.1 (D) Arg. Graphite 0.2 (E) Arg. Graphite 0.04 (F).

Figure 5 - Test results: a) Flexural tensile strength; b) resistance to axial compression

(a)



(b)



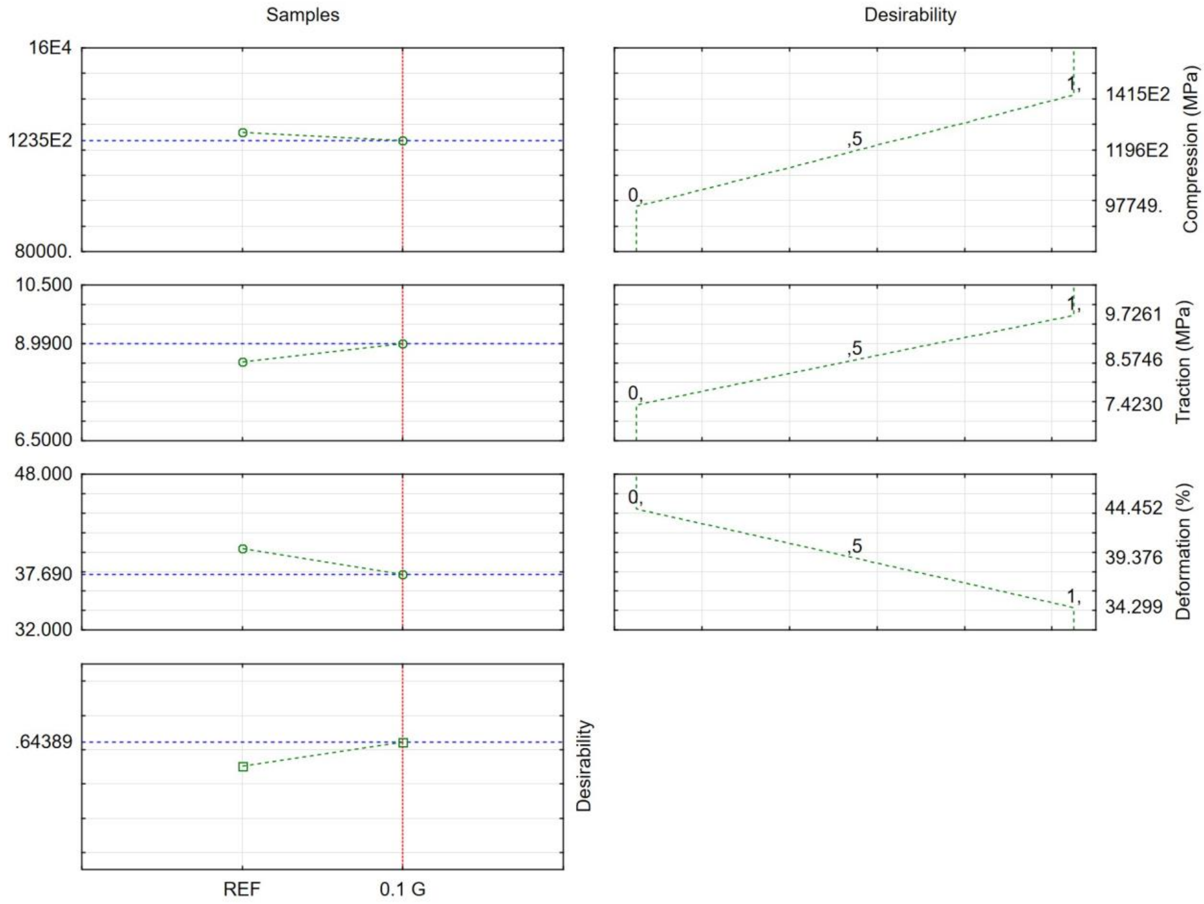
Source: The authors, 2022.

Sample REF; Sample with Graphene Oxide 0.1% (A); Sample with Graphene Oxide 0.2% (B); Sample with Graphene Oxide 0.04%(C); Sample with Graphite 0.1% (D); Sample with Graphite 0.2% (E); Sample with Graphite 0.04% (F).

Wang *et al*. (2013) and Song and Li (2021) also found an increase in flexural strength and compression in cement pastes with the addition of GO, even with the reduction in fluidity; this is because graphene oxide promotes a robust interfacial interaction in the carboxyl groups and hydration of the cementitious filament (SILVA *et al*., 2021).

In addition, as nanomaterials have an ultrafine nature, they act by filling the pores between the cement grains, promoting a denser microstructure and better resistance to penetration of harmful material, thus resisting the detrimental forces of the environment (SONG; LI, 2021).

Figure 6 - Result of statistical analysis and feasibility of results



Source: The authors, 2022.

In this way, the statistical analysis and viability of all the mechanical and deformation test results can be verified (Figure 6) that sample D was the best sample, presenting the best results in the flexion traction test, compression, and less deformation after printing. Applying the desirability function in optimizing analytical methods brings numerous advantages, such as efficiency, economy, and objectivity in optimizing procedures with multiple responses (CANDIOTI *et al.*, 2014), making it possible to analyze the feasibility of applying the mixtures performed.

4 CONCLUSION

Based on the analysis of the results, it was verified that the printer extruded all the samples, and the samples modified with graphite had better results in extrudability, as they supported up to the fourth layer of printing. The modified samples had different results from the reference sample in the hardened state; sample F, sample D, and sample C had the best results in the flexural tensile test. In the axial compression test, the samples added with graphene oxide had the best results about the reference sample because it contributes to the hydration process and fills the pores of the cementitious matrix.

Through statistical and feasibility analysis, sample D was the best sample, presenting the best results in the tensile test in flexion, compression, and less deformation after printing.

In this way, it becomes feasible to use cementitious filament reinforced with graphene and graphite oxide for application in additive manufacturing in the civil construction sector since it is possible to point out that the new cementitious matrix will contribute to economic, social, and environmental development. Consequently, it will strengthen the Sustainable Development Goals (SDGs) to foster sustainable growth and justify the adoption of this technology and its cleaner processes.

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