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Research Article

Determining the Surfactant Consistent with Concrete in order to Achieve the Maximum Possible Dispersion of Multiwalled Carbon Nanotubes in Keeping the Plain Concrete Properties

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A new surfactant combination compatible with concrete formulation is proposed to avoid unwanted air bubbles created during mixing process in the absence of a defoamer and to achieve the uniform and the maximum possible dispersion of multiwalled carbon nanotubes (MWCNTs) in water and subsequently in concrete. To achieve this goal, three steps have been defined: (1) concrete was made with different types and amount of surfactants containing a constant amount of MWCNTs (0.05 wt%) and the air bubbles were eliminated with a proper defoamer. (2) Finding a compatible surfactant with concrete compositions and eliminating unwanted air bubbles in the absence of a common defoamer are of fundamental importance to significantly increase concrete mechanical properties. In this step, the results showed that the polycarboxylate superplasticizer (SP-C) (as a compatible surfactant) dispersed MWCNTs worse than SDS/DTAB but unwanted air bubbles were removed, so the defoamer can be omitted in the mixing process. (3) To solve the problem, a new compatible surfactant composition was developed and different ratios of surfactants were tested and evaluated by means of performance criteria mentioned above. The results showed that the new surfactant composition (SDS and SP-C) can disperse MWCNTs around 24% more efficiently than the other surfactant compositions.

1. Introduction

In recent years, the use of cementitious composites has led to significant improvements in the construction and transport industry [1]. Since tensile strength, modulus of elasticity, and thermal and electrical conductivity of MWCNTs are far superior to those of ordinary fibers, they have received much attention in the preparation of cementitious composites [2–4]. The challenge of effective and uniform dispersion of MWCNTs in the mixture is due to the natural tendency of agglomeration of MWCNTs and their high surface activity [5, 6]. Although the issue of effective dispersion of MWCNTs in the concrete mixtures has been investigated in many studies, there are some difficulties to achieve the effective dispersion of MWCNTs [1]. Since water plays an essential role in preparing cementitious materials reinforced with MWCNTs, a convenient solution for making cementitious materials containing MWCNTs is to homogeneously disperse MWCNTs in water prior to their combination with the cement [7]. One of the most common mechanical methods for dispersing MWCNTs in water is the use of an ultrasonic device. The ultrasonic method is not sufficient to uniformly disperse MWCNTs in the water [7], but it can be used as a complement to the chemical method [8]. One of the most widely used methods for the effective dispersion of MWCNTs in water is the use of surfactants so that the formation of CNT agglomerates is prevented and MWCNTs are uniformly dispersed in the water [1, 7]. This noncovalent method is straightforward and classically used to disperse both organic and inorganic particles in aqueous solutions [9]. The concentration of surfactant and type of interaction with it are known to play a significant role in the phase behavior of classical colloids [10] as well as carbon nanotubes [11, 12]. Basically, surfactants due to electrostatic and steric

Table 1: Properties of MWCNTs.

Type of additive	Characteristic (research center)	MWCNTs diameter (nm)	MWCNTs length (μm)	Tensile strength (GPa)	Purity	Specific gravity	Electrical conductivity (Ohm-cm)
CNT	MWCNT (Research Institute of Petroleum Industry)	10–20	10 ± 3	About 100	≥95%	About 1.5	$<10^{-2}$

repulsion forces prevent the formation of CNT agglomerates [13]. In this regard, the researchers have attempted to enhance dispersion of MWCNTs in water using different surfactant materials including sodium dodecylbenzenesulfonate (NaDDBS), sodium deoxycholate (NaDC), Triton X-100 (TX100), Gum Arabic (AG), and cetyltrimethylammonium bromide (CTAB) [14]. They came to the conclusion that the NaDDBS and TX100 with a mixing ratio of 3 : 1, respectively, by weight of cement and with the concentration of 0.02 grams per mL exhibit the best dispersion capability [15, 16]. In order to decrease CNT aggregative tendency in water, the ionic surfactants, sodium dodecyl sulfate (SDS) [17–19] and dodecyl-benzene sodium sulfonate (NaDDBS) [20, 21], were commonly used. The high dispersive efficiency of NaDDBS [22] and even better efficiency of Dowfax surfactant (anionic alkyldiphenyl-oxide disulfonate) which has twice the charge of NaDDBS and a dibenzene group [20] are mainly due to the benzene ring along the surfactant. Also, aerosol OS surfactant (sodium diisopropylnaphthalene sulfonate) showed higher fractions of individual tubes compared to the results obtained with NaDDBS and Dowfax surfactants, as confirmed by UV-vis spectroscopy analysis [20]. In another study conducted by Sharifi et al., they used SDS and dodecyltrimethylammonium bromide (DTAB) with a mixing ratio of 9 : 1, respectively, by weight of cement and with the concentration of 8 mMol/L in order to disperse MWCNTs in water. They reported this combination as the best combination for dispersing MWCNTs in water compared to other surfactants [23, 24]. It should be noted that the surfactant creates foam when making concrete which may lead to problems for the cement hydration and concrete strength. Therefore, MWCNTs being well dispersed in water are not sufficient to promote concrete properties as well and the effect of surfactants on the properties and stability of cementitious materials should be examined [16]. In this regard, researchers found that the excessive amounts of NaDDBS and TX100/SDS in the mixture have a negative effect on the hydration of cement and may cause more trapped air in the concrete [7, 25]. The primary additives compatible with concrete and used as a surfactant in the process of dispersing MWCNTs in water are the water-reducing additives, plasticizers, and polycarboxylate superplasticizers capable of facilitating the uniform dispersion of MWCNTs and preventing unwanted voids in the cement paste and subsequently in concrete [25]. The researches have demonstrated that, using the ultrasonic method, a small amount of MWCNTs can be dispersed in water containing 5 wt% superplasticizer [7]. Shah et al. achieved an efficient dispersion of MWCNTs with various lengths and concentrations in cementitious materials by employing polycarboxylate-based superplasticizers [8, 26]. In the meantime, Han et al. reported that the efficient dispersion of MWCNTs by means of superplasticizers can be assigned to their double-dispersion effect on MWCNTs/CNFs (carbon nanofibers) and cement particles [27]. In this paper, the influence of various surfactants on the dispersion of MWCNTs in the water and on the concrete performance is investigated. A new combination as surfactant compatible with concrete is presented and provides the best dispersion of MWCNTs whereas the concrete mechanical properties remain constant. This will help also to fabricate a concrete sensor with the same mechanical properties as plain concrete and will lead to a cast concrete sensor that can evaluate structural and mechanical properties of plain concrete.

The optimal amount of the new surfactant composition is calculated using UV-visible spectroscopy and turbidimetry to measure the dispersion quality of MWCNTs in the aqueous phase. The mechanical properties of the concrete such as compressive and flexural strength have been chosen as the concrete performance criteria to illustrate the effect of MWCNTs dispersion quality and surfactant interaction on the concrete composition and hydration process compared to the plain concrete.

2. Materials and Methods

2.1. Materials. In this study, multiwalled carbon nanotubes (MWCNT) obtained from Research Institute of Petroleum Industry (RIPI) were used; their properties are given in Table 1. In order to evaluate the different surfactants effect on dispersion quality of MWCNTs in aqueous phase, the amount of MWCNTs was assumed constant at the value of 0.05 wt% which was used in many previous studies to prepare smart concrete [28]. The used mix design is shown in Table 2. Type II Portland cement was obtained from Abyek Cement Company. In order to disperse MWCNTs in the mixture, different surfactants were used (Table 3). Also, 0.02 wt% tributyl phosphate made by Sigma-Aldrich Co. was used as a defoamer. A dark brown solution of polycarboxylate-based superplasticizer (Sarapush construction chemical manufacture) with 36% solid contents and a density of 1.1 g/cm^3 was used for workability purpose. SP-C are composed of a methoxy-polyethylene glycol copolymer (side chain) grafted with a methacrylic acid copolymer (main chain). The carboxylate group –COO–Na$^+$ dissociates in water, providing a negative charge along the SP-C backbone.

Table 2: Mix design.

Mix design	Cement	Small sand*	Big sand*	Small gravel*	Big gravel*	Water	W/C
Percentage	15.34	39.58	6.96	23.19	7.72	7.21	0.47
kg/m^3	380	980.40	172.40	574.42	191.22	178.60	

*Sand and gravel are silica based.

Table 3: The optimal amount of different combinations of surfactant.

Surfactant	Manufacturer	Purity	Dosage (gr/mL)
SDS/DTAB (9 : 1) [23, 24]	Sigma-Aldrich	SDS ≥ 98%, DTAB ≥ 98%	SDS = 2.272, DTAB = 0.2524
SDS**	Sigma-Aldrich	≥98%	SDS = 2.524
NaDDBS [15]	Merck Group	—	NaDDBS = 4.88
TX-100/NaDDBS (3 : 1) [14]	Merck Group	—	TX-100 = 6.672, NaDDBS = 2.224
TX-100/NaDDBS (2.25 : 1)**	Merck Group	—	TX-100 = 5.004, NaDDBS = 1.668
Superplasticizer (0.4 wt*%) [29]	Sarapouch Co.	≥97%	Superplasticizer = 0.0098
Superplasticizer (0.5 wt*%)**	—	≥97%	Superplasticizer = 0.0123
Superplasticizer (0.7 wt*%)**	—	≥97%	Superplasticizer = 0.0173
Superplasticizer/SDS (9 : 1)**	—	SDS ≥ 98%, Superplasticizer ≥ 97%	SDS = 0.0014, Superplasticizer = 0.0123

∗: Weight of cement used per cubic meter of concrete (wt).
∗∗: These compositions were tested for further research.

2.2. Methods. SDS/MWCNT and NaDDBS/MWCNT solutions were prepared by mixing 0.05 wt% MWCNTs with an aqueous individual surfactant solution at concentrations 2.254 and 4.88 (gr/mL), respectively. Also mixtures of NaDDBS/TX-100 and SDS/DTAB at concentrations 8.896 and 2.524 (gr/mL), respectively, were prepared. Then 0.05 wt% MWCNTs were added to each mixed solution. All solutions were stirred for 10 min with a magnetic stirrer (model, WIFESTEER MSH-20B) and then sonicated for 130 min with an ultrasonic cleaner set (model NISONIX 3000) at an average power of 275 W. The temperature was controlled during sonication to prevent warming of the solutions. Subsequently, the solutions were centrifuged for 10 min at 8000 rpm to remove the bigger clusters. The result shows that the supernatant was well separated from the sediment after centrifugation stage. After 24 h resting, the characterization of MWCNs solutions was performed (see Figure 1). UV-vis absorption spectra were recorded using a Shimadzu-2100 UV-visible spectrophotometer (model, Japan) with a matched pair of glass cuvettes, 1 cm in optical path length, placed in a thermostated cell holder.

The final samples were diluted to three times their original volume with pure water to decrease the original darkness of solution for UV-vis test. Given that the absorption spectrum of MWCNTs is in the 500–700 nm wavelength range, the UV-vis test can be used to evaluate the dispersion of MWCNTs in the water [9, 13]. The UV test scaled from 0 to 5 where the maximum value means the dispersion quality is at the maximum and the minimum value means the dispersion quality is low. The UV test was conducted at the wavelength of 650 nm. Finally, the results obtained from the UV-vis test and the turbidimetry which is the process of measuring the loss of intensity of transmitted light due to the scattering effect of particles suspended in it were compared to determine the best surfactant for the uniform and effective dispersion of MWCNTs in the aqueous phase.

The cement was added to the mixture of water and MWCNTs in a high-speed mixer (3600 rpm, Pars-Khazar Co.) to be uniformly mixed. After adding the aggregate to the mixer, the concrete was placed in preoiled molds (Figure 2) and by applying appropriate vibration, any air that may have been trapped was released. Concrete specimens were removed from the molds after 24 hours and cured in water at 25°C for 28 days [30]. The 6 specimens were tested for compressive ($5 \times 5 \times 5\,cm^3$) and flexural ($4 \times 4 \times 16\,cm^3$) stresses in accordance with ASTM C39 and ASTM C78 standards, respectively [31, 32]. SEM images which were captured with ZEISS SUPRA™ 40 Field Emission Scanning Electron Microscope (FESEM) were also used to assess the dispersion quality of the MWCNTs in the concrete.

3. Results and Discussion

The degree of dispersion of MWCNTs in aqueous media has been investigated using the UV-vis spectra of the dispersions [33]. Individualized MWCNTs are active in the UV-vis region and exhibit characteristic bands corresponding to additional absorption due to 1D Van Hove singularities [23, 34]. However, bundled MWCNTs are hardly active in the wavelength

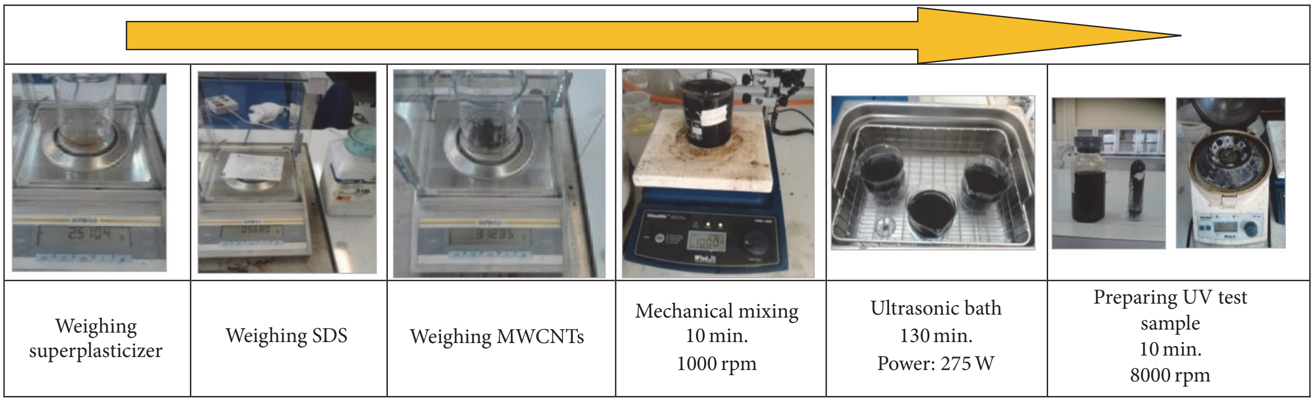

Figure 1: Dispersion of MWCNTs in aqueous phase.

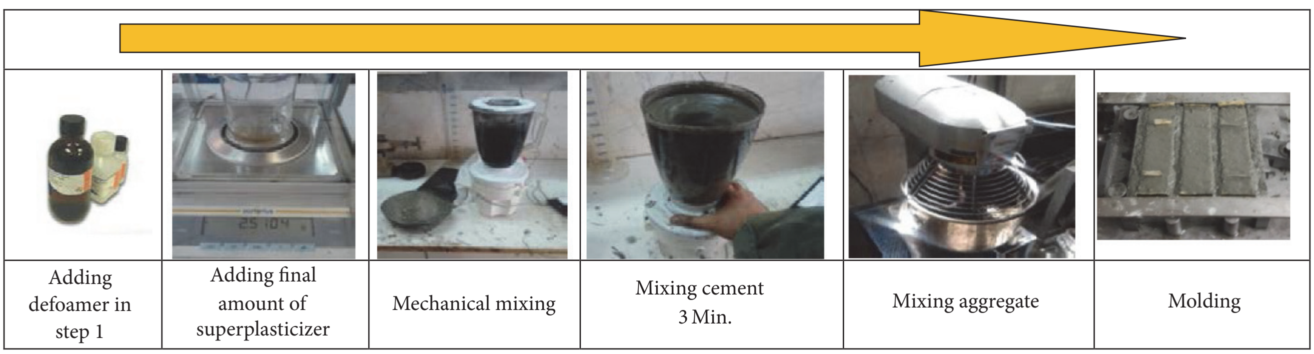

Figure 2: Dispersion of MWCNTs in cement and fabricate the sensor.

region between 200 and 1200 nm [34]. Therefore, it is significant to compare the amounts of individually dispersed MWCNTs in the solution through the absorption intensity.

Step 1. The UV-vis spectra of aqueous MWCNTs dispersions using different systems are shown in Figure 3. According to Figure 3, the maximum absorbance was obtained for SDS/DTAB mixture, indicating high dispersion of MWCNTs. The dispersion of MWCNTs with SDS, NaDDBS, and TX_100/NaDDBS solutions was low compared to SDS/DTAB mixture. Therefore, by mixing anionic and cationic surfactants, a higher dispersion of MWCNTs can be achieved at a lower concentration of surfactants. Also, all other parameters such as the sonication time and power and MWCNTs content were held constant during the test. It has been remembered that the solution was diluted to three times its original volume with pure water to achieve high resolution spectra. So, the absolute values of the absorbance are higher than the results shown in Figure 3, but relatively, these results are reliable. Also, based on the test procedure, the solution without surfactant was not stable and the MWCNTs were completely sedimented after centrifugation, so that, after dilution step, the absorbance result was near zero.

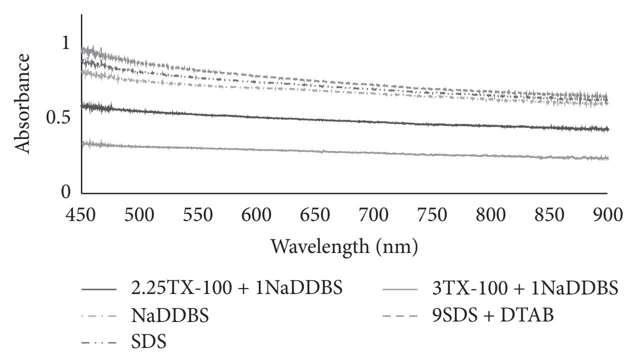

Figure 3: UV-vis spectra of an aqueous (0.05 wt%) MWCNTs dispersion using different surfactants.

The results showed that when a mixture of water, dispersed MWCNTs, and the surfactant material is used to make concrete, the mechanical properties of concrete are severely reduced due to the foam resulting from the surfactant in the concrete mixing process. Figure 4 illustrates the impact of not using a defoamer in the concrete mixing.

In this case, the defoamer, tributyl phosphate, can be used to resolve this problem. So far, the exact mechanism of the antifoams is not well understood [35]. Nevertheless, the general function of the antifoams is that they disrupt the stability of the liquid films in the foam, increasing the rate of the liquid drainage and thus enhancing foam destruction [36]. Azhari and Banthia observed that methylcellulose

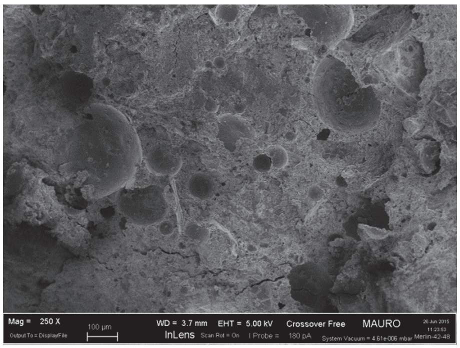

Figure 4: Air bubbles in the concrete with SDS as surfactant in the absence of defoamer.

(which has an air-entraining effect and increases the amount of porosity) and tributyl phosphate defoamer (which has the ability to decrease the amount of porosity) cause a change in the content of the void phase [37].

Figure 5 illustrates the impact of using tributyl phosphate defoamer (0.02 wt%) on the concrete mixing process when SDS is used as surfactant.

Comparing the SEM micrographs (Figures 4 and 5) demonstrates that the elimination of the defoamer in the concrete mixing process results in a lot of unwanted air bubbles and may decrease the mechanical properties of concrete. It can also be observed that air bubbles reduction can promote MWCNTs dispersion uniformity in the concrete mixture. The compressive and flexural test results on the concrete specimens containing 0.05 wt% MWCNTs and 0.02 wt% defoamer are illustrated in Figures 6 and 7.

As can be seen in Figures 6 and 7, the mechanical properties of concrete reinforced with 0.05 wt% MWCNTs and containing different types of surfactant as well as 0.02 wt% defoamer are enhanced with respect to plain concrete, as the dispersion quality of MWCNTs is increased. The maximum compressive and flexural stresses which are related to SDS and DTAB surfactant composition with a mixing ratio of 9 : 1 are improved by 60.3% and 51.2%, respectively, with respect to plain concrete (without MWCNTs). So, it was evident that the combination of SDS/DTAB resulted in the best dispersion capability.

Step 2. Another approach to resolving the problem of unwanted air bubbles in the concrete mixing process without the use of defoamers is the use of a surfactant compatible with concrete. One of the approaches proposed here is to use compounds that reduce the amount of water needed in the mixture such as plasticizers and superplasticizers [27, 29, 38]. In this study, different amounts of polycarboxylate-based superplasticizer were used to disperse MWCNTs in the water. The UV-vis spectra of aqueous CNT dispersions using different concentrations of polycarboxylate are measured to determine their optimal concentration (C_{opt}). As shown in Figure 8, the increase in SP-C concentrations up to the concentration of 0.5 wt% leads to the increase in the absorbance intensity; however, further increases in SP-C concentrations cause the reduction in absorbance intensity. Thus, the value of C_{opt} for SP-C is determined to be 0.5 wt%. Therefore, it is assumed that most SP-C in the suspensions adsorbed onto the surfaces of the MWCNTs. Further increase in SP-C concentrations above the C_{opt} results in wasting materials and may lead to undesired results. Since the new surfactant (superplasticizer) is compatible with concrete and does not require the use of a defoamer, the unwanted air bubbles are less, as shown in Figure 9. However, UV-Vis test results have shown that the dispersion power of DTAB/SDS mixtures is higher than that of the superplasticizer. As shown in Figure 9 by black ring, dispersion quality of MWCNTs dispersed with a superplasticizer is not proper and there is agglomeration of MWCNTs in concrete.

Step 3. Based on the results presented in Figure 3, the combination of SDS + DTAB (9SDS + 1DTAB) produced mildly better results in dispersing multiwalled carbon nanotubes than SDS alone. To minimize the cost of the new surfactant combination which will be used in large amounts in the process of fabricating smart concretes and because of the ratio between SDS and DTAB is also challenging when this surfactant combines with SP-C, SDS is substituted with a combination of SDS + DTAB to promote SP-C surfactant effect. So, in order to obtain a high dispersion ability, the dispersing power of SP-C/SDS mixtures as new surfactant composition was examined. To compare the dispersing powers of SP-C/SDS mixtures, the UV-vis spectra of aqueous MWCNTS dispersions for different ratios of SP-C/SDS mixtures were measured. As shown in Figure 10, decreasing the ratio SP-C/SDS in the aqueous solution, the absorbance in the characteristic wavelength region changes to higher values, indicating that the amount of individually dispersed MWCNTs is increased. Hence, according to our experimental results, the maximum dispersion is obtained with the ratio of 5 for SP-C/SDS mixtures. The results showed that the combination of these two surfactants can develop the new combination which can disperse MWCNTs in aqueous phase much higher than each one did alone with the same dosage. The synergic effect of these two surfactants is due to attractive interactions between these surfactant molecules when dispersing MWCNTs in water [13, 20].

To evaluate the effects of the new surfactant composition on the mechanical properties of concrete, 6 specimens ($5 \times 5 \times 5\,\text{cm}^3$) were fabricated and after 28 days of curing, they were tested according to ASTM C39 standard. The results showed that the compressive strength of concrete varies from −10% to +33% of the plain concrete compressive strength (30 MPa) as the ratio of SP-C to SDS is decreased (Figure 10). Since the amount of foam formed in the concrete is increased as the amount of SDS is increased, this, in turn, would lead to the decline in the mechanical properties of the concrete. Therefore, considering the intended purpose of making concrete containing MWCNTs, the optimum ratio of SP-C/SDS should be concerned. If the goal is to make concrete with the maximum strength, the ratio of SP-C/SDS equal to 12 is suggested, but if the goal is to make concrete containing the largest amount of well-dispersed MWCNTs, the ratio of SP-C/SDS equal to 5 may be suggested. A ratio of

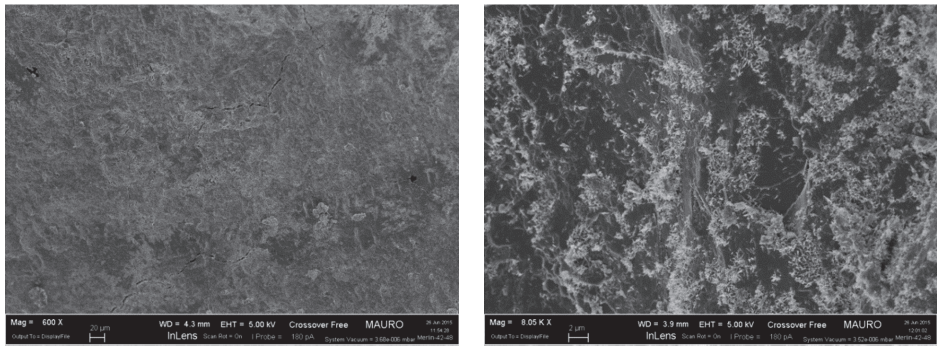

Figure 5: Concrete reinforced with 0.05 wt% MWCNTs with SDS as surfactant and tributyl Phosphate defoamer.

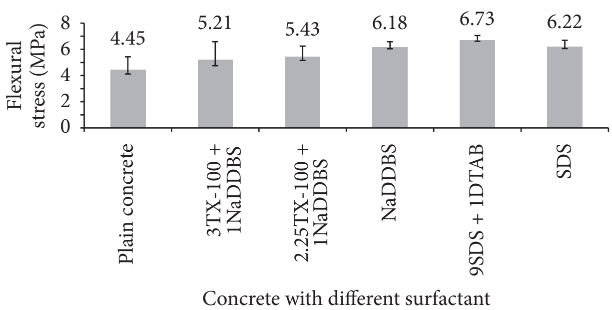

Figure 6: Modulus of rupture of concrete specimens containing 0.05 wt% MWCNTs and different surfactants.

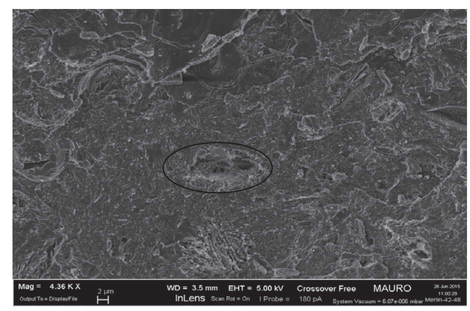

Figure 9: Concrete reinforced with 0.05 wt% MWCNTs dispersed with superplasticizer in the absence of defoamer.

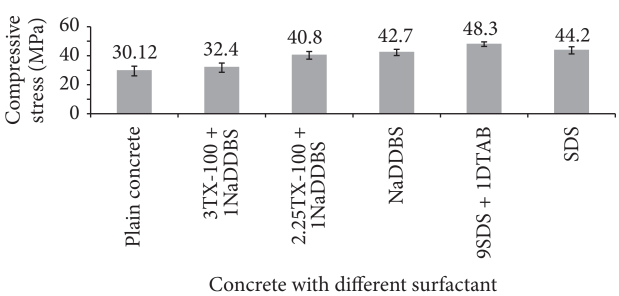

Figure 7: Compressive strength of concrete specimens containing 0.05 wt% MWCNTs and different surfactants.

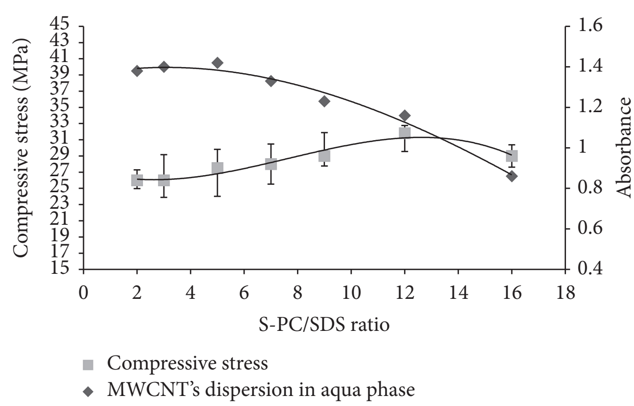

Figure 10: The effect of different mixing ratios of SP-C to SDS on the dispersion quality of MWCNTs in aqueous phase and the compressive strength of specimens containing MWCNTs.

