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Comparative Study between Nanoparticles Incorporated with Magnetite added in Concrete for Civil Construction

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Abstract

Civil construction considerably developed in the last years and technology has become an ally in the quality improvement of the materials, mixtures and structures. Reinforced concrete, the main construction method employed, admits variations in properties and strengths according to the composition and additives adopted, aiming to meet project specifications. Increasingly, nanotechnology has been inserted as an application for the engineering to improve properties such as the porosity and mechanical resistance, mainly due to characteristics of high surface area and nanometric dimension. This study evaluates the effect of added magnetite derivative nanoparticles in the concrete for comparative purposes of mechanical strength. In this context, graphene oxide (GO) and nanosilica (NS) were synthesized and, together with graphite nanosheets (GR) and nanocellulose (NC), incorporated with magnetite (Fe_3O_4) in the proportions 1:5 and 1:10 (in mass: mass ratio) (Figure 1). The nanoparticles were characterized by the methods XRD and FTIR. When added in concrete, in the 0.03% proportion by weight of the cement according to the referring regulations, tests of workability (Slump Test) and resistance to the compressive forces were carried out. The Slump Test demonstrates that the mixture remained workable in acceptable way in all samples. The compression test showed that NC incorporated with magnetite reduced the mechanic resistance by up to 11% when compared to the reference, while GR magnetite incorporated in the proportion 1:5 increase the resistance values in 8%, as well as the NS, $NS \cdot Fe_3O_4$ 1:10, GR and $GR \cdot Fe_3O_4$ in a 1:10 proportion, also resulted in significant increases.

Keywords: Engineering; Cement; Resistance increase; Nanoparticles

Introduction

The environment of civil construction mobilizes several sectors of material supply, production, commercialization, and manpower. Pastes used in civil works have a common component, Portland cement, which, added to the aggregates, additives, and water from the concrete; When combined with steel bars it gives rise to reinforced concrete. Among structural design options, reinforced concrete is the most used arrangement in Brazil [1]. Different compounds have been included in reinforced concrete aimed to improve their basic properties, such as workability and durability. In addition, another important property that can be enhanced is mechanical resistance, considering that an increase in strength can reduce steel consumption as well as construction costs [2]. Thus, nanoscience and nanotechnology are useful tools for the synthesis and modification of compounds in order to produce stronger, more flexible, and stable materials [3,4]. Additionally, nanocomposites are promising materials, as the combination of two or more compounds can reduce the individual limitations [5]. At the same time, magnetic nanocomposites have potential application in civil construction due to the compatibility and expansion after the formation of cracks, filling the empty spaces [6]. It is well reported that the mechanical resistance of concrete, an important parameter for civil construction, can be improved with the use of carbon nanomaterials, nanosilica, titanium dioxide, iron oxide, calcium hydroxide, and zeolite [7]. This increase is mainly related due to effective pore filling of the material, considering the nanometric dimension of the NPs [8]. Graphene oxide (GO) is an oxidized product of graphite formed by a honeycomb-like carbonic network with numerous oxygenated functional groups on the surface. This nanomaterial presents potential properties for addition to concrete that makes it possible to improve the characteristics (mechanical resistance and durability) [9,10]. Recently, studies reported that the GO may increase 33% and 58% the compressive resistance and cement paste bending, respectively. This increases concerns about the interaction between the empty spaces (concrete) and GO, hydration reaction, as well as a strong bond with carboxylic groups [11]. The addition of graphite nanosheets and carbon nanofibers in high-performance concrete resulted in an increase of 187% in absorption capacity, 56% in tensile resistance in compression, 59% in flexural tensile strength, and 276% in tenacity, using 0.3% of nanoparticles [12]. Tabatabaei [13] reported that the production of graphite nanosheets emits 80% less CO_2 than cement. At the same time, the incorporation of 5% nanoparticles in material can reduce the effects of global warming by 21%. Another material with the potential to replace the amount of cement is nanosilica due to its low cost and sustainable source. This nanoparticle reacts with calcium hydroxide during the hydration of Portland cement, not only increasing the resistance and pore reduction but also decreasing the pH, which is related to the protection of reinforcement against corrosion. Therefore, this process densifies the microstructures, reducing the rate of entry of water and chloride ions [14]. The incorporation of polymeric nanoparticles into cement can also improve corrosion resistance and decrease permeability and thermal conductivity. Studies revealed that nanocellulose can alter the fracture behavior of cement pastes. By adding 0.2% and 1.0% of cellulose nanocrystals to the cement, the compressive strength increased by 10% and 17%, respectively. These results can be explained by the surface area of the nanoparticle as well as the transfer of tension with cement components [15]. Considering that only few studies reported the use of nanoparticles in reinforced concrete, this work aimed to investigate the influence of the incorporation of nanoparticles and magnetic nanocomposites $(NC \cdot Fe_3O_4, NS \cdot Fe_3O_4, , GR \cdot Fe_3O_4 \text{ and } GO \cdot Fe_3O_4)$ and the effect of the amount of magnetite in composite material on the mechanical resistance.

Methodology

Synthesis and characterization of Magnetite Incorporated Nanoparticles

Table 1: Samples definitions.

Nanoparticle	GO	$GO \cdot Fe_3O_4$	GR	$GR \cdot Fe_3O_4$	NS	$NS \cdot Fe_3O_4$	NC	$NC \cdot Fe_3O_4$
Code	X	1:5 1:10	X	1:05 1:10	X	1:5 1:10	X	1:5 1:10

The graphite and nanocellulose were purchased commercially from Sigma-Aldrich and Delaware, respectively. The graphene oxide and nanosilica was synthesized at Laboratório de Materiais Magnéticos Nanoestruturados (LaMMaN). (Table 1) shows the informations of nanomaterials and magnetite incorporation proportions. Graphene oxide (GO) was synthesized according to Salles et al. [10]. Graphite in flakes (1 g) (Sigma-Aldrich ®) and 60 mL of 98% H_2SO_4 (Synth ®) were added in an erlenmeyer, under magnetic stirring (120 rpm). The mixture remained under stirring for 20 minutes at room temperature. 6 g of $KMnO_4$ (Synth ®) was slowly added over 10 minutes. Sequentially, the reaction was heated to 40 °C and kept under stirring for 5 h. After this time, 180 of distilled water was added in the reaction and kept under stirring for 10 h at room temperature. Finally, the solution was heated at 40 °C for 1 h and added 300 mL of distilled water and 10 mL of H_2O_2 35% (Synth ®) to reduced Mn (VII) species. The product was washed consecutives times with distilled water and dried at 50 °C. The nanosilica synthesis was carried out according to Ong et al. [16]. The process was separated into three steps: acid leaching, calcination, and top-down process to reduce particle size. The incorporation of magnetite into (NS, NC, GO, and GR) were accomplished according to the methodology described by Rhoden et al. [17]. In a 250 mL round-bottom flask containing 100 mL of ultrapure water previously deoxygenated, 100 mg of (NS, NC, GO, and GR) were added singly with different amounts of iron chloride II ($FeCl_2$) (Sigma-Aldrich ®) i.e., 500 mg for $(NS \cdot Fe_3O_4 \text{ 1:5}, NC \cdot Fe_3O_4 \text{ 1:5}, GO \cdot Fe_3O_4 \text{ 1:5 and } GR \cdot Fe_3O_4 \text{ 1:5})$, 1000 mg for $(NS \cdot Fe_3O_4 \text{ 1:10}, NC \cdot Fe_3O_4 \text{ 1:10}, GO \cdot Fe_3O_4 \text{ 1:10 and } GR \cdot Fe_3O_4 \text{ 1:10})$. Then, the pH of the middle was adjusted for ($pH \approx 9.0$), with ammonium hydroxide (Synth ®) allowing the creation of oxidant middle, favoring the iron incorporation. Afterward, the mixture was submitted alternately to ultrasonic irradiation (Elma, power 150W) and heating (50 °C) for 180 minutes. When the sample showed magnetic field responsiveness, the solution was poured with the assistance of acetone (Synth ®). Lastly, the material was dried in an oven (DeLeo) at 50 °C for total evaporation of the solvent.

Concrete Assay

(Table 2) shows the proportions and information which were used to release to experiments (compress tensile strength and slump teste). The specifications: Cement Portland II with filler addition II (CP II F) Votoran; water treated to Water Treatment Plants of region; sand with dimension of 1.2 mm. Gravel 0 (between 4.9 and 9.5 mm); Gravel 1 (between 9.5 and 19 mm); multifunctional additive Grace (GCP); nanoparticles describe in (Table 1). The concrete dosage was employed by Brazilian Regulation [18]. For the experiments with nanoparticles, 0.03%

(mass related) of NPS in cement. According to the literature, increments higher than 3% result in more resistance. However, non-represent compensatory gain, and with this value the nanoparticles can agglomerate, harming the filling of cracks [11].

Table 2: Concrete dosage.

Dosage to 20 L - 0,5 m^3 - relation water/cement 0,55 - total weight 46,20 kg							
Material	**Brand**	**Specific mass kg/m^3**	**Unit weight kg/m^3**	**Unity**	**Dosage 1 m^3**	**Dosage kg**	**% of weight**
Cement	CPII F40 VOTORAN	3009	1500	kg	312	6.42	13.9
Sand	Supertex	2610	1530	kg	790.5	15.81	34.2
Gravel 0	Compasul	2640	1220	kg	301.7	6.034	13.1
Gravel 1	Compasul	2630	1270	kg	74	14.08	30.5
Water	-	1000	1000	kg	192.7	3.85	8.3
Additive % mass density							
1	Grace - GCP	0.85%	1118	g	288g	54.6g	-
2	NPs	0.03%	1118	g	9.5g	1.9g	-

Workability Assay - Slump Test

The conical slump test was completed as per Brazilian Standard [19,20], i.e., 20 cm in the base and 10 cm in the top with 30 cm the total height of the cylinder. The cylinder was filled with the sample, the top of the cylinder was smoothed over, and the cylinder lifted slowly and evenly. The change in height between the cylinder and deformed material was measured. The midpoint of the slumped material was taken as the representative height. Heights were measured with a ruler to the nearest 0.5 mm [21].

Simple axial compressive strength test

The simple axial compressive strength test was accomplished according to Brazilian Regulations [22], and consists of compressing a specimen, generating a linear deformation. The specimen was introduced in cylindrical models of 10 cm of diameter and 20 cm of height, according to Brazilian Regulations [23,24], and used a press (EMIC) of 2000 kN.

Results and Discussion

Synthesis of nanoparticles with different amount of magnetite

The yields of magnetite incorporation are shown in (Table 3) For the process 100 and 200 mg of nanomaterials (GO, GR, NS, and NC) were employed, and obtained 70% of average yield. This results, agree with Rhoden et al. [17] that synthesized GO with different amounts of magnetite incorporated, using a similar method. The methodology employed for magnetization proved to be highly efficient, with good yields, low energy requirements, and a reduced time process.

Table 3: Yield of magnetite incorporation.

1:05								
Nanoparticle	**Graphene Oxide (GO)**		**Graphite nanosheets (GR)**		**Nanosilica (NS)**		**Nanocellulose (NC)**	
Amount (mg)	100	200	100	200	100	200	100	200
Magnetite Incorporation(%)	72.8	74.66	76.53	64.4	70.4	74.4	63.46	64
Yield (mg)	273	560	287	483	264	560	238	480
1:10								
Nanoparticle	**Graphene Oxide (GO)**		**Graphite nanosheets (GR)**		**Nanosilica (NS)**		**Nanocellulose (NC)**	
Amount (mg)	100	200	100	200	100	200	100	200
Magnetite Incorporation(%)	69.53	60.1	65.69	68	74.3	71.84	86.61	67.53
Yield (mg)	452	783	427	884	483	934	563	878

X-ray diffraction

The XRD of nanomaterials (GO, GR, NS, NC, and nanocomposites with different amounts of incorporated magnetite) are shown in (Figure 2). For graphene oxide (Figure 2a), it is possible to observe a signal at $2\theta \approx 11.10^\circ$ (001), characteristic of GO [25]. The XRD of graphite nanosheets (Figure 2b) peaks around $2\theta \approx 26.4^\circ$ (002) and 54° (004), attributes of graphite [26]. The XRD pattern of nanosilica shows a broad peak around $2\theta \approx 22^\circ$ (100), corresponding to an amorphous structure [27]. In the nanocellulose (NC), peaks at $2\theta \approx 14.7^\circ$, 16.4°, 22.5°, and 34.2°

were assigned to the $(1\bar{1}0)$, (110), (002), and (040) planes respectively, presenting the typical diffraction of nanocellulose [28]. For magnetic nanomaterials, the appearance of signals at 2θ ≈ 30.2, 35.3, 43.5, 57.10, and 63.3°, corresponding to the indices (220), (311), (400), (511), and (440), which are compatible with magnetite (JCPDS card no. 19-0629). However, the peaks of GO and NS in $GO \cdot Fe_3O_4$ 1:5, $GO \cdot Fe_3O_4$ 1:10, $NS \cdot Fe_3O_4$ 1:5 and $NS \cdot Fe_3O_4$ 1:10, are constrained due to the large amount of Fe_3O_4, indicating the recovery of nanomaterials surfaces and agglomerate of magnetite [5,17]. For $NC \cdot Fe_3O_4$ 1:5 and $NC \cdot Fe_3O_4$ 1:10, the presence of peak (002) and disappearance of others inferred a decrease in the crystallinity of the polymer, which may result from the magnetization process.

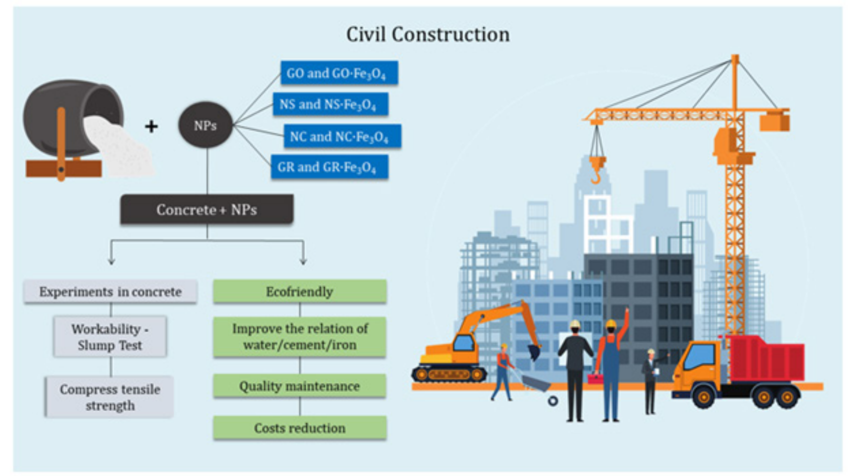

Figure 1: Graphical Abstract.

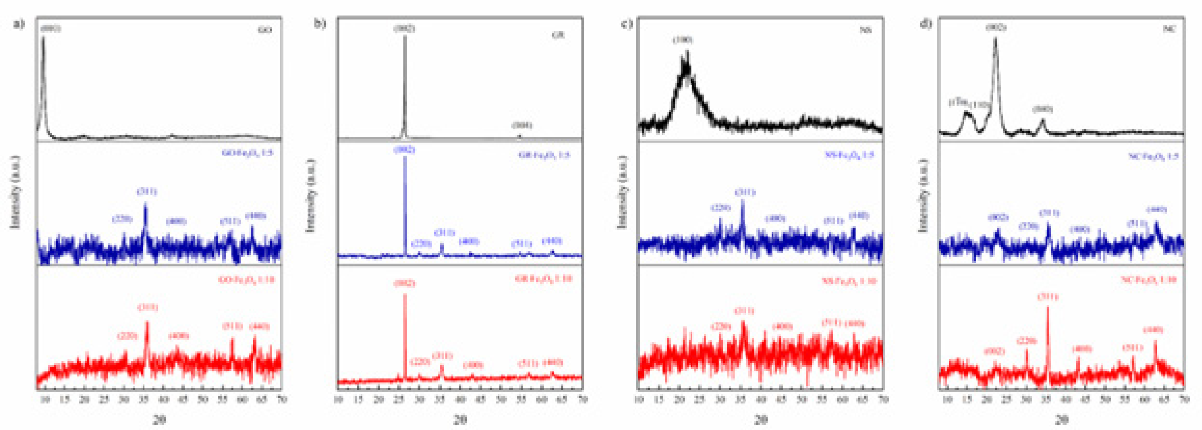

Figure 2: XRD of GO, GR, NS and NC with different amounts of magnetite.

FTIR

(Figure 3) shows the FTIRs spectrums of nanomaterials. For graphene oxide (Figure 3a), an intense band in 3400 cm^{-1} refers to the axial deformation of the hydroxyl group, showing the presence of OH on the nanomaterial's surfaces. The signals at 1497 and 1452 cm^{-1} correspond to C=O and C=C groups, respectively [10]. (Figure 3b), indicates a water absorption peak at 3460 cm^{-1} of graphite nanosheets, and around 1500 and 1000 cm^{-1}, corresponding to C-H and C-O groups [29]. According to (Figure 2c) (NS FTIR spectrum), the peaks at 801 and 474 cm^{-1} indicate the symmetrical Si-O-Si, and 1011 cm^{-1} to the asymmetric vibration of Si-O [30]. (Figure 2d) shows the spectrum of NCs, it is possible to observe around 3486 cm^{-1} the band related to vibrational stretching of OH bond, and 1600 cm^{-1} indicates the water absorption by NC molecules. Moreover, at 1400, 1100, and 1050 cm^{-1}, are attributed to CH, C-C, and C-O stretching, and Calquil-O, respectively [31]. For magnetic nanomaterials, the appearance of a peak at 600 cm-1 infers the presence of Fe-O stretching, which originated from magnetite [17].

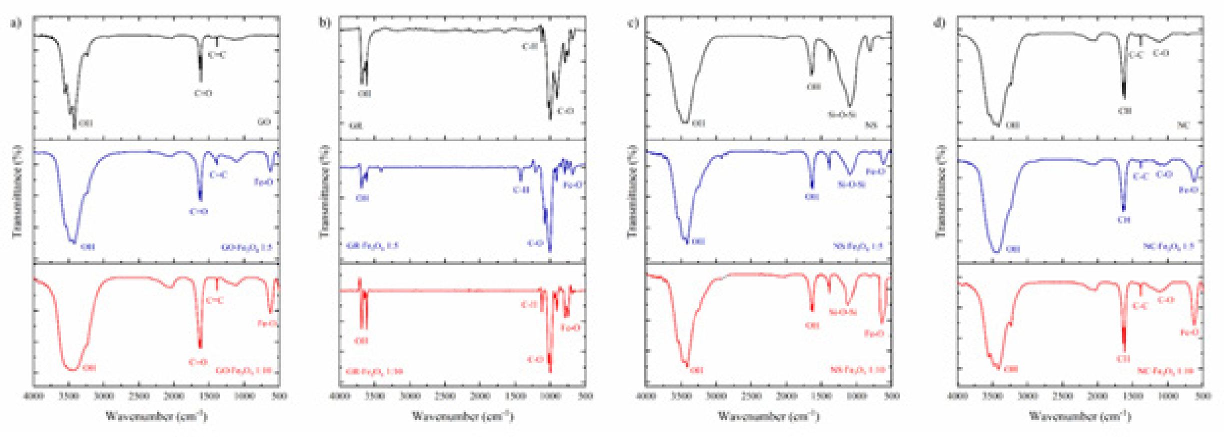

Figure 3: FTIR of GO, GR, NS and NC with different amounts of magnetite.

Experiments in concrete

Workability - Slump Test

The slump experiments were performed to verify the paste homogeneity. The sample of reference (REF) obtained 16 cm in workability tests. Besides, particularly $GO \cdot Fe_3O_4$ 1:5, $GR \cdot Fe_3O_4$ 1:5 and $GO \cdot Fe_3O_4$ 1:10 earned results different in REF, 15.5, 15.5, and 14.5 cm, respectively. However, the standard derivations agree with Brazilian Regulation [19], permitting ± 2 cm. (Table 4) shows the results of the slump test and compressive tensile compression. The results are agreed with Brazilian Regulations [1]. Also, the values found are comparable with Wu et al. [11] and Du et al. [14], where the REF samples present low values when compared with those containing nanosilica and graphite nanosheets.

Table 4: Workability and tractive force results in concrete.

Compression Results									
Slump Test (cm)		3 days (MPa)	7 days(MPa)	28 days (MPa)	Increase 3 to 7 days	Increase 7 to 28 days	Increase 3 to 28 days	Gain/loss in relation to concrete REF (MPa)	% Gain/loss in relation to concrete REF
REF.	16	35,42	46,32	51,28	31%	11%	45%	x	x
GO	16	25,02	38,03	45,6	52%	20%	82%	-5,68	-11%
$GO \cdot Fe_3O_4$ 1:5	15,5	25,02	39,17	46,9	54%	22%	87%	-4,38	-9%
$GO \cdot Fe_3O_4$ 1:10	16	27,53	38,43	47,27	40%	23%	72%	-4,01	-8%
GR	16	29,65	46,21	54,89	56%	19%	85%	3,61	7%
$GR \cdot Fe_3O_4$ 1:5	14,5	34,03	46,63	55,39	37%	19%	63%	4,11	8%
$GR \cdot Fe_3O_4$ 1:10	15,5	31,14	47,32	52,80	52%	12%	70%	1,52	3%
NS	16	31,27	44,37	54,18	42%	22%	73%	2,9	6%
$NS \cdot Fe_3O_4$ 1:5	16	29,5	47,14	53,87	60%	14%	83%	2,59	5%
$NS \cdot Fe_3O_4$ 1:10	16	30,9	47,72	54,58	57%	14%	80%	3,3	6%
NC	16	27,79	41,16	46,05	48%	14%	69%	-5,23	-10%
$GR \cdot Fe_3O_4$ 1:5	16	25,47	39,11	46,89	54%	20%	84%	-4,39	-9%
$NC \cdot Fe_3O_4$ 1:10	16	27,22	41,65	48,87	53%	15%	77%	-2,41	-5%

Compress tensile strength

The cylindrical standard used for compressing tensile experiments was measured in two different directions, obtaining 9.94 and 10.25 cm. (Table 4) and (Figure 4) show the results of compress tensile strength. For 3 days, none of the samples with nanoparticles obtained higher results than REF (35.42 MPa). The $GR \cdot Fe_3O_4$ 1:5 presented 34.03 MPa. However, GO and $GO \cdot Fe_3O_4$ 1:5 showed the lowest strength

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values, 25.05 MPa for both nanomaterials. On the other hand, it is possible to observe an increase in compression resistance with higher amount of employed iron in the magnetization process. At 7 days, the REF broke at 46.32 MPa, overcome by $NS \cdot Fe_3O_4$ 1:10 (47.72MPa), $GR \cdot Fe_3O_4$ 1:5 (46.63 MPa) and $GR \cdot Fe_3O_4$ 1:5 (47.32 MPa).

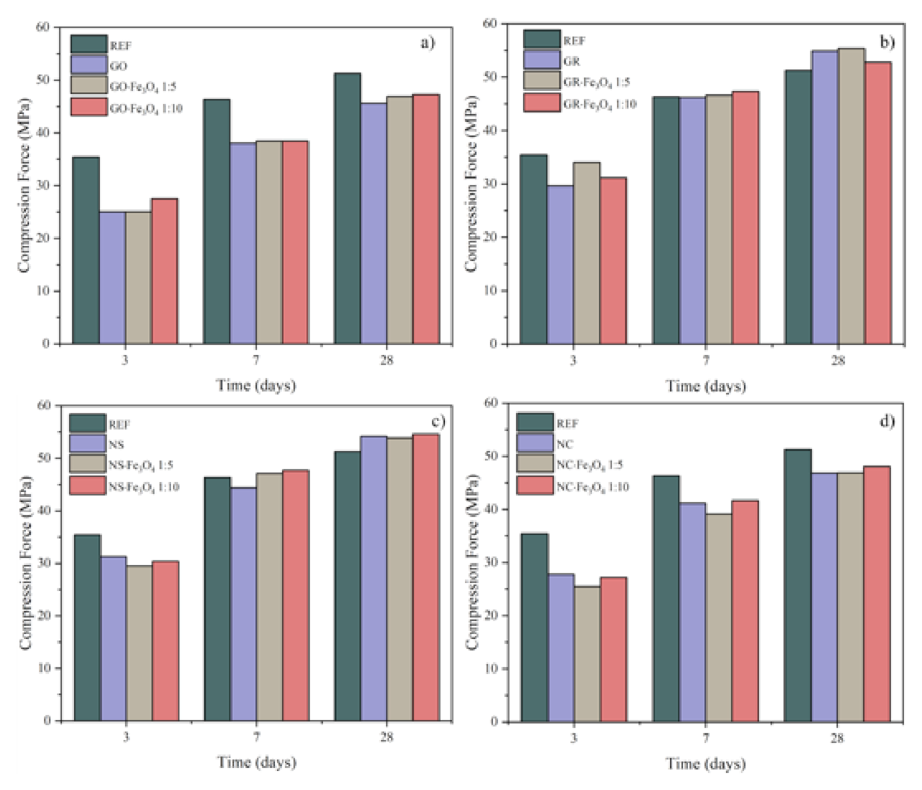

Figure 4: Compress tensile strength of nanoparticles.

Thereafter 28 days, the REF reached 51.28 MPa, which was taken by GR, $GR \cdot Fe_3O_4$ 1:5, $GR \cdot Fe_3O_4$ 1:10, NS, $NS \cdot Fe_3O_4$ 1:5, $NS \cdot Fe_3O_4$ 1:10 , presenting 54.89, 55.39, 52,80, 54.18, 53.87 and 54.58, respectively. However, GO, NC, and their nanocomposites with different amounts of magnetite incorporated do not overcome the compress results found by the REF sample. The increase in resistance is related to the more effective filling of interstices by the mixture of nanoparticles (nanomaterials and magnetic nanoparticles) in the concrete. Along with this, it is possible to observe that with increasing in magnetite incorporation the samples present more resistance when compared to REF. On the other hand, GO, NC and their nanocomposites, present low values than REF, probably to the pore dispersion of nanoparticles along the cylindrical scaffold [32,33,34].

Conclusion

This study aimed experimentally evaluate the effects of the addition of magnetite incorporated nanoparticles in graphene oxide, graphite nanosheets, nanosilica and nanocellulose, in construction cement, in order to compare their performance in relation to the mechanical properties of concrete, a mixture used in the main constructive method for civil engineering buildings. The nanoparticles were characterized by the methods of X-ray Diffraction (XRD) and Fourier-transform Infrared Spectroscopy (FTIR). By comparing the nanoparticles added in concrete with a referential sample (without NPs). The study reveals that all analyzed material maintained the workability, reducing around 10%, remaining in accordance with the tecnical regulation. The Slump test revealed that only the samples containing GO and NC decrease the compression resistance, although no significant workability reduction was observed. The graphite nanosheets and nanosilica, with and without incorporated magnetite, showed increasing resistance in concrete by about 8% compared to the reference by 28 days test. These results are mainly related to the interactions of the NPs and water dispersion fulfilling the cement pores due its nanometric dimensions. Considering the initial results regarding the increase in the concrete resistance with the conservation of the workability, the addition of the nanoparticles arises the possibility of further studies from application NPs in civil construction, improving the relation of water/cement/iron, aiming conservancy and costs reduction without environmental damage and quality maintenance.

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