Dispersion of Single Wall Carbon Nanotubes by Bath and Tip sonication

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INTRODUCTION

In order to make use of carbon nanotubes (CNTs) for circuit wiring or incorporation into composite materials, bundles of CNTs need to be dispersed, categorized (insulator, semiconductor, conductor, size, etc.) and aligned. This study was focused on dispersion of single wall carbon nanotubes (SWCNTs) in various solvents agitated by tip and bath sonication. The dispersion states of single wall carbon nanotubes were characterized with optical microscope (OM) and atomic force microscope (AFM). It was found that the dispersion states were not only depended on type of solutions but also on the sonication power.

EXPERIMENTAL

A bath sonicator (690DA, Crest Ultrasonics, 42-45 kHz, average sonic power 270W) and a tip sonicator (Ultrasonic Processor, 20kHz±50Hz, 750 W, Model QEX750-5C; amplitude set at 40%) were used in this study to disperse the carbon nanotubes. During tip sonication, the solutions were cooled by ice.

The solvents used in this study included water (demineralised), methanol 99.8%, reagent alcohol (RA), chloroform stablized 99+%, acetone p.a.99.5%, isopropyl alcohol extra pure (IPA), N,N-dimethylformamide (DMF) 99%, and 1,1,1-trichloroethane (TCA) 95.7%. According to the vendor, cheaptubes.com, the SWCNTs purity is more than 90 wt%.

The concentration of 0.1 mg/ml of SWCNTs in solutions was prepared in 25 ml of solvents. The different sets of SWCNTs/solvents were sonicated by tip and bath sonicator for 30 minutes and 72 hours, respectively. The samples were taken at every 24 hours for bath sonication and every 10 minutes for tip sonication. The samples were deposited on mica without centrifugation and characterized by OM and AFM (Multimode, Nanoscope IIIa, Veeco).

RESULTS AND DISCUSSIONS

Figure 1 a) shows the solutions of SWCNTs after 30 minutes of tip sonication and left in quiescence condition for 14 weeks. Figure 1 b) shows the solutions after 72 hours of bath sonication and left in quiescence condition for 12 weeks. For tip sonication, the solutions of SWCNTs in chloroform, acetone, IPA, DMF and TCA appeared to be dark. This suggested that they were in dispersed state even though the solution of chloroform and acetone appeared a little lighter. The solution of water, methanol, and RA appeared clear and SWCNTs were swollen. It was also noticed that the size of swollen SWCNTs in methanol and RA were bigger than those in water.

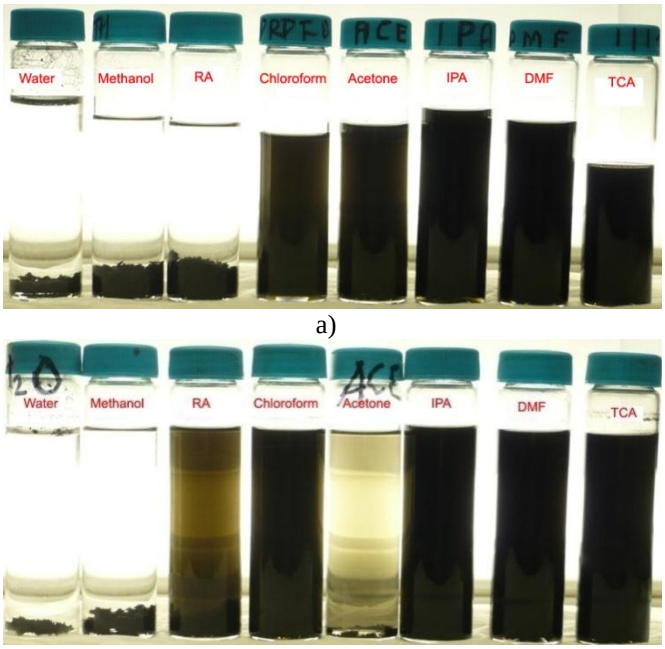

Figure 1. 0.1 mg/ml of SWCNTs in solutions after: a) tip sonication for 30 min.; and b) bath sonication for 72 hours.

From AFM images, the only solution that showed fully dispersion state was TCA, as shown in Figure 2 a). SWCNTs in IPA, chloroform, and DMF were found in a state between dispersed and swollen, and for those in acetone between swollen and sediment. This was unexpected for DMF, as from previous experiments DMF was able to fully disperse SWCNTs after 30 minutes of tip sonication (30% amplitude) [2]. It might be partially caused by water moisture in the air that were condensed on the side wall of vials during the tip sonication and accumulated in the solutions. Figure 3 shows AFM images after 10 minutes of tip sonication in: a) chloroform; and b) DMF, which showed the better results than those for 30 minutes of sonication. It was noted that TCA was not miscible in water, chloroform neither likely, but DMF was able to be so. This also supported the assumption of water accumulation in the solutions during tip sonication.

