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ORIGINAL ARTICLE

Analysis of Portland cement consumption reduction by using functionalized multiwalled carbon nanotubes in mortars

Análise da redução do consumo de cimento Portland por meio da utilização de nanotubos de carbono de paredes múltiplas funcionalizados em argamassa

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Abstract: The use of carbon nanotubes in cementitious materials improves the mechanical properties of these composites and increases their compressive and flexural strength. The main objective of this study was to evaluate the technical feasibility of reducing the Portland cement content by using functionalized multi-walled carbon nanotubes (MWCNT) in mortar samples, using mechanical properties as parameters (compressive strength, flexural strength and modulus of elasticity). Experimental results allowed us to conclude that the addition of MWCNT in 0.1% by weight of Portland cement, to the mortar samples allowed a reduction of the Portland cement content by up to 10%, while the water/cement ratio remained unchanged without affecting the mechanical behavior.

Keywords: multiwalled carbon nanotubes, reduction of the Portland cement, mechanical behavior.

Resumo: A utilização de nanotubos de carbono em materiais cimentícios melhora as propriedades mecânicas destes compósitos, aumentando a sua resistência à compressão e à flexão. O principal objetivo deste estudo foi avaliar a viabilidade técnica da redução do teor de cimento Portland através da utilização de nanotubos de carbono de paredes múltiplas funcionalizados (MWCNT) em amostras de argamassa, utilizando como parâmetros as propriedades mecânicas (resistência à compressão, resistência à flexão e módulo de elasticidade). Os resultados experimentais permitiram concluir que a adição de MWCNT em 0,1% em peso de cimento Portland, às amostras de argamassa, permitiu uma redução do teor de cimento Portland em até 10%, mantendo-se inalterada a relação água/cimento, sem afetar o comportamento mecânico.

Palavras-chave: nanotubos de carbono de paredes múltiplas, redução do cimento Portland, comportamento mecânico.

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1 INTRODUCTION

The use of nanotechnology in cementitious materials to improve the properties of composite materials has been the subject of numerous studies in the last decade. Among the nanomaterials, carbon nanotubes (CNT) have been highlighted for their high ability to improve mechanical strength.

Many studies have shown that the use of MWCNT in cementitious materials offers numerous advantages due to their mechanical properties and durability [1]–[5]. The authors pointed out that this improvement is due to the fact that MWCNT form interconnected bridges with the cementitious matrix, resulting in better stress distribution, as well as

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Data Availability: the data that support the findings of this study are available from the corresponding author, [AVSR], upon reasonable request.

reducing, or even mitigating the propagation, of microcracks and reducing porosity. The improvement of the mechanical properties of cementitious materials by using this nanomaterial makes it possible to reduce the consumption of Portland cement to obtain the same mechanical strength.

It is well known that Portland cement is the main contributor to the high greenhouse gas (CO2) emissions in the construction industry. According to [6], for every ton of Portland cement produced, about one ton of carbon dioxide (CO2) is released. The fact that it is possible to reduce the consumption of Portland cement by using MWCNT makes this material a very important factor in reducing environmental impact. However, the major challenge in the use of MWCNT is to ensure complete dispersion in the cement matrix since agglomeration phenomena may occur due to the high attractive force between the nanoparticles and van der Waals forces [7]. When agglomeration occurs, the nanomaterials do not perform effectively.

As described in [8], the most commonly used techniques to improve the dispersion quality of these nanomaterials are (1) insertion of functional chemical groups, (2) use of dispersing chemical admixtures; and (3) ultrasonic treatment. These techniques can be used either alone or in combination. Often, CNT are dispersed in an aqueous medium with an amount of dispersing chemical admixture, and the dispersion is assisted by ultrasound.

As for the dosage of MWCNT, most studies have considered their incorporation into the cement matrix in addition to the weight of Portland cement [9]–[11], which means that the same cement mixture is used with the same amounts of the constituents, only with the addition of the nanomaterial. Therefore, this study aims to evaluate the technical feasibility of reducing the consumption of Portland cement by adding MWCNT in mortars, using the compressive strength at 28 d as a parameter, and using the sample without MWCNT as a reference. Mortars with 5% and 10% reduced cement content in weight and addition of MWCNT were prepared and compared with a reference sample.

A literature search revealed that there are no studies that analyze the potential of reducing Portland cement content by adding MWCNT as a fixed parameter of compressive strength at 28 d. In addition, few studies have used MWCNT as a replacement for Portland cement in mortar and concrete [12]. There are not many studies on MWCNT in concrete due its better performance in the interfacial transition zone of mortars than in concretes. The possibility of reducing the amount of Portland cement used in concrete and mortar is considered an environmentally correct practice, since Portland cement is the material responsible for most of the generation of greenhouse gases in construction. Thus, the originality of this research is the produce and evaluation of mortar with reduced cement content, using MWCNT to obtain the same mechanical strength as reference mortar (without MWCNT). The fresh state analysis complements the study.

2 MATERIALS AND EXPERIMENTAL PROGRAM

2.1 Materials

The materials used in the study were Brazilian Portland cement CP V – ARI, MWCNT functionalized with the carboxyl group (COOH), length 10 µm -30 µm, diameter 5 – 15 nm, from Nanostructured & Amorphous Materials (Nanoamor), fine aggregate (natural sand and crushed sand), and a superplasticizer admixture based on polycarboxylate from MC-Bauchemie. The cement type CP V – ARI was selected for its high purity among the cements commercially available in Brazil.

The fine aggregates were characterized by testing the particle size and specific gravity tests according to the Brazilian standards ABNT NBR NM 248 (2003) and ABNT NBR 6508 (1984), respectively. Figure 1 shows the particle size distribution of the sand. The volumetric sand ratio for the production of mortar samples was set to 70% natural sand and 30% crushed sand.

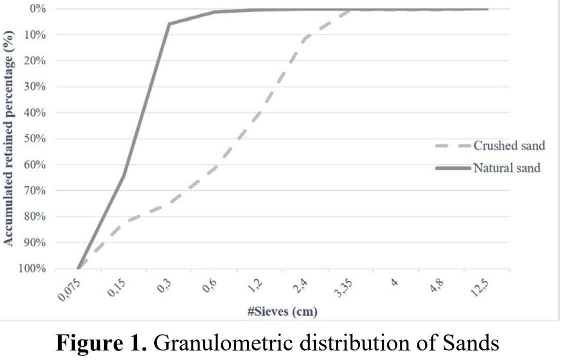

Figure 1. Granulometric distribution of Sands

2.2 Mortar composition and mixing procedures

The mortars were prepared in a volumetric ratio of 1:3 (cement/aggregate). The volumetric composition was used to produce the same volume of mixture for each mortar sample. Therefore, when there was a decrease in the Portland cement content, the amount of aggregate was increased to maintain the same volume of mixture. The composition of the mixtures is given in Table 1, according to: (1) REF, reference; (2) MWCNT, addition of 0.1% of MWCNT, by weight of Portland cement; (3) 5SW, reduction in Portland cement content by 5% (without MWCNT); (4) S5, reduction in Portland cement content by 5% (with MWCNT); (5) SW10, reduction in Portland cement by 10% (without MWCNT); (6) S10, reduction in Portland cement by 10% (with MWCNT). The w/c ratio was fixed (0,50), and the total volume of mortar produced was remained unchanged, with more aggregate added to the samples with Portland cement reduced. The incorporation of MWCNT was normalized at 0.1% in relation to the weight of Portland cement of the reference mix.

Table 1. Quantitative of mortars produced

Materials	Mixture Superplasticizer fixed					
	REF	**MWCNT**	**SW5**	**S5**	**SW10**	**S10**
Cement (g)	557,42	557,42	529,55	529,55	501,68	501,68
Natural sand (g)	990,95	990,95	1031,52	1031,52	1073,63	1073,63
crushing sand (g)	458,18	458,18	476,94	476,94	496,42	496,42
water (g)	279,54	279,54	264,77	264,77	250,84	250,84
MWCNT (g)	-	0,57	-	0,57	-	0,57
admixture (g)	1,67	1,67	1,59	1,59	1,51	1,51
Materials	**Mixture Fixed Spread**					
	REF	**MWCNT**	**SW5**	**S5**	**SW10**	**S10**
Cement (g)	557,42	557,42	529,55	529,55	501,68	501,68
dune sand (g)	990,95	990,95	1031,52	1031,52	1073,63	1073,63
crushing sand (g)	458,18	458,18	476,94	476,94	496,42	496,42
water (g)	279,54	279,54	264,77	264,77	250,84	250,84
MWCNT (g)	-	0,57	-	0,57	-	0,57
admixture (g)	1,67	2,12	1,82	1,92	3,02	3,12

The mortars were evaluated according to two main groups: (1) fixed superplasticizer content of 0.3% by weight of cement and (2) predefined spread of 230 ± 20 mm (variable superplasticizer content).

The mortars were prepared using a conventional multi-speed planetary mortar mixer. Dry materials, cement, and sand were premixed for 1 min, and then the mixing water and superplasticizer were added gradually and mixed for 4 min. For samples with MWCNT, 40% mixing water and 1 g superplasticizer were used to disperse the nanomaterial for hand mixing for 1 min [13]. An ultrasonication process was not used to disperse the MWCNT, in order to simplify the use of the nanomaterial in a future industrial process. Furthermore, due to the fact that the nanomaterial is functionalized, the use of a superplasticizer admixture and the mixing energy are sufficient to obtain the necessary dispersion.

2.3 Testing procedures

The properties of the fresh state were evaluated for spread according [14], and entrapped air in accordance with the Brazilian Standard NBR 13276 and ASTM C1437 - 20. Afterwards, six cylindrical specimens of 50 mm × 100 mm were prepared for each mixture, three of which were used to measure the dynamic modulus of elasticity and compressive strength according [15] and ASTM C39 [16], and the other three to determine water absorption and pore volume according to ABNT NBR 9778 and ASTM C642 [17]. Three prismatic specimens (40 mm × 40 mm × 160 mm) were fabricated to evaluate the flexural strength. The samples were kept in wet curing, submerged in a tank with water and lime at a controlled temperature of 21°C ± 1°C until the test age.

The modulus of elasticity was measured by a nondestructive testing method, the excitation impulse, using Sonelastic software according to with ASTM E1876 [18]. Flexural strength evaluation was performed using an Instron Universal Testing Machine after the specimens had been wet curing for 28 d. The results were analyzed by ANOVA one-way statistical. This method allows you to check whether there are differences between the averages of a given variable in relation to a treatment with two or more categorical levels.

3 RESULTS AND DISCUSSIONS

3.1 Mortar behavior in the fresh state

The values of consistency and entrapped air are shown in Figure 2, Figure 3 and Figure 4. The consistency of the mortars was affected by the presence of MWCNT, and a decrease was observed compared to the samples without MWCNT.

