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Short Communication

Preparation and characterization of TiO_2 Fe_2O_3 nanocomposite by sol gel method

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ABSTRACT

Nanocomposites have improved aspect ratio and better mechanical properties when compared to the composites which had structures less than 100nm They are 1000 times tougher compared to conventional composites. Besides mechanical properties, nanocomposites also have improved electrical conductivity, thermal stability, chemically resistant, flame retardant and low permeability. TiO_2 finds its best application in the field of photo catalytic activity and it is used in sunscreen lotions since it is resistant to UVA(315-400nm) and UVB(280-315nm). Fe_2O_3 is used as the pigment in construction of roof tiles, pavers, plaster etc. It is used as a polishing agent for glass, diamonds and it is dental abrasive. It is reported that a nanocomposites of TiO_2- Fe_2O_3 have improved visible water light splitting, and photocatalytic degradation of pollutants.The acute toxicity of the oxide nanoparticles make them suitable for their applications.The objective of the present work is to synthesize composites of titanium dioxide -iron oxide by a simple sol gel route in laboratory scale and to validate them as nanoparticles for preparation of nanofluids.

KEYWORDS: Sol gel, TiO_2, Fe_2O_3,Nanocomposite

1. INTRODUCTION

The study of nanocomposites started about 1950 when they were first referenced .For the past 70 years there has been considerable research in nanomposites through the change in matrix or fiber phase and other geometrical variations. The unique properties of nanomaterial have further attracted scientists and engineers to manufacture nanocomposites. Nanocomposites show promising potential applications in battery cathode material,non linear optics,ionics,nanowires and sensors[1]. Among the several nanocomposites that are tailored,oxide nanocomposites have been mostly investigated because of easy preparation, less cost with minimum toxicity. αFe_2O_3 TiO_2 nanocomposites have been synthesized by two step hydrothermal method which require titanium sheets [2]. Fe doped TiO_2 nanoparticls have been prepared by hydrothermal method with $TiCl_4$ and $FeCl_3$ as precursor solution. Interesting results of pure TiO_2 with 10-15nm and Fe doped TiO_2 with 50-100nm was reported [3].In the present study,an attempt was made to synthesize Fe doped TiO_2 nanocomposite of different morphology and size using Titanium Tetra Iso propoxide and $FeCl_3$ as precursors. But large nanocomposite particles of 100-150nm of TiO_2-Fe_2O_3 was obtained.

2. EXPERIMENTAL

Titanium Tetra Iso Propoxide (TTIP) 4.2gm is mixed with 5ml of distilled water and mixed in a magnetic stirrer for about 2 hrs. 5 ml of Polyethylene glycol is added to the mixture. The color turned milky white after 2 hrs of stirring.. 2 ml of $FeCl_3.6H_2O$ was added in the solution to give a yellow colour The colour changed to dark yellow by adding 4 ml of Dilute Hydrochloric acid. 1 pellet of Sodium hydroxide (NAOH) was further added to maintain the pH value .Stirring was continued for one hr.This was followed by drying in a hot plate. A colour change to orange is an indication of the final phase transition. The solution was left isolated for 96 hrs. The excess solvent was filtered to prepare the wet powders for drying. A primary heat treatment was done in the muffle furnace for one hour till the temperature reached 200C. The powders were dried at room temperature and hand milled for half an hour. Finally the powders were sintered at 600C for two hours to yield the material ready for characterization.

The prepared powders were characterized by XRD to identify the phase and approximate particle size.SEM studies were performed to investigate morphological aspects.EDS studies were performed to investigate the elements present.

3.RESULTS AND DISCUSSION:

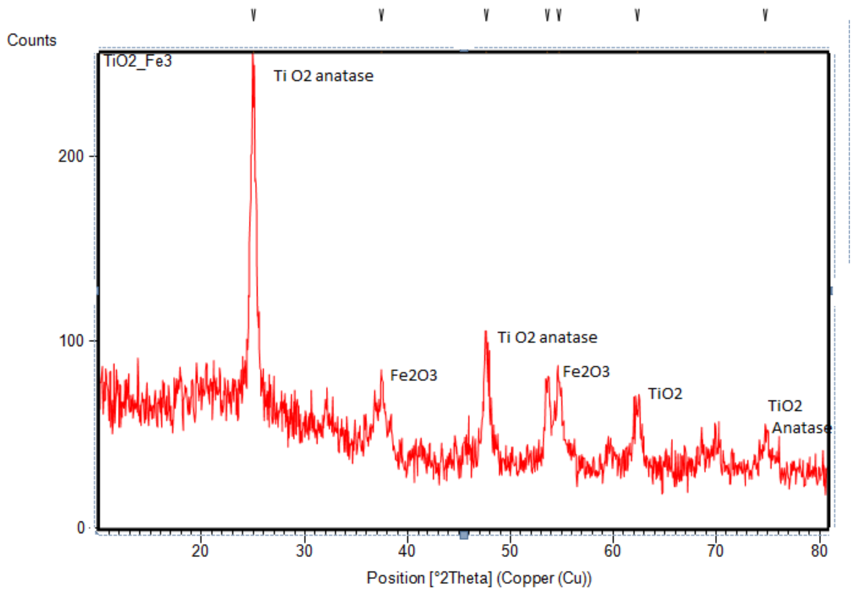

Fig1.XRD of TiO_2 Fe_2O_3 nanocomposite

Powder X Ray diffraction studies were performed with XPERT-PRO diffraction system using a characteristic Kα radiation of 1.54A in continious slow scanning mode starting from 10 to 20.The XRD pattern of the prepared nanocomposite is shown in Fig.1. The diffraction peaks present at 25,48 and 74 indicate the presence of anatase phaseTiO_2.The peaks located at 38 53 and 55 indicate the presence of Fe_2O_3.The peak present at 53,55 indicate the formation of maghemite phase (α- Fe_2O_3)[4].In comparison to the XRD pattern of Fe_2O_3, the composite shows lattice strain as indicated by the peak at 53 and 55.The diffraction pattern also reveals the partial crystalline phase of TiO_2 and higher amorphous phase of Fe_2O_3. The above effect could be due to the sintering of the composite at 600C which is close to the crysallisation temperature of anatase phase of TiO_2The average grain size calculated using Debye Scherrer equation is 143 nm.The dislocation density calculated using Williamson –Smallman equation if 4.89 $x10^{13}$ $/m^2$. The obtained density is lower than that for nanometals which is typically $10^{15}/m^2$.

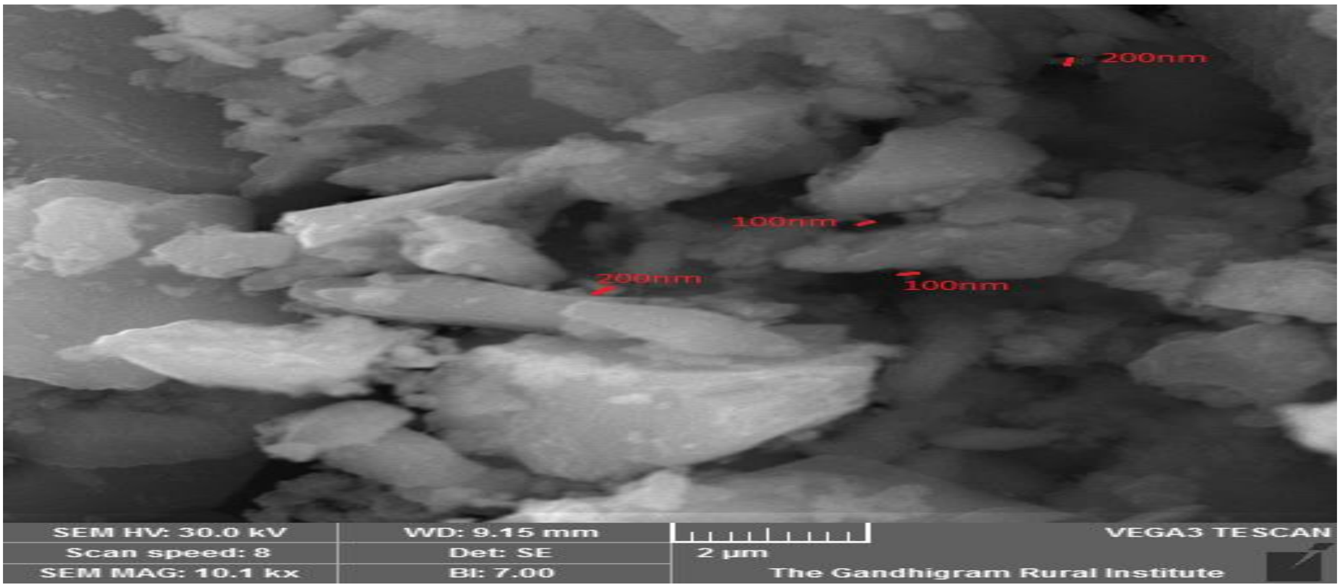

Fig2.SEM of TiO_2 Fe_2O_3 nanocomposite

SEM analysis was performed at a voltage of 30.KV . The SEM image of the prepared nanocomposite is shown in Fig.1.The results idicate the presence of micro and nano size particles with an average size of 150nm.Irregular large micro size flakes with nanorods of the composite are seen.

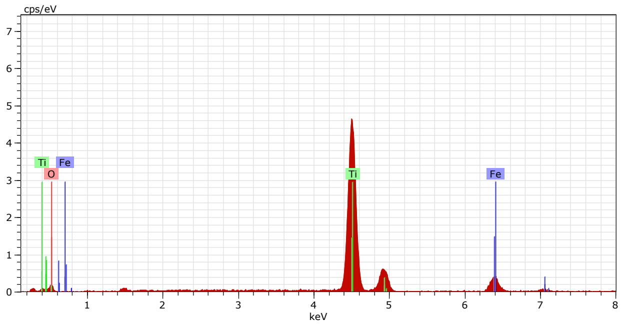

Fig3.EDX image of TiO_2 Fe_2O_3 nanocomposite

The EDX image (Fig.3) reveals the presence of Oxygen ,Titanium and iron indicating the purity of the prepared nanocomposite.Fe is present in lesser atomic and weight percent as indicated by Table 1.

Table 1. EDX analysis of TiO_2 Fe_2O_3 nanocomposite

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	25.04	24.63	49.94	8.83
Ti	22	K-series	66.09	65.00	44.04	1.93
Fe	26	K-series	10.53	10.36	6.02	0.42
		Total:	101.66	100.00	100.00	

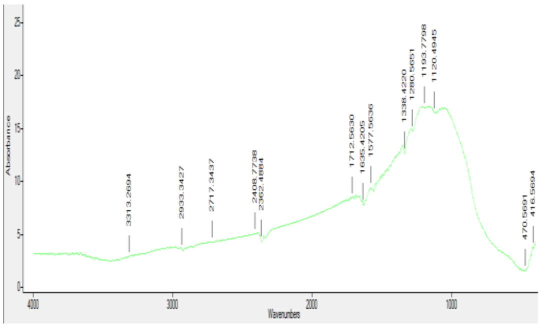

Fig4. FTIR absorbance of TiO_2 Fe_2O_3 nanocomposite

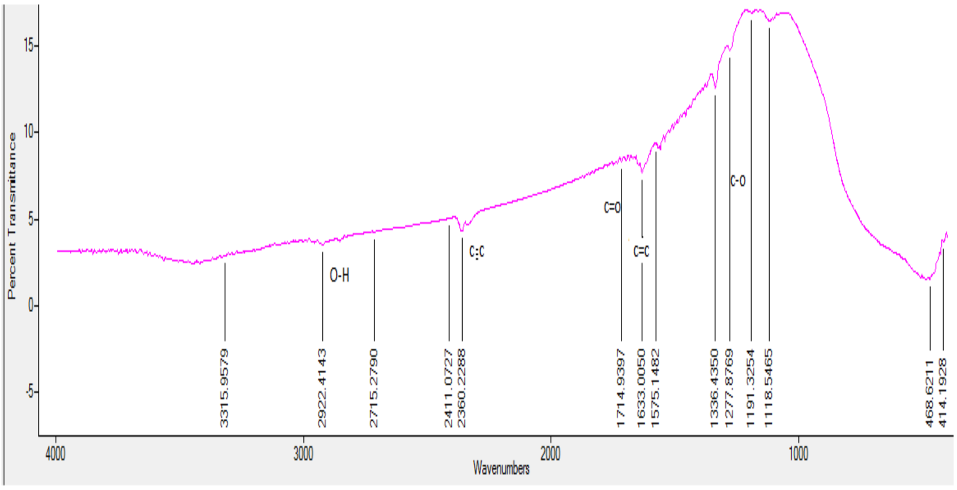

Fig5. FTIR transmittance of TiO_2 Fe_2O_3 nanocomposite

The FTIR analysis was performed for a range of wavelength from 400 cm^{-1} to 4000 cm^{-1} in absorption and transmission mode.The FTIR Figure 5 shows the FTIR transmittance spectrum of the prepared nanopowders . The band around 1633 cm^{-1} relates to bending modes of water TiOH . The band at 1336 cm^{-1} is related to the Ti-O modes.The broad band around 2900 cm^{-1} shows the O-H bond. The bands observed at 468 cm^{-1} is related to the magnetite phase of iron oxide.

The prepared TiO_2 Fe_2O_3 was dispersed in water by ultrasonication method at a frequency of 42 KHz for 10 mins.The naoparticle concentration was 0.1 wt% in water. The zeta potential was measured using

Malvern Panalytical zetasizer instrument with water as a dispersant at a temperature of 25C.The count rate was 72.5kcps for 10 zeta runs.The zeta potential is about -34.7 mV .This indicates good stability of the nanofluid.The conductivity of the nanofluid is 0.0531 mS/cm and the zeta deviation is around 4.30 mV

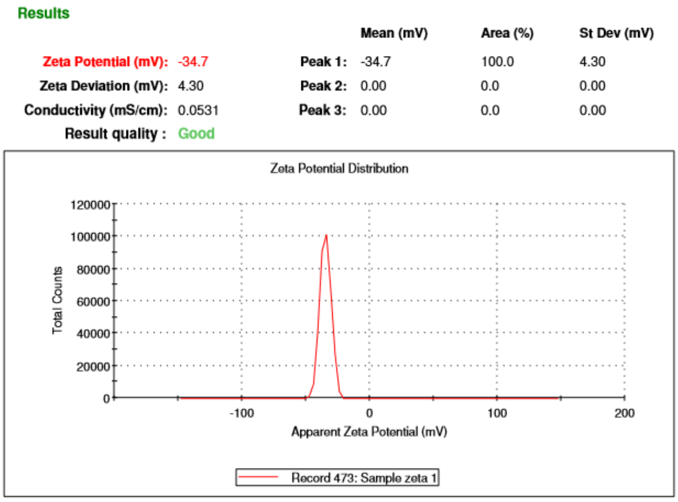

Fig6. DLS of TiO_2 Fe_2O_3 nanocomposite nanofluid

3. Conclusions

Nanocomposites of TiO_2 Fe_2O_3 have been prepared by sol gel route and characterized. Average grain size obtained is 143nm and SEM tests reveal the morphology as flakes.EDAX confirms the presence of Fe,Ti and O.FTIR reveals the presence of Fe-O and Ti-O bonds. The prepared nanocomposites also exhibit the property of stable nanofluids with base fluid as water at a weight fraction of 0.1%.We suggest TiO_2 Fe_2O_3 nanocomposite nanofluid as a better medium for heat transfer with high stability .

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