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Rheological Properties of Carbon Nanotube Infused Cementitious Composites with Various Amounts of CNT

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Abstract: The addition of carbon nanotubes (CNTs), which are hydrophobic materials, significantly influences the rheology of cementitious materials but requires important mix modifications in order to provide proper flowability for further use. This paper investigates the influence of various dosages of carbon nanotubes (0.05 wt.%, 0.1 wt.%, 0.2 wt.%, 0.5 wt.%, and 1 wt.%) on the flowability, rheological parameters, air content, and volume density of cement mortars. The results show an increase in the yield stress parameter with an increment in CNT dosage up to the threshold of 0.5 wt.% for mixes with an increased amount of cement. For standard proportions, it was on a stable level for all mixes except for 0.2 wt.%. The plastic viscosity parameter also increased with the CNT dosage; mixes with standard proportions of components were not higher than the reference, and mixes with an increased amount of cement were lower than the reference for dosages up to 0.5 wt.% of CNT. The addition of a superplasticizer and modifications of the ratio of the components were employed to achieve proper flowability and measure the rheological parameters. The presented results show that regardless of the negative influence of carbon nanotubes on the properties of fresh mortar, it is possible to achieve a stable flow and workability using simple modifications of the composition.

Keywords: carbon nanotubes; cementitious nanocomposites; rheology; flowability

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1. Introduction

Carbon nanotubes (CNTs) are currently among the most widely researched carbon nanofillers for cementitious materials [1]. Besides improvement in the mechanical properties of the cement matrix, their elongated shape, very high conductivity, and aspect ratio make them a potent choice for electrically conductive smart cementitious materials, which are used as structural health monitoring sensors [2–5]. Since CNTs are not a material used traditionally in cement and concrete engineering, a wide range of properties of cementitious materials that can be influenced by the addition of CNTs should be considered. Depending on multiple factors, including type, size, dispersion quality, and dosage, the addition of carbon nanotubes in cement material can cause both beneficial and negative effects on the composite's properties. Generally, the addition of carbon nanofiller can be beneficial, with improvements in mechanical strength, tightness, and electrical conductivity of the naturally dielectric cement material. On the other hand, negative effects on workability and other rheological parameters can occur [6], and an improper dispersion quality can lead to the devaluation of all the abovementioned beneficial factors [7]. Attempts have been made to model and predict the behavior of carbon nanomaterials, such as reduced graphene oxide (rGO) paired with cement hydration products, in order to better understand their interaction at the atomic level [8]. The results established a connection between rGO and the chemical composition of the CSH phase and confirmed the possibility of extracting pristine graphene from rGO. This approach to modeling atomic-scale interactions using density functional theory has been proven to properly reflect the mechanical properties of cement phases [9] and, more recently, interactions between cement hydrates and rGO nanomaterial. In the latter example, the properties of a composite were mostly dependent on the interface between the rGO and CSH phase and the properties of functional groups that are part of the nanomaterial [10]. If applied to carbon nanotubes and their interaction not only with hydration products but fresh cement in the presence of water, a model could highlight their ability to adsorb or trap water molecules, which could be one of the main influences they have on the rheological properties of cement-based materials.

As carbon nanotubes are strongly hydrophobic, their agglomerations, caused by strong van der Waal's forces, might trap water molecules in between the clusters of CNTs. Therefore, it is likely that high amounts of CNTs in cementitious materials will reduce their flowability [11]. Jiang et al. [12] compared the influence of multiple types of nanofillers on the rheological properties of cement pastes and pointed out the large aspect ratio and shape of nanomaterials as the main cause for the reduced flowability of the cement pastes. This effect was mostly noted in nanofillers with elongated shapes, CNTs, and nanofibers. The comparison was made for nano silica, nano titanium, CNTs, and carbon nanofibers at different w/c ratios, dispersion techniques, and superplasticizer content.

There are multiple methods of increasing the flow of cement-based materials, among which the most popular are increasing the water-to-cement ratio and adding superplasticizers or fine mineral additions in the form of fly ash, micro silica, or others. Each of these techniques has drawbacks that can mitigate the beneficial effects of the addition of carbon nanotubes. In the study of Jiang et al. [9], rheological parameters of cement pastes with CNT were sensitive to changes in w/c ratio at very low levels between 0.2 and 0.22. They also pointed out that for tested CNTs and their content of 0.5 wt.% there is a saturation point of 1 wt.% for the superplasticizer to have a significant influence on the fluidity of the cement paste. Andrare Neto et al. [13] investigated the influence of CNT dosages of 0.05 wt.%, 0.075 wt.%, and 0.1 wt.% along with metakaolin as supplementary cementitious material on the rheological parameters of cement pastes. They reported an increasing influence on the yield stress for the CNT water suspensions alone; however, in combination with metakaolin, the yield stress increased over ten times. This phenomenon was attributed to the conflict between CNTs and metakaolin, both having a negative impact on their dispersion in the cement matrix. Silvestro et al. [14] investigated the influence of water dispersion of CNTs, mainly the sonication parameters, and on the rheological parameters of cement pastes with 0.05 wt.% and 0.1 wt.% of functionalized CNTs. The results showed an increase in yield stress for higher dosages of CNT and for higher sonication amplitude. The authors implied that better dispersion of CNTs in water suspension causes a higher area of the nanomaterial to be in contact with water. Therefore, considering the large specific surface area of individual carbon nanotubes, the amount of water absorbed by them is higher than in the case of worse dispersed, agglomerated CNTs. No significant differences were found for plastic viscosity. An important factor regarding the rheological parameters of cement pastes was pointed out by Mendoza Reales et al. [15]. After testing cement pastes with a constant 0.15 wt.% of CNT and a variety of different surfactants to aid the dispersion, they concluded that the interaction between the surfactant used for water dispersion of CNTs and components of the cement material needs to be considered in the context of the rheological properties. Some surfactants might not be compatible with cement, especially if they are industrial cleaning products and not concrete admixtures.

To counteract the flowability and CNT dispersion problem, De Souza et al. [16] proposed the synthesis of carbon nanotubes on clinker to avoid introducing them along with water solutions. Results obtained for 0.15 wt.% and 0.30 wt.% of CNT revealed proper flowability and rheological parameters for cement pastes, which were achieved via a relatively low addition of a superplasticizer of 0.2 wt.% and for a low w/c ratio of 0.4. However, it was also pointed out that optimal amounts of CNT and superplasticizer, chosen with regard to flowability, hydration mechanics, and mechanical strength, were valid only for the specific composition and materials used in the study. MacLeod et al. [17] proposed a solution for rheological and dispersion issues by introducing CNTs in the form of a premade liquid admixture. In this form, even a high dosage of 5 wt.% and 10 wt.% of CNTs did not negatively affect the slump flow and even reduced the yield stress of the concrete mix. A prolonged mixing time of the concrete mix was also denoted as an important factor for the proper dispersion of CNTs in the matrix.

