Bull. Mater. Sci., Vol. 37, No. 6, October 2014, pp. 1439–1445. © Indian Academy of Sciences.

Statistical analysis of thermal conductivity of nanofluid containing decorated multi-walled carbon nanotubes with TiO_2 nanoparticles

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MS received 16 June 2013; revised 1 September 2013

Abstract. In this paper, we report for the first time the statistical analysis of thermal conductivity of nanofluids containing TiO_2 nanoparticles, pristine MWCNTs and decorated MWCNTs with different amounts of TiO_2 nanoparticles. The functionalized MWCNT and synthesized hybrid of MWCNT–TiO_2 were characterized using transmission electron microscopy (TEM). TEM image confirmed that the ends of MWCNTs were opened during their oxidation of them in HNO_3 and TiO_2 nanoparticles successfully attach to the outer surface of oxidized MWCNTs. Thermal conductivity measurements of nanofluids were analysed via two-factor completely randomized design and comparison of data means is carried out with Duncan's multiple-range test. Statistical analysis of experimental data show that temperature and weight fraction have a reasonable impact on the thermal conductivity of all tested nanofluids ($\alpha = 0{\cdot}05$). The results also show that increased temperature and weight fraction leads to the increased thermal conductivity.

Keywords. **MWCNT; TiO_2 nanoparticles; thermal conductivity; nanofluid.**

1. Introduction

Dispersing the particles with a typical size of < 100 nm in the conventional heat transfer fluids such as oil and water leads to the production of new classes of heat transfer fluids, which are called nanofluids (Arani and Amani 2012). The application of particles with nanometer size reduced the clogging of flow channels, eroding of pipelines and pressure drop. In addition, compared with millimetre- or micrometer-sized particle suspensions, nanofluid have better long-term stability and greater thermal conductivity. Up to date, various types of nanoparticles such as metallic, non-metallic, polymeric and nanotubes were suspended in base fluids (Duangthongsuk and Wongwises 2009). Among the various kinds of nanoparticles, carbon nanotubes (CNTs), owing to their unique structure, high aspect ratio and excellent thermal conductivity, have attracted much attention. As a result, dispersing CNTs in a base fluid can enhance the thermal conductivity of nanofluid (Phuoc *et al* 2011). Dispersibility of nanoparticles in the base fluid is a key factor in the stability of nanofluids. Therefore, the application of dispersing agent such as surfactant can enhance the stability of nanofluids (Assael *et al* 2005). The previous study reported that the surfactant molecules which introduce to the outer surface of nanoparticles and CNTs increased the thermal resistance. Thus the application of surfactant decreased the thermal conductivity of nanofluids (Huxtable *et al* 2003). Introducing the oxygen-containing groups such as hydroxyl and carboxyl groups to the outer surface of carbon nanotubes has a significant effect on the high stability of CNTs nanofluids, which leads to the enhancement of thermal conductivity (Kyotani *et al* 2001).

Murshed *et al* (2005) reported the thermal conductivity of TiO_2/water-based nanofluids as a function of the shape of nanoparticles. They investigated TiO_2 nanoparticles of rod shapes of 10 nm × 40 nm (diameter by length) and in sphere shapes of 15 nm. Their experimental results show that the thermal conductivity of nanofluids was much higher than that of base fluid even in a small amount of nanoparticles. Meanwhile, they observed that volume fraction, particle size and shape of nanoparticles also influence the thermal conductivity enhancement of nanofluids.

Zhang *et al* (2007) investigated the heat transfer performance of TiO_2/water nanofluid for various volume fractions and temperatures. They observed that the effective thermal conductivities of nanofluids have not shown any anomalous enhancements.

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Chen *et al* (2008) reported the thermal conductivity of nanofluids containing multi-walled carbon nanotubes (MWCNTs), which were treated using mechanochemical reaction method to enhance their dispersibility. Their results showed the thermal conductivity enhancement up to 17·5% at volume fraction of 0·01.

Raykar and Singh (2010) investigated the thermal conductivity of nanofluids containing carbon nanotubes as a function of temperature and Brownian motion. They observed that Brownian motion played a key role in the thermal conductivity of nanofluids.

Talaei *et al* (2011) studied the thermal conductivity of nanofluids containing MWCNTs with various concentrations of functional group. They reported that the concentration of functional groups which attached to the surface of MWCNTs in different methods is varied. Meanwhile they observed that thermal conductivity of nanofluids increased with respect to the functionalized group concentration.

Xie and Chen (2009) investigated the thermal conductivity of nanofluids of MWCNs into ethylene glycol base fluid. Their experiments showed that the thermal conductivity was enhanced with nanotube loading and temperature.

