Sonochemical Synthesis of TiO_2 and TiO_2-SiO_2 Nanocomposites and their Photocatalytic Properties

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Abstract - *TiO_2 and TiO_2- SiO_2 nanocomposites were synthesized using simple sonochemical technique and their photocatalytic properties were investigated. The nanomaterials were further characterized by XRD and FESEM studies. Measurement of photocatalytic degradation of Rhodamine B dye was carried out under visible light and the photocatalytic efficiency of the ssynthesized nanocomposites was calculated. XRD studies of TiO_2 suggest the formation of a mixed structure of TiO_2 Anatase and Rutile while that of TiO_2- SiO_2 nanocomposite shows predominant orientation along TiO_2 anatase along with some low intensity peaks corresponding to planes of TiO2 rutile and SiO_2 .FESEM measurements indicate the formation of a rod like structure along with some spherical particles with a cloudy appearance.*

Key Words: **Photocatalysis, Nanocomposites, Sonochemical, Sol-Gel, Photocatalytic Degradation.**

1. INTRODUCTION

This document is template. Nanocrystalline Titania (TiO2) has been an area of intensive investigation due to its large area of applications like photocatalysis, photovoltaic cells and gas sensors. It is also chemically stable, cheasp, abundant, and a multi-functional material. TiO2 also shows properties like biocompatibility and strong oxidizing power. Particle size has a great impact on its structure and properties [1,2.]

TiO2 is a widely used nanomaterial as a photocatalyst when irradiated with UV light wherein under UV light, the organic contaminants are adsorbed on the surface of the material. This property of TiO_2 can be utilized in various fields like air and water purification system[2]. The photocatalytic process is basically initiated by the generation of electron-hole pairs by absorption of UV light with energy which may be higher than or equal to the band gap of TiO_2 (3.2eV). The photocatalytic performance of TiO_2 can be enhanced by forming composites using support materials like SiO_2.[4] Shifu and Gengyu[5] have reported that addition of *TiO_2* to *SiO_2* improves the photocatalytic performance of TiO_2 as it increases the adsorbability of the photocatalsyts. This redsuces the effect of temperature at which TiO_2 anatase changes to rutile and prevents agglomeration of TiO_2 particles thereby increasing its surface area and thus helping in photocatalytic drgradation.[6] Several synthesizing technique have been suggested by different researchers including sol-gel [9-14], sonochemical [8], hydrothermal[15] and electrochemical techniques. Zhu et.al [7] used ultrasonic irradiation for preparing TiO_2 nano tubes and whiskers. They showed that direct sonication of TiO_2 and NaOH at high power forms Titania whiskers. However not much work has been reported in the synthesis of TiO_2-SiO_2 nanocomposites by sonochemical technique. The present paper reports the synthesis of TiO_2 and TiO_2-SiO_2 nanocomposite by simple Sonochemical technique. The photocatalytic properties of the synthesized nanocomposites were studied and compared with that of TiO_2. XRD and FESEM studies were carried out to study the structural and morphological properties of TiO_2 and TiO_2-SiO_2.

2. EXPERMENTAL TECHNIQUES

2.1 Sample Preparation: TiO_2 and TiO_2-SiO_2 Synthesis using Sonochemical Method-

10M NaOH solution was mixed with 0.5gm TiO_2 and the obtained mixture was then allowed to undergo vigorous stirring for 2hrs and then irradiated in an ultrasonic bath (5.3 kHz / 80W) for 2hrs. The resulting precipitate was then centrifuged and washed with de-ionized water several times and dried at 700C for 6 hrs. TiO_2-SiO_2 Nanocomposites were also synthesized using sonochemical technique in the same manner as that of TiO_2 wherein SiO_2 prepared by sol gel method [9] was added just before the vigorous stirring of 2hrs.

2.2 MEASURING INSTRUMENTS:

The absorption spectral studies were carried out using ELICO SL: 210 UV:VIS Spectrophotometer. XRD studies were done using PANanalytical 3KW Powder X Ray Diffractometer at NIT Raipur. FESEM and EDS studies were done at VNIT Nagpur using JSM 7610F Scanning Electron Microscope.

2.3. PHOTOCATALYTIC ARRANGEMENT:

Photocatalytic degradation of Rhodamine B solution was carried out in visible light. For this 0.003% dye solution was taken to which an appropriate quantity of TiO_2 / TiO_2-SiO_2 was added and the mixture is magnetically stirred for 30 minutes to obtain the absorption-desorption equilibrium. The absorbance of the solution was measured before exposing it to visible light and subsequently after every 30 minutes for 2hrs. The degradation the dye and change in its absorbance was studied with respect to time.

