On the efficiency of UV–vis spectroscopy in assessing the dispersion quality in sonicated aqueous suspensions of carbon nanotubes

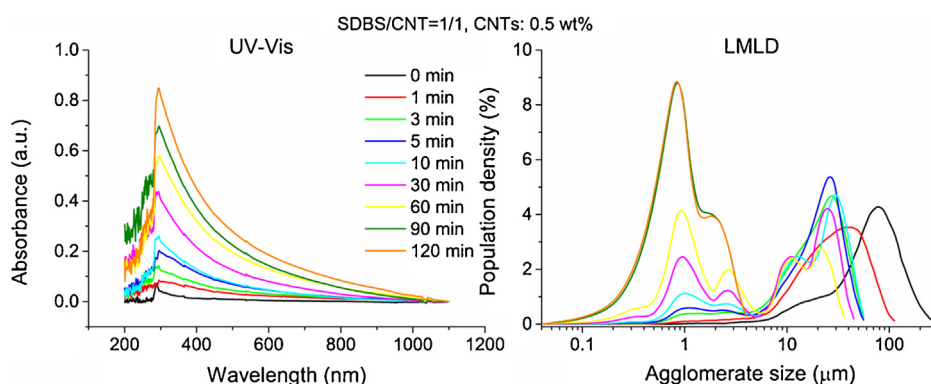P. Alafogianni [a], K. Dassios [a], S. Farmaki [a], S.K. Antiohos [b], T.E. Matikas [a], N.-M. Barkoula [a,*]

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HIGHLIGHTS

- UV–vis spectroscopy is not inferior to LMLD for dispersion monitoring.
- Peak intensity is a convolution of the mean agglomerate size and the PDI.
- Suspensions with low SDBS concentrations require $7.7\,J\,min^{-1}\,ml^{-1}$ for 90 min.
- Suspensions with high SDBS concentrations achieve homogeneity after only 30 min.
- Viscocrete offers lower dispersion quality even when used in higher concentrations.

GRAPHICAL ABSTRACT

ARTICLE INFO

ABSTRACT

The present study evaluates carbon nanotube (CNT) dispersion quality in sonicated aqueous suspensions assisted by two types of dispersive agents, an ionic surfactant commonly used in nanocomposite development and a plasticizer-type dispersant relevant to cementitious matter. Dispersion quality is assessed by UV–vis spectroscopy and related to experimental parameters such as sonication duration/energy, dispersant type and concentration, and CNT loading. The results obtained from the versatile and straightforward methodology are compared to those obtained by the more complex and analytical Liquid Mode Laser Diffractometry (LMLD). The efficiency of UV–vis spectroscopy in effectively following the sonication process and in capturing its different stages is investigated and conclusions concerning the relation between surfactant concentration and sonication duration for achievement of monodisperse suspensions are drawn. The efficiency of the two assistive agents with regard to dispersion quality is compared and discussed in the text.

1. Introduction

The discovery of carbon nanotubes (CNTs), the nanoscale-sized one-dimensional allotropes of carbon, signalled the dawn of a new era in materials science [1]. These incredible structures exhibit a series of remarkable electronic, magnetic and mechanical properties, extremely high aspect ratios while being at least 100 times stronger than steel, but only one-sixth as heavy [2–4]. However, due to the high Van der Waals attractive forces acting on their surfaces, of the order of $500\,eV/\mu m$ per μm of tube–tube contact [5], their very high surface areas and high aspect ratios, CNTs tend to entangle even without external stimulus hence appearing highly

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tortuous in their native form [5,6]. This tortuosity combined with their hydrophobic surfaces leads to the formation of agglomerates when the tubes come in contact with most solvents [7]. This causes great problems to their incorporation in continuous media and matrices where CNT dispersion homogeneity is indispensable for the full exploitation not only of the tube's properties but also of their stress transfer and sensing potentials. Not surprisingly, achievement of well dispersed CNT suspensions is currently one of the most sought after endeavours in the field of nanocomposite materials [8].

To overcome dispersion issues, different approaches based on physical and chemical processes have been proposed and evaluated [9–12]. In the physical route, shear forces are applied via sonication or high-shear mixing on the nanotubes in order to separate them from each other. The key drawback of this approach is related to the fragmentation and scission of nanotubes [13,14] under the forces exerted by the vibrating liquid which results in substantial reduction of their high initial aspect ratio. The second approach is based on the use of surfactants or chemical moieties for the alteration of the surface energy of the nanotubes, hence improvement of their wetting or adhesion characteristics and ultimately of their dispersibility in solvents [15]. The major disadvantage of this particular approach is that aggressive chemical functionalization usually increases the defects on the external sidewalls of the CNTs hence altering their electrical and mechanical properties [16,17]. Recently, the noncovalent approach is gaining ground wherein adsorption of chemical moieties on the surface of CNTs occurs by π–π stacking or through coulombic attraction in case of charged chemical moieties [18,19]. This method is physical and does not affect the chemical structure of the tube's graphene walls; conditionally it can even improve the interfacial properties between the tubes and the dispersing medium [20].

A wide variety of surfactants, such as sodium dodecyl-benzene-sulphonate (SDBS) [21], octyl phenol ethoxylate (Triton X-100) [22], hexadecyl-trimethyl-ammonium bromide (CTAB) [23], and sodium dodecyl sulfate (SDS) [24], in combination with sonication, have been suggested for optimisation of CNT dispersion in aqueous solutions under the noncovalent approach. The dispersive efficiency of the surfactants is linked with the length of their alkyl chain, the presence of benzene rings and the type of head group(s) [24,25]. It has been shown that not only surfactant type, but also surfactant concentration is a key parameter towards improved dispersion efficiency [24,26,27]. Relevant literature currently reviews a vast selection of surfactant-to-CNT ratios ranging from 0.2:1 up to an excessive 520:1 [24,27,28]. While higher surfactant concentrations normally aid achievement of CNT suspension homogeneity, it should not be overlooked that such loadings are inconvenient for further CNT processing into composite materials [29].

For the special case of aqueous CNT dispersions intended for use in cement paste, use of surfactants has been documented to relate to cement hydration inhibition and enablement of air entrapment [30]. Usage of surfactants in low concentrations combined with addition of defoaming agents such tributyl phosphate (TBP) has been suggested as countermeasure [26,31]. However, even with the addition of defoaming agents, the amount of entrapped air still remains uncertain. Thus, for cement applications, dispersion of CNTs in aqueous solutions must be assisted by other types of dispersant agents, like plasticizers and superplasticizers, dispersants which would otherwise be secondary choices for CNT suspensions [32,33]. Although literature reviews superplasticizer/CNT ratios up to 10/1 [33,34], it should be noted that there is an upper limit in the amount of the superplasticizer that can be used in the

suspension without deterioration of the physical properties of the cement material (workability, consistency) and without segregation. This upper limit for most of commercially available superplasticizers is 2 wt% of cement which, for a water/cement ratio of 0.5, translates to 4 wt% water.

Another difficulty in selecting appropriate dispersion conditions and agents arises from the perplexity of assessing the dispersion state of CNTs in aqueous solutions in a straightforward, reliable and quantitative manner. To date, a number of methodologies have been suggested including atomic force microscopy (AFM) [25,35,36], scanning and transmittance electron microscopy (SEM & TEM) [24,37], UV–vis scanning [24], fluorescence and Raman spectroscopy [38–41], small angle neutron scattering (SANS) [42,43], and dynamic light scattering (DLS) [15,44]. Among these methodologies, UV–vis spectroscopy, applicable to any type of suspension, is one of the most robust and straightforward techniques for quantifying the portion of light lost as it travels through a sample, relative to its blank counterpart. In UV–vis measurements, CNTs display characteristic peaks in the ultraviolet spectral region [22,23] while only individual disentangled CNTs are active in the UV–vis region; their characteristic bands correspond to additional absorption due to 1D van Hove singularities [45,46]. Based on this principle, the absorbance measured at a specific wavelength can be related to the degree of disentanglement [7,47]. In bundled CNTs the photoluminescence that is normally detected by the UV–vis spectroscopy is easily quenched by other CNTs or from the concentric walls of the same nanotube, thus they hardly absorb the light in the wavelength region between 200 and 1200 nm [24,48]. Therefore, it is possible to establish a relationship between the amounts of individually dispersed CNTs in suspension and the intensity of the corresponding absorption spectrum [49]. It must be however noted, that especially in the case of CNT suspensions where agglomerates can be formed at sizes comparable or even greater than the incident wavelength, Mie scattering theory applies and the UV–vis technique measures not only the light attenuation due to absorption of individual CNTs but also due to the scattering of incident radiation on the surface of CNT bundles. The relevant measure is then extinction, the sum of absorbed and scattered light. In order to minimize the contributions of scattering to the measured extinction few studies were performed using an integrating sphere setup [50–54].

Next to that UV–vis spectroscopy does not provide information regarding the size of the agglomerates nor does it allow for quantification of the effects of sonication parameters on tube length and sample polydispersity. To address such complex issues, a novel method relying on temporal CNT agglomerate size analysis by Liquid Mode Laser Diffractometry (LMLD) [21], was recently suggested by our research group. LMLD is applied herein for the purpose of benchmarking the effectiveness of UV–vis spectroscopy in assessing the dispersion quality of the suspensions under investigation.

Based on the above argumentation, the aim of the current study is threefold. The first goal is to define the dispersion quality of sonicated CNT suspensions as a function of: (a) sonication duration, i.e. sonication energy and (b) surfactant relative concentration, i.e. surfactant/CNT ratio, using UV–vis spectroscopy. The second objective is to evaluate the resolution of the UV–vis methodology by comparison of obtained data with those of LMLD. The ultimate goal of the study is to assess the dispersion quality achieved using surfactant- and plasticizer-based dispersant agents, under identical experimental conditions. The effect of CNT loading (0.4–1.6 wt%) on the UV–vis spectra in superplasticizer-based suspensions is also investigated and discussed.

2. Materials and methods

2.1. Materials

Multi-walled CNTs synthesized via catalytic chemical vapour deposition by Shenzhen Nanotech Port Co., Ltd., China, available under the commercial name "Long MWNT 2040", were used in the present study. Nominal tube diameter ranged from 20 to 40 nm while tube length ranged between 5 and 15 μm. Nominal sample purity was higher than 97% with less than 3% amorphous carbon and 0.2 wt% ash. Nominal special surface area of the tubes was 80–140 m^2/g while their tapped density ranged from 0.15 to 0.28 g/cm^3. SDBS, Sigma–Aldrich code 289957, a surfactant particularly popular for CNT separation, was chosen on the basis of its high dispersive efficiency. A polycarboxylate-based polymer of the superplasticizer family, Viscocrete Ultra 300 supplied by Sica Hellas SA, specific to suspensions targeted for cement-based composites, was selected as the second dispersant agent.

2.2. Preparation of CNT based suspensions

Ultrasonic processing for dispersion of CNTs in suspension was performed using a Hielscher UP400S 24 kHz device (Hielscher Ultrasonics GmbH, Teltow, Germany), power capacity of 400 W, equipped with a Ø 22 mm cylindrical sonotrode. Output power was regulated by means of manual adjustment of wave amplitude within 25–100% of the device's maximum capacity. The actual power transmitted to the suspension was measured using a Hielscher PowMet230 digital ultrasound energy meter. All suspensions where sonicated at 50% of the maximum ultrasound wave amplitude corresponding to an energy input rate of 7700 J/min [21]. Dispersant agent-assisted aqueous suspensions of CNTs were prepared in fume hood environment according to the following strategy: A predetermined dispersant agent mass was initially added to 1000 ml of tap water in 2000 ml capacity low-form Pyrex glass beakers and an opalescent solution indicating complete dissolution was achieved after 60 s of magnetic stirring. The sonotrode was subsequently fully immersed into the suspension, sonication was commenced and CNTs were gradually introduced into the sonicated liquid. To investigate the effect of surfactant concentration on dispersion quality and identify the optimal surfactant/CNT ratio required to avoid surfactant stagnation or saturation, suspensions with SDBS/CNT ratios of 0.5/1, 1/1 and 2/1 were prepared by varying the initial surfactant mass within 2.5, 5 and 10 g. For SDBS-assisted suspensions, two CNT loadings were used, 0.5 and 1 wt% CNTs. For viscocrete-assisted suspensions, the corresponding ratio was 1.5/1, while CNT loading varied between 0.4 and 1.6 wt%.

2.3. Dispersion characterization of CNT based suspensions

UV–vis measurements were conducted in a Shimadzu UVmini-1240 spectrophotometer (Shimadzu Corp., Kyoto, Japan). Pure SDBS and Viscocrete samples were initially recorded for baseline purposes. A 10 μl droplet of the CNT suspension was diluted in 3 ml of distilled water in PMMA cuvettes.

