Spectroscopic analysis of CNT dispersed by electron-beam irradiation

Seung Hwa Yoo*, Cheol Hyeon Lee
Department of Quantum System Engineering, Jeonbuk National University, Jeonju-si, 54896, Republic of Korea
**Corresponding author: seunghwayoo@jbnu.ac.kr*

1. Introduction

It is clear that CNT has very high utilization potential in various fields due to its excellent physical and electrical properties. In our previous study, we confirmed that simply irradiating electron-beam to CNT-solvent dispersion can helps to improve dispersibility. In this experiment, following the last study, we quantitatively measured how much electron-beam irradiation affects the improvement of dispersibility and investigated the effect of electron-beam irradiation on CNT and CNT dispersion.

2. Methods and Results

2.1 Preparation of CNT dispersion solution

In this experiment, multi walled CNTs (Product of Nanocyl, NC 7000) were used. CNTs were added to each solvent (DI water, NMP, DMF, DMAc and DMSO) [1] and tip sonicated for 2min. Samples were irradiated using an electron accelerator with 10 MeV energy, and absorbed doses were 50, 100, and 150 kGy. After irradiation, samples were stabilized for 2h and bath-sonicated for 30 min.

2.2 Zeta-potential

We measured the zeta potential of the sample to quantitatively evaluate the dispersion. Supernatants were assessed after 1, 3, 7 and 14 days of settling time. As a result, it was confirmed that dispersion was improved by electron-beam irradiation in three of the five solvents we used. This difference was shown 3 or 7 days after the irradiation. In the case of DMF and DMAc, the significantly higher value (Max. <-50mV) of zeta-potential were shown with very stable dispersion. Although NMP showed slight difference compared to the case of DMAc and DMF, it was also confirmed that the dispersion stability was clearly improved after 14 days. [2]

2.3 Structural analysis

Various spectroscopy (FT-IR, Raman, XRD, and XPS) were conducted to determine whether CNT surface changes or structural damage occurred by electron beam irradiation. Samples were filtered to remove the solvent, by washing with DI water of a total of 1 liter or more. The remaining CNTs powders were dried at 80°C for more than 96 h.

As a result of the comprehensive analysis, it was found that there was no surface or structural damage by the electron-beam irradiation, therefore it is estimated that the dispersion by the electron-beam irradiation is not caused by the change in CNT.

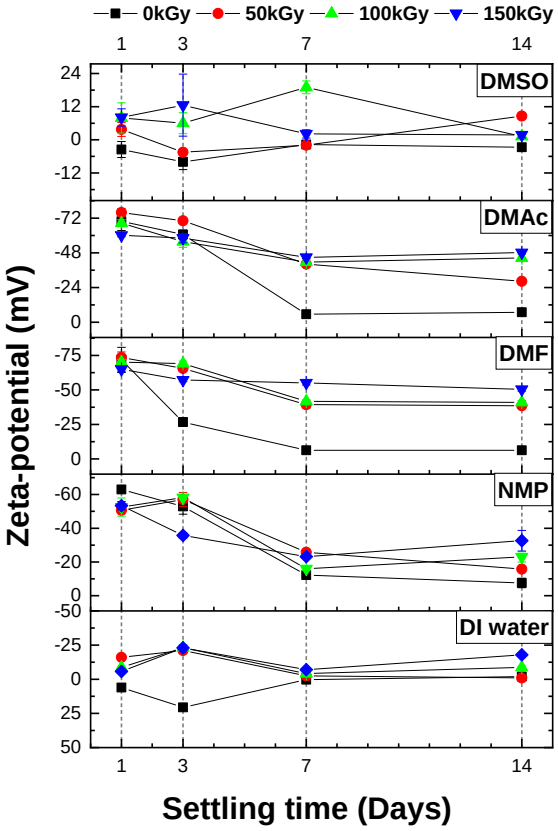

Fig. 1. Zeta-potential of samples after 1, 3, 7 and 14 days of settling time.

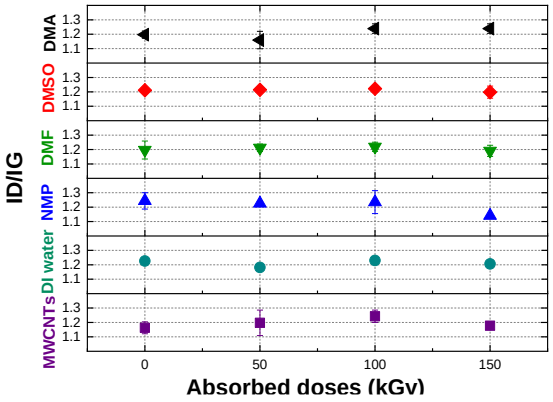

Fig. 2. ID/IG of Raman spectrum of samples.

2.3 NMR spectroscopy

NMR measurement was attempted to analyze the dispersion mechanism of CNT by electron-beam

irradiation. By adding a small amount of our sample to chloroform with H substituted with D, we were able to obtain NMR spectrum using 600 MHz NMR spectrometer.

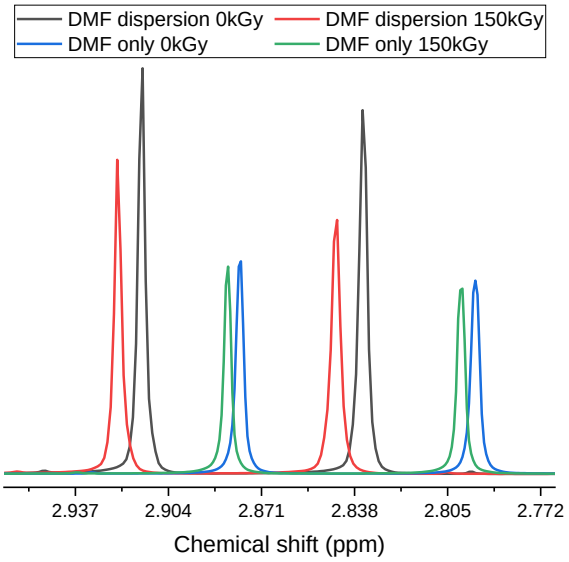

Fig. 3. Comparison of methyl group NMR spectra of irradiated and non-irradiated DMF dispersion and DMF.

The NMR spectra we measured showed that the pick corresponding to the methyl group of DMF was clearly shifted. This shift is not only caused by the addition of CNT, but also by the addition of the shift after the electron-beam irradiation. This is presumed to be caused by the interaction of the CNT with the solvent before and after the electron beam irradiation.[3]

3. Conclusions & Future work

In this study, the improved dispersion using electron-beam irradiation was evaluated, and CNT and its dispersion were analyzed through various spectroscopy.

As a follow-up study, Hansen Solubility Parameter will be introduced to understand the dispersion mechanism of CNTs by electron-beam irradiation. For more various solvents, dispersion by electron-beam irradiation will be checked, and additional measurement and analysis will be conducted accordingly.

Acknowledgement

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