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Measurement of the dispersion stability of pristine and surface-modified multiwalled carbon nanotubes in various nonpolar and polar solvents

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Abstract

A qualitative and rapid measurement technique based on multiple light scattering was employed to analyze the dispersion stability of black multiwalled carbon nanotube (CNT) suspensions. Pristine and chemically oxidized CNTs were dispersed in various polar and nonpolar solvents. The change in the transmission of near-infrared light from the suspensions was periodically measured along the height of a sample cell at room temperature. Using this method, it was possible to obtain the variation of the dispersion stability within only a day. Pristine and surface-modified CNTs dispersed in nonpolar media aggregated within 2 h and sedimentation progressively proceeded with time. As the polar component of the solubility parameter and the solubility in water decreased, faster aggregation and severe sedimentation occurred and vice versa. When the CNTs were modified with carboxylic anion groups, the dispersibility in polar solvents was significantly enhanced due to the combination of polar–polar affinity and electrostatic repulsion, with the result that the transmission flux remained unchanged. The origin of electrostatic repulsion can be found from the increased zeta potential and conductivity of CNTs with carboxylic anion groups.

Keywords: carbon nanotube, surface modification, oxidation, dispersion, stability

1. Introduction

Currently multiwalled and singlewalled carbon nanotubes (CNTs) have come into the spotlight in nanoscience and nanotechnology since they were discovered in 1991. Their enforced mechanical [1], electrical [2] and optical properties [3] have attracted the material industry and academic society. Owing to their great possibilities, CNTs are expected to substitute a variety of classical materials in the near future.

However, CNTs with their high van der Waals force, surface area, high aspect ratio inevitably cause self-aggregation. The improvement of dispersion has become a challenge to maximize the properties of CNTs [4]. In order to overcome self-aggregation, chemical modification of the CNTs' surface or utilization of surfactants is regarded as an effective way to improve their wettability and adhesion to host matrix materials [5]. When surfactants are employed in CNT dispersions, surfactant molecules

work by adsorption at the interface and self-accumulation into supramolecular structures, which help CNT dispersion retain a stable colloidal state. Coulombic or hydrophobic attraction plays a key role in achieving stable colloidal systems in ionic or nonionic surfactants, respectively [6–8].

Besides surfactants, gigantic polymeric molecules are also adopted where steric stabilization is a concrete reason for stable colloidal systems. For this purpose, noncovalent and covalent functionalization techniques are used. In noncovalent modification, wrapping CNTs with polymer molecules using the interfacial activity of CNTs is the method to treat the surface of CNTs with minimum damage to CNTs. For this noncovalent adsorption, various hydrophilic uncharged polymers have been tried including poly(vinylpyrrolidone) [9], poly(vinyl alcohol) [10] and poly(ethylene oxide) [11]. On the other hand, the surface of CNTs can be covalently modified by various polymeric chains to endow permanent steric stabilization. Those polymer brushes can be generated by the ‘grafting-from’ or ‘grafting-onto’ approach [12]. Without doubt, suitably chosen surfactant-coated or polymer-treated carbon nanotubes are known to form an exfoliated structure of nanotubes. However, it should be noted that the electrical conductivity of polymeric CNT composites prepared with auxiliary dispersants becomes remarkably lower, i.e. five orders of magnitude lower [13], which now embarrasses researchers working with CNTs for a decade. Certainly the reduction of the electrical conductivity originates from the inclusion of impurity substances, i.e., dispersants which are deliberately added to obtain exfoliated carbon nanotubes.

CNTs uniformly dispersed in solvents can be utilized in direct conductive coating, further sol–sol process and nanocomposite manufacturing. However, the evaluation of the dispersion state of CNTs in liquids is quite difficult due to its blackness even at a very low concentration. The most frequently and generally used technique is to ‘just wait’ until CNTs settle down and observe the dispersion with the naked eye or a digital photograph. However, it takes too long a time, even several months, and one cannot obtain quantitative or qualitative data during the sedimentation process.

The above-mentioned dilemmas became the motivation of this research. In order to obtain stable aqueous dispersion of CNTs without any auxiliary dispersant, we modified multiwalled CNTs to possess carboxylic acid (–COOH) or carboxylic anion (–COO[–]) groups by a well-established chemical oxidation reaction [14] followed by treatment with an aqueous NaOH solution. The CNTs with carboxylic acid and carboxylic anion were dispersed in various hydrophobic and hydrophilic solvents. The dispersion stability of the pristine and modified CNTs in the liquids was qualitatively analyzed by employing a multiple light scattering technique which enables one to investigate the colloidal stability of a black CNT dispersion only within a day.

2. Experimental section

2.1. Materials

Multiwalled CNTs (95% pure by TGA analysis) were purchased from Iljin Nanotech, Korea. 63% nitric acid (HNO₃)

and 98% sulfuric acid (H₂SO₄) from Duksan Pure Chemical, Korea were used as received. Styrene, toluene and methanol purchased from Samchun Pure Chemical, Korea were used as dispersion media.

2.2. Surface modification of CNTs

CNTs (2 g), sulfuric acid (8 mL) and nitric acid (22 mL) were charged in a 250 mL round bottom flask equipped with a condenser and a 50 mm plain-type stirrer. The flask was sonicated for 15 min with a horn-type ultrasonic apparatus. The chemical oxidation reaction was carried out at 110 °C for 24 h. After acid treatment the MWNTs are functionalized with the carboxylic acid groups (COOH) on the surface [14]. After cooling down to room temperature, the mixture was diluted with 1000 ml of deionized water, then vacuum filtered through a 0.2 μm Teflon filter (Adventec MFS). The CNTs were re-dispersed in 1000 ml deionized water and filtered again. For the substitution of the carboxylic acid groups with carboxyl anion groups (–COO[–]), the obtained carboxylated CNTs were mixed under ultrasonication with 50 ml of 10 wt% aqueous NaOH solution three times. Filtering, washing with DDI water and redispersion were repeated at least ten times until the pH of the filtrate reached near 7.0. 1.65 g CNTs with carboxylic anions were finally obtained after complete drying.

2.3. Characterizations

A transmission electron microscope (TEM) (CM200, Philips) was used for the observation of the morphology of CNTs. Thermal gravity analysis (TGA) in order to verify the degree of oxidation was performed using TGA Q50 (TA Instrument, USA) in a nitrogen atmosphere at a heating rate of 20 °C min^{–1}. 0.025 g functionalized CNTs were dispersed into 25 g deionized water and the conductivity was measured with GM-115, KEM Co., Japan. The zeta potential (ζ) of the CNT dispersion in water was obtained with a Malvern Zetasizer 4 (UK) at an angle of 90° at room temperature. The ζ values are calculated from the particle velocity by means of the Helmholtz–Smoluchowski equation [15]

$$\zeta = 4\pi\mu\eta/D,$$

where μ is the electrophoretic mobility and η and D, respectively, stand for the velocity and the dielectric constant of the liquid at the boundary layer. 0.1 or 0.01 wt% pristine and modified CNTs were dispersed by means of ultrasonication (Bandelin, HD2200) in various media including styrene, toluene, methanol and deionized water and the dispersion stability was measured by Turbiscan (Formulation, France) for 24 h. The migration behavior of the CNTs in various liquids was monitored by measuring the backscattering and transmission of monochromatic light (λ = 880 nm) from the suspension. Suspensions in flat-bottomed cylindrical glass tubes (70 mm height, 27.5 mm external diameter) were placed in the instrument and the transmission of light from suspensions was then periodically measured along the height at room temperature. The transmission detector receives the light going out of the sample at 0° from the incident beam, while the backscattering detector receives the light scattered by the sample at 135° from the incident beam. The results from transmission are presented as the sedimentation profile,

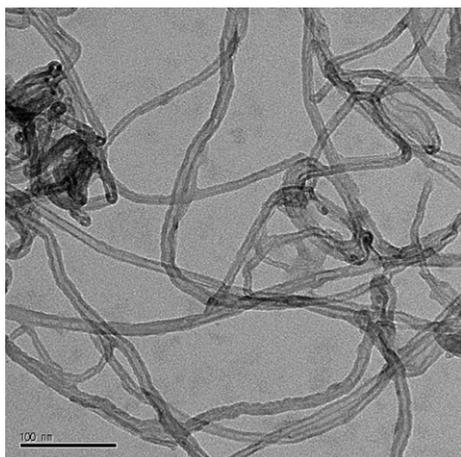


Figure 1. TEM microphotograph of pristine CNTs used in this study.

i.e., Δ transmission flux versus time. The backscattering (BS) and transmittance (T) of incident light are measured by calculating the transport mean free path of photons (l^*) through the medium. Based on Mie theory, the BS and T can be obtained for a concentrated suspension as follows [16]:

$$BS \approx (1/l^*)^{1/2} \quad (1)$$

$$T \approx \exp(-r/l) \quad (2)$$

where r is the internal radius of a measurement cell. The photon transport mean free path (l^*) and photon mean free path (l) are defined as follows:

$$l^* = \frac{2d}{3\Phi(1-g)Q_s} \quad (3)$$

$$l = \frac{2d}{2\Phi Q_s} \quad (4)$$

where d , Φ , g and Q_s denote the particle mean diameter, the volume fraction of a dispersed phase, the asymmetry factor and scattering efficiency factor, respectively.

3. Results and discussion

Figure 1 shows the TEM morphology of pristine CNTs. The multiwalled CNTs were synthesized by a thermal chemical vapor deposition (CVD) process using an aluminum nitrate catalyst. The CNTs have a diameter of 10–20 nm and a length of 10–50 μm . The average diameter is found to be 11.8 nm measured from the TEM microphotograph.

Thermogravimetric analysis (TGA) was performed for the quantitative analysis of the degree of modification of CNTs by chemical oxidation using a mixture of nitric acid and sulfuric acid. The weight loss curves of raw CNTs and oxidized CNTs are represented in figure 2. For raw CNTs, a constant weight is maintained up to 600 $^\circ\text{C}$ and complete decomposition is achieved at 700 $^\circ\text{C}$, while the surface-treated CNTs have approximately 100 $^\circ\text{C}$ lower complete decomposition temperature due to possible destruction of perfect CNT structure during the oxidation process, as reported elsewhere [17]. Approximately 12 wt% of surface acid

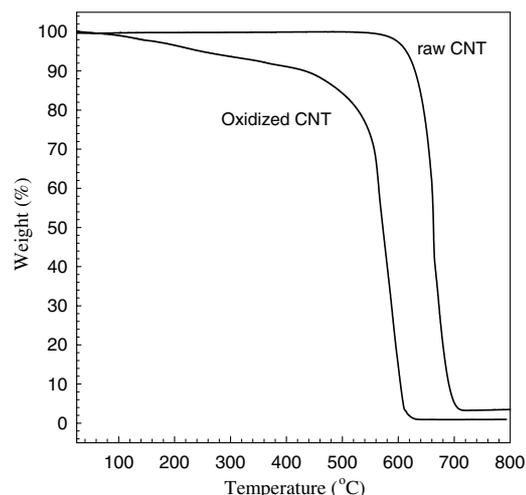


Figure 2. TGA weight loss curves of raw CNTs and oxidized CNTs.

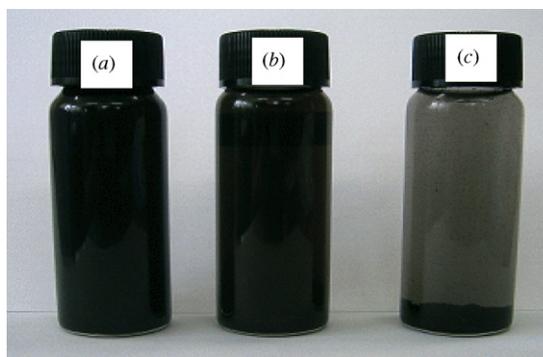


Figure 3. Digital photographs of 0.01 wt% raw and surface-modified carbon nanotubes dispersed in water. Pictures were taken after 2 months: (a) CNT-COOH, (b) CNT-COO⁻ and (c) CNT.

groups are developed on the surface of CNTs by chemical oxidation.

CNTs tend to cohere in aqueous solutions due to their high surface energy and lack of chemical affinity with the dispersing medium. Conventionally, the investigation of CNTs' dispersibility in a liquid phase is difficult due to its opaque blackness. So, one has to just wait until distinguishable visible change of the darkness of the CNT suspension takes place. Numerous previous studies on the dispersion stability have depended on the naked eye in order to investigate the dispersion of CNTs in solvents [18–22]. For a more quantitative analysis, UV-Vis spectroscopy [22–24] and small-angle polarized light scattering combined with optical microscopy [25, 26] have also been utilized for a few studies.

Figure 3 shows digital photographs of the 0.1 wt% pristine and modified CNT dispersions in water. It is noted that we had to wait 2 months for a clear distinction of the sedimentation of CNTs in the dispersions. The pristine CNTs dispersed in water severely aggregated within a few hours and sedimentation started in a day. After 2 months, most CNTs settled down in the dispersion. In contrast, the modified CNTs bearing carboxylic acid and carboxylic anion showed much improved stability in water due to the chemical affinity between the polar modified groups and water. Judging from the photographs, it is

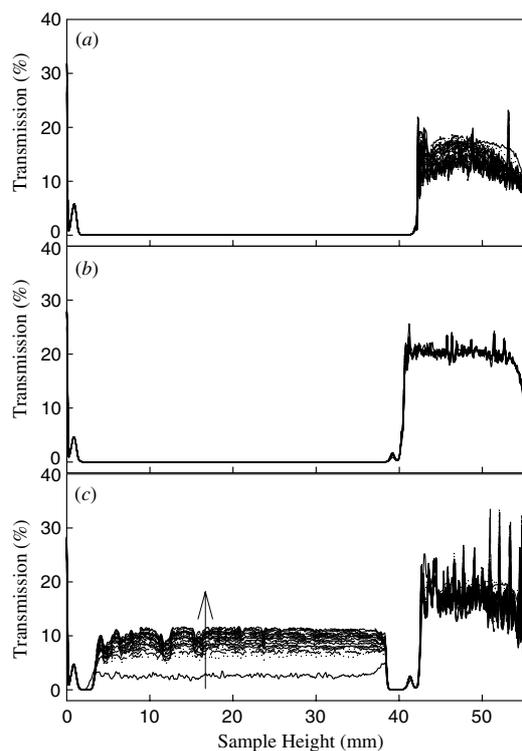


Figure 4. Transmission intensity versus the height in the sample at different times for the 0.01 wt% different CNTs suspended in H₂O at room temperature: (a) CNT-COOH, (b) CNT-COO⁻ and (c) CNT.

impossible to distinguish the difference between the dispersion stability of CNT-COOH and CNT-COO⁻ samples in water. In fact, a substantial amount of CNT-COOH settled down for a prolonged time span up to 2 months, whereas CNT-COO⁻ stays in water without agglomeration or sedimentation. This observation strongly suggests that an improved technique is required for a proper analysis of the black CNTs dispersion.

A recently developed optical analyzer, Turbiscan[®], was successfully employed to investigate the dispersion stability of the black pristine and surface-modified CNTs in an aqueous solution. The instrument was originally used to study emulsion stability. It measures any change in the sample by monitoring the transmission or backscattering of near-infrared light every 40 μm along the sample cell. Using Turbiscan, dispersion destabilization phenomena, including particle size variation kinetics such as coalescence or flocculation and particle migration kinetics such as creaming or sedimentation, are readily investigated even in concentrated or opaque emulsions or suspensions [27]. In this experiment, the sample cell containing CNTs suspended in an aqueous solution was placed in the instrument and its aggregation behavior is monitored every 1 h where transmittance intensity versus the height in the sample at periodic times is obtained in figure 4. Using the primary data, the change in transmittance versus time, indicating the aggregation kinetics, is calculated from the embedded software, as seen in figure 5. For pristine CNT dispersion, the transmission flux drastically increases within 1 h and steadily increases with time. In contrast to the behavior of pristine CNTs, no discernable change in the transmission flux with time is observed for the treated CNTs

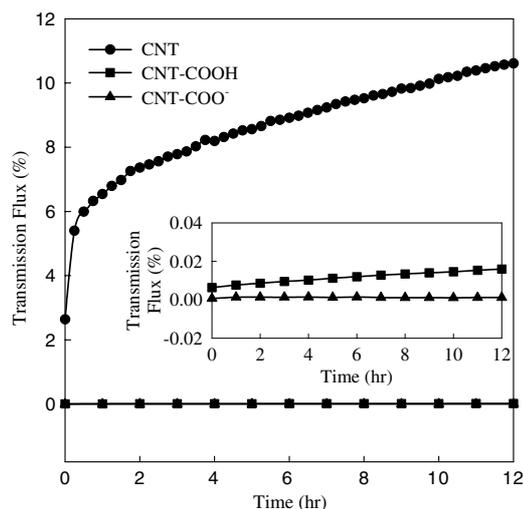


Figure 5. Aggregation behavior of the 0.01 wt% pristine and modified CNTs in water.

having carboxylic acid and carboxylic anion groups. When the transmission variation is magnified for CNT-COOH and CNT-COO⁻ (inset to figure 5), the transmission flux is slightly increased for CNT-COOH, while it remains nearly unchanged for CNT-COO⁻ due to electrostatic repulsion. This result suggests that the surface-treated CNTs, especially CNTs with carboxylic anion groups, have a much improved dispersion stability in an aqueous solution so that the aggregation of each CNT can be minimized. The surface chemical nature of pristine and carboxylated CNTs explains the difference between dispersion states in water. The zeta potential is an important indicative of ionically stabilized colloid systems. Greater magnitude (usually ± 25 mV) of the zeta potential endows the colloid system with an improved stability against coagulation. The variation of the surface properties of the colloids stabilized by an ionic mechanism also causes an electrical property to change. In this regard, conductivity measurement also provides useful information with high accuracy for the ionically stabilized colloid system [28]. The pristine CNTs have a conductivity $0.02 \mu\text{S cm}^{-1}$ and zeta potential of -8.3 mV at pH 7. The conductivity sparingly increases to $0.03 \mu\text{S cm}^{-1}$ and the zeta potential increases to -34.8 mV for CNT-COOH. When the carboxylic acid group is further modified with a carboxylic anion group, the conductivity jumps to $0.13 \mu\text{S cm}^{-1}$ and the zeta potential is increased to -53.2 mV (table 1). It is noted that the zeta potential values are consistent with previously reported values for pristine and oxidized CNTs [29]. The enlarged values of the conductivity and zeta potential of oxidized CNTs, especially CNT-COO⁻, well account for the much improved dispersibility in water. Therefore, the dispersion stability of pristine and modified CNTs in water has a strong relationship with the conductivity and zeta potential.

The pristine CNTs were dispersed in several solvents including styrene and toluene for non-polar solvents, and water as a polar solvent. The concentration of CNTs was fixed at 0.01 wt%. Aggregation and sedimentation occur in the increasing order of water, toluene and styrene. In all cases, the transmission flux suddenly increases with time, as shown in

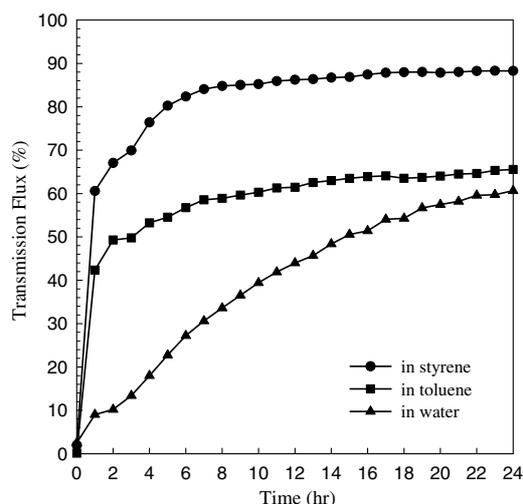


Figure 6. Aggregation behavior of the 0.01 wt% pristine CNTs in polar and nonpolar solvents.

Table 1. The conductivity and zeta potential at pH 7 of raw and surface-modified carbon nanotubes in water.

Sample	Conductivity ($\mu\text{S cm}^{-1}$)	Zeta potential (mV)
CNT	0.02	-8.3
CNT-COOH	0.03	-38.6
CNT-COO ⁻	0.13	-53.2

Table 2. The solubility in water and the Hansen solubility parameter of each liquid.

Solvent	Solubility in water (mol dm^{-3})	Solubility parameter, δ ($\text{cal}^{1/2} \text{cm}^{-3/2}$)	Polar component, δ_p ($\text{cal}^{1/2} \text{cm}^{-3/2}$)
Toluene	7.2×10^{-3}	8.9	0.7
Styrene	4.3×10^{-3}	9.3	0.5
Methanol	Complete solubility	14.5	6.0
Water	-	23.5	11.1

figure 6. In nonpolar solvents, the pristine CNTs severely aggregate to settle down within 2 h. On the other hand, aggregation and sedimentation take place almost linearly with time in a polar solvent. Digital photographs and the variation of transmission show clear phase separation between CNTs and solvents for all cases. The relation between aggregation and the following sedimentation phenomena and the type of solvent is found from the solubility parameter and the degree of polarity as listed in table 2. It has been reported that the total solubility parameter (δ) cannot explain the degree of pristine CNT dispersion in liquids, whereas the dispersion term (δ_d) is more appropriate to explain the dispersibility [30]. Herein we have found that the total solubility parameter has little importance accordingly, whereas the polar component (δ_p) together with the degree of polarity as can be expressed as solubility in water is more significant. As the polar component, δ_p , of the solubility parameter and the solubility in water decrease, faster aggregation and severe sedimentation occur.

Now, the dispersion stability of CNT-COO⁻ is examined in various nonpolar and polar solvents, as shown in figure 7.

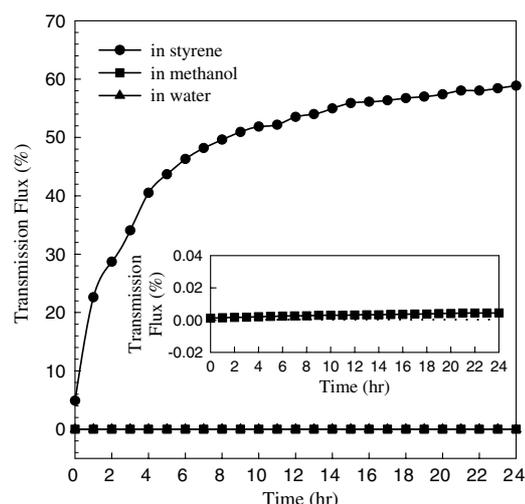


Figure 7. Aggregation behavior of the 0.01 wt% CNTs modified with carboxylic anion groups in polar and nonpolar solvents.

The concentration of CNTs was again fixed at 0.01 wt%. Due to the polar carboxylic anions on the surface of CNTs, the transmission flux rapidly increases up to 4 h, followed by a steady increase with time in a nonpolar solvent, styrene. An excellent dispersion stability is obtained for CNT-COO⁻ in polar solvents, methanol and water. Although the solubility parameters of methanol and water are very different, the stable dispersion state is achieved by the combination of polar-polar affinity and electrostatic repulsion in polar solvents. Finally, the improved dispersion of CNTs in water can be effectively utilized to prepare well-dispersed water-soluble polymer/CNT nanocomposites [31].

4. Conclusion

A novel measurement technique based on multiple light scattering is successfully employed to analyze the dispersion stability of black CNT suspensions. Pristine and chemically oxidized CNTs were dispersed in various polar and nonpolar solvents and the change in the transmission of near-infrared light from the suspensions was periodically measured along the height at room temperature. This method enables one to measure the state of CNT dispersion in both qualitative and quantitative ways. Furthermore, the change in dispersion stability can be rapidly achieved within a day. An improved dispersion stability results for oxidized CNTs in polar media. When the CNTs are modified with carboxylic anion groups, the dispersion stability in polar solvents was significantly enhanced due to the combination of polar-polar affinity and electrostatic repulsion. The presence of electrostatic repulsion can be found from the conductivity and zeta potential of the modified CNTs. The dispersion stability can be interpreted by means of the solubility parameter and solubility of a chosen solvent in water. As the polar component of the solubility parameter and the solubility in water decrease, faster aggregation and severe sedimentation occur and vice versa. Using this principle, it would be possible to fabricate nanocomposites consisting of water-soluble polymers and CNTs modified to possess electrostatic repulsion.

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