For benchmarking purposes, the dispersion quality of suspensions was independently evaluated by LMLD following the experimental and interpretation methodologies developed previously [21]. In brief, the operational principle of LMLD relies on the observation that the spatial distribution of light scattered from a liquid is proportional to the sizes of particles in suspension therein. Based on the size distribution curve of particles in suspension, the mean particle size, expressed by the average agglomerate diameter, Dmean, and the polydispersity index, PDI, of the distribution, were calculated. The PDI value of each distribution was used for evaluating dispersion quality; as a general rule index values higher than 0.5
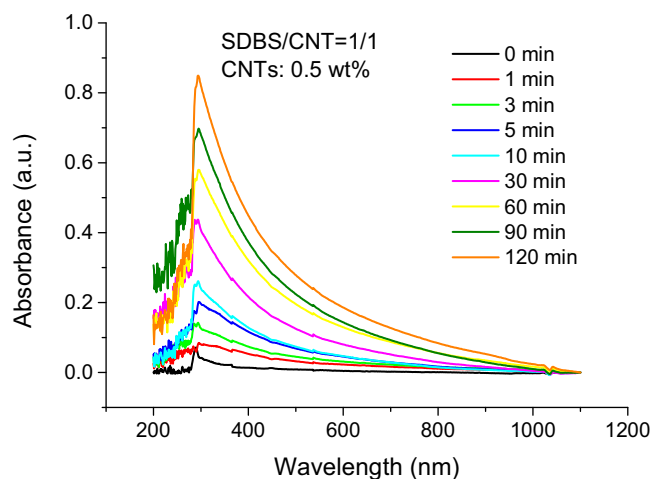

Fig. 1. UV–vis spectrum of aqueous 0.5 wt% CNT suspensions (SDBS/CNT = 1/1) as a function of sonication time.

are considered associated with poly-disperse/non-homogeneously distributed samples.

3. Results and discussion

3.1. Effect of sonication duration

Fig. 1 displays the effect of sonication duration on the UV–vis spectra of CNT suspensions with SDBS/CNT ratio of 1/1. Data from non-sonicated solutions (0 min) are also included in the graph for comparison purposes. It is observed that all suspensions exhibit a characteristic peak in their UV–vis spectrum at a wavelength of ca. 300 nm. Next to that, it is noted that the higher the sonication time, the higher the intensity of the characteristic peak, while the intensity of the measurement gradually decreases from UV to near IR. These results are in accordance with literature findings indicating that individual multi-walled CNTs exhibit their characteristic peak around 260 nm. Since the measured absorbance at specific wavelength can be related to the degree of de-bundling of the suspensions [7,24,47,49,54], the peak intensity of the obtained spectra can be correlated to sonication time as a tool to monitor the dispersion process. As already discussed, the absolute value of this peak contains the contribution from both scattering and absorption phenomena. Since the sonication process contains two mutually antagonistic effects, one being the de-agglomeration of CNT agglomerates and the other being the fragmentation of individual CNTs [13,55], while UV–vis absorbance data cannot assess mean agglomerate size, it is not possible to infer, from the particular data alone, whether the provided sonication energy is optimum for de-agglomeration of the particular system.

As aforementioned, the dispersion quality of the suspensions under investigation was independently evaluated by LMLD following the experimental and interpretation methodologies developed previously [21]. Thus, the CNT agglomerate size distributions based on this methodology are provided in Fig. 2. As demonstrated previously for the particular system, average agglomerate diameter above ca. 11.2 μm may correspond to entangled states only whereas diameters below that threshold may correspond to either un-shortened individually dispersed tubes, or to agglomerates of much shortened tubes [21]. Based on Fig. 2, it can be seen that unsonicated suspensions present a characteristic peak at app. 80 μm, which shifts to 30 μm after only 5 min of sonication. A characteristic peak below the threshold of 11.2 μm is first evidenced after 10 min of sonication, while further sonication results in the appearance of additional peaks at 0.3, 0.9 and 2.5.
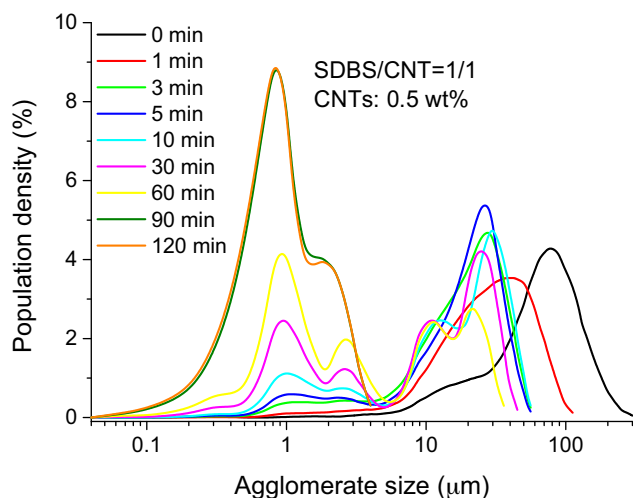
Fig. 2. Agglomerate size distribution curves of aqueous 0.5 wt% CNT suspensions (SDBS/CNT = 1/1) as a function of sonication time.
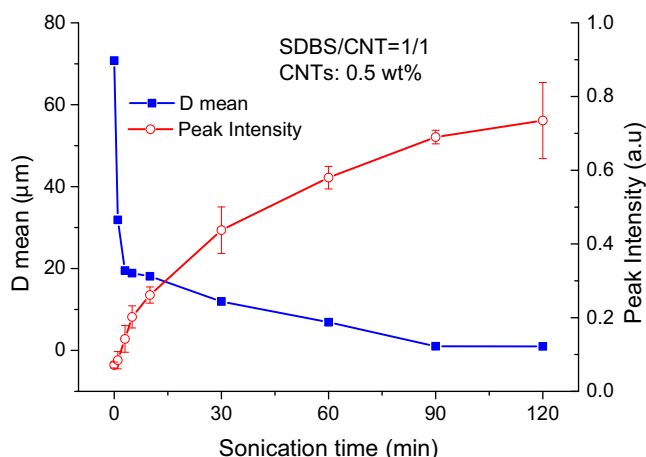(Adapted from [21], Copyright © 2015, American Chemical Society, with permission.)

Fig. 3. Average agglomerate diameter (D mean) and UV–vis peak intensity of aqueous 0.5 wt% CNT suspensions as a function of sonication time (SDBS/CNT = 1/1).

Table 1
PDI index of aqueous 0.5 wt% CNT suspensions (SDBS/CNT = 1/1) as a function of sonication time.

Sonication time (min)	SDBS/CNT	PDI
0	1/1	1.05
1	1/1	0.69
3	1/1	0.47
5	1/1	0.49
10	1/1	0.51
30	1/1	0.69
60	1/1	1.25
90	1/1	0.53
120	1/1	0.56

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In order to shed more light into the dispersion process, the peak intensity obtained using the UV–vis methodology and the average agglomerate diameter (Dmean) based on the LMLD analysis are plotted against each other in Fig. 3, under identical experimental conditions. PDIs of the corresponding suspensions are provided in Table 1. The graph indicates an inverse, albeit highly compatible, relationship between the results from the two independent characterisation methods. During early sonication times (1–10 min), a rapid decrease in average agglomerate diameter is seconded by a

sharp increase in UV–vis peak intensity. Thus both methodologies imply that at this stage of the sonication process, disentanglement of CNTs is the dominant phenomenon at this stage, leading to smaller bundle sizes and improving the absorption of the measured samples, while light attenuation due to scattering is prevailing. An almost linear relationship between UV–vis absorbance and mean agglomerate size is being observed at intermediate sonication durations (30–90 min). The mean agglomerate size drops below 11.2 μm while the PDI values (Table 1) vary between 1.25 (60 min) and 0.53 (90 min). This signifies that disentanglement is progressing and suspensions move towards mono-dispersity. At higher sonication durations (120 min) peak intensity increase levels off while Dmean values reach a plateau value of approximately 1 μm. Looking into the PDI values of the suspension, it can be concluded that an acceptable 0.5 value is obtained after 90 min of sonication, a value which does not change considerably with further energy transmission to the suspension. Thus, the extra amount of energy delivered by sonication to the system after 120 min can only cause fracture of the tubes and levels off the peak intensity and the Dmean of the suspension. Comparing LMLD to UV–vis, it can be argued that the primer is more quantitative and provides information on the mean agglomerate size whereas the latter is more qualitative and provides information on the disentanglement process. However both methods are effective in following the sonication process and capturing its different stages. Thus, UV–vis independently validates the conclusion that an energy density rate of 7.7 J min^{-1} ml^{-1} applicable for a duration of 90 min, is sufficient for achievement of homogeneous distribution of CNTs in the suspensions under investigation.

3.2. Effect of SDBS/CNT ratio

Another key factor for achieving homogenous and temporally stable CNT suspensions is the surfactant to CNT ratio. Surfactant concentration in the suspension is a critical parameter because small amounts of dispersant agent will not efficiently cover and separate the tubes while excessive amounts will lead to the formation of redundant surfactant micelles in the solution bulk. It is hence imperative to keep the ratio as low as possible in order to maximize dispensability while minimizing potential side effects that the excess amount of surfactant may induce to the host matrix [26,56]. Since sonication duration may act complementarily to surfactant to CNT ratio, in the sense that longer durations may facilitate dispersion at lower ratios, an investigation of the effect of varying SDBS/CNT ratio on dispersion quality appeared highly relevant. Three different ratios were studied, namely 0.5/1, 1/1 and 2/1; the corresponding peak intensity (UV–vis) and Dmean (LMLD) dispersion efficiency data are compared in Fig. 4 for sonication durations of 30, 60 and 90 min.

As observed in Fig. 4a, a 100% increase in peak intensity is obtained when SDBS/CNT ratio rises from 0.5 to 1, while further increase of the ratio to 2 leads to an additional increase in peak intensity of only 25%. At the same time, a small decrease of the order of 5%, in Dmean of corresponding suspensions is found related to the initial increase of SDBS/CNT ratio from 0.5 to 1, while an SDBS/CNT ratio of 2 leads in a tenfold drop in Dmean. Based on the PDI values of the suspensions under investigation (Table 2), it is observed that a SDBS/CNT ratio of 2/1 results in mono-dispersed samples, while low ratios relate with polydispersity conditions. It is hence entailed that both methodologies manage to efficiently highlight the significant role of the SDBS/CNT ratio on dispersion quality and that, for sonication durations as low as 30 min, use of high amounts of surfactants is necessary for obtainment of homogenous, mono-dispersed suspensions.

Fig. 4b illustrates the variation of UV–vis peak intensity and mean agglomerate size as a function of SDBS/CNT ratio after 60 min
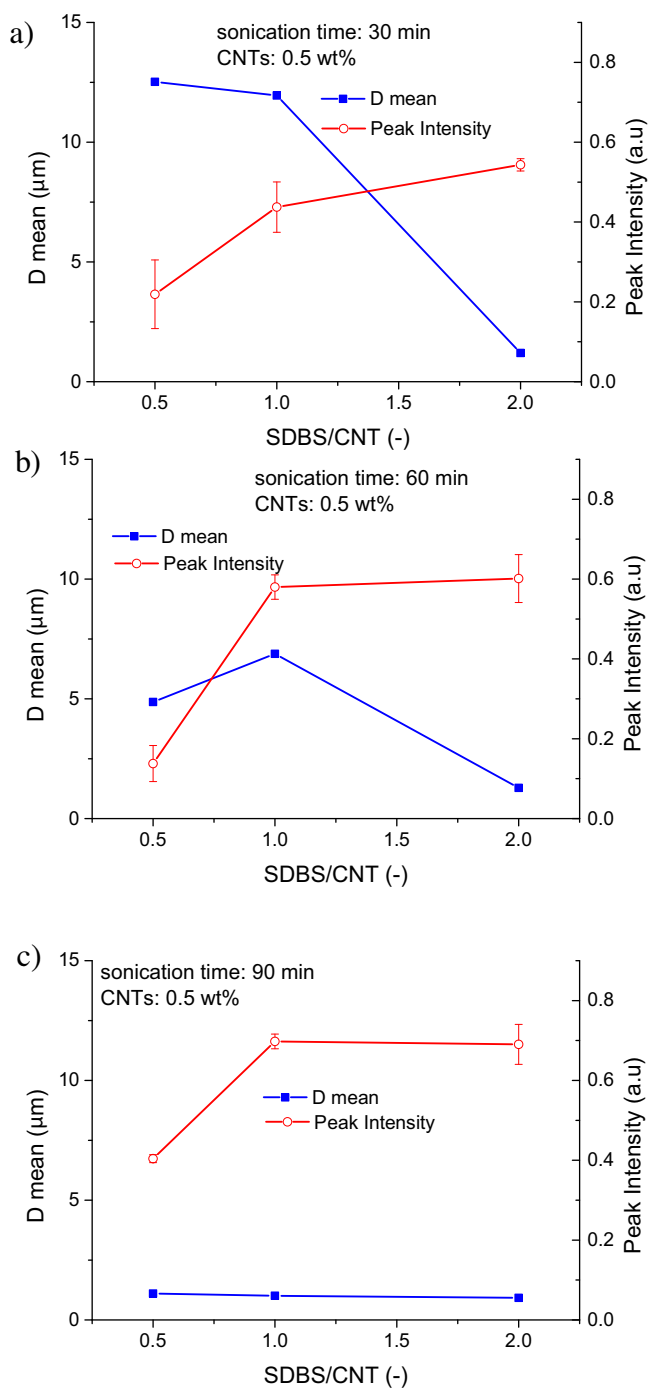
Table 2
PDI index of aqueous 0.5 wt% CNT suspensions after 30, 60 and 90 min of sonication, as a function of SDBS/CNT ratio.

Sonication time (min)	SDBS/CNT	PDI
30	0.5/1	0.63
30	1/1	0.69
30	2/1	0.33
60	0.5/1	1.73
60	1/1	1.25
60	2/1	0.84
90	0.5/1	0.74
90	1/1	0.53
90	2/1	0.25

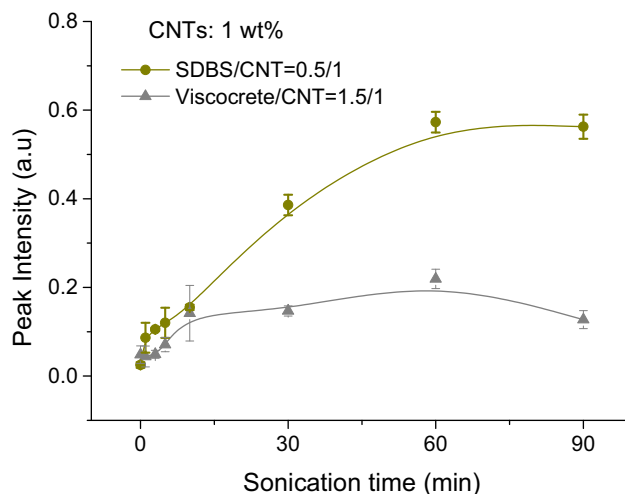(Adapted from [21], Copyright © 2015, American Chemical Society, with permission.)

Fig. 5. UV–vis peak intensity of aqueous 1 wt% CNT suspensions as a function of dispersant agent (SDBS vs. Viscocrete) and sonication duration.

Fig. 4. Average agglomerate diameter (D mean) and UV–vis peak intensity of aqueous 0.5 wt% CNT suspensions after (a) 30 (b) 60 and (c) 90 min of sonication, as a function of SDBS/CNT ratio.

of sonication. A significant increase in peak intensity is obtained with the increase of SDBS/CNT ratio from 0.5 to 1 while further increase in surfactant concentration again does not improve the peak intensity greatly. An interesting increase in Dmean is observed at a ratio of 1, the property reduces significantly at a corresponding ratio of 2. By observation of PDI data, it is found that all suspensions are poly-disperse after 60 min of sonication. After 90 min of sonication, a parallel shift in peak intensity towards higher values is obtained, while Dmean remains unchanged with respect to the relative surfactant concentration (Fig. 4c). Based on poly-dispersity data, it can be concluded that low SDBS concentrations

result in poly-dispersed samples while intermediate and high concentrations lead to mono-dispersed ones with the same mean agglomerate size.

Comparing the UV–vis and LMLD techniques, it is observed that the UV–vis signal exhibits higher sensitivity to SDBS/CNT ratio than Dmean. For instance, Dmean does not change with surfactant concentration when the provided energy is high (90 min of sonication); for the same conditions peak intensity does vary indeed. The same variations are observed also in PDI values suggesting that the UV–vis peak intensity is essentially a handy convolution of the double information, i.e. Dmean and PDI, provided by the LMLD methodology. Based on the behaviour depicted in Fig. 4, it can be concluded that low SDBS concentrations require at least 90 min of sonication to provide homogeneous suspensions, whereas acceptable results can also be obtained only after 30 min of sonication at high SDBS concentrations.

3.3. Effect of dispersant agents

In order to compare the dispersion qualities achieved by surfactant- and plasticizer-based dispersant agents, UV–vis measurements were carried out at the optimal sonication parameters established in the previous paragraph, i.e. 7.7 J min^{-1} ml^{-1}, and durations up to 90 min and the results are presented in Fig. 5. As aforementioned, the selection of dispersive agent for cementetious applications is a challenging task, since surfactant based suspensions are linked with side effects on the physical properties of the cement paste even at low surfactant concentrations, while plasticizers/superplasticizers show limited dispersion quality and may result in deterioration of the physical properties at
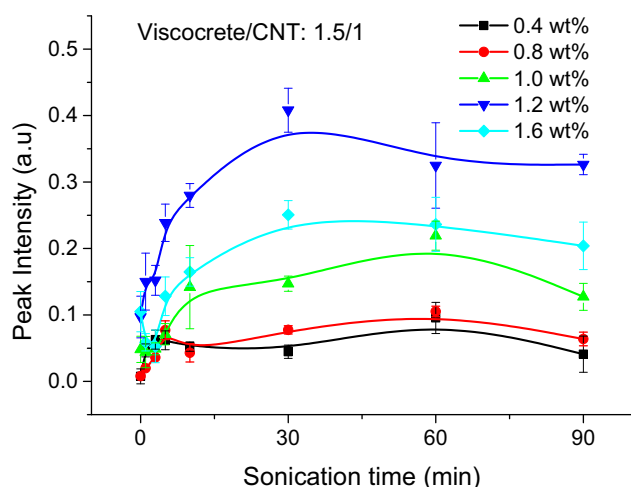
Fig. 6. Effect of CNT loading on the UV–vis peak intensity of viscocrete assisted sonicated aqueous CNT suspensions.

high concentrations (above ca. 4 wt%). Thus, the conditions that are set for this comparison, are: (i) minimum SDBS content for adequate dispersion, and (ii) maximum allowable plasticizer concentration determined by the manufacturer specifications. To this end suspensions with low concentration of SDBS (SDBS/CNT = 0.5) are compared with higher concentration Viscocrete suspensions (Viscocrete/CNT = 1.5).