Meibodi *et al* (2010) have reported the thermal conductivity and stability of CNT/water nanofluids as a function of nanoparticle size and concentration, surfactant type and concentration, pH, temperature, power of ultrasonication and elapsed time after ultrasonication and their interactions. Their results revealed that, as a result of the statistical analysis of the thermal conductivity, elapsed time after ultrasonication had the most significant effect and pH was not more significant.

Jha and Ramaprabhu (2009) investigated the thermal conductivity of nanofluids containing MWCNTs which were decorated with Pd, Au and Ag crystalline metal nanoparticle. They used water and ethylene glycol as base fluids without any surfactant. Their experiments corroborated that the thermal conductivity of these nanofluids increases with increase in concentration of metal-MWNT and with temperature.

Nevertheless the effect of many factors such as temperature, pH, concentration, size, shape and type of nanoparticles on the thermal conductivity of nanofluids have been reported by many researchers in the previous studies. In addition, a few papers have been published on the thermal conductivity of nanofluids containing decorated CNTs with nanoparticles. Although the thermal conductivity enhancement in the nanofluid containing decorated MWCNT with Pd, Au and Ag crystalline metal nanoparticle was investigated by previous investigators, it is clear that these nanoparticles have high intrinsic thermal conductivity. Therefore, the hybrid of these nanoparticles and MWCNTs can enhance the thermal conductivity of nanofluids. In this paper, we decided to choose TiO_2 nanoparticles with low intrinsic thermal conductivity, such that we could investigate whether the thermal conductivity enhancement of nanofluids containing the hybrid of TiO_2 nanoparticles and MWCNTs are considerable or not.

Till now, no paper has reported the effect of decorated MWCNTs with metal oxide such as TiO_2 nanoparticles with different amounts of nanoparticles loading. Meanwhile, the statistical analysis of the thermal conductivity of these nanofluids has never been investigated before. Therefore, in this paper, we want to investigate, for the first time, the statistical analysis of the thermal conductivity of nanofluid containing modified MWCNTs with various amounts of TiO_2 nanoparticles.

2. Materials and experimental

TiO_2 nanoparticles were prepared by the hydrolysis of titanium tetrachloride ($TiCl_4$, M = 189·79, 99%, Merck). The synthesis procedure of TiO_2 nanoparticles is as follows. A certain amount of $TiCl_4$ was dissolved in distilled water at ambient temperature and stirred for about 5 h. Then the temperature of the solution was increased to 80 °C and stirred for 24 h. Finally, the solution was filtrated using the Whatman filter paper (Ashless, circies, 125 mm) and the separated TiO_2 nanoparticles dried at room temperature and calcined at 370 °C for 3 h.

For the synthesis of decorated MWCNTs with TiO_2 nanoparticles, the following materials are needed. MWCNTs (average diameter of 40–60 nm and lengths ranging from 5 to 15 μm), nitric acid (HNO_3, M = 63, 65%, Merck), hydrochloride acid (HCl 37 wt%, Merck) and titanium tetrachloride which is mentioned earlier. The details of the synthesis procedure of MWCNT–TiO_2 has been described in our previous work (Abbasi *et al* 2013). Only a brief description is given here. The synthesis includes two steps. First, oxidation of MWCNTs with nitric acid for about 2 h in an ultrasound bath and 2 h in a stirrer at high speed to open the end of MWCNTs and introduce the oxygen-containing groups to their outer surface. Then, the oxidized MWCNTs were washed with distilled water until the pH value of the drained water reached neutral and dried overnight. Secondly, a certain amount of $TiCl_4$ was dissolved in 100 mL distilled water, followed by adding a little HCl (37 wt%) to the distilled water before $TiCl_4$ was dissolved in the water. Then, ~ 75 mg of the functionalized MWCNTs were added to the solution and dispersed using ultrasound bath for 2 h and stirred for 22 h. Finally, the temperature of solution increased to 80 °C and stirred for 3 h. then filtered, dried at 80 °C for 1 h and calcined at 370 °C for 3 h.

Thermal conductivity of nanofluids was measured using KD2 Prothermal property analyser purchased from Decagon Devices Inc. The measurements were carried out using the single needle (KS-1) of 60 mm length and 1·3 mm diameter and accuracy $\pm 0{\cdot}01\ \mathrm{Wm^{-1}\,K^{-1}}$ from

$0{\cdot}02$ to $0{\cdot}2$ Wm^{-1} K^{-1}. The effect of temperature and concentration was studied in the range of 25–70 °C and 0·25–1·5 wt%, respectively. For controlling the temperature during the measurements, a water bath (Thermo Haake K10 TT4310) at constant temperature which circulated the water around the double-walled cylindrical container of samples was connected to the KD2 Pro device. For accurate results, the measurements were repeated several times and only three measurements which had the mean correlation coefficient $r^2 > 0{\cdot}9998$ were considered. Transmission electron microscopy (TEM) micrographs of acid-treated and modified MWCNTs with TiO_2 nanoparticles were obtained using a LEO 912AB system which operated at 120 kV.

Statistical analysis of the thermal conductivity of nanofluids in different nanofluids was performed with a two-factor completely randomized design using Mstac (Ver. 1·42) and significant differences between means were determined by Duncan's multiple range test. P values $< 0{\cdot}05$ were considered statistically significant. For the investigation, the combined effects of temperature and weight fraction on the thermal conductivity of nanofluids, response surface method was measured out using Minitab Release software (Ver. 11.12).

3. Results and discussion

3.1 *Oxidation of MWCNTs*

As a result of oxidation of MWCNTs in nitric acid, the ends of tubes which closed with catalyst particles were opened. Furthermore, the acid treatment of MWCNTs leads to the cutting of MWCNTs to a shorter length. During oxidation of MWCNTs, the sp^2 hybridization of MWCNTs can be changed to sp^3. Therefore, the outer surface and open ends of MWCNTs functionalized with oxygen-containing groups such as acid carboxylic (–COOH) and hydroxyl (–OH). Figure 1 shows TEM image of acid treatment of MWCNTs in nitric acid.

3.2 *Decoration of MWCNTs*

Hydrolysis of $TiCl_4$ in the solution of functionalized MWCNTs leads to the production of titanium ions. These ions can be absorbed by hydroxyl and acid carboxylic groups which introduce to the outer surface of MWCNTs during the acid treatment process. These functional groups act as active sites and titanium ions attach due to electrostatic attraction. Therefore, the nucleation of TiO_2 nanoparticles carried out and the calcination of the powder leads to the formation of nanocrystalline TiO_2 on the outer surface of MWCNTs. It should be mentioned that the amount of $TiCl_4$ has a key role in the nucleation and formation of TiO_2 nanoparticles. Therefore, in this study we synthesized MWCNTs–TiO_2 with 34 and 61% of TiO_2 nanoparticles by changing the amount of $TiCl_4$. Figure 2 illustrates TEM image of decorated MWCNTs.

3.3 *Data analysis and statistical studies*

3.3a *Effect of temperature on thermal conductivity of nanofluids*: Figure 3 depicts the effect of temperature

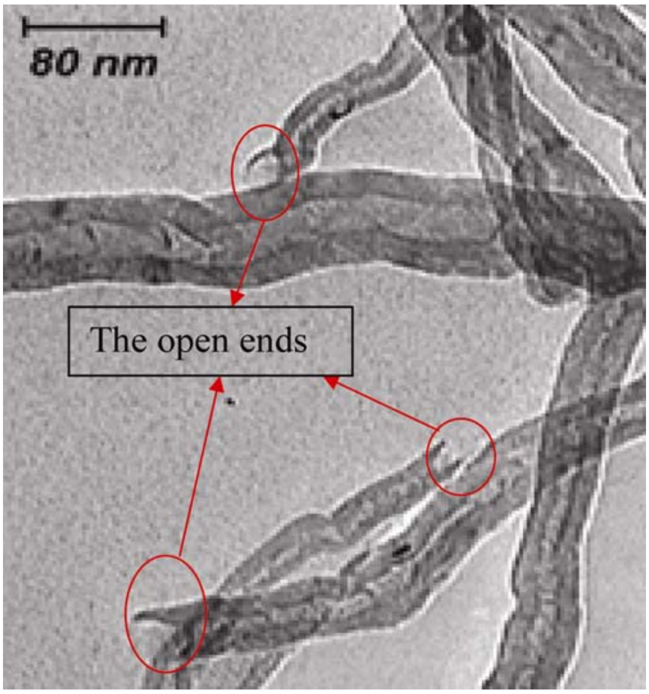

Figure 1. TEM image of acid-treated MWCNTs.

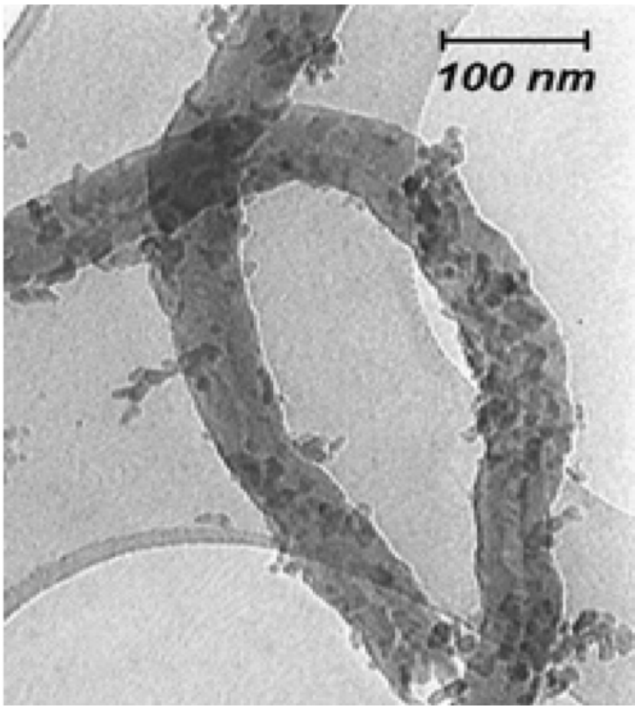

Figure 2. TEM image of MWCNTs–TiO_2.

on the thermal conductivity of TiO_2 nanofluids. As can be deduced, there was a completely significant difference among different temperatures from the aspect of thermal conductivity. By increasing the temperature from 25 to 70 °C, the thermal conductivity of TiO_2 nanofluid was increased. Index was increased from 25 °C (0·5907 $Wm^{-1} K^{-1}$) to 40 °C (0·6426 $Wm^{-1} K^{-1}$) equal to 8·78%; from 40 to 55° C (0·6623 $Wm^{-1} K^{-1}$) equal to 3·06% and from 55 to 70 °C (0·686 $Wm^{-1} K^{-1}$) equal to 3·57%. In addition, the thermal conductivity of TiO_2 nanofluid during the increase of the temperature, from 25 to 70 °C, was increased to ~ 16%.

The influence of temperature on the thermal conductivity of pristine MWCNTs nanofluid is shown in figure 4. It is clear that there was a significant difference among four temperatures for thermal conductivity of pristine MWCNTs nanofluid ($\alpha = 0{\cdot}05$). Moreover, it can be observed that by increasing the temperature, thermal conductivity also increased. Minimum thermal conductivity was related to a temperature of 25 °C equal to 0·6181 $Wm^{-1} K^{-1}$. The value of this quality at 40 °C was increased to 0·6852 $Wm^{-1} K^{-1}$, e.g. 10·85%, at 55 °C increased to 0·7202 $Wm^{-1} K^{-1}$, e.g. 5·10% and at 70 °C increased to 0·7572 $Wm^{-1} K^{-1}$, e.g. 5·13%. Therefore, the augmentation of the thermal conductivity of pristine MWCNTs nanofluid from 25 to 70 °C is equal to 22·50%. Meanwhile, comparing the augmentation of the thermal conductivity of pristine MWCNTs nanofluid and TiO_2 nanofluid revealed that increasing thermal conductivity of nanofluid containing pristine MWCNTs nanofluid (22·5%) is > TiO_2 nanofluid (16%). This can be due to the excellent intrinsic thermal conductivity of MWCNTs. In other words, the augmentation of the thermal conductivity of pristine MWCNTs nanofluid is 40·62% greater than that of TiO_2 nanofluid. Because in the nanofluids containing sphere metal or metal oxide nanoparticles, the Brownian motion of nanoparticles affected the variation of thermal conductivity with temperature (Das *et al* 2003). Nevertheless, in the nanofluids containing MWCNTs, in addition to the Brownian motion of MWCNTs, the chemical functionalized groups have a key effect on the amount of energy which transfers into the nanofluids by changing the temperature (Chen and Xie 2010).

It is clear that an increase in the temperature leads to the weakness of the hydrogen bond of water molecules. As a result, the number of free water molecules in the nanofluid increases. Therefore, the produced free water molecules arrange around MWCNTs' surface as a liquid layer due to the chemical surfaces of MWCNTs and van der Waals force between the water molecules. The thermal conductivity of the produced layer is higher than that of bulk liquid. Therefore, it can be deduced that the chemical functionalized groups of MWCNTs have a significant effect on the enhancement of thermal conductivity of nanofluid.

Figures 5 and 6 illustrate the effect of temperature on the thermal conductivity of nanofluids containing MWCNTs–TiO_2 with 61 and 34% of TiO_2 nanoparticles, respectively. It is clear that in both the nanofluids, there was a significant difference among four temperatures for thermal conductivity of MWCNTs–TiO_2 nanofluid ($\alpha = 0{\cdot}05$) and the thermal conductivity of both nanofluids increased with respect to the temperature. From figures 5 and 6, it can be inferred that the least thermal conductivity was recorded for temperature of 25 °C equal to 0·6342 $Wm^{-1} K^{-1}$ and 0·652 $Wm^{-1} K^{-1}$ for nanofluids containing MWCNTs–TiO_2 with 61 and 34% of TiO_2 nanoparticles, respectively. In addition, maximum thermal conductivity was related to the temperature of 70 °C

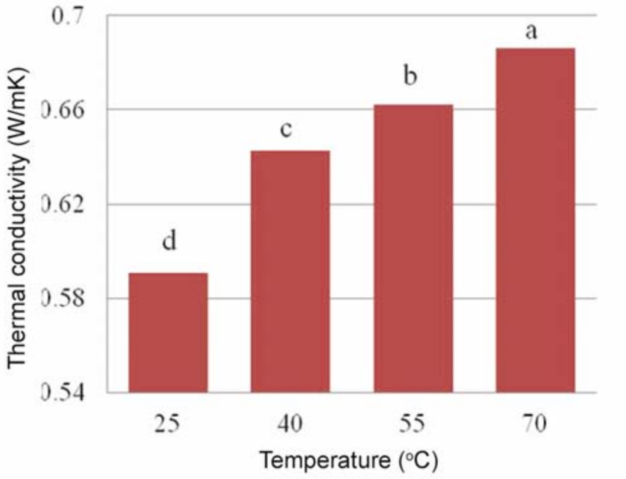

Figure 3. Effect of temperature on thermal conductivity of TiO_2 nanofluid, means with different letters are significantly different based on Duncan's multiple range test ($\alpha = 0{\cdot}05$).

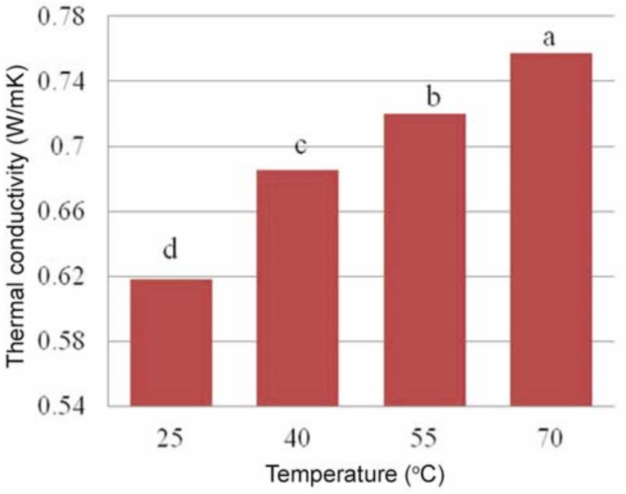

Figure 4. Effect of temperature on thermal conductivity of MWCNT nanofluid, means with different letters are significantly different based on Duncan's multiple range test ($\alpha = 0{\cdot}05$).

equal to 0·7737 $\mathrm{Wm^{-1}\,K^{-1}}$ and 0·7896 $\mathrm{Wm^{-1}\,K^{-1}}$ for nanofluids containing MWCNTs–TiO_2 with 61 and 34% of TiO_2 nanoparticles, respectively.

Meanwhile, it can be deduced that in all the tested temperatures, by increasing the loading of TiO_2 nanoparticles on the surface of MWCNTs, the thermal conductivity of MWCNT–TiO_2 nanofluids also increased. It is due to the decreasing TiO_2 nanoparticles content in the hybrid of MWCNTs–TiO_2, the amount of MWCNTs which have higher intrinsic thermal conductivity increased.

3.3b *Effect of concentration on thermal conductivity of nanofluids*: The results of data variance showed that there was a significant difference among four weight fractions in statistical level of 5% on aspect of thermal conductivity of TiO_2 nanofluids. Maximum thermal conductivity of TiO_2 nanofluids was 0·6512 $\mathrm{Wm^{-1}\,K^{-1}}$ at weight fraction of 1·5 wt% but its difference in comparison with thermal conductivity at weight fraction of 1 wt% (0·6475 $\mathrm{Wm^{-1}\,K^{-1}}$) and 0·5 wt% (0·6437 $\mathrm{Wm^{-1}\,K^{-1}}$) was insignificant. Minimum thermal conductivity was related to the weight fraction of 0·25 wt% (0·6393 $\mathrm{Wm^{-1}\,K^{-1}}$) that it was 1·18% less than that of weight fraction of 1·5 wt% (figure 7).

There was a significant difference among different weight fractions from the viewpoint of thermal conductivity of MWCNT nanofluids. It was observed that by increasing the weight fraction of MWCNT, thermal

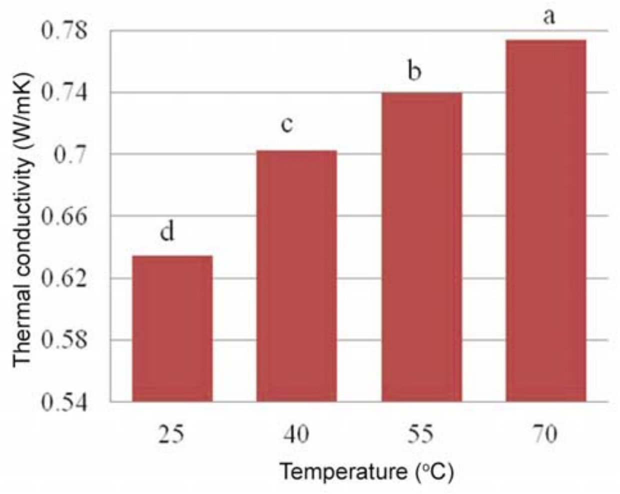

Figure 5. Effect of temperature on thermal conductivity of MWCNT–TiO_2 (61%) nanofluid, means with different letters are significantly different based on Duncan's multiple range test ($\alpha = 0·05$).

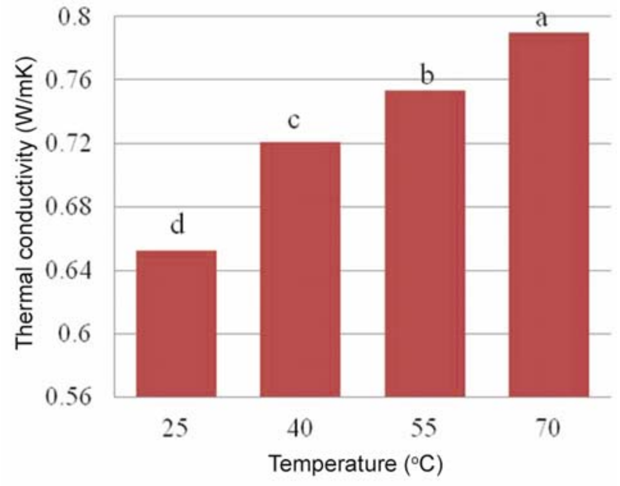

Figure 6. Effect of temperature on thermal conductivity of MWCNT–TiO_2 (34%) nanofluid, means with different letters are significantly different based on Duncan's multiple range test ($\alpha = 0·05$).

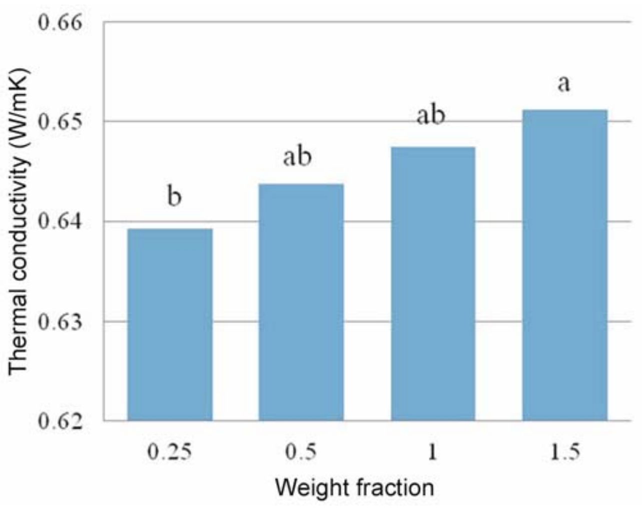

Figure 7. Effect of weight fraction on thermal conductivity of TiO_2 nanofluid, means with different letters are significantly different based on Duncan's multiple range test ($\alpha = 0·05$).

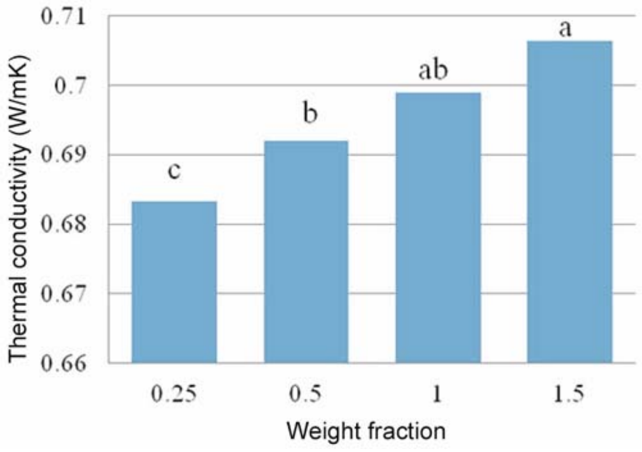

Figure 8. Effect of weight fraction on thermal conductivity of MWCNT nanofluid, means with different letters are significantly different based on Duncan's multiple range test ($\alpha = 0·05$).

conductivity also increased. Maximum thermal conductivity of MWCNT nanofluids was 0·7063 $Wm^{-1} K^{-1}$ at weight fraction of 1·5 wt% but its difference in comparison with thermal conductivity at weight fraction of 1 wt% (0·699 $Wm^{-1} K^{-1}$) was insignificant. Meanwhile, the thermal conductivity of MWCNT nanofluids during the increasing the weight fraction, from 0·5 to 1·5 wt% was increased equal to 3·36% (figure 8). Comparing the augmentation of the thermal conductivity of pristine MWCNTs nanofluid with temperature and concentration revealed that the increasing of thermal conductivity of nanofluid with temperature (22·5%) is higher than that of with concentration (3·36%), which is consistent with the results obtained by Ding *et al* (2006).

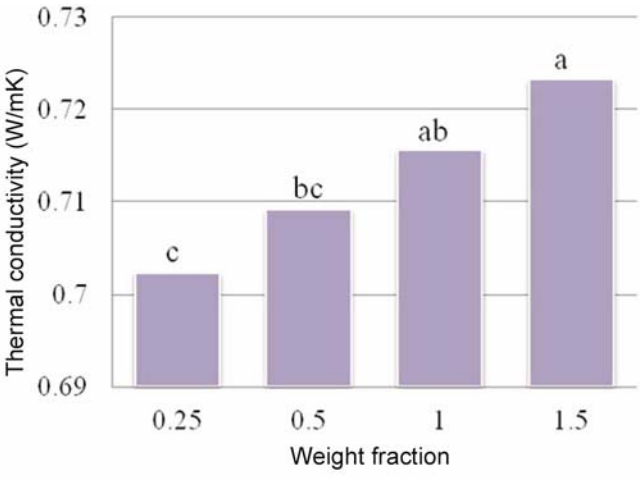

Figure 9. Effect of weight fraction on thermal conductivity of MWCNT–TiO_2 (61%) nanofluid, means with different letters are significantly different based on Duncan's multiple range test ($\alpha = 0·05$).

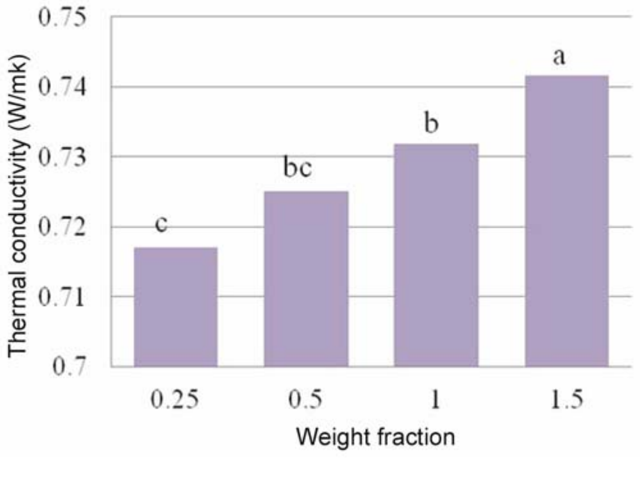

Figure 10. Effect of weight fraction on thermal conductivity of MWCNT–TiO_2 (34%) nanofluid, means with different letters are significantly different based on Duncan's multiple range test ($\alpha = 0·05$).

The influence of weight fraction on the thermal conductivity of nanofluids containing MWCNT–TiO_2 is shown in figures 9 and 10 for MWCNT–TiO_2 with 61 and 34% TiO_2 nanoparticles, respectively. It can be inferred that the minimum and maximum of thermal conductivity of both MWCNT–TiO_2 nanofluids were related to weight fraction of 0·25 and 1·5 wt%, respectively. According to the Duncan's multiple range test ($\alpha = 0·05$), there was a significant difference among the highest and lowest weight fractions for thermal conductivity of both MWCNT–TiO_2 nanofluids, but difference between the weight fraction of 0·5 and 1 wt% was insignificant. In addition, increasing the weight fraction from 0·25 to 1·5 wt% led to the augmentation of thermal conductivity equals to 2·975 and 3·416% for MWCNT–TiO_2 nanofluids with 61 and 34% TiO_2 nanoparticles, respectively. Therefore, it can be deduced that increasing the thermal conductivity of MWCNT–TiO_2 nanofluids with 34% TiO_2 nanoparticles is higher than that of MWCNT–TiO_2 nanofluids with 61% TiO_2 nanoparticles. It is due to the

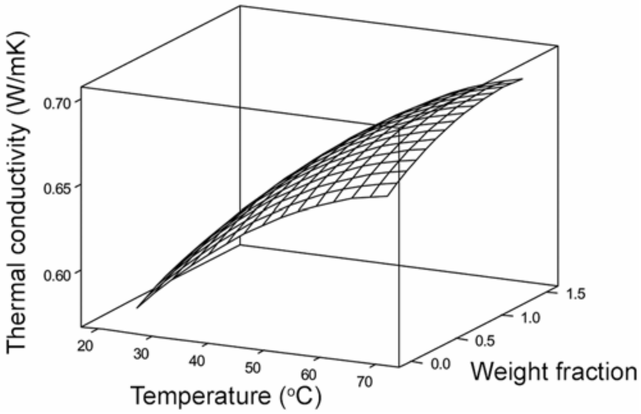

Figure 11. Response surface of thermal conductivity of TiO_2 nanofluid vs temperature and weight fraction.

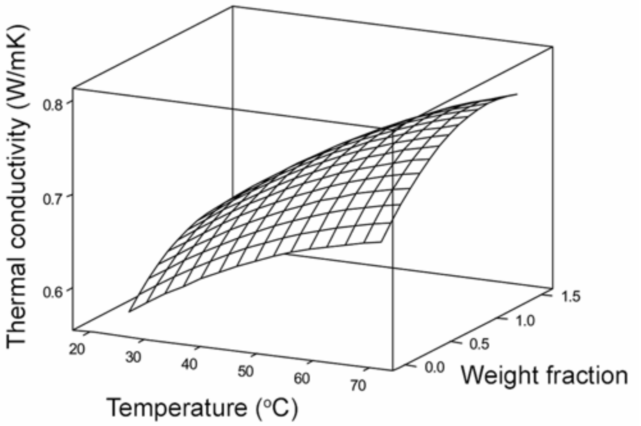

Figure 12. Response surface of thermal conductivity of MWCNT nanofluid vs temperature and weight fraction.

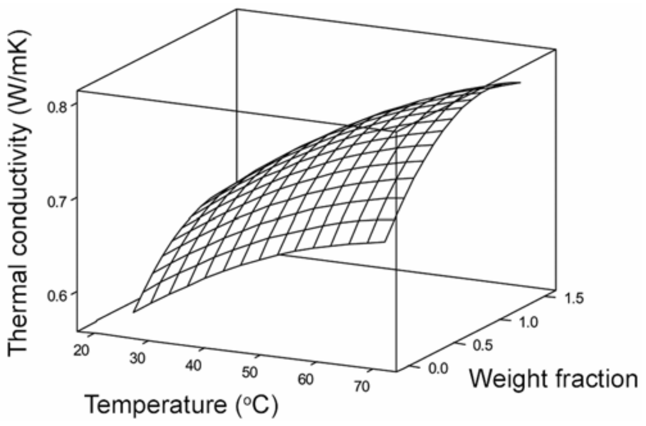

Figure 13. Response surface of thermal conductivity of MWCNT–TiO_2 (61%) nanofluid vs temperature and weight fraction.

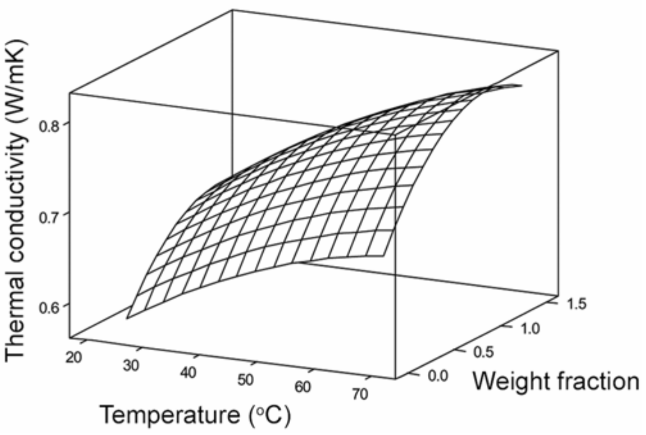

Figure 14. Response surface of thermal conductivity of MWCNT–TiO_2 (34%) nanofluid vs temperature and weight fraction.

increasing portion of MWCNT with higher intrinsic thermal conductivity in the hybrid of MWCNT–TiO_2.

3.3c *Response surface study*: In this study, the response surface fitting method was used in order to study the combined effect of temperature and weight fraction on the thermal conductivity of nanofluid. Figure 11 shows the surface response of thermal conductivity of TiO_2 nanofluid, which implies that the thermal conductivity of TiO_2 nanofluid increases with increasing temperature and weight fraction, although the weight fraction variations of the thermal conductivity is less than that of temperature.

With respect to figure 12, which shows the surface response of thermal conductivity of MWCNT nanofluid with temperature and weight fraction, thermal conductivity increases with increasing temperature and weight fraction.

Figures 13 and 14 show the surface response of the thermal conductivity of MWCNT–TiO_2 nanofluids with 61 and 34% TiO_2 nanoparticles, respectively. In both the cases, it is observed that thermal conductivity value increases with temperature and weight fraction. However, the influence of temperature is more considerable than that of weight fraction.

4. Conclusions

The present study investigated the thermal conductivity nanotubes containing TiO_2 nanoparticle, pristine and decorated multi-walled carbon with TiO_2 nanoparticles nanotubes. Statistical analysis of the experimental data shows that the temperature and weight fractions have a significant effect on the thermal conductivity of nanofluids. However, the influence of temperature on the thermal conductivity of nanofluids is more reasonable than that of weight fraction. Meanwhile, the results revealed that in all the tested temperatures, by increasing the loading of TiO_2 nanoparticles on the surface of MWCNTs, the thermal conductivity of MWCNT–TiO_2 nanofluids also increased.

Acknowledgements

The authors gratefully acknowledge the help by Mrs R Pesyan from Central Research Laboratory of Ferdowsi University of Mashhad and Mrs T Saeedi from Par-e-Tavous Research Institute.

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