Collins et al. [18] tested the influence of 0.5 wt.%, 1 wt.%, and 2 wt.% of CNTs, along with different admixtures and water-to-binder ratios, on the dispersion and workability of cement pastes. Results from the mini-slump test showed a flow reduction of 48.9% and 38.3% for the 2 wt.% CNT samples with a w/c ratio 0.5 and 0.6, respectively. They also reported a decrease in mechanical strength for samples with higher dosages of CNTs on respective water-to-binder levels.

Hong et al. [19] described changes in the conductivity and flowability of cement-based composites in relation to the material's moisture. They noticed that the flowability of the mortars dropped linearly with the increment in CNT dosages from 0.1 wt.% to 1 wt.%. Similar properties were investigated by Tafesse et al. [20], where they attempted to correlate the flowability and electrical conductivity of cementitious composites with carbon nanotubes. The tests employed two different forms of CNTs, powdered and pelletized, at low water-to-cement ratios between 0.15 and 0.30. Their main conclusions stated that the cement mortar's flowability and resistivity increased with an increment in the w/c ratio; however, they were also highly dependent on the type of CNT used and the quality of dispersion in the cement matrix, which was confirmed with SEM imaging. In the study by Konsta et al. [21], the flowability and setting time of fresh cement mortar were also attributed mainly to the type of carbon nanotubes used, their size, bulk density, and possible functionalization. CNTs with higher bulk density decreased the flow of the mortar by 10% more than samples with lower bulk density.

Typical methods for solving problems with the flowability of cementitious materials include increasing the water-to-cement ratio or the addition of admixtures such as plasticizers or superplasticizers. The following study focuses on methods of providing proper flowability of cement mortars containing varying dosages of multi-walled carbon nanotubes of 0.05 wt.%, 0.1 wt.%, 0.2 wt.%, 0.5 wt.%, and 1 wt.%. The influence of CNT dosage on rheological parameters and the feasibility of optimizing the mix design in accordance with the flowability requirements were studied. The aforementioned properties were studied using two techniques—the flow table method and rheometer measurements. Lastly, the air content and volume density of the fresh mortars were measured to determine CNTs' influence on these values and correlate them with the rheological parameters.

The paper is divided into four sections. Section 1 describes the theoretical background of the research, Section 2 lists materials and testing methods that were used, Section 3 presents the results, and Section 4 concludes the research.

2. Materials and Methods

All the tested specimens were prepared using Portland cement CEM I 42,5. Standard sand, compliant with the PN-EN 196-1 standard [22], was used as an aggregate. The characteristic of standard sand is its specific grain size distribution, which ranges between 0.08 and 2.00 mm. In detail, the cumulated sieve residue in % vs. squared mesh size in mm is as follows: 99 ± 1% (0.08 mm); 87 ± 5% (0.16 mm); 67 ± 5% (0.50 mm); 33 ± 5% (1.00 mm); 7 ± 5% (1.60 mm); and 0% (2.00 mm). The chemical and mineral composition of the tested cement is presented in Tables 1 and 2, respectively, while the basic physical and mechanical properties of the cement are depicted in Table 3.

Table 1. Chemical composition of Ordinary Portland cement CEM I 42.5R (OPC).

Component	Content (%)
Loss in ignition	2.66
Insoluble residue	0.73
SiO_2	20.16
Al_2O_3	5.30
Fe_2O_3	2.69
CaO	63.37
MgO	1.41
SO_3	2.63
Na_2O	0.17
K_2O	0.81
Cl	0.095

Table 2. Mineral composition of Ordinary Portland cement CEM I 42.5R (OPC).

Component	Content (%)
Portland clinker	95.7
-C_3S	62.8
-C_2S	14.9
-C_3A	10.4
-C_4AF	8.3
-Free Cao	1.1
Limestone	4.3

Table 3. Physical properties and strength of Ordinary Portland cement CEM I 42.5R (OPC).

Property	Value
Compressive strength (MPa)	
-after 2 days	28.9
-after 28 days	57.8
Water demand (%)	27.6
Specific surface (cm^2/g)	3510
Initial setting time (min)	175
Soundness (mm)	0.4

Five dosages of multi-walled carbon nanotubes (CNTs) of 0.05 wt.%, 0.1 wt.%, 0.2 wt.%, 0.5 wt.%, and 1 wt.% were added into the mortars through water suspensions. CNTs used in this study were NANOCYL NC7000, with an average length of 1.5 µm, an average diameter of 9.5 nm, and a purity of 90% [23]. CNTs used in the study were pure and were not functionalized. The remaining 10% comprised transition metals that persisted from the chemical vapor deposition production method. These metallic particles, constituting a relatively small proportion of the final product, were not expected to affect the dispersion and rheological properties of the mortars. The basic properties of Nanocyl NC7000 multi-walled carbon nanotubes are listed in Table 4.

Table 4. Properties of Nanocyl NC7000 [20].

Property	Value
Average diameter (nm)	9.5
Average length (µm)	1.5
Carbon purity (%)	90
Transition metal oxide (%)	<1
Specific area (m^2/g)	250–300
Volume resistivity (Ω cm)	10^{-4}

The water suspensions were prepared in 100 g of distilled water (200 g for mixes with a CNT content of 0.5 wt.% and 1.0 wt.%) by the addition of a measured amount of CNTs and naphthalene-based superplasticizer in the ratio of 1:5 by mass. Then, without mechanical stirring, the suspensions were sonicated for 30 min using a Hielscher UP200St ultrasound processor [24] operating at 40% of maximum amplitude. The amplitude was constant and translated into 18 ± 2 W of power. The instrument was equipped with an automatic power control system for the set amplitude, which adjusts power to provide optimal working conditions. The suspensions were placed in an ice bath, and the total sonication time was divided into 30 s intervals to avoid water evaporation.

The exact composition of the prepared samples is given in Table 5.

Table 5. Composition of tested mortars.

Sample	CNT (wt.%)	Water/Cement Ratio	Cement (g)	Water in Total (g)	Standard Sand (g)	Superplasticizer (g)
CNT 0	0	0.5	450	225.0	1350.0	0
CNT 0.05	0.05					0.5
CNT 0.1	0.1					0.7
CNT 0.2	0.2					1.0
CNT 0.5	0.5					5.0
CNT 1	1.0					10.0
2C CNT 0	0		675	337.8		0
2C CNT 0.05	0.05					
2C CNT 0.1	0.1					
2C CNT 0.2	0.2					
2C CNT 0.5	0.5					
2C CNT 1	1.0					

The preparation process of the cement mortars was compliant with the PN-EN-196-1 standard [22]. Water used for the preparation of the suspension was accounted for in water/cement ratio calculations; the remaining water was tap water. Based on preliminary tests, two series of samples were prepared. The first series had standard proportions of cement to sand equal to 1:3, which is compliant with the PN-EN-196-1 standard [22]. The second group had this proportion changed to 1:2 in order to leverage the lubricating effect caused by an increased amount of cement. To improve the flowability of mixes with standard proportions of the components, a polycarboxylate ether-based (PCE) superplasticizer (SP) in powdered form was added into the mix along with the dry components. However, in the case of sample CNT 1.0, the amount of superplasticizer needed to achieve any flow was so high that it was decided to exclude that sample from further tests. The addition required to provide any flow of that mix was 10 g, which is over two times more than the maximum dosage recommended by the manufacturer, which is 4.5 g. For samples with increased amounts of cement, there was no need for the addition of PCE SP in order to achieve flowability that allows for testing in the rheometer.

Immediately after mixing, the flowability of the mortars was tested using a flow table (Figure 1). Based on the PN-EN 1015-3:2000/A2:2007E standard [25], a truncated cone was filled with the tested composites and placed at the center of the flow table. Next, the truncated cone was removed, leaving the sample, which was dropped continuously 15 times at the rate of around 1 drop per second. The flow of the mortar was measured in orthogonal directions, and the value of the flow was calculated as a mean value of these two measurements. The main target for this preliminary test was to assess the required dosage of PCE SP and to establish a reference point for further tests. For samples with standard proportions of the components, the main objective was to obtain a flow possibly close to the reference mix, except for sample CNT 0.5, for which it was more important to just achieve a flow that would allow for testing in the rheometer without using too high a dosage of PCE SP.

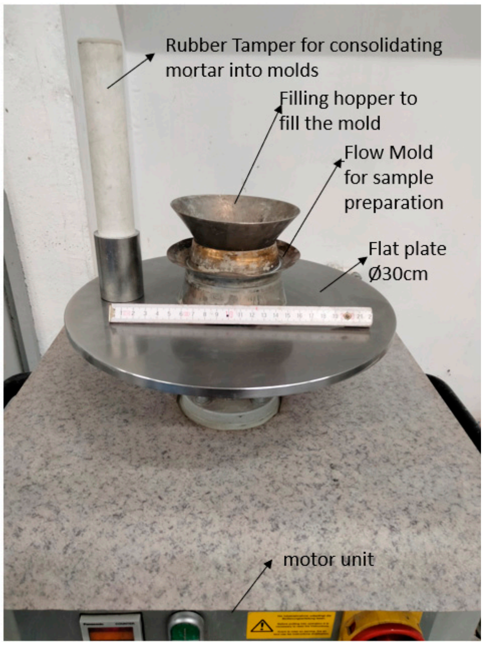


Figure 1. Flow table.

After the flow table test, samples were placed in a rheometer Viskomat NT (Figure 2) for measurement of the rheological parameters. The measurement was made using a stationary probe submerged in mortar, which was placed in a steel cylinder. The cylinder rotated at an increasing speed up to 80 rpm and then was slowed down again. During that time, the torque was measured on the probe and recorded in the time domain. The constant temperature of the mortar was kept equal to 20 °C throughout the duration of the test and in between measurements. Measurements were made 5 min and 30 min after mixing to observe changes in the rheological parameters. The result of the test is the relationship between the rotational speed (N) and torque (M). By fitting a linear function with the results, using the smallest squares method, the rheological parameters g and h can be obtained according to Equation (1). In most cases, the mortar flow is characterized by a hysteresis loop, which contains an upward curve when the shearing speed increases and a down curve when the rheometer starts to slow down. Values needed to calculate the rheological parameters are often taken from the down curve as it contains more stable and reliable values. In the device used, the rheological parameters were calculated using flow curves plotted directly from the rheometer data:

$$M = g + h\,N, \tag{1}$$

where M—Torque in (Nmm), g—yield stress parameter (Nmm), h—plastic viscosity parameter (Nmms), and N—rotational speed (1/s).

The parameters *g* and *h* correspond with the values of yield stress and plastic viscosity in the Bingham model and are used in case of an unknown constant of the specific rheometer. The Bingham model used to describe the cement mortar as viscoplastic material, from which the relation is derived, is represented by Equation (2):

$$\tau = \tau_0 + \eta_{pl}\,\gamma, \tag{2}$$

where τ—shearing stress, τ_0—yield stress, η_{pl}—plastic viscosity, and γ—shearing speed.

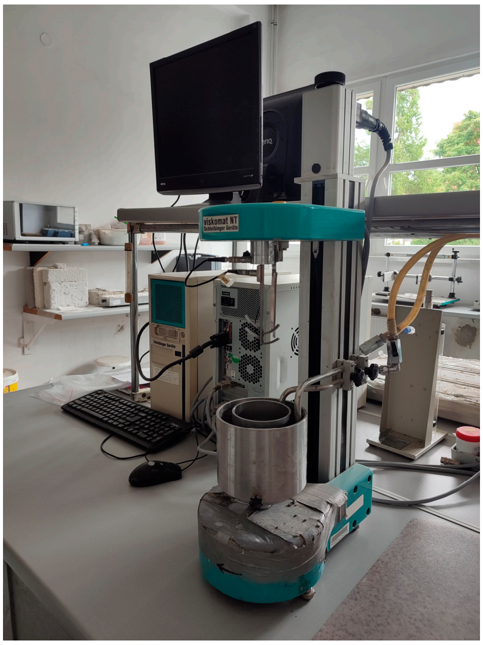


Figure 2. Rheometer Viskomat NT.

The yield stress and the corresponding parameter describe the minimal shear stress needed to initiate the mortar's flow. In the flow table test, this phenomenon takes place under the weight of the material and through consecutive shakes of the table, both of which initiate the flow of the sample. The lower the yield stress, the easier it is to initiate the flow of the mortar up to the point when it can be initiated solely by the material's weight, creating a self-compacting mortar. Therefore, the yield stress can accurately describe the flowability of the mortar in a more precise way, which derives from the material's mechanics. Moreover, the shaking table test is more susceptible to testing conditions, preparation, and accuracy of the flow measurement. Such factors have a lower impact on rheometer measurements.

Plastic viscosity, and by extension, the plastic viscosity parameter, can be used to describe the workability of the mortar. These values represent the shearing stress needed to keep the flow of the mortar at a specific shearing speed and after the flow was initiated by surpassing the yield stress. With workability being often a subjective term, calculating the plastic viscosity parameter can describe it in a quantitative way and allow for a more precise comparison.

After the rheometer measurements, the mortars were tested for air content in accordance with the PN-EN 413-2:2016-11 standard [26]. The method is based on the measurement of the volume change in the fresh mortar under pressure. The measurement is performed using the apparatus presented in Figure 3, which also allows for measuring the volume density of the mortar. During the procedure, fresh mortar is placed in the steel cylinder, which has a volume of 0.75 L, and the mass measurement is taken to calculate the volume density. Then, after thoroughly cleaning the edge of the cylinder, the upper part is placed on top and sealed. Through side valves, the water is injected into the apparatus until it leaves through the opposite valve without air bubbles. The valves are closed, and the air is pumped under pressure to the level designated on the manometer. With the gauge of the manometer set to zero, the pressure is released, and the air content in the fresh mortar can be read directly from the gauge.

Figure 3. Apparatus for measuring air content in fresh mortar.

The volume density of the mortar was calculated directly from Equation (3) using the mass of the mortar measured in the steel cylinder and the known volume of the cylinder equal to 0.75 L:

$$\rho_V = m/V, \quad (3)$$

where ρ_V—volume density, m—mass of the mortar, and V—volume of the cylinder.

3. Results

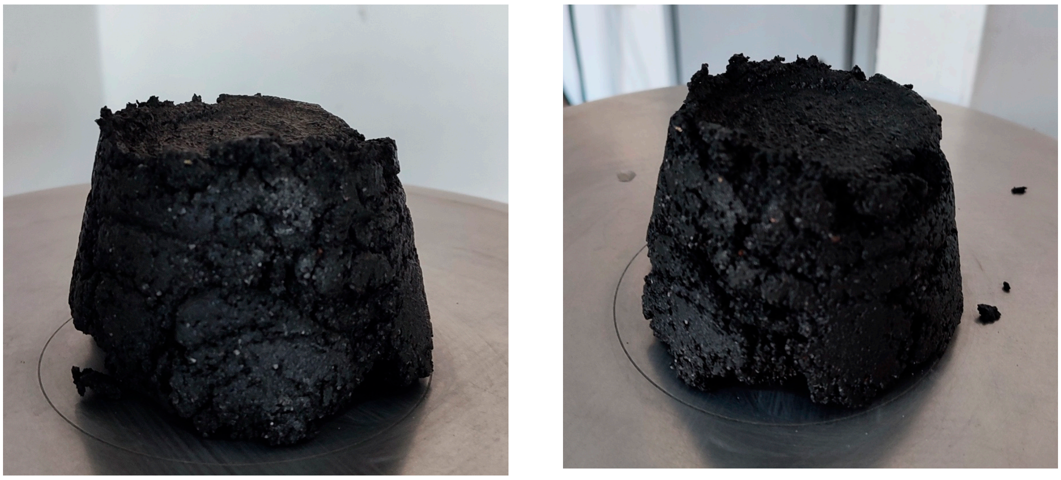
Flow tests and rheometer measurements were conducted to assess the influence of CNTs on the rheological parameters of the mortars. The rheological parameters were calculated using flow curves, which can be plotted directly from the rheometer data. A curve consists of an up curve when the container with mortar accelerates and a down curve when it slows down. From the lower part of the resulting loop, the parameters g and h can be derived according to Equation (1). In order to aid with correlating the results, the air content and volume density were measured.

3.1. Flowability

Results of the flowability test of cement mortars are given in Table 6. The amount of superplasticizer (SP) added into each mix was targeted to achieve the proper flowability of the mortars. It is important to note that the maximum recommended dosage of the PCE superplasticizer (PCE SP) declared by the manufacturer is 1 wt.% of the binder, which is 4.5 g. For the standard mix group, it was possible to achieve a proper flow similar to the reference mix using a relatively small amount of PCE SP. In the case of the mix denoted as CNT 0.5 (Table 5), the flow and SP dosage were a compromise between the possibility of conducting measurements in the rheometer and the usage of a possibly small dosage of PCE SP. Even if the dosage is higher than the recommended dose, no negative effect or segregation was observed. For sample CNT 1.0, it was impossible to achieve any flow at a w/c ratio of 0.5. The addition of a large amount of 10 g PCE SP, which is over two times higher than recommended, did not improve the flow of the mix. It is possible that with such a high amount of CNTs and standard w/c ratio, mixing water is absorbed by the CNT agglomerations and trapped in between the clumped structure of the mortar, rendering the PCE SP useless. The example of the failed consistency of a mortar with 1 wt.% of CNT is shown in Figure 4. This kind of consistency is not suitable for testing on a flow table as consecutive shakes do not cause flow, but rather, scattering of the mortar, and it is not feasible to test in the rheometer. Therefore, mix CNT 1.0 was excluded from the measurement of rheological parameters.

Table 6. Flowability test results.

Sample	CNT (wt.%)	Water/Cement Ratio	PCE Superplasticizer (g)	Average Flow [cm]
CNT 0	0	0.5	0	17.60
CNT 0.05	0.05		0.5	18.60
CNT 0.1	0.1		0.7	18.50
CNT 0.2	0.2		1.0	18.30
CNT 0.5	0.5		5.0	15.30
CNT 1.0	1.0		10.0	n/a
2C CNT 0	0		0	20.70
2C CNT 0.05	0.05			21.10
2C CNT 0.1	0.1			20.20
2C CNT 0.2	0.2			22.10
2C CNT 0.5	0.5			23.55
2C CNT 1.0	1.0			23.60



(**a**) (**b**)

Figure 4. Cement mortar with water trapped in between clumped structures. CNT dosage equal to 1.0 wt.%: (**a**) exemplary sample no.1, (**b**) exemplary sample no.2.

For mixes with a modified composition and cement-to-aggregate ratio of 1:2, a stable flow was achieved for all the mortars, including ones with 0.5 wt.% and 1.0 wt.% of CNT. The flow was obtained without using any PCE SP, with most values close to the reference sample 2C CNT 0. This kind of result could be an incentive to prepare these mixes with lower w/c ratios in future studies without impacting their flow.

3.2. Rheological Parameters

The rheological parameters were measured for all the mixes after the flow table test to investigate the influence of CNTs on the flowability of the mortars in a more precise and in-depth way. The results were compared with flow table measurements in order to look for a correlation in the rheological behavior of the tested mixes.

3.2.1. Yield Stress Parameter

The results of the yield stress parameters are presented in Figures 5 and 6. The measurements were taken 5 min and 30 min after mixing the mortars.

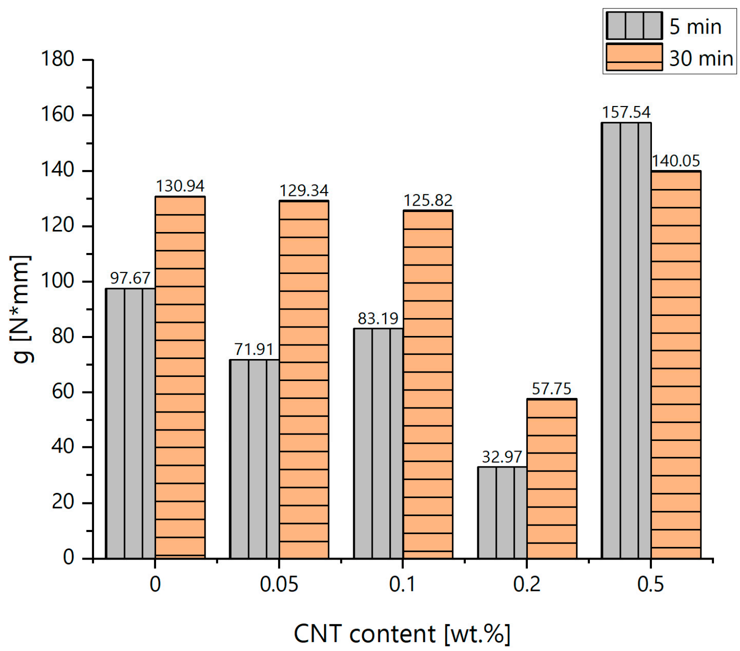


Figure 5. Yield stress parameters for mixes with standard proportions of components.

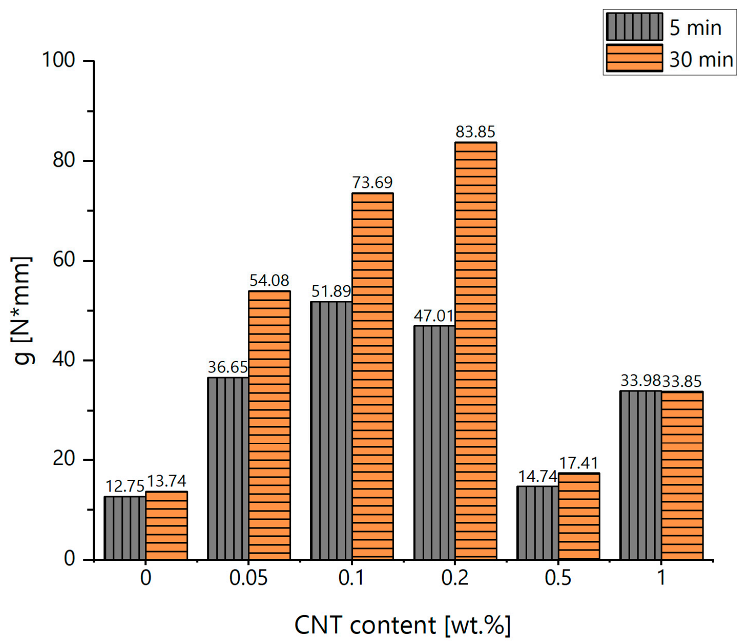


Figure 6. Yield stress parameters for mixes with increased amount of cement.

Figure 5 presents the results of the yield stress parameters for samples with standard mix proportions. Measured values for all mixes besides CNT 0.5 and CNT 0.2 were similar 30 min after mixing. Such a result was expected because the composition of mortars regarding PCE SP dosage was chosen to achieve similar flowability. The value for CNT 0.5 was higher by 6% than the reference, which, considering a significantly lower flow value in the flow table test, can be considered a positive result. For CNT 0.2, there was a significant decrease in the yield stress parameter compared to all the other mixes, which was approximately 55% lower than for samples with lower CNT content. It is possible that at that specific or slightly lower content of CNTs, the additional PCE SP is not hindered by the nanotubes' addition, causing it to influence the flowability in a stronger way. Another thing to consider is the water adsorption of the CNTs. It is possible that for certain lower dosages, they are dispersed better, so more of their large surface area reacts with water and PCE SP, causing the adsorption of water and PCE SP. A possible balance between the superplasticizer promoting the flow and CNTs degrading the flow might also be a factor. In the presented results, for a dosage of 0.2 wt.%, this balance, regarding mutual hindering and adsorption of PCE SP by CNTs, could go in favor of the superplasticizer and improve the flow. For a high amount of CNTs of 0.5 wt.%, the large dosage of CNTs overcame the positive influence of PCE SP and reduced the flow because of the adsorption effect. This explanation could also be valid for the failed mix of CNT 1.0, which exhibited no flow, even with a large dosage of PCE SP, which excluded it from being tested. For the lower dosages of CNTs, their dispersion was good enough that they were able to adsorb water and PCE SP with a larger area.

The yield stress parameter results are also compliant with flow values from the previous test for all samples, except CNT 0.2, with the relationship between mixes similar to results with the flow table method. Results measured 5 min after mixing showed different trends, with higher differences between the mixes, with the only small deviation in this trend at 0.1 wt.%. The yield stress parameter values seemed to decrease with the increase in CNT dosage up to 0.2 wt.%. It is possible that at an early age, CNTs adsorb water and PCE SP slowly, allowing for an easier flow. The process of water adsorption might be slow enough to allow the PCE SP to work in the early phase. The large dosage of 0.5 wt.% overcomes the positive influence of PCE SP in the early phase, allowing it to work after a prolonged time. For all samples besides CNT 0.5, the yield stress parameter was lower than at 30 min after mixing, which is an expected result since, with time, the setting of the mortars begins and reduces its flowability. For CNT 0.5, it is possible that the high amount of PCE SP caused a saturation effect in the early phase and slowly activated with time, which was evident with delayed results.

It Is important to note that the main goal for this series of samples was to overcome the negative influence of CNTs on the flowability of the mortars, so the results are also influenced by the addition of PCE SP and show the influence of CNTs in an indirect way. The results should be interpreted considering the synergistic influences of CNTs and PCE SP, which might add up or counteract each other depending on the dosages.

Results for the yield stress parameter of the mixes with increased amounts of cement are given in Figure 6. For these mixes, without the addition of PCE SP, the impact of CNTs was much more visible. There was a clear trend of the yield stress parameter increasing with the increment of dosage of CNTs 30 min after mixing. All the mixes with CNT, except for 2C CNT 0.5, had significantly higher yield stress parameters than the reference, which shows the impact of CNTs on the flowability of the mixes. The influence of CNTs was mostly visible in the mixes 2C 0.05, 2C 0.1, and 2C 0.2, which had yield stress parameters higher than the reference by 294%, 436%, and 506%, respectively. Such a huge difference can be caused by CNTs' entrapment and adsorption of water. For mixes with an increased amount of cement, the lubricating effect of increased cement content was enough to provide proper flow in the flow table test; therefore, the influence of CNTs was evident only in a more precise rheometer measurement. For mixes 2C CNT 0.5 and 2C CNT 1.0, the difference compared to the reference sample was smaller, with 26% and 146%, respectively. For these large dosages of CNTs, the previous trend of linear increase of the yield stress parameter dropped and started anew. This result could suggest that there is a threshold up to which the flowability of the mortars decreases and then returns to levels similar to the reference sample. There was no visible segregation for these mixes caused by the superplasticizer used for CNT dispersion; however, it cannot be ruled out that a large amount of superplasticizer was not fully adsorbed on CNTs and caused a decrease in the yield stress parameter. Looking at just the CNT dosage, it is possible that larger amounts of

CNTs were not dispersed properly, and their agglomerations presented an overall smaller surface area than the well-dispersed, individual nanotubes, causing the water adsorption effect to be less impactful. When CNTs agglomerate, they tangle together into large clumps. Such agglomerations cause a reduction in the total surface area because areas of CNTs that are in physical contact with each other cannot be considered as an area that is free to adsorb water. Therefore, the total area of clumped nanotubes is not equal to the sum of their individual surface areas, which, in turn, causes a smaller surface area to adsorb water.

Measurements 5 min after mixing showed a similar trend, besides a small deviation for mixes 2C CNT 0.1 and 2C CNT 0.2. The yield stress parameter values increased for measurements taken after 30 min with the reference sample, 2C CNT 0.5 and 2C CNT 1.0, staying nearly the same. Such a result might imply that the lower dosages of CNT of 0.05 wt.%, 0.1 wt.%, and 0.2 wt.% disperse better, and with a lower dosage of superplasticizer in the suspension, they adsorb water due to a larger surface area, which influences the setting time of the mortar, causing it to stiffen quicker. It is important to note that mortars with a large dosage of CNT and the reference-maintained fluidity 30 min after mixing. It could be possible that when there are large amounts of CNT poorly dispersed in the cement matrix, they bundle, and such agglomerations have a lower total surface area, causing them to adsorb and trap less water. For mixes with increased amounts of cement, there is no direct correlation between results from the rheometer and flow table. In the flow table test, all the mixes had similar results, close to the reference, while yield stress parameter results showed significant differences in flowability. Compared to the series with standard proportions, all the mixes have significantly lower yield stress parameters, which is an expected result considering a generally higher fluidity of mixes with increased cement content.

3.2.2. Plastic Viscosity Parameter

Figure 7 presents results for the plastic viscosity parameters of mixes with standard proportions of components. For both measurements, after 5 min and 30 min, there is an increasing trend for plastic viscosity parameters for all the mixes. For the samples CNT 0.05 and CNT 0.1, there was a drop in this trend compared to other mixes. It is possible that during the testing, these mortars were clumped around the probe, therefore having no flow in the material, while the measured value was taken from friction between the clumped mortar and the container. The aforementioned samples have high-yield stress parameters, which could confirm this theory. The flow was initiated at high shearing stress and did not proceed. The plastic viscosity parameter increased for all samples except CNT 0.2 and CNT 0.5; however, the results stayed in line with the trend of other results at their respective measurement times. For all the mixes, besides CNT 0.5 and the first measurement of CNT 0.2, the value of plastic viscosity parameters was lower or close to the reference, which implies a good workability for all of the mixes.

The results for the plastic viscosity parameter of samples with an increased dosage of cement are presented in Figure 8. The measured value for both 5 min and 30 min measurements increased sharply at 0.2 wt.% of CNT dosage and decreased up to that point. At 0.2 wt.%, the plastic viscosity parameter was similar to the reference mix 5 min after mixing. For mixes 2C CNT 0.05 and 2C CNT 0.1, which had high yield stress parameters significantly higher than the reference, the plastic viscosity parameters were significantly lower than the reference, which could suggest that smaller, better-dispersed dosages of CNTs do not hinder the flow of the mortar even if the stress needed for initiation of the flow is high. For large dosages, the plastic viscosity parameter sharply increases, which could indicate that only a high content of CNT hinders the flow of the mortars in a significant way. There were also no large changes in the plastic viscosity parameters throughout the samples after 5-min and 30-min measurements, which can be attributed to the lack of PCE SP in these mixes.

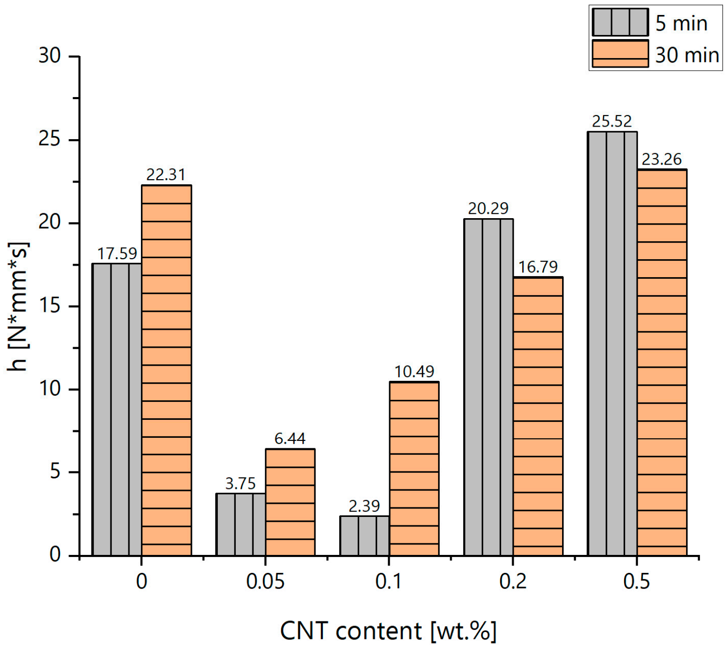


Figure 7. Plastic viscosity parameters of mixes with standard proportions of components.

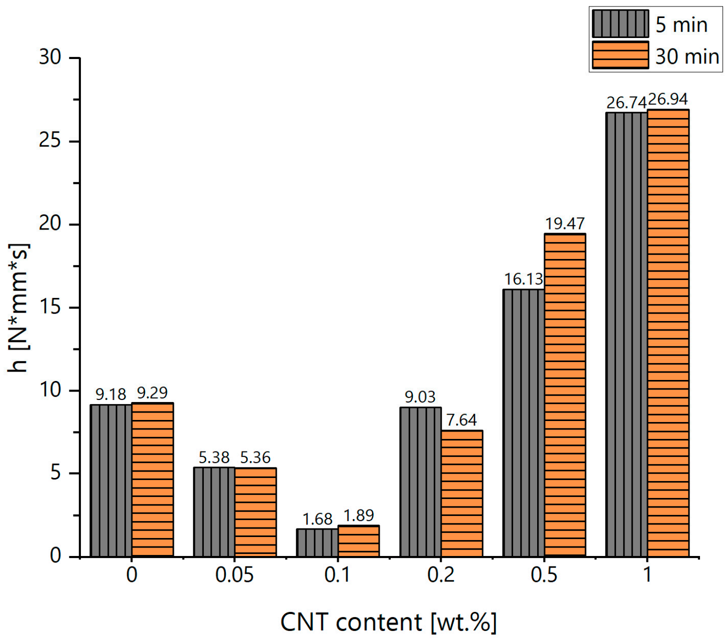


Figure 8. Plastic viscosity parameters of mixes with the increased amount of cement.

Summarizing these results, a high CNT content increased plastic viscosity. The viscosity of cement mortars is influenced by the interactions between solid particles (cement grains, CNT, aggregate) dispersed in the liquid phase, which in this case is water. The forces occurring at the phase boundary can be modified by fluidizing the admixtures (SP), which usually reduces the yield point and viscosity. The effectiveness of fluidizing the admixtures depends on the degree of their adsorption on the hydrating surfaces of the cement grains. Due to their large surface area, nanotubes affect the hydration process and the system of adsorption fields for fluidizing the admixtures, influencing the effectiveness of their operation, and resulting in the overall effect of the content of carbon nanotubes on the plastic viscosity of mortars, which is quite difficult to generalize.

3.3. Air Content

The air content measured for fresh mortars is given in Table 7. In cement-based materials, the air content in the fresh mix can be influenced by the mixing procedures and air entraining admixtures. In the presented mixes, no specific admixture was added from the material side, which implies that differences in air content depend only on CNT content and possible PCE SP. For samples with a standard ratio of the components, there is a visible correlation between flow and air content. Mortars with larger flow also had a higher air content. In that case, the influence of CNTs on the air content might be secondary and expressed just through their influence on the flow value of the mortars and the amount of PCE SP needed. Moreover, all the samples with CNT, except CNT 0.5, had higher air content than the reference, while their flow was similar. Mix CNT 0.5 had both lower flow and air content than the reference. For samples with increased amounts of cement, there was no clear correlation between the air content and either flow or CNT content; however, again, all the mixes had higher air content than the reference. It might be possible that air that entered the mortar during mixing was trapped in between the CNT agglomerations, increasing the air content compared to the reference mix. This conclusion may not be universal since there was no clear correlation between the air content and CNT dosage for samples with increased amounts of cement. The difference compared to the reference was relatively small—between 1.0% and 2.0%. Such a small difference can also be attributed to measurement uncertainty denoted in the PN-EN 413-2:2016-11 standard [26], which is equal to 1.0%. For samples with standard proportions, up to 0.5 wt.% there seems to be a slight correlation; however, since they contain superplasticizers, there is a possibility that PCE SP also contributes to differences in air content.

Table 7. Air content of fresh mortars.

Sample	CNT (wt.%)	Average Flow (cm)	Air Content (%)
CNT 0	0	17.60	4.9
CNT 0.05	0.05	18.60	6.5
CNT 0.1	0.1	18.50	8.0
CNT 0.2	0.2	18.30	8.5
CNT 0.5	0.5	15.30	4.2
CNT 1.0	1.0	n/a	5.7
2C CNT 0	0	20.70	0.9
2C CNT 0.05	0.05	21.10	1.9
2C CNT 0.1	0.1	20.20	2.9
2C CNT 0.2	0.2	20.00	1.6
2C CNT 0.5	0.5	23.55	1.0
2C CNT 1.0	1.0	23.60	2.0

3.4. Volume Density

The volume density of the fresh mortars is given in Table 8. In the case of standard component proportions, it can be noticed that all the mixes had a lower density than the reference. This difference does not seem to depend on CNT dosage in any direct way; however, the results are compliant with air content measurements. Mixes with the highest density had the lowest air content. In the case of mixes with an increased amount of cement, almost all the samples with CNT had a similar density to the reference. These samples had significantly higher flow than samples with standard proportions of components. It is possible that at this high level of fluidity, the air content does not influence the density in a noticeable way. The behavior of samples with increased amounts of cement, which resembles self-compacting mortars, might cause the excessive air to be pushed out under its weight, therefore reducing the influence on density. Considering the previous conclusion about the similarity of air content in mixes with increased amounts of cement, the similar density of the mortars might confirm that conclusion since the density of the mortar is directly related to its air content in relation to other components. A similar relation was also evident when compared to the flow of the tested mixes. For mixes with standard proportions of components, the average flow was higher for mortars with lower density, except for CNT 0.05. For series with increased amounts of cement, all the samples had a similar flow and density.

Table 8. Volume density of fresh mortars.

Sample	CNT (wt.%)	Volume Density (g/cm^3)	Average Flow (cm)
CNT 0	0	2.22	17.60
CNT 0.05	0.05	2.18	18.60
CNT 0.1	0.1	2.07	18.50
CNT 0.2	0.2	2.06	18.30
CNT 0.5	0.5	2.12	15.30
CNT 1.0	1.0	2.17	n/a
2C CNT 0	0	2.16	20.70
2C CNT 0.05	0.05	2.17	21.10
2C CNT 0.1	0.1	2.17	20.20
2C CNT 0.2	0.2	2.21	20.00
2C CNT 0.5	0.5	2.17	23.55
2C CNT 1.0	1.0	2.16	23.60

4. Conclusions

The presented paper aimed to contribute to the knowledge of the influence of carbon nanotubes on the rheology of cement mortars. Five different dosages of CNTs were chosen: 0.05 wt.%, 0.1 wt.%, 0.2 wt.%, 0.5 wt.%, and 1.0 wt.%. Properties of the fresh mixes of two series, with standard proportions of components and with an increased amount of cement, were tested using the flow table method and a rheometer, with their complimentary measurements of air content and volume density.

Assessment of the results was conducted with several goals in mind. First, the influence of CNTs on flow and rheological parameters, with a superplasticizer in the standard mix and without a superplasticizer in the modified mix, was measured. A combined influence of CNTs and PCE SP was considered in the case of the standard mix, and the influence of just CNTs and their suspension was the main factor in modified mixes. Besides the general influence on the rheology of fresh mixes, the goal for standard proportions of components was to assess the possibility of fabricating mortars with standard proportions of components and stable flow controlled by practically viable dosages of superplasticizer. For mixes with the modified ratio of components, the influence of the CNTs was more evident, as their flow was assured through leveraging the lubricating mechanism of the increased amount of cement. In both cases, a negative influence of CNTs on the flowability and flow of the mortar was revealed. As the sonication time of the suspensions was relatively short and a naphthalene-based superplasticizer was used to aid the dispersion, there was a low probability that CNTs were damaged during the process and that any damage to them influenced the results.

Standard mixes required increasing dosages of PCE SP to maintain a stable flow, similar to the reference sample. This result was achieved using a relatively practical dosage of PCE SP, in line with the manufacturer's recommendations for all dosages up to 0.5 wt.%. The dosage of 1.0 wt.% was not feasible for standard proportions and required a change in cement to aggregate ratio. The results for samples with 0.2 wt.% and 0.5 wt.% of CNT indicate that a more complex mechanism might be involved in the flowability assessment. The relation between the positive influence of the PCE SP and the hindering effect of CNTs can be considered, and for some dosages, the sum of these influences can turn the balance of flowability in an unexpected way. For measurements of the yield stress parameters after 5 min from mixing, the values dropped for all of the mixes besides CNT 0.5, which suggests that the aforementioned balance is in favor of PCE SP in the early phase and water adsorption and hindering effects of CNTs activate with time. On the other hand, a dosage as high as 0.5 wt.% seems to hinder the PCE SP more in the early phase, with the beneficial effects of the superplasticizer revealed 30 min after mixing. The influence on the plastic viscosity parameters and, by extension, on the workability of the mortars was increasingly negative for all of the mixes; however, it stayed lower or close to the reference mix, which implies that the composition was correct and the influence of CNTs was negated. Regarding the compliance of the results from the rheometer measurement and flow table, for samples with standard proportions, they stayed in good correlation, as did the air content and density measurements. It can be concluded that for standard mortars with CNTs, a flow table test is a viable method for the assessment of composition modifications required with regard to flowability.

Results for mixes with increased amounts of cement revealed the influence of CNTs in a clearer way. It cannot be ruled out that the superplasticizer used for water dispersion of CNTs also influenced the rheological parameters, however, lack of PCE SP in these mixes means that the influence of CNTs is much more evident. The flowability of mixes with increased amounts of cement measured through yield stress parameters decreased steadily with higher dosages of CNT up to 0.2 wt.%. Following, there was a sharp decrease to levels similar to the reference for 0.5 wt.% and another increase for 1.0 wt.%. Such a phenomenon is explained by the different qualities of dispersion for smaller and larger CNT contents. With further trend increasing again, it can be concluded that CNTs clearly influence the flowability in a negative way up to a certain dosage, and after that, the decrease of flowability starts to degrade again. A similar result was obtained for the plastic viscosity parameter, which was increasing with the dosage of CNTs.

For samples with increased cement content, there was no clear correlation with flow table tests, which did not reveal significant differences in flow that were observed in the rheometer measurements. Air content and volume density of the mortars showed no significant differences between CNT dosages and the reference.

With regards to CNT content, there was no clear trend in both air content and density for both series of samples. It could be concluded that the influence of CNT dosage on these properties was secondary and expressed through their influence on the flow and rheological parameters of the mortars.

Comparing the results of all the conducted tests, the correlations were clearer for samples with standard proportions of components. The higher density of the mortars was a partial result of lower air content, and both of these factors correlated to a decrease in flow. For samples with an increased amount of cement, high flow, similar across the series, also meant a similar density and air content.

The influence of carbon nanotubes at different dosages on the rheological properties of the mortars is a complex mechanism that strongly depends on the dosage and general composition of the mortar. Water adsorption of the CNTs themselves, their reaction and mutual influence with superplasticizer, and the possible influence of the superplasticizer used in aiding the dispersion are all factors that need to be considered separately and synergistically. For the tested mixes, the influence of water adsorbed by CNTs was deemed the most impactful factor considering the flowability and flow of the cement mortars and was also linked with the quality of the dispersion and total surface area of CNTs that are exposed to water. Moreover, the interaction between CNTs and PCE SP used in mortars with standard proportions of components was also considered as the trends for yield stress parameter differed between 5 min and 30 min measurements, and unexpected results were obtained for mix with 0.2 wt.% of CNTs. For mixes with an increased amount of cement, the dispersion and water adsorption of CNTs was probably the main factor influencing the flowability of the mixes. Differences in dispersion quality might result in different total surface areas of the CNTs in contact with mixing water and, therefore, a reduction in flowability. As tested CNTs had a large specific surface area of 250–300 m^2/g, it is possible that individual, well-dispersed nanotubes could present more of that surface area to interact with water than bundles of intertwined CNTs. Considering such a large specific surface area, this difference can add up to a significant value.

It can be concluded that the influence of the CNTs on the rheological parameters of fresh mortars is negative and causes a reduction in the mix flowability and flow of the mixes. Carbon nanotubes lower the flowability of the cement mortars, which is evident in both the flow table test and by increment of yield stress parameters. The correlation between the flow table test and more precise rheometer measurements was good only for one of the tested series. Despite the negative influence of CNTs, it is possible to obtain a stable flow of the mortar using relatively low dosages of PCE SP or by simple modification of the components' ratio. The usage of superplasticizer can be seen as a safer and more stable method for providing proper flowability, as results from the rheometer complied well with an easier and simpler flow table method. This conclusion could make the design of mixes easier to use in practice. On the other hand, a cost estimation could rule in favor of a less reliant but cheaper method of increasing the amount of cement. Another thing to consider while assessing the viability of the presented methods could be their influence on other properties of cement mortars, e.g., mechanical strength, which could elevate one of the techniques despite the abovementioned considerations from only a flow perspective. Moreover, even if the type and dosage of superplasticizer used were found to perform well in the tested mixes, it cannot be ruled out that a different superplasticizer, with a different working principle, could provide better results or similar results at lower dosages. This could further add to the economic aspect of the first of the proposed methods. Such a consideration could be a subject of further study. As a practical side is considered, it could be possible that mortars with CNT and increased cement content could be used as a component for self-compacting concrete mixes. A possibility of preparation of mix with stable flow could be used for creating concrete enhanced with carbon nanotubes but also a practical usage during the construction process. Providing proper workability could be utilized for smaller-scale applications such as repair mortars or functional layers. All of these applications are also dependent on economic factors and the influence of CNTs on different properties of cement mortars.

In order to better understand the influence of CNTs on the rheological parameters of the cement mortars, it would be important to also assess the influence of the dispersion method. There is a need for a volumetric method to assess CNT dispersion in the cement matrix. Even if such assessment is not difficult for water suspensions, it is much more difficult after combining water dispersion with cement. The use of UV-vis spectroscopy would not be sufficient for a non-transparent material of fresh mortar. It might be possible to use electrochemical impedance spectroscopy and link the dispersion rate with the conductivity of the fresh mortar. Such an idea needs a reliable testing method confirmed by reliable tests. The use of a different surfactant, which has a proven neutral effect on the flowability of the mortars, should be considered. Moreover, analyses of other properties that can be influenced by water adsorption of CNTs, such as shrinkage or porosity, along with microstructural analyses of the matrix, could help in the assessment of the scale of water adsorption of CNTs, its dependence on dispersion quality and the influence on the cement mortars. This kind of long-term influence, connected with CNT addition, could be an important factor in finding the optimal mix proportions from a rheological perspective. Moreover, as carbon nanotubes could influence the microstructure, hydration dynamics, and mechanical properties of cement mortars, the influence on all of these important factors should be balanced with the rheological properties.

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