It is observed that short sonication durations (up to 10 min) result in very low peak intensity values for both dispersant agents. This implies that, independently of the agent used, 10 min of sonication are insufficient to effectively disperse the suspensions under investigation. A considerable difference in the dispersive efficiency of the two agents is being observed after 30 min of sonication, where SDBS-based suspensions exhibit ca. 160% higher peak intensity values than Viscocrete-based ones. As sonication proceeds peak intensity increases in a much steeper manner in SDBS-assisted solutions, compared to Viscocrete, suspensions of the latter agent appear to obtain very low peak intensity values throughout the sonication process. A sonication duration of 90 min results in reduction of the peak intensity of the suspension, which is most probably related to the degradation of the superplasticizer at such prolonged sonication durations. Thus it can be suggested that Viscocrete results in the formation of big agglomerates and the origin of the peak intensity is most probably due to scattering of CNT bundles rather than absorption of individual CNTs. These findings are in total accordance with literature reports that plasticizers present very poor efficiency as dispersant agents even at concentrations three times higher than the surfactant's [57].

The dispersive efficiency of the plasticizer agent, Viscocrete, was further investigated as a function of CNT loading within 0.4–1.6 wt%; relevant results for a fixed Viscocrete/CNT ratio of 1.5/1 are displayed in Fig. 6. In agreement with the behaviour depicted in Fig. 5, best results were obtained after intermediate sonication durations (30–60 min) while higher sonication durations lead to small decreases in peak intensity values. It was further observed that an increase in CNT loading up to 1.2 wt% resulted in higher peak intensity values which deteriorated at higher CNT concentrations, a finding consistently observed for all sonication durations. According to the Beer–Lambert's Law [26] in an ideal suspension the absorbance of a sample can be related to its molar concentration and the path length via the molar extinction coefficient. Based on Fig. 6 it can be observed that the peak intensity of the measured suspensions is not linearly related to its mass concentration, at any sonication time. This is mainly due to the fact that the

formation of agglomerates diminishes the contribution of absorption in the peak intensity of the suspensions while Mie scattering prevails. Unlike the case of Rayleigh scattering where light is disseminated in all directions, in Mie scattering, light dissemination is much more profound in the incident direction light (forward scattering) than in the opposite one (back scattering). Moreover, Mie scattering by larger particles provides a narrower forward scattering pattern, than scattering by smaller ones [58], resulting in lower light attenuation at the detector. This fact might explain why peak intensity drops at 1.6 wt% Viscocrete-based CNT suspensions, where CNT concentration may be too high for the particular dispersive agent, to surmount the attractive forces between the tubes, hence resulting in bigger agglomerates.

Based on UV–vis peak intensity values it can be concluded that a Viscocrete/CNT ratio of 1.5/1 is not adequate to effectively separate the agglomerates in the suspensions and leads to low dispersion quality. A careful selection of the dispersive agent type and its relative ratio to CNT is therefore recommended when looking to substitute surfactant-based dispersive agents.

4. Conclusions

In the present study, the dispersion quality of sonicated CNT suspensions as a function of sonication duration and surfactant relative concentration was investigated using UV–vis spectroscopy. The dispersion qualities achieved using a surfactant (SDBS) and a plasticizer (Viscocrete), independently but under identical experimental conditions, were assessed. In order to evaluate the resolution of the UV–vis technique, results were compared to data obtained via a newly-suggested LMLD-based methodology.

Based on the obtained results the following conclusions can be drawn:

- UV–vis spectroscopy is not inferior to LMLD in effectively following the sonication process and capturing its different stages.
- UV–vis peak intensity is a versatile convolution of the two-fold information contained in LMLD, namely the mean agglomerate size and the polydispersity characteristics of the suspension.
- For SDBS-assisted suspensions, an energy density rate of 7.7 J min^{-1} ml^{-1}, applicable for a duration of 90 min, is sufficient for homogeneous distribution of the suspension under investigation.
- Low SDBS concentrations require at least 90 min of sonication for achievement of homogeneity of suspensions, while acceptable results can be obtained even after only 30 min of sonication provided the SDBS concentration is high enough.
- Compared to the surfactant, Viscocrete plasticizer, offers lower dispersion quality and cannot adequately separate the agglomerates in the suspensions even when used in higher concentrations.

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