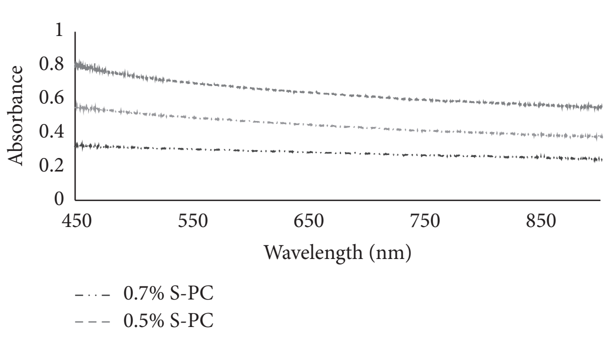

Figure 8: UV-vis spectra of an aqueous (0.05 wt%) MWCNT dispersion using different concentrations of SP-C.

SP-C/SDS equal to 9 is suggested when making concrete with similar properties to the plain concrete (without MWCNTs) is concerned.

In order to compare the amounts of dispersion of MWCNTs, turbidity measurements of colloidally stable dispersed MWCNTs solutions containing pure surfactants, superplasticizer, and mixtures of DTAB/SDS, NaDDBS/TX-100, and SP-C/SDS are performed. The UV and turbidity results of all systems are shown in Figure 11 and Table 4.

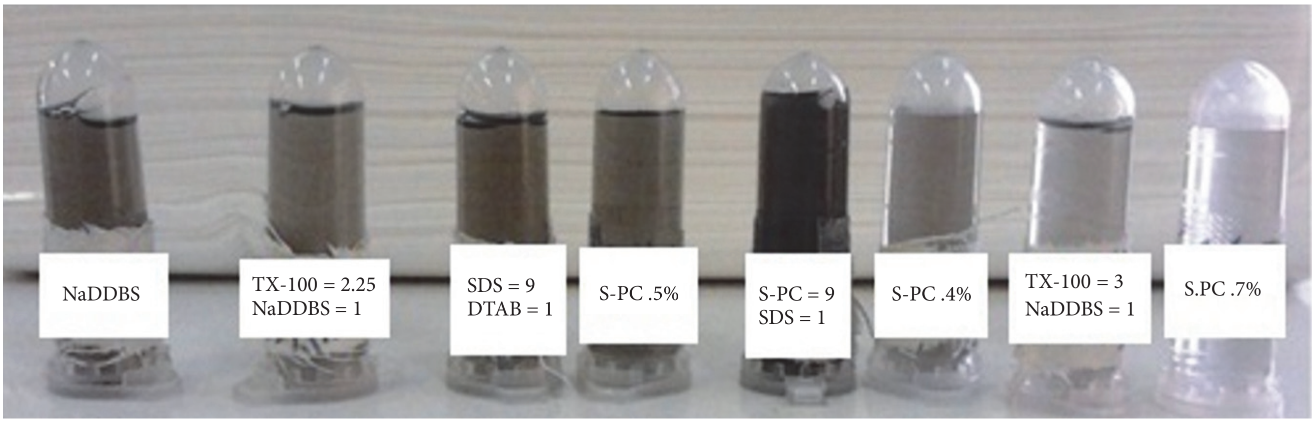

Figure 11: Comparing MWCNTs dispersion in water using turbidity meter.

Table 4: The turbidity of water containing MWCNTs using different surfactants.

Surfactant	Absorbance (out of 5)	Turbidity (NTU)
SDS/DTAB (9 : 1)	0.76	1320
SDS	0.71	1086
NaDDBS	0.68	579
TX-100/NaDDBS (3 : 1)	0.28	27.6
TX-100/NaDDBS (2.25 : 1)	0.48	245
Superplasticizer (0.4 wt* %)	0.29	89.7
Superplasticizer (0.5 wt* %)	0.65	376
Superplasticizer (0.7 wt* %)	0.45	187
Superplasticizer/SDS (9 : 1)	1.23	1835

As can be seen from Figure 11, the mixture containing the combination of superplasticizer and the SDS with a mixing ratio of 9 : 1, respectively, by weight of cement exhibits the best dispersion capability than the other systems.

4. Conclusions

In this paper, the combination of SDS and superplasticizer was introduced as a new surfactant composition in order to effectively disperse MWCNTs in aqueous phase whereas the mechanical properties of reinforced concrete were similar to the ones of plain concrete. Also, the following conclusions may be drawn from this study:

(1) The mechanical properties of concrete are improved with the use of a defoamer and as the amount of MWCNTs remains constant (0.05 wt%), the compressive and the flexural strengths of concrete containing MWCNTs are improved by 60.3% and 51.2%, respectively, with respect to plain concrete.

(2) Omitting tributyl phosphate as the defoamer, some little foam is produced in concrete due to SDS presence in the compatible surfactant composition and this causes a reduction of concrete mechanical properties with respect to the concrete with the defoamer. Then, a higher SDS amount leads to a higher dispersion quality in aqueous phase but also more unwanted air bubbles in concrete and a higher reduction of the concrete mechanical properties too.

(3) The optimal dosage of superplasticizer as the surfactant is 0.5 wt% in order to achieve the maximum dispersion of MWCNTs in aqueous phase when the amount of MWCNTs is 0.05 wt%.

(4) The combination of SDS/DTAB or SP-C/SDS causes a synergic effect while dispersing MWCNTs in water.

(5) A combination of superplasticizer and SDS with a mixing ratio of 9 : 1, respectively, by weight of cement is suggested when making concrete with similar properties to the plain concrete is mentioned.

(6) A combination of superplasticizer and SDS with a mixing ratio of 12 : 1, respectively, by weight of cement is suggested when making concrete with higher compressive strength is targeted.

(7) A combination of superplasticizer and SDS with a mixing ratio of 5 : 1, respectively, is suggested when the largest amount of well-dispersed MWCNTs in aqueous phase is required.

Competing Interests

Mostafa Adresi on behalf of the other authors confirms that there is not any known conflict of interests associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

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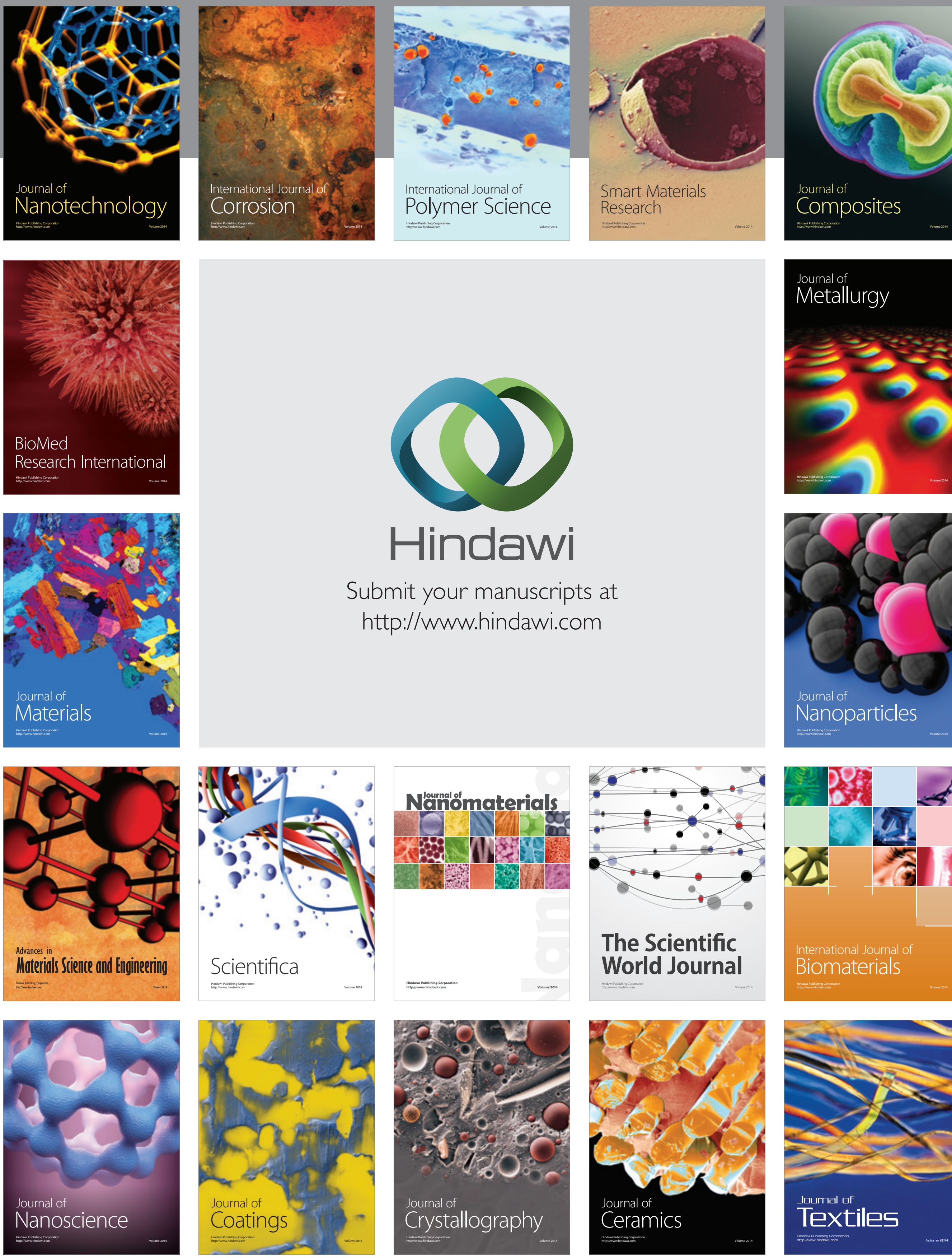
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