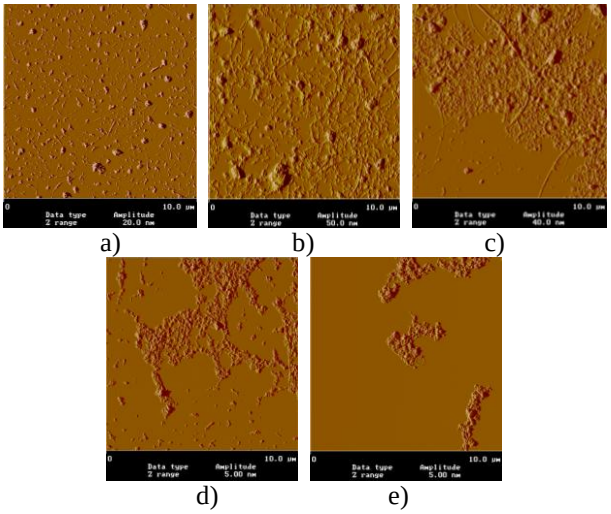

Figure 2. AFM images of SWCNTs/solvents: a) TCA; b) IPA; c) chloroform; d) DMF; and e) acetone, after tip sonication for 30min.

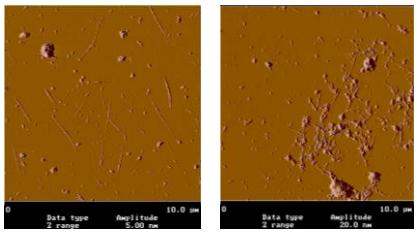

Figure 3. AFM images of: a) SWCNTs/chloroform; and b) SWCNTs/DMF, after tip sonication for 10 min.

For the bath sonication, the visible appearance shown in Figure 1 b) was similar to those in tip sonication (Figure 1 a), except the sizes of swollen SWCNTs in water and methanol by the bath sonication were smaller than those in tip sonication. Interestingly the acetone solution was quite clear, instead of dark color as in tip sonication. These might subject to the difference in sonication power between the tip and bath sonication. The RA solution was dark, instead of clear as in the case of tip sonication. This might be due to the water accumulation during tip sonication. From AFM images shown in Figure 4, the solutions that showed fully dispersion were DMF, TCA and chloroform. SWCNTs in IPA were not fully dispersed. And RA solution was in a state between swollen and dispersed. It was noted from AFM images of both tip and bath sonication that only TCA and chloroform were able to disperse SWCNTs well with relatively "clean" nanotubes. This was believed to be related to their similar atomic structures and solubility parameters (TCA - δ_t:17.4, δ_d:16.8, δ_p :4.3, δ_h :2.0; chloroform - δ_t:19.0, δ_d:17.8, δ_p :3.1, δ_h :5.7 $MPa^{1/2}$. Here δ_t is Hildebrand solubility parameter, and δ_d,δ_p,δ_h are dispersion, polar and hydrogen bonding component of Hansen solubility parameters, respectively[3]).

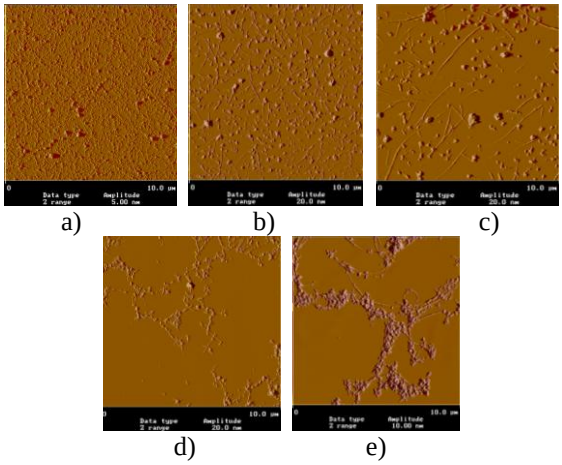

Figure 4. AFM images of SWCNTs/solvents: a) DMF; b) TCA; c) chloroform; d) IPA; and e) RA, after bath sonication for 72hours.

Dispersion of SWCNTs in solvents blending with a second solvent, acetone, was also conducted by bath sonication for 24 hours. DMF/acetone 60%/40% solution with SWCNTs appeared to be dark and AFM images showed similar results to those of non-blending DMF. The visible appearance of the different proportion of chloroform/acetone mixtures, ranging from 50 v% to 70 v% of chloroform, showed a clear distinguish in solutions' color between 52 v% and 54 v% of chloroform, with the former appearing light grey and the latter at dark color. During the experiments it was noted that the solution with 62 v% chloroform appeared to be in swollen state. But after changed position of the vial closer to an agitation source in bath sonication tank, it was turned to be dark. This effect caused by a small change in agitation power indicated that sonication power would be an important factor for SWCNTs dispersion.

CONCLUSIONS

The results of this work suggested that SWCNTs in water, methanol and acetone, while they were previously classified as in sedimented dispersion state [1], were able to be classified as swollen state. This might be caused by a difference in agitation power. The assumption was supported by comparison of the solution dispersion states in tip and bath sonication. Among the solvents, only TCA and chloroform were able to disperse SWCNTs with relatively "clean" nanotubes. This might be related to their similar atomic structures and solubility parameters. It was also found that sonication power would be an important factor for SWCNTs dispersion.

REFERENCES

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