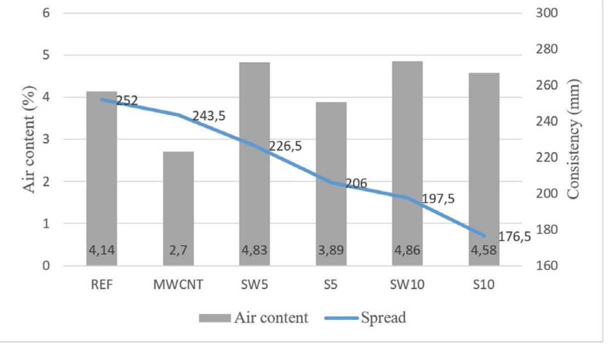

Figure 2. Results of fixed superplasticizer mortars

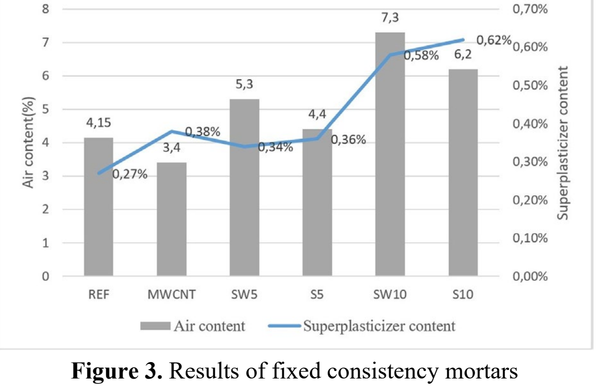

Figure 3. Results of fixed consistency mortars

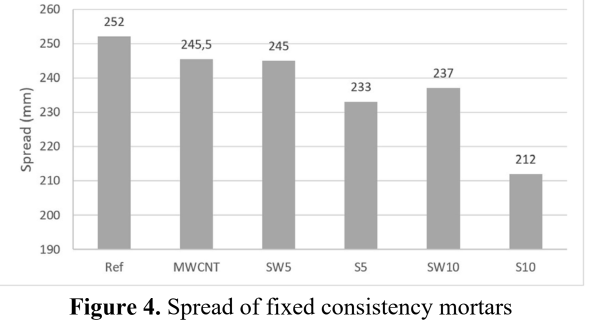

Figure 4. Spread of fixed consistency mortars

Regardless of the main mortar group, the samples without MWCNT exhibited a greater amount of entrained air than the mixtures with nanomaterials. Two secondary effects explain this phenomenon. First, according to [19], [20], MWCNT also interact with the superplasticizer admixture, so fewer molecules are available to interact with the cement and produce entrained air. Second, the presence of MWCNT leads to densification of the cementitious matrix and fills empty spaces with the nanomaterial [9]–[11].

As can be seen in Figure 3, there is a tendency to increase the amount of superplasticizer in the samples when the cement content is reduced, to achieve the same consistency. This phenomenon is due to the reduction in the amount of mixing water required to obtain the same w/c ratio in all samples. As the cement content was reduced, the friction between the aggregates increased due to the lower available cement paste content, which reduced the flowability of the mixture [21]. In addition, the aggregate has more angular morphology than the Portland cement particles, which contributes to an increase in friction. According to [22], the rheology of Portland cement paste is determined by colloidal forces. However, in the presence of aggregates (mortar and concrete), the rheology is affected by more complex processes such as friction, volume of solid particles, and granular structure.

Finally, it can be seen in Figure 3 that the mortars without MWCNT required a smaller amount of superplasticizer to obtain a fixed consistency. Figure 2 confirms this, as the mortars with the nanomaterial and a fixed superplasticizer content gave lower consistency values. According to [23]–[25], this can be explained by the high specific surface area of MWCNT and the interaction between superplasticizer-nanomaterial-water molecules.

3.2 Compressive strength

Figures 5 and 6 show the results of compressive strength at 28 d of age for the two main groups of mortars produced. To compare the two main groups, ANOVA one-way statistical analysis was performed to determine if there was a significant difference. Table 2 shows the results.

