



The experimental study of the effect of microwave on the physical properties of multi-walled carbon nanotubes



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ABSTRACT

This paper reports the effect of microwave on the physical properties of multi-walled carbon nanotubes (MWCNTs) where different power levels of microwave were applied on MWCNTs in order to apprehend the effect of microwave on MWCNTs distinctly. A low energy ball milling in aqueous circumstance was also applied on both MWCNTs and microwave treated MWCNTs. Temperature profile, morphological analysis by field emission scanning electron microscopy (FESEM), defect analysis by Raman spectroscopy, thermal conductivity, thermal diffusivity as well as heat transfer coefficient enhancement ratio were studied which expose some strong witnesses of the effect of microwave on the both purification and dispersion properties of MWCNTs in base fluid distilled water. The highest thermal conductivity enhancement (6.06% at 40 °C) of MWCNTs based nanofluid is achieved by five minutes microwave treatment as well as wet grinding at 500 rpm for two hours.

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1. Introduction

Carbon nanotubes (CNTs), first experimentally observed in the early 1990s [1,2], are one of the most attractive nanomaterials for its some extraordinary properties such as one of the lightest [3], strongest [4], stiffest [4], electrically conductive [5] nanoparticles with high thermal conductivity [6]. Individual CNTs have mobilities in excess of 100,000 cm²/Vs [7], current carrying capacity of 10⁹ A/cm² [8] which is three orders of magnitude higher current than copper [9] and ON/OFF current ratios higher than 10⁵ [10]. The applications of CNTs have been reported in many works of literature [11–13]. For example, Liao et al. [11] conducted the Al-CNTs mixture for powder metallurgy use where they showed that a small addition of CNTs undoubtedly improved the tensile strength as well as hardness of the composite by comparing with the pure matrix. However, there are some problems in making CNTs based nanofluids where water is used as base fluid. Beside the

long, winding shapes and hydrophobic surfaces [14], commercially available raw CNTs contain different impurities such as amorphous carbon and metallic nanoparticles [15–17]. There are several reports have been published in order to overcome the aforementioned issues using various methods, such as mechanical grinding [6,18], ultrasonication [19,20], physical separation [21], liquid-phase oxidation [22] and combinational purification [23]. Among them, liquid-phase oxidation is carried out with acid solutions such as HNO₃ or mixture of HNO₃/H₂SO₄ or H₂SO₄/KMnO₄, which gives a better dispersion of MWCNTs in aqueous circumstance. For example, Yen et al. [24] showed the better dispersibility of acid oxidized MWCNTs in the base fluid in their experiment. However, this conventional high concentrated acid treatment causes defects on the CNTs' side walls which results in damaging the CNTs [25]. For example, Liu et al. [26] conducted concentrated nitric acid based oxidation process of CNTs and observed that CNTs were severely damaged which results in the degradation of the composite. As a solution to this problem, many researchers suggest that microwave treatment will be an efficient method for the purification of CNTs [17].

The microwave is a rapid, nonuniform and volumetric heating process [9]. Moreover, it is an attractive choice for chemical

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applications in recent nanoscience research [7]. For example, Va'zquez et al. [27] observed microwave assisted functionalization of CNTs, which gave a reduction in reaction time and a higher degree of functionalization than that obtained by the conventional thermal method. In the conventional heating method, thermal energy is absorbed on the surface of the workpiece and then the heat energy is transferred through the thermal conductivity mechanism which is slow [28]. On the other hand, microwave heating, applied at the most famous of the frequencies allowed for ISM applications [29], offers sintering at a much lower temperature and shorter time than that required in the conventional method [28]. The principle of the microwave heating is abstruse. Simply, at too lower frequency of radio wave, the permanent dipoles of dielectric material immediately follows the direction of electric field and at too much higher frequency of radio wave, dipole does not able to follow [30]. For these two cases, no heat generates. However, at microwave frequency, the dipole changes a little behind the electric field [30] and during this time, the material absorbs the energy of the microwave and yields heat. On the other hand, the skin effect is theorized as the primary cause of heating of metal by microwave. In this present experiment, MWCNTs has been taken into consideration that is always metallic.

The objective of this study was three-fold: (I) to observe the thermal profile and its effect (II) to analyze the purification state and (III) to observe the change in thermal properties of nanofluid by microwave treatment of MWCNTs. A special step had been taken into consideration for measuring the temperature of the MWCNTs sample which was heated in microwave. Moreover, in this regard, distance to spot ratio of the infrared thermometer was taken into account. In this paper, thermal profile and thermography of each level of microwave oven (Section 3.1), morphological analysis by FESEM (Section 3.2), defect analysis by Raman spectroscopy (Section 3.3) of microwave assisted heated MWCNTs, thermal conductivity (Section 3.4), thermal diffusivity (Section 3.4) and heat transfer coefficient enhancement ratio (Section 3.4) have been presented carefully and precisely. Here, it should be noted that the experimental approach and the results of this paper are only for observation and comparison and do not suggest the optimum conditions.

2. Experimental details

2.1. Materials

MWCNTs measuring ~ 20 nm diameter and ~ 5 μm length and with greater than 95% purity, less than 3% impurities and a specific surface area of $40\text{--}300\text{ m}^2/\text{g}$ (purchased from Carbon Nanomaterial Technology Co., Ltd., South Korea) was used. Distilled water (DW) was used as the base fluid for making the nanofluids. Surfactant Sodium dodecyl benzene sulfonate (SDBS, $\text{C}_{18}\text{H}_{29}\text{NaO}_3\text{S}$) with hard type, 348.48 molecular weight (Tokyo Chemical Industry Co., Ltd.) was used for better dispersion of MWCNTs in the base fluid.

2.2. Microwave oven

A domestic microwave oven (magic oven, MWO-20M7) with 2.45 GHz frequency was used to execute microwave treatment of MWCNTs. The microwave oven was slightly customized for the purpose of temperature measurement. For measuring the temperature of the sample inside the microwave oven by the infrared thermometer, a hole was shaped in the top surface of the microwave oven. The temperature for calculating the power was measured by a T-type thermocouple, data logger (MV 106-1-2-1F, YOKOGWA, Japan) and cool tap water. The thermocouple's temperature range is -250°C to 350°C . Digital stopwatch was

used to record the time. Since, the microwave has non-uniform heating effect, the heated water was stirred for 10s, and the temperature was measured by the thermocouple. The full process was done as follows [31].

In each microwave heating treatment performance, an infrared thermometer (AR330*, $-32^\circ\text{C}\sim 330^\circ\text{C}$) was used to measure the yielded temperature as well as the temperature during the convective heat dissipation of the heated sample MWCNTs into the surroundings of the microwave oven. In this experiment, the diameter of the porcelain sample container was 4.3 cm. The distance to spot ratio for this infrared thermometer is 12:1, and the distance from the top surface to the sample holder inside the microwave oven is 22 cm. So, it was necessary to use at least a sample container with a diameter of 1.83 cm (Fig. 1). At first, time for microwave treatment on MWCNTs was set for one minute, three minutes, and five minutes. Since, during five minutes, the effect of microwave on MWCNTs occurred more clearly i.e. more increase in time gives more clear appearance of effect of microwave on MWCNTs, however, there is also generation of more heat which will be risky to handle the experiment. So, heating for five minutes was chosen for the overall experimental approach of microwave treatment of MWCNTs

Portable infrared camera (Ti45, Fluke Inc.) was also applied in order to capture the infrared images as well as check the original temperature profile of the heated samples which could not be determined by infrared thermometer.

2.3. Grinding of MWCNTs

In this experimental study, a planetary ball mill (HPM-700, Haji Engineering, Korea) was used to shorten the length of the multi-walled carbon nanotubes by a simple grinding process. At first, dry grindings at 200 rpm and 400 rpm for one hour were done for making samples to check the effect of microwave on the ground as well as dried MWCNTs with different particles sizes. Later, wet grinding at 500 rpm for 2 h was chosen for dispersing MWCNTs into base fluid distilled water. The reason for choosing wet grinding is that, unlike dry grinding, almost all ground nanoparticles can be extracted after wet grinding process. As shown in Fig. 2, after dry grinding, most of the nanoparticles have agglomerated with grinding balls and pot firmly whereas after wet grinding the MWCNTs nanoparticles have loosely agglomerated, and it is easier to extract them all within minimum loss of nanoparticles. Moreover, Tang et al. [32] conducted the wet ground MWCNTs ultrasonically dispersed in chitosan solution and reported that wet-grinding could improve the water wet-ability of MWCNTs. And the reason for choosing 500 rpm is that Munkhbayar et al. [33] conducted their wet grinding of MWCNTs at various rotation speeds (200–500 rpm) and reported that the best dispersion

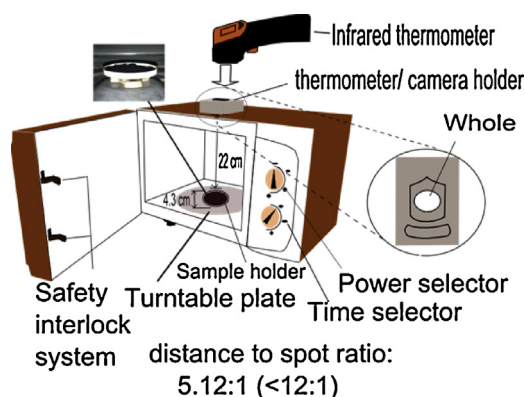


Fig. 1. Schematic diagram.

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