3. RESULTS AND DISCUSSIONS:

3.1 Absorbance spectra and photocatalytic studies:

Fig 1 Shows the absorbance spectra of TiO2 and TiO2-SiO2 nanocomposite. Increased absorption in the visible region from 400-700nm is observed in both the cases.

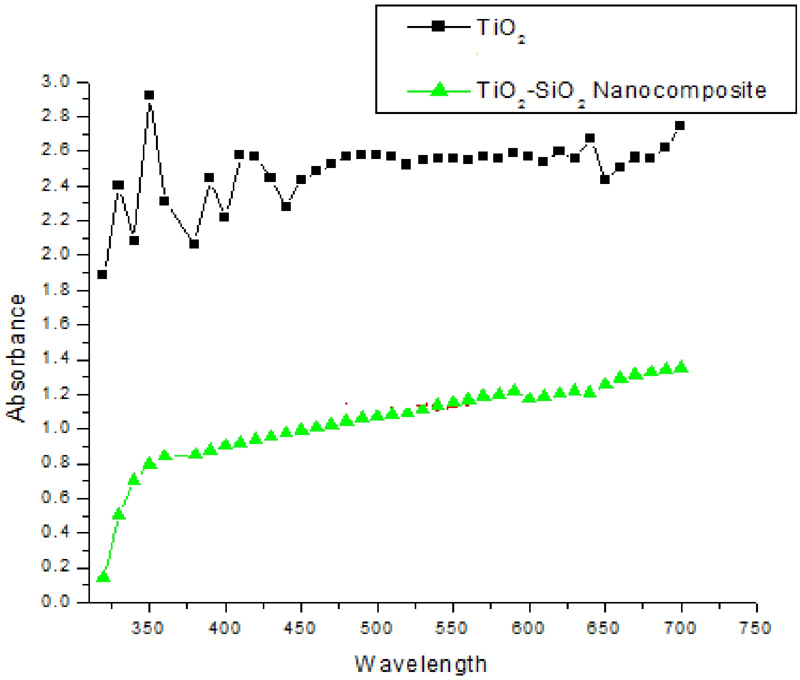

Fig -1 Absorbance spectra of TiO_2 and TiO_2-SiO_2 nanocomposite

3.2 PHOTOCATALYTIC DEGRADATION

Figs.2 (a) and 2 (b) show the Absorbance vs wavelength plot of degraded dye solution after different time intervals with TiO_2 and TiO_2-SiO_2 nanocomposite respectively. Rhodamine B has a prominent absorption peak at 554nm and the absorbance at this wavelength decreases with decrease in concentration [15]. It can be observed from both the figures that the absorbance measured at 0 minutes was comparatively higher than that measured at different time intervals which implies that photocatalytic degradation of the dye has occurred due to its adsorption by TiO_2 and TiO_2-SiO_2 nanocomposite. Enhanced photocatalytic degradation is observed in the case of TiO_2-SiO_2 nanocomposite in comparison to that of TiO_2 which can be seen from the concentration vs time plot as shown in fig.2(c) The photocatalytic efficiency of the TiO_2 and TiO_2-SiO_2 was calculated using the relation [15]

[(C0-C) / C0] x 100 -------------------------- (1)

Where C0 is the initial concentration and C is the concentration after different times

The values of photocatalytic efficiency were found to be 49% in the case of TiO_2 and 61% in the case of TiO_2-SiO_2.

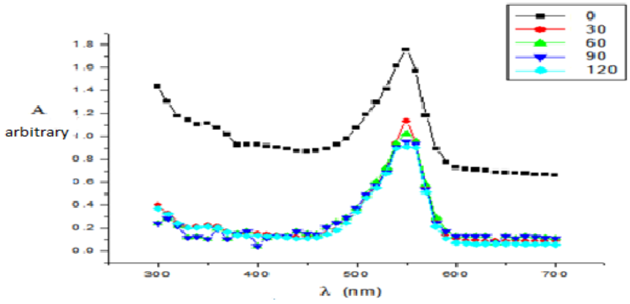

Fig -2 (a): Absorbance vs wavelength plot of degraded dye solution after different time intervals with TiO2

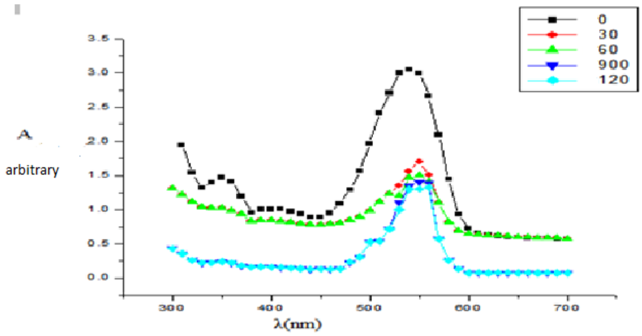

Fig -2 (b) