

Degree of MWCNT suspension dispersity and its influence on rheology of cement pastes

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Abstract. Modification of cement pastes by MWCNT (multi-walled carbon nanotubes) suspension enables to manage by their rheological properties due to the nanometer size of MWCNT. Dispersity degree of MWCNT suspension may be determined by the number of factors such as type and intensity of homogenization equipment, duration of homogenization and some other. The aim of present research consists in the establishment of optimal methodology of MWCNT suspension homogenization and investigation of dependence between dispersity degree of MWCNT suspension and rheological properties of cement pastes with that suspension. Two types of homogenization methods of MWCNT were tested such as ultrasonication and homogenization based on rotor-stator principle. The ultrasonication treatment about 6 min was established as optimal homogenization technique in the course of the current research. The yield stress and plastic viscosity of cement paste modified by MWCNT suspension increased in comparison with cement paste modified by polycarboxylate ether plasticizer (PCE) in 5 and 30 min after cement pastes mixing. Time of ultrasonication of MWCNT suspension more than 6 min did not have the influence on rheological behaviour of cement pastes. Addition of MWCNT decreased the volume coefficient of water bleeding of cement paste with PCE by 18%.

Keywords: multi-walled-carbon nanotubes, cement paste, rheology, dispersity degree, homogenization.

Introduction

Fundamental rheological parameters of cement systems, such as yield stress, thixotropy and viscosity, depends on water content, cement composition, aggregates particle size distribution, type of applied additives and admixtures, time after mixing of cement systems, type of mixing during the test and some other properties (Yahia, Mantellato, & Flatt, 2016; Jiao et al., 2017; Secrieru et al., 2018).

The particle size of constituents in cement systems and interactions between them are one the key factors which determine the rheological behavior and technological properties of mixtures. Hydrodynamic, colloidal, inertial, gravitational forces and Brownian motion underlie the interactions between particles in cement systems (Roussel, Lemaître, & Coussot, 2010; Ma, Qian, & Kawashima, 2018a).

The size, shape, distance between aggregates in concrete and mortars have an influence on the rheological properties of cement systems. The influence of sand content on rheological properties of concrete mixtures was analyzed in the (Dauksys & Skripkiunas, 2018). The increase of sand content from 50 to 65% increased the yield stress approximately 1.5 times, decreased viscosity and dilatancy index and led to higher workability of concrete mixture.

Widespread application of mineral additives and chemical admixtures for generation of high workability and self-compacting concrete in construction practice requires the understanding of their action mechanism in cement systems (Bentz, Ferraris, Galler, Hansen, & Guynn, 2012; Štefančíč, Mladenovič, Bellotto, Jereb, & Završnik, 2017; Vance, Kumar, Sant, & Neithalath, 2013; Zhang, Luo, Kong, Wang, & Gao, 2018; Qian et al., 2018; Smirnova, 2018a, 2018b). Development of self-compacting concrete (SCC) requires not only application of effective polymer plasticizers to reach high flowability, but also chemical admixtures to provide mixture stability (Isik & Ozkul, 2014). Nanoadditives with nanometer dimensions of particles, high aspect ratio and unique physical, chemical, electrical

properties may be used as a perspective tool to improve the stability of concrete mixture. With an increase of content of graphite nanoplatelet and carbon nanofiber from 0.05 to 0.30% by weight of binder, W. Meng et al. obtained the increase of plastic viscosity (Meng & Khayat, 2018).

Nano-silica can be used as thickening agent of cement paste (Lavergne, Belhadi, Carriat, & Ben Fraj, 2019). E. Garcia-Taengua et al. observed the increase of viscosity of fresh mortar modified by 2% of nano-silica (García-Taengua, Sonebi, Hossain, Lachemi, & Khatib, 2015).

Application of cellulose filaments (CF) with diameter of 30–400 nm, length of 100–2000 nm and aspect ratio of 100–1000 nm for modification of cement pastes and SCC demonstrated the significant viscosity modifying effect in dosage of CF from 0.05 to 0.30% by weight of binder (Hisseine, Basic, Omran, & Tagnit-Hamou, 2018).

Carbon nanotubes (CNT) increase all rheological parameters in cement mortar. However, their application in blended systems may have the opposite effect on rheology (Ma, Qian, & Kawashima, 2018b). Frequently, carbon nanotubes are used in combination with dispersants applied for their better distribution. The researchers (Nadiv et al., 2016; Mendoza Reales et al., 2018) highlighted the importance of interactions between dispersing agent and CNT.

The main barrier in wide application of nanomaterials in construction practice caused by their distribution inside volume of water suspension and cement systems. The effectiveness of homogenization is directly correlated with dispersion degree of generated suspension and has the further influence on rheological properties of cement systems modified by nanoadditives. Homogenization of nanomaterials can be provided by different methods, such as bead milling process (Yoshio et al., 2011), hydrodynamic, acoustic cavitation (Chai, Yusup, Chok, Arpin, & Irawan, 2016; Kiu, Yusup, Soon, Arpin, & Samion, 2016) and ultrasonication technique, which is more frequently used nowadays (Zou et al., 2015; Konsta-Gdoutos, Metaxa, & Shah, 2010). The listed methods allow to avoid agglomeration process in water suspension of nanoparticles and make it possible to use them for modification of cement systems in fresh and hardened state.

The aim of current research is to compare ultrasonication method and method based on rotor-stator principle for homogenization of MWCNT suspension with establishment of optimal technique and further investigation of dependence between dispersity degree of MWCNT suspension and rheological properties of cement pastes modified with that suspension.

Materials and Methods

Materials

The Portland cement without mineral additives CEM I 42.5 R conforming to EN 197-1 with water consumption 26.6% and fineness by Blain of 3552 cm²/g was used as a binder.

The curve of cement particle size distribution, determined by CILAS 1090, is presented in Figure 1a. The main content of particles of cement is placed in the range from 1 to 80 μm in size with average value of cement particles size about 14.28 μm.

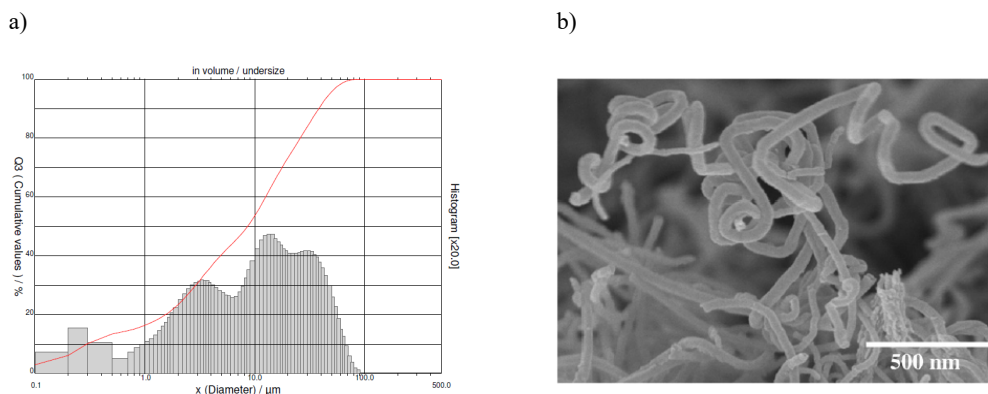


Figure 1. The particle size distribution of Portland cement without mineral additives(a); SEM image of MWCNT (b)(source: (Zou et al., 2015))

The MWCNT suspension was prepared from the masterbatch pellets “Graphistrength CW 2-45” produced by the company “Arkema” (France). The masterbatch contains 45 wt. % of MWCNT and 55 wt. % of carboxymethylcellulose (CMC). The MWCNT was characterized by filament length of 100 – 10000 nm and diameter of 15–20 nm. SEM image of MWCNT is presented in Figure 1b. Distilled water was used as dispersion medium for MWCNT. The polycarboxylate ether of MPEG type (PCE) with specific gravity 1.100 g/cm³, pH up to 5 at 20 °C and dry content equal to 50.5% was applied in the research as dispersant to distribute the nanotubes in the volume of suspension.

Table 1. Composition of MWCNT suspensions

Specimen	MWCNT in volume of suspension, %	PCE in volume of suspension, %	Ratio between MWCNT and PCE	Time of ultrasonication /rotor-stator homogenization, min
Ultrasonication				
S0	0.28	1.12	1:4	0
S3	0.28	1.12	1:4	3
S6	0.28	1.12	1:4	6
S9	0.28	1.12	1:4	9
S12	0.28	1.12	1:4	12
Rotor-stator homogenization				
ST6	0.28	1.12	1:4	6
ST30	0.28	1.12	1:4	30

The compositions of generated MWCNT suspensions are presented in Table 1. The ratio of 1 to 4 between MWCNT and PCE was set based on results of literature review.

Methods

The MWCNT suspension was prepared in following way. The masterbatch pellets “Graphistrength CW 2-45” were immersed in hot water with temperature about 75 °C and stirred by standard mixer agitation about 1000 rpm during 5 min.

After that the MWCNT suspension was subjected to ultrasonication for a duration of 3, 6, 9, 12 min. The ultrasonication was performed by Bandelin Sonopuls HD 3400 ultrasonic homogenizer (400 W, 20 kHz) with probe VS 200 T (Ø 25 mm, amplitude – 82 µm). The time of ultrasonication was taken according to the data presented in (Liebscher et al., 2017), where the same ultrasonication equipment was used.

The ultrasonication technique was compared with method based on rotor-stator principle, which was recommended by nanotubes producer “Arkema”. Rotor-stator homogenization was performed by Ultra-Turrax Dispersers T25 digital with frequency 50/60 Hz at speed 5000 rpm for a duration 6 and 30 min. The duration of treatment was selected based on the technical data sheet.

The ultrasonication treatment is based on generation of ultrasonication waves, which promote to the dispergation of nanoparticles in the volume of water suspension. On the other hand, the rotor-stator equipment for homogenization consists of a rotor within a stationary stator. The high speed and minimal gap between the rotor and stator provide the generation of strong shear forces which lead to homogenization of particles.

The prepared MWCNT suspension was characterized by pH, electrical conductivity tests, measurements of surface tension, particle size analysis and measurements of zeta potential. pH test was performed by pH-meter Mettler-Toledo EL20 with limits of error ±0.01 pH. The electrode with gel electrolyte was used. Electrical conductivity was measured by Mettler-Toledo EL30. The measurement range of it is 0.1 to 199.1 mS/cm with limits of error ±0.5%. The electrode for medium to high conductivities (10 µS/cm ... 500 mS/cm) was applied.

Surface tension of MWCNT suspension was measured based on stalagmometric method by stalagmometer-Traubewith measurement accuracy 1 mN/m. The stalagmometric method consists in determination of drops number which are formed in the course the flowing of liquid from capillary tube of known volume. The surface tension of studied liquid is calculated based on formula (1):

$$\sigma_x = \sigma \cdot \frac{N}{N_x} \cdot \frac{d_x}{d}, \quad (1)$$

where: σ – surface tension, mN/m; N – number of drops; d – density, g/cm³ of water; σ_x , N_x , d_x – the same for studied liquid.

Particle size analysis and zeta potential measurements of MWCNT suspension were performed by particle size and zeta potential analyser Delsa Nano C, Beckman Coulter. Particle size analysis was based on the principle of photon correlation spectroscopy with resolution of measurements placed from 0.6 nm to 7 µm and measurement accuracy of 0.05 nm. Zeta potential measurement was based on the electrophoretic light scattering principle with range of measurement from –150 mV to +150 mV and measurement accuracy of 0.005 mV. Polydispersity index (PI) was calculated as ratio between the square of standard deviation and the square of mean diameter.

The cement pastes with composition presented in Table 2 were prepared for rheological test.

Table 2. Content of materials for 1 m³ in tested cement pastes

Specimen	Cement, kg	Water, kg	W/C ratio	MWCNT, % bwoc*	PCE, % bwoc*
REF	935	280	0.30	0	0
PCE	935	280	0.30	0	0.3
S3	935	280	0.30	0.08	0.3
S6	935	280	0.30	0.08	0.3
S9	935	280	0.30	0.08	0.3
S12	935	280	0.30	0.08	0.3

*bwoc – by weight of cement.

The MWCNT suspensions with composition presented in Table 1 were used as mixing liquid and mixed together with cement for 180 s at high speed by standard mixer according to EN 196-1.

Rheological properties of cement pastes were tested in different time after pastes mixing: 5 and 30 min. The rheology tests were carried out at temperature 20±2 °C. The rotational rheometer Rheotest RN 4.1 with coaxial cylinders was used for rheological tests. The scheme of cylinder measuring system of rheometer is presented in Figure 2a. The assembled measuring cup (1) with coupling is poured by cement paste (3), and fixed in the equipment stand. The rotating cylinder rotor (2) is placed inside the measuring cup (1) with gap between them of 1.5 mm and rotates during the test. Shear rate ranged from 100.0 s⁻¹ to 0.1 s⁻¹ during testing time. The shear rate mode presented in Figure 2b was used in the current experiments.

Cement pastes after mixing between tests were placed in the plastic containers and carried in the laboratory conditions with temperature 20±2 °C and relative humidity not less than 65%. The cement pastes were premixed before rheological test by hands in plastic container using metallic scoop for 1 min.

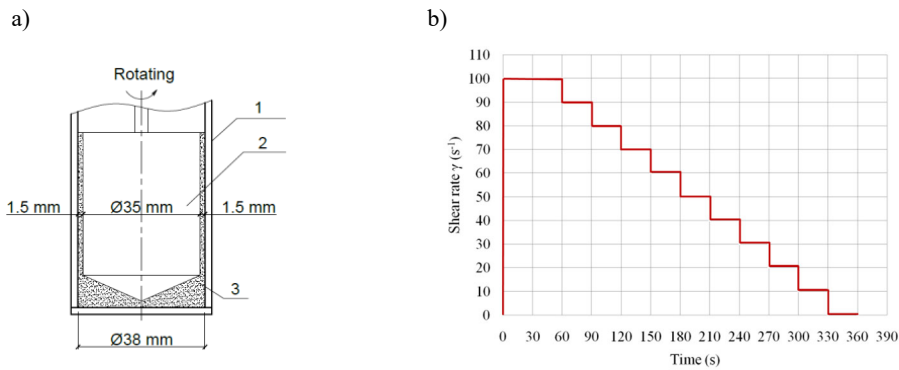


Figure 2. Scheme of cylinder measuring system Rheotest RN 4.1 (a) and shear rate mode of rheological test (b)

The yield stress (τ_0) and plastic viscosity (K) were determined in the course of approximation of flow curve (dependence between shear stress (τ) and shear rate (γ) in the range from 0.1 to 100 s⁻¹ based on Herschel-Bulkley model. The Herschel-Bulkley model is more widely used model for description of flow behavior of cement systems, which is expressed by the Eq. (2):

$$\tau = \tau_0 + K \cdot \gamma^n, \quad (2)$$

where: τ – shear stress, Pa; τ_0 – yield stress of the cement paste, Pa; K – plastic viscosity, Pa·s; γ – shear rate, s⁻¹; n – shear thinning or shear thickening index if $n < 1$ or $n > 1$, respectively (Yahia, Mantellato, & Flatt, 2016).

Amount of bleeding water was determined according to the following method. 50 g of MWCNT suspension was poured into the 100 ml container, after that 50 g of cement was added for 1 min. Subsequently, MWCNT suspension and cement were mixed continuously for 4 min in container and were poured into a graduated cylinder of 30 ml. The graduated cylinder with cement paste was placed onto the table and the initial volume of cement paste was written. The graduated cylinder was standing without moving and shaking during the test.

The volume of sedimented cement in cement paste after water bleeding was determined after 2 hours since the reference point and every 30 min after it. The observation of water bleeding was performed till the moment when the volume of cement paste did not change. Water separation (water bleeding) was calculated according the Eq. (3):

$$K = \frac{a-b}{a} \cdot 100, \quad (3)$$

where: K – coefficient of water bleeding, %; a – initial volume of cement paste, cm³; b – volume of sedimented cement, cm³.

Results and discussion

Analysis of MWCNT suspension homogeneity

The results of particle size analysis of MWCNT suspensions subjected to ultrasonication and rotor-stator homogenization are presented in Figure 3a.

The standard mixer agitation during 5 min are not enough to distribute the particles with nanometer size and elongated shape. The average particle size diameter of MWCNT suspension reached 869.5 nm as it is presented in Figure 3a. Ultrasonication for 3 min demonstrated the decrease of average diameter from 869.5 to 300.8 nm, what is the evidence of ultrasonication effectiveness. Ultrasonication for 6 min enables to get the average particle size diameter equal to 245.2 nm. The further increase of ultrasonication time did not revealed the significant changes in particle size distribution. In other words, ultrasonication for 6 min was obtained as optimal homogenization time.

Figure 3a presents that homogenization by rotor-stator technique for 6 min allows to obtain the particles with average diameter up to 423.3 nm. The increase of homogenization time to 30 min did not decrease the average particle size significantly.

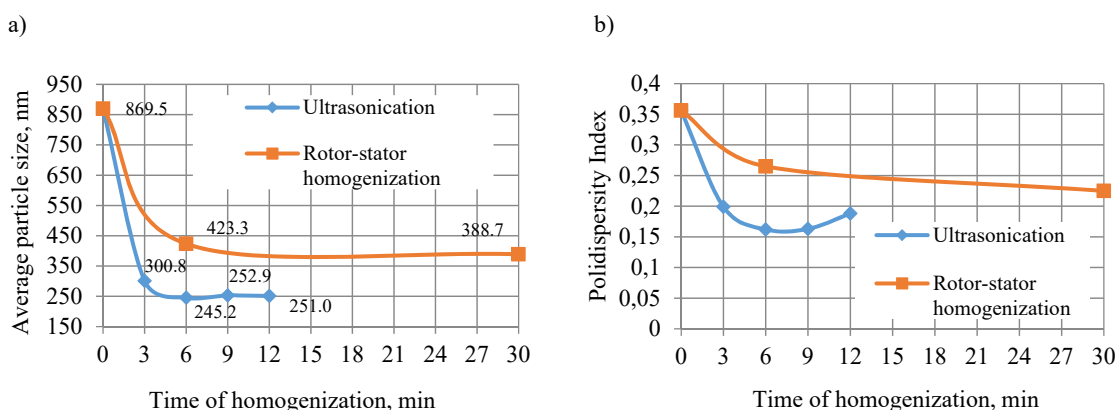


Figure 3. Particle size analysis of MWCNT suspension subjected to ultrasonication and rotor-stator homogenization (a); Poldispersity Index of MWCNT suspensions (b)

As it is seen in Figure 3, the ultrasonication treatment is more effective in comparison with rotor-stator homogenization in chosen time intervals and frequency. The difference in average diameters of particles may be caused by difference in principles of homogenization, which were described above. The changes of poldispersity index (PI), which presents the dimensionless measure of the broadness of the size distribution, are showed in Figure 3b. The value of PI closer to 0 confirms the better homogeneity of nanoparticles in water suspension (Rane & Choi, 2005). The MWCNT suspension after 6 min of ultrasonication had the smaller average diameter of particles and PI of 0.15, which confirms the homogeneous distribution.

The results of pH, electrical conductivity, zeta potential and surface tension measurements for MWCNT suspension with different ultrasonication time are available in Table 3.

Table 3. Properties of MWCNT suspensions with different ultrasonication time

Specimen	pH	Electrical conductivity, $\mu\text{S}/\text{cm}$	Surface tension, mN/m	Zeta potential, mV
S3	5.07	0.0744	67	-52.09
S6	5.05	0.0754	67	-51.29
S9	4.84	0.0755	67	-54.46
S12	4.87	0.0698	67	-53.24

Results presented in Table 3 revealed that pH, electrical conductivity, surface tension and zeta potential of MWCNT suspension subjected to different time of ultrasonication remained on the same level.

To sum up, ultrasonication for 6 min was obtained as optimal homogenization method in the course of the current research. The observation of changes in pH, electrical conductivity, surface tension and zeta potential did not reveal the significant dependency between them and duration of ultrasonication.

Rheological properties of cement pastes with MWCNT suspension

Figure 4 and 5 present the flow behavior of cement pastes modified by MWCNT suspension with different duration of ultrasonication. The equations derived based on the Herschel-Bulkley model, which describe the flow behavior of cement pastes, are listed in Table 4. The thixotropy indexes are listed in Table 4 as well.

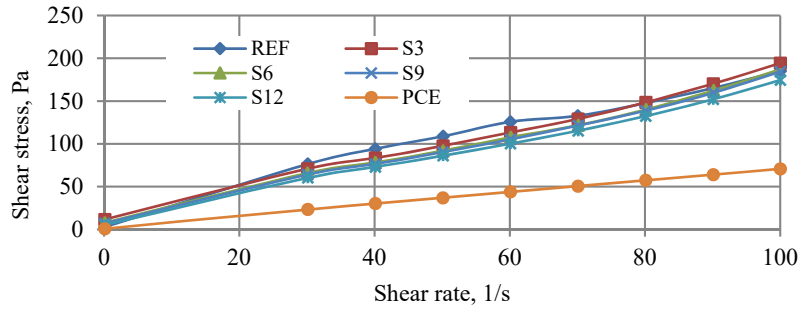


Figure 4. Flow curves of nanommodified cement pastes after 5 min of cement paste mixing

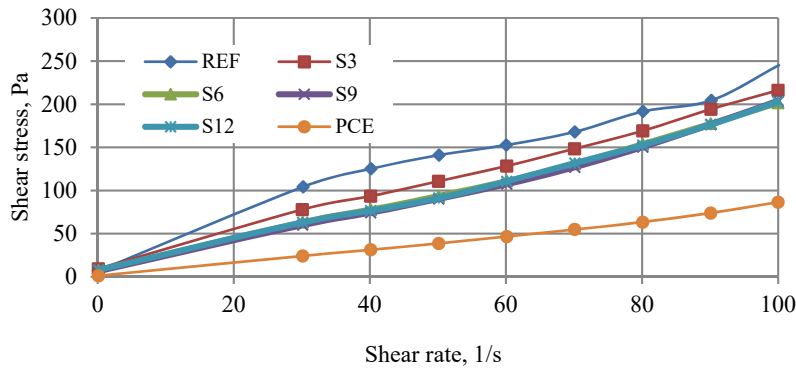


Figure 5. Flow curves of nanommodified cement pastes after 30 min of cement paste mixing

Figure 4 demonstrates that modification of cement pastes by MWCNT suspension leads to the increase of shear stress and consistency factor in comparison with cement pastes modified by PCE in 5 min after mixing of cement pastes. Time of ultrasonication did not influence on rheological behaviour of cement pastes in 5 min after mixing. The cement pastes modified by PCE and MWCNT suspensions with different duration of ultrasonication treatment demonstrated the shear thickening behaviour in comparison with reference sample without admixtures characterized by shear thinning behaviour.

As it seen from Figure 5, the flow behaviour of studied cement pastes did not change significantly in 30 min after their mixing. The duration of ultrasonication treatment did not have the significant effect on the rheological properties of cement pastes in 30 min after their mixing.

Table 4. Low curves equations based on Herschel-Bulkley model and thixotropy indexes of cement pastes

Specimen	Flow curves equations		Thixotropy Indexes	
	After 5 min	After 30 min	After 5 min	After 30 min
REF	$\tau = 2.96 + 1.97\gamma^{0.996}$	$\tau = 6.9 + 5.91\gamma^{0.790}$	0.996	0.790
PCE	$\tau = 0.85 + 0.62\gamma^{1.034}$	$\tau = 1.24 + 0.37\gamma^{1.177}$	1.034	1.177
S3	$\tau = 11.75 + 1.73\gamma^{1.011}$	$\tau = 9.3 + 2.07\gamma^{1.002}$	1.011	1.002
S6	$\tau = 7.8 + 1.72\gamma^{1.009}$	$\tau = 7.2 + 0.91\gamma^{1.160}$	1.009	1.160
S9	$\tau = 7.2 + 1.71\gamma^{1.008}$	$\tau = 6.5 + 0.90\gamma^{1.165}$	1.008	1.165
S12	$\tau = 5.3 + 1.64\gamma^{1.008}$	$\tau = 7.98 + 0.91\gamma^{1.165}$	1.008	1.165

Figure 6a demonstrates the changes in yield stress. The addition of PCE plasticizer led to the decrease of yield stress in comparison with reference sample without plasticizer. Modification of cement systems by MWCNT

suspensions with different ultrasonication treatment increased the yield stress in comparison with sample modified by PCE plasticizer at the same dosage and reached the values closer to reference cement paste.

The similar rheological behaviour was observed in 30 min after cement pastes mixing. The addition of MWCNT suspension increased yield stress in comparison with sample modified only by PCE plasticizer. The changes in duration of ultrasonication treatment did not have the significant influence on yield stress of cement pastes in 5 and 30 min after cement pastes mixing.

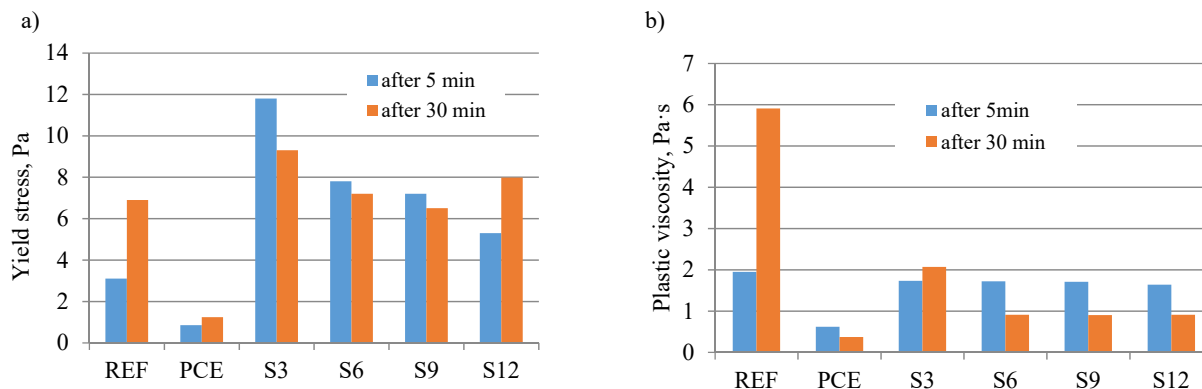


Figure 6. Yield stress (a) and plastic viscosity (b) of nanomodified cement pastes in 5 and 30 min after mixing

Figure 6b presents the changes in plastic viscosity of cement pastes modified by PCE plasticizer and MWCNT suspension. Modification of cement pastes by MWCNT suspension revealed the increase of plastic viscosity. The nanomodified cement pastes in 5 min after mixing had the same plastic viscosity. However, plastic viscosity was different for nanomodified cement pastes in 30 min after mixing. The maximum increase of plastic viscosity was observed for cement pastes modified by MWCNT suspension with 3 min ultrasonication treatment. The plastic viscosity increased from 0.37 to 2.07 Pa·s in comparison with cement paste modified by PCE.

The increase of yield stress and plastic viscosity of cement pastes modified by MWCNT suspensions with different ultrasonication treatment may be caused by colloidal forces, such as Van-der-Waals, electrostatic and steric forces, which appear between cement particles in the course of their modification by plasticizer and MWCNT. The time of ultrasonication treatment of MWCNT suspension did not reveal the significant influence on rheological behaviour of cement pastes.

Bleeding of cement pastes modified by MWCNT suspension

Figure 7 presents the results of bleeding for cement pastes modified by PCE and MWCNT suspensions with different duration of ultrasonication.

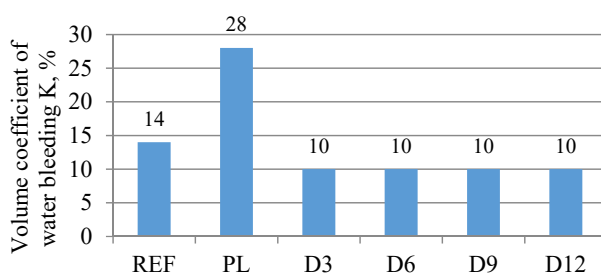


Figure 7. Bleeding of nanomodified cement pastes

Modification by MWCNT suspension leads to decrease of volume coefficient of water bleeding by 4% in comparison with reference sample without admixtures. Addition of MWCNT enables to decrease the volume coefficient of water bleeding for cement paste with PCE by 18%. The observed changes may be explained by the fine size of MWCNT and their high specific surface.

Conclusions

The following conclusions are drawn in this study:

1. The particle size was decreased from 869.5 nm to 388.7 and from 869.5 nm to 245.2 nm in the case of rotor-stator homogenization and ultrasonication, respectively. Ultrasonication of MWCNT suspension for 6 min

was obtained as optimal homogenization method in the course of the current research performed by Bandelin Sonopuls HD 3400 ultrasonic homogenizer (400 W, 20 kHz) with probe VS 200 T (\varnothing 25 mm, amplitude – 82 μ m). The observation of changes in pH, electrical conductivity, surface tension and zeta potential of MWCNT suspensions with different time of ultrasonication treatment did not revealed the significant dependency between them and duration of ultrasonication.

2. The yield stress and plastic viscosity of cement paste modified by MWCNT suspension increased in comparison with cement paste modified by PCE plasticizer in 5 and 30 min after cement pastes mixing. The increase of yield stress and plastic viscosity for cement pastes modified by MWCNT suspensions with different ultrasonication treatment may be caused by Van-der-Waals, electrostatic and steric forces, which appear between cement particles in the course of modification by plasticizer and MWCNT. Time of ultrasonication of MWCNT suspension more than 6 min did not have the influence on rheological behaviour of cement pastes.
3. Modification by MWCNT suspension leads to decrease of volume coefficient of water bleeding by 4% in comparison with reference sample without admixtures. Addition of MWCNT enables to decrease the volume coefficient of water bleeding for cement paste with PCE by 18%.
4. Effect of MWCNT suspension on the rheological properties of cement pastes is one of the fundamental step in understanding of their action mechanisms, however the further researches on hydration kinetics, mechanical properties and durability of nanomodified cement composites are necessary.

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