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Preparation and Investigation of Thermal Properties of Titanium Dioxide (TiO2) Nanostructures

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ABSTRACT

Nano titanium oxide (TiO_2) powders were successfully prepared by sol-gel process by employing ethylene glycol which was heat-treated in conventional oven to obtain distinct crystalline structures. The as-synthesized titanium oxide nanoparticles were also characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscope (TEM), UV-Visible spectroscopy, band-gap analysis, and Fourier Transformed Infrared Spectroscopy (FTIR). This research was conducted to formulate stable nanofluids containing titanium dioxide (TiO_2) nanoparticles in ethylene glycol (EG) as a base fluid. The thermal conductivity of nanofluids was measured. Two-step method was employed for nanofluids preparation. The thermal conductivity was enhanced by increasing the concentration of nanoparticles and temperature due to effect of Brownian motion. The research showed that TiO_2 in EG nanofluids has the potential to be employed as a heat transfer fluids compared to the conventional fluids.

Keywords: Titanium dioxide(TiO_2), Sol-gel method, X-ray Diffraction, Scanning Electron Microscope, Transmission Electron Microscopy, UV-Visible, Thermal Properties

1. INTRODUCTION

Titanium dioxide (TiO_2) is a very useful semiconducting transition metal oxide material and exhibits unique characteristics such as low-cost, easy handling, non-toxicity and resistance to photochemical and chemical erosion [1-3]. These advantages make TiO_2 a material in solar cells, chemical sensors, for hydrogen gas evolution, as pigments, as additives in nanofluid, self-cleaning surfaces and environmental purification applications [4-7]. Xu and his team has reported the oxide nanoparticles synthesized by several methods appear more and more useful, because these nanoparticles have good electrical, optical and magnetic properties that are different from their bulk counterparts [8].Titanium dioxide exists in both crystalline and amorphous forms and mainly exists in three crystalline polymorphous, namely, anatase, rutile and brookite. Anatase and rutile have a tetragonal structure, where as brookite has an orthorhombic structure. Mahshid demonstrated the immobilization of TiO_2 nanoparticles on an appropriate support has been widely accepted since it could help to eliminate the costly phase separation processes and to promote the practicality of such catalysts as an industrial process [9].

Several methods of TiO_2 preparation have been reported in literature based on the hydrolysis of acidic solutions of Ti (IV) salts. Also, oxidations of $TiCl_4$ on gaseous phase [10-12] and hydrolysis of titanium alkoxides [13, 14] have been used to generate finely divided with a high purity TiO_2 powders. The sol-gel method is an attractive method for the synthesis of TiO_2. Since this method is carried out in solution, tailoring of certain desired structural characteristics such as compositional homogeneity, grain size, particle morphology and porosity is possible. A uniform distribution of the particles is important for optimal control of grain size and micro structure to maintain high reliability. In the present work, we have prepared high efficient TiO_2 nanoparticles using sol-gel process having a large surface area and to characterize the prepared sample using XRD, SEM, TEM, and UV-Visible spectroscopy. This study was conducted to meet the following objectives: (i) to formulate nanofluids that contain titanium dioxide nanoparticles (TiO_2) in ethylene glycol (EG) as a base fluid, (ii) to measure thermal conductivity of nanofluids at different temperature and concentration of nanoparticles.

2. EXPERIMENTAL PROCEDURES

A. Materials and Methods

Analytical-grade reagents of Titanium tetraisopropoxide($Ti\{OCH(CH_3)_2\}_4$), hydrochloric acid (HCl), Ethylene glycol (EG), ethanol and deionized water were used in all experiments. All the chemicals were used as received without further purification.

B. Synthesis of TiO2 Nanoparticles

The Titanium tetraisopropoxide($Ti\{OCH(CH_3)_2\}_4$) was used as a precursor and was mixed with HCl, ethanol and deionized water mixture, stirred for half an hour, in pH range of 1.5. 10 ml of deionized water was added to the above mixture and stirred for 2 hours at room temperature. Finally the solution was dried at temperature and the powder was heated at 120ºC for 1 hour and white crystalline TiO_2 nanopowder obtained.

C. Preparation of Nanofluid using TiO_2 Nanostructures

Two-step method was used for this study since it is a simple and less costly compared to the single-step method. The as-prepared TiO_2 nanoparticle was dispersed in EG. The nanofluids samples were prepared and consist of different concentration of nanoparticles which are 0.25 wt%, 0.50 wt%, 0.75 wt % and 1.00 wt %. All samples were dispersed in 50 mL EG and were sonicated in ultrasonic homogeniser for 2 h.

D. Thermal conductivity measurement

In this study, KD2 Pro Thermal Properties Analyzer (Decagon Devices, Inc.) was used to measure the thermal conductivity of nanofluids. The samples of nanofluids were analysed at different temperature of 30°C, 40°C, 50°C, and 60°C. Hot water bath and heating plate was used in order to increase the temperature of nanofluids.

3. RESULTS AND DISCUSSION

A. Structural analysis

X-Ray Diffraction measurements were carried out by powder X-Ray Diffraction (PXRD Bruker D8 Advance) to investigate and quantify the purity and crystalline nature of materials using Cu-Kα wavelength (λ =1.54059 Å) as source, where the voltage and current were held at 40kV and 30mA respectively.

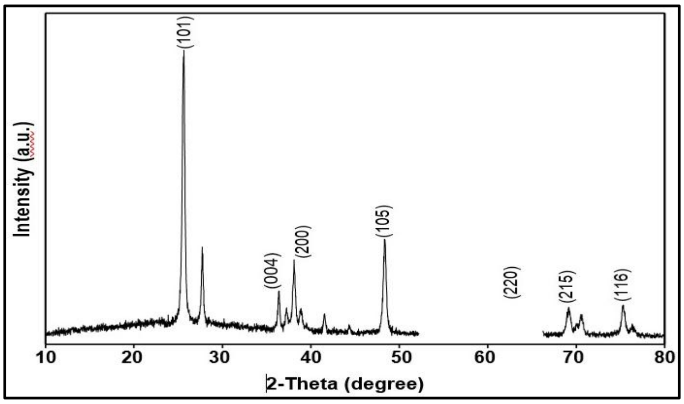

Figure 1: X-Ray Diffraction graph of titanium dioxide nanoparticles.

The peaks appeared at 2θ value ranging The diffraction peak at 2θ with 27.76°, 36.5°, 41.68°, 54.58°, 57.0°, 69.22° corresponds to the crystal planes of (110), (101), (111), (211), (220) and (301) respectively, indicating the formation of anatase phase of TiO_2 .The XRD pattern of TiO2 is perfectly equivalent with JCPDS file no. (84-1286), which is identify as tetragonal structure (a=b=3.7822Å, c=9.5023Å). The peaks were found appreciably sharper with very high intensity, maximum peak intensity located at 25.48 (2θ) that belongs to (1 0 1) hkl planes. The average crystalline size of synthesized anatase phase was found to be 20.54 nm by Debye-Scherrer's formula.

Table 1: Structural characteristics of titanium dioxide nanoparticles.

2θ (degree)	Intensity (a.u.)	h k l	FWHM (β)	d (Å)	Crystallite Size D (nm)
27.76	6140	1 1 0	0.2312	3.211	35.27
36.50	3336.69	1 0 1	0.2393	2.459	34.84
41.68	1822.21	1 1 1	0.2409	2.164	35.17
54.58	4056.62	2 1 1	0.2698	1.679	33.03
57.00	1413.96	2 2 0	0.2994	1.634	30.09
69.22	1473.22	3 0 1	0.3427	1.355	28.07

B. Morphological and Topographical Analysis

The morphology of the samples was observed with a scanning electron microscope (Zeiss EVO MA-10 SEM) at an accelerating voltage of 10.0 kV. The SEM analysis was used to determine the structure of the reaction products that were formed. Thin films of the sample were prepared on a carbon coated copper grid by just dropping a very small amount of the sample on the grid, extra solution was removed using a blotting paper and then the film on the SEM grid were allowed to dry by putting it under a mercury lamp for 5 min. SEM image has roughly spherical spongy shape and agglomeration nanoparticles given in Figure 2.