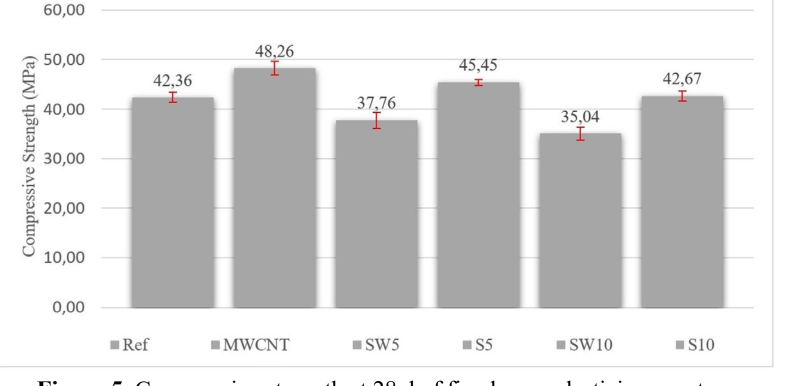

Figure 5. Compressive strength at 28 d of fixed superplasticizer mortars

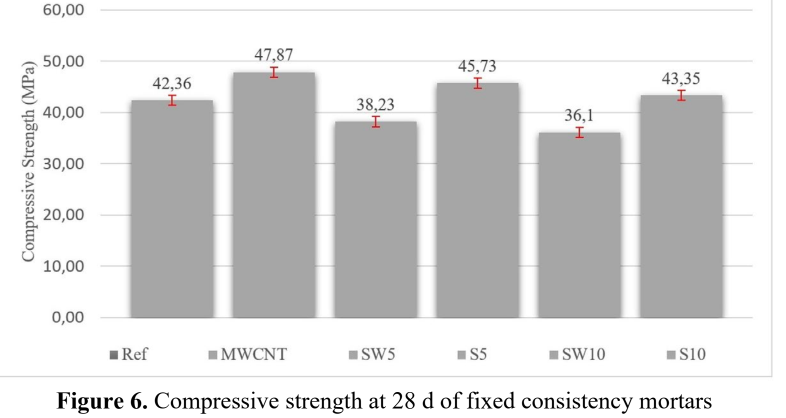

Figure 6. Compressive strength at 28 d of fixed consistency mortars

Table 2. Statistical analysis of the main groups of mortars

Type of mixture	Result
MWCNT	No significant different
SW5	No significant different
S5	No significant different
SW10	No significant different
S10	No significant different

For this study, with 95% confidence, it was determined that the use of fixed superplasticizer content or fixed consistency did not significantly affect the compressive strength at 28 d. Therefore, for the remainder of this study, the results of the mortars prepared using the fixed superplasticizer method were adopted.

The same statistical method was used to verify the influence of the use of MWCNT on the compressive strength of the mortars at 28 d. With 95% confidence, it was determined that the mortars with the nanomaterial, S5 and S10, increased the average compressive strength compared to mortars REF, SW5 and SW10, respectively. The results of the analysis are shown in Table 3.

Table 3. Statistical analysis of compressive strength at 28 d of fixed superplasticizer mortars

Mixtures	Result
REF-MWCNT	Significant different
SW5 – S5	Significant different
SW10 – S10	Significant different
REF – S5	Significant different
REF – S10	No significant different

The improvement in the compressive strength of the samples with MWCNT was 7.5% for the reference mortar and 9.5% for the mortar with a 10% reduction in cement content. This increase in compressive strength is due to matrix densification and reduced air entrapment, which is consistent with the results of previous studies [26]–[29]. It is important to emphasize that carbon nanotubes can promote a better distribution of internal stresses in the cementitious matrix, improving the mechanical properties [1], [2], [4]. Results similar to those found in this study were reported by [27], where the compressive strength with the use of MWCNT increased up to 15% in relation to the reference mortar.

Moreover, the S10 mortar was not statistically significantly different from the REF mortar, which means that in the presence of MWNCT, it was possible to produce a mortar with a compressive strength equal to that of the reference, reducing the Portland cement content by 10%. The feasibility of reducing the Portland cement content by adding MWCNT is evident in Figure 7, which shows for each sample the amount of cement used for each MPa of compressive strength at 28 d. These data were determined according to [30]. The mortars with MWCNT yielded in the lowest kg/MPa indices, improving the compressive strength potential of the cement. The lower consumption of Portland cement brings the advantage of cement-based materials with a lower environmental impact.

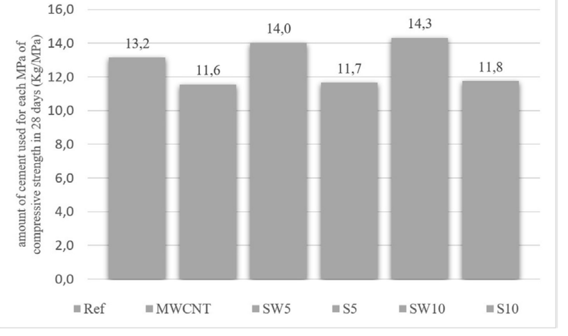

Figure 7. Portland cement content for 1MPa compressive strength

3.3 Flexural strength

Figure 8 shows the results of the flexural strength of the mortars at 28 d, which were statistically compared using the ANOVA one-way analysis to verify whether the use of MWCNT significantly affects the flexural strength. The results of the statistical analyzes are shown in Table 4.

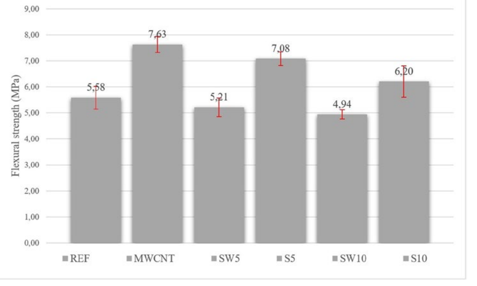

Figure 8. Flexural strength at 28 d

Table 4. Statistical Analysis

Type of mixture	Result
Ref - MWCNT	Significant different
SW5 – S5	Significant different
SW10 – S10	Significant different
REF – S5	Significant different
REF – S10	No significant different

From the results of the statistical analysis, there is 95% confidence that the samples with MWCNT have 25-35% higher flexural strength than the mortars without nanomaterials. Compared with REF, only sample S10 showed no significant difference, which means that its flexural strength was equivalent.

The improvement in flexural strength can be attributed to the fact that the nanomaterial acts as a reinforcing fiber in the cementitious matrix, bridging the cracks and leading to an increase in flexibility [31]. However, the same authors suggested that short and very strong bonds between C and C of MWCNT can form bridges between nanocracks and, therefore, increase the flexural strength. Moreover, the nanomaterials could have filled the pores and voids in the cementitious nanostructure [32].

It is important to emphasize that, in addition to the compressive strength, the presence of MWCNT also resulted in a flexural strength statistically equivalent to that of REF, when the cement content was reduced by 10% (S10), proving the feasibility of reducing the cement content of the mortar.

3.4 Dynamic modulus of elasticity

The results of the modulus of elasticity tests of the mortar are shown in Figure 9. The results were evaluated using ANOVA-one-way statistical analysis (Table 5) to determine whether the presence of MWCNT significantly affects modulus elasticity.

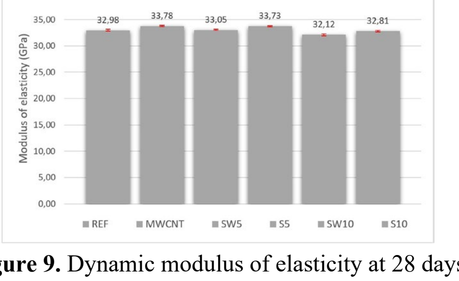

Figure 9. Dynamic modulus of elasticity at 28 days

Table 5. Statistical Analysis

Type of mixture	Result
Ref - MWCNT	Significant different
SW5 – S5	Significant different
SW10 – S10	Significant different
REF – S5	Significant different
REF – S10	No significant different

The mortars with the addition of MWCNT had slightly higher elastic modulus values than the mortars without the nanomaterial. This increase in the modulus values is to be expected since the modulus of MWCNT itself is greater than that of the other mortar components. Despite the low content of nanomaterial in the mortars, it was sufficient to cause in a significant increase in the dynamic modulus of elasticity.

Moreover, as shown in the results of [33], MWCNT can fill the gaps between the layers of C-S-H, reducing the nanoporosity of the composite, and consequently improving its mechanical properties [34]. In addition, the increase in the dynamic modulus values may be due to the decrease in the entrained air in the samples with nanomaterials. The presence of entrained air in cementitious materials reduces the modulus of elasticity.

Statistically, there was no significant difference between the moduli of the REF and S10 mortars; i.e., even with a 10% reduction in Portland cement content, there was no decrease in the value of the modulus of elasticity of the mortar with the nanomaterial.

3.5 Absorption and percent voids

Water absorption and percent voids in the mortars were evaluated according to ASTM C642-13. The test results are shown in Figure 10. The mortars prepared with MWCNT had a lower percentage of voids and water absorption than the mortars without MWCNT.

These results are consistent with the evaluation of the compressive strength. The samples with increased compressive strength (with MWCNT) were the same, that reduced porosity. According to [32], [35], the use of MWCNT in mortars reduces water absorption due to the narrowing of pores, resulting in a more compact microstructure.

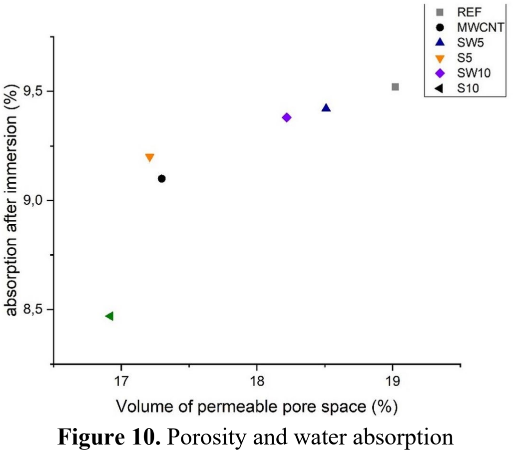

Figure 10. Porosity and water absorption

According to [36], the main factor affecting the permeability coefficient of cementitious matrices is the size and connectivity of the pores. The authors also suggested that permeability and absorption are related to porosity, connectivity, and pore size. Thus, mortars with lower permeability and absorption reduce the internal transport of potentially aggressive materials and increase their durability.

Cementitious matrices with a higher percentage of voids lead to a deterioration of mechanical properties. Thus, it can be verified that the mortars without MWCNT have a higher percentage of voids, higher water absorption, and lower compressive strength than the mortars prepared with the nanomaterial.

4 ANALYSES

Analyses of the results indicates that the use of functionalized MWCNT-COOH in mortars increases the compressive strength, modulus of elasticity, and flexural strength at 28 d. In addition, it was found that reducing the Portland cement content by up to 10% in the samples with nanomaterials had no effect on the mechanical properties. This could be related to the reduction of porosity and densification of the cement matrix due to the better internal stress distribution in the cement matrix caused by the MWCNT.

It appears that the mortars produced with lower consumption of cement S5 and S10, have higher proportions of MWCNT than the MWCNT mortar, this may also have contributed to the mechanical properties, rheological properties and porosity of these mortars. The decrease in Portland cement consumption in SW5 and SW10 mortars was offset by the use of aggregate in the same volume, so that the total mix volume was not altered. It is important to point out that the samples with lower cement content resulted in lower mechanical properties than the reference mixture, and the gain in mechanical properties in the mortars (S5 and S10) was due to the use of MWCNT. Therefore, higher MWCNT contents may result in higher mechanical properties and less spreading in the fresh state.

All mortars produced with the nanomaterial had lower entrained air content, a lower percentage of pores, and lower water absorption compared to mortars without nanomaterials. These results show the potential to reduce porosity, which increases mechanical properties and durability. Since the tensile strength of MWCNT is much higher than that of the cementitious matrix, the improvement in the flexural strength of the mortars can be explained by the formation of bonds in the microstructure of the cementitious matrix. The resulting improvement in mechanical properties and increase in flexural and compressive strengths allowed the cement content to be reduced. In addition, the reduction in entrained air and porosity improved the microstructure of the mortars.

It is important to highlight that the increase in the modulus of elasticity due to the use of MWCNT was low. Therefore, the increase in modulus of elasticity could be related to mixture heterogeneity and equipment/operational error. However, it is interesting to note that none of the samples without MWCNT had a higher modulus of elasticity compared to the samples with the nanomaterial. Thus, the low increase in the modulus of elasticity may be related to the use of MWCNT.

These results indicate an improvement in the microstructure of cementitious materials with MWCNT, which also increases their durability potential. It is important to note that complex dispersion methods were not used, which increases the feasibility of their use in civil construction. Finally, through the presented results, it is concluded that the use of MWCNT, without an ultrasonication process, provided mortars with a 10% reduction in the Portland cement content without damage to the mechanical properties of samples in relation to the reference mortar. It is important to highlight that the demand for reductions in the cement content of mortars and concretes is important for the sustainability of civil construction.

5 CONCLUSIONS

From the results, materials, and methods used in this study, the following conclusions can be drawn.

- There was less entrained air in the fresh mortars with MWCNT, which was also confirmed by the decrease in total pore volume and water absorption in the hardened state.
- The addition of MWCNT to the mortars resulted in a decrease in consistency as well as a higher consumption of superplasticizer additive to achieve the same consistency value.
- The mechanical properties of the mortars prepared with MWCNT were improved. Statistically, there was no significant difference between the reference mortar and the mortar with a 10% reduction in cement content and the addition of MWCNT. The effects of the nanomaterial on the microstructure offset the reduction in Portland cement content and allowed the production of cementitious materials with lower environmental impact.
- It was demonstrated that the use of MWCNT provided satisfactory results without a complex dispersion method such as ultrasonic treatment. The combination of techniques, such as the functionalization of CNTs and the use of a superplasticizer admixture with manual mixing, was sufficient to obtain important results in terms of improving mechanical properties, which is consistent with the results observed by other authors. This facilitates the application of MWCNT in civil construction projects.

ACKNOWLEDGEMENTS

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