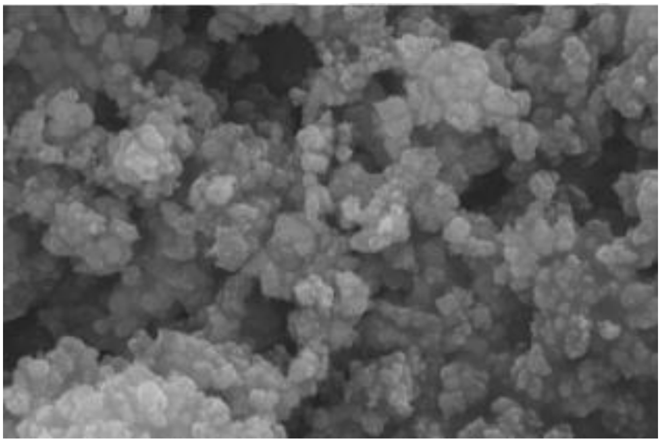

Figure 2: SEM image of titanium dioxide nanoparticles

HRTEM analysis was gives structural information on atomic size level is known as lattice imaging and has become very important for interface analysis. It has further capacities of exploring deeper information like the crystalline orientation, lattice spacing, structural defects present in the materials. It has also the potentials to predict that if a certain nanostructure is in solid form or has hollowness inside them. Figure 3 shows the lattice fringe image of the synthesized sample, which is highly crystalline, with regular spacing. The d-spacing of the sample is 0.24 nm with the corresponding plane of orientation is (004) and which is in accordance with the XRD data where the peak with the intensity at $2\theta = 37.90$ have this plane. Some amorphous region is present in the material, which is clearly visible in the specified area and is shown in figure 3. Frequently extended areas containing numerous small overlapping grains are observed leading to the different periodicity shown in figure 3. These may occur due to the rotation of the two crystals with respect to each other at the interface.

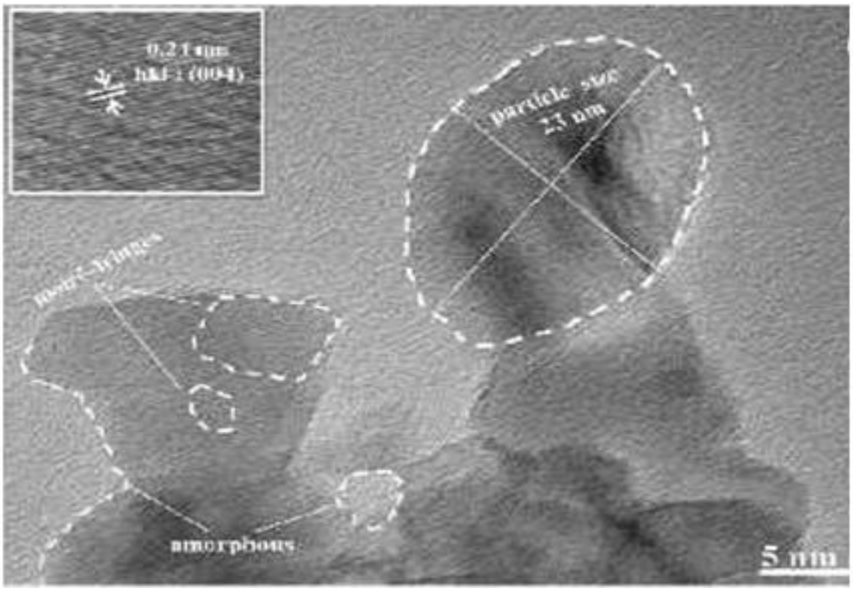

Figure 3: TEM image of titanium dioxide nanoparticles

C. UV-Visible analysis

The UV-Visible spectra of all the samples were investigated by UV-Visible spectrometer (UV-2401 PC, Shimadzu Corporation Japan). UV-Vis absorption spectroscopy is one of the most important methods to analysis the energy structures and optical properties of the metal oxide nanostructures. Figure 4 shows the UV-Vis absorption spectra of the titanium dioxide nanostructures. And it depicts the absorbance of TiO_2 nanoparticles at 250 nm.

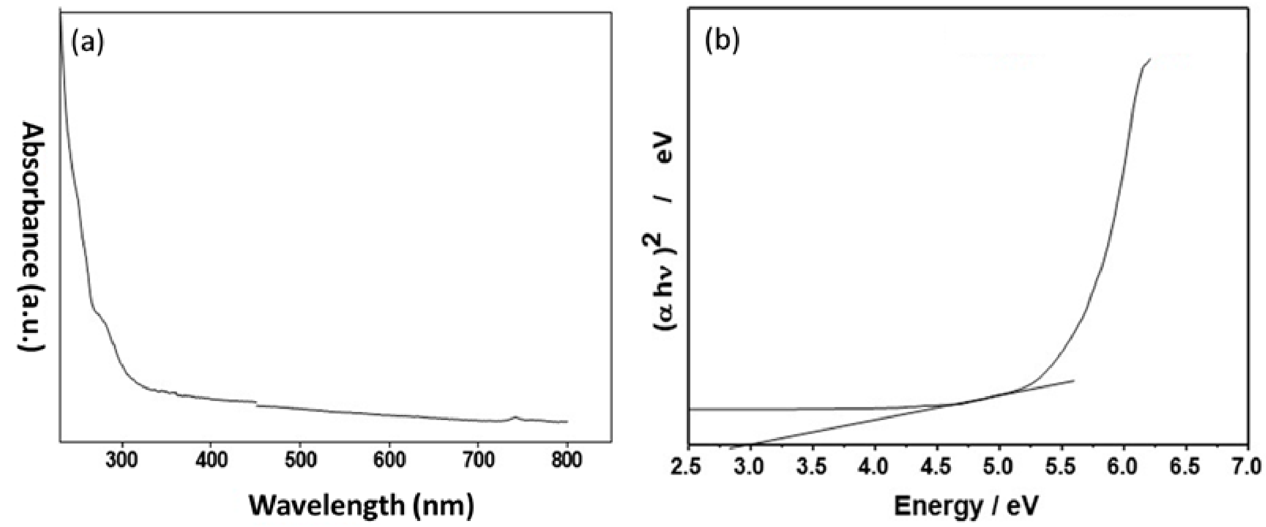

Figure 4: (a) UV-Visible spectra of titanium dioxide nanoparticles, (b) Tauc plot analysis of titanium dioxide nanoparticles.

The Tauc plot analysis is used for the calculation of band gap. The band-gap of as-prepared TiO_2 nanoparticles is about 3.15 eV at 250 nm absorption.

D. Fourier Transformed Infrared Spectroscopy (FTIR)

In figure 5, many absorption bands of the organic groups and alkane group were appeared. In TiO_2 as prepared sample, the peak 948 cm-1 was indicated to aromatic (C-H) deformations.

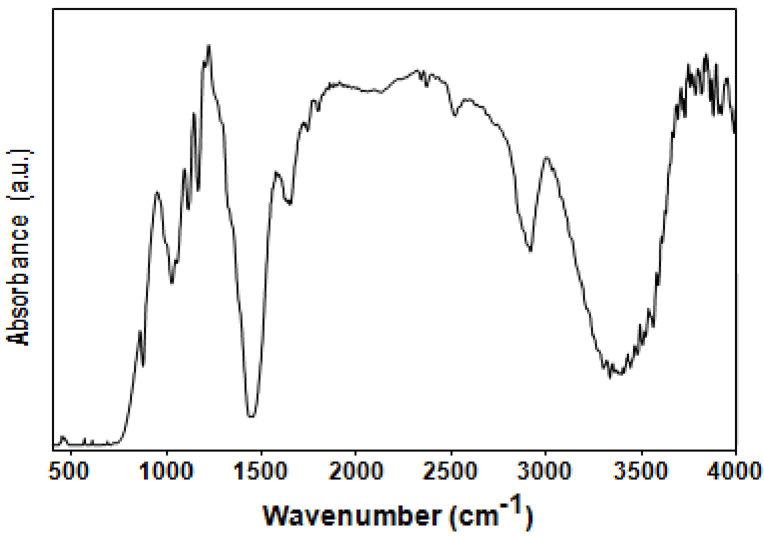

Figure 5: FTIR spectrum of as-prepared TiO_2 nanoparticles.

The other peak at 1212 cm-1 was indicated to stretching of (C-O) bond. The peak 1578 cm-1 were indicated to stretching of (C=C) bond. The peak 2310 cm-1 were indicated a nitrile stretching mode and peak 3008 cm-1 were indicated to stretching mode of (=C-H) bond.

E. Conductivity Analysis

The improvement in thermal conductivity becomes significant parameter for nanofluids when used as effective heat transfer fluids. The concentration of nanoparticles is one of the factors that can affect the thermal conductivity of nanofluids. Figure 6 shows the relationship between thermal conductivity and concentration of nanoparticle at room temperature. From Figure 6, it can be seen that the thermal conductivity was improved by increasing the concentration of Nanostructures TiO_2. The reason is for usual solid-liquid mixture, the thermal conductivity increases due to higher thermal conductivity of solid particles. The nanofluids thermal conductivity is the average thermal conductivity of the solid and liquid [15]. When the nanoparticle concentration increases, it means more solid particles were present and will lead to more Brownian motion and increases the thermal conductivity.

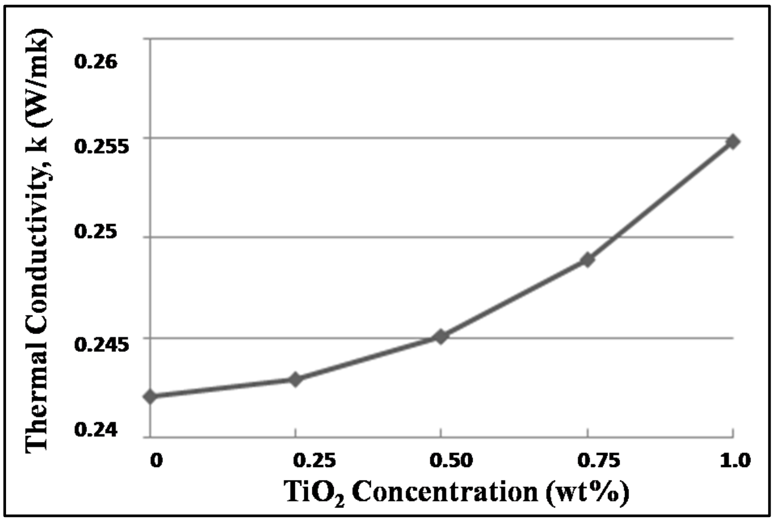

Figure 6: Thermal conductivity of nanofluids as a function of TiO2 concentration at room temperature.

By increasing the concentration of nanoparticles, there is greater possibility of collision between particles and that makes energy transfer becoming quicker. This condition also can be related to the clustering of nanoparticles in the base fluids. As heat transport in solid-fluid suspension occurs at the particle-liquid interface, interfacial area increases can lead to more efficient heat transport properties [16]. This clustering will give a major effect on increasing the thermal conductivity. Finally, thermal conductivity seems to be significantly changed by changing the temperature of nanofluids. The effect of temperature on the enhancement of effective thermal conductivity of nanofluids was investigated by measuring the thermal conductivity of nanofluids for different temperatures ranging from 30°C to 60°C.

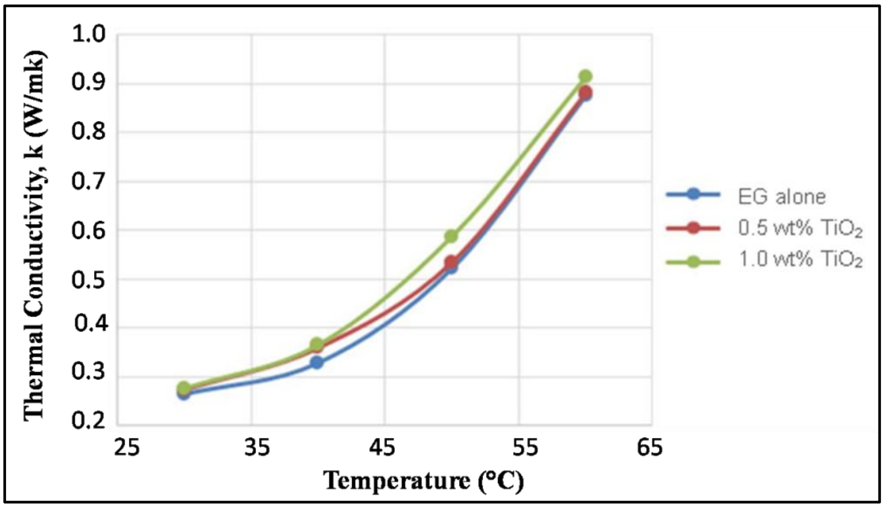

Figure 7: Thermal conductivity of nanofluids as a function of temperature and TiO2 concentrations.

Figure 7 depicts that the relationship between the temperature and effective thermal conductivity of nanofluids. The thermal conductivity against temperature profile at different concentration are much closed to each other, thus only two results are shown here. The result shows that for 1 wt% TiO_2, the thermal conductivity was enhanced by 31 %, 112 %, and 230 % for temperature 40°C, 50°C and 60°C. Temperature increase leads to the reduction of the particle surface energy and decreases the agglomeration of nanoparticles. The smaller size leads to the more intensive Brownian motion, then increase the thermal conductivity.

CONCLUSION

Titanium dioxide (TiO_2) nanoparticles have been successfully synthesized using a sol-gel technique. The formation of the TiO_2 nanoparticles was confirmed by powder X-ray diffraction (XRD). The size, morphology, and topography of the samples were characterized using SEM and TEM. The nearly spherical shaped particles and 20.4 nm particle sizes were confirmed through the SEM analysis. The band-gap of TiO_2 nanoparticles is about 3.15 eV at 250 nm absorbance. The formulation of nanofluids containing TiO_2 nanoparticles in EG base fluids was performed. The thermal conductivity was enhanced by varying the amount of TiO_2 concentration, and temperature of the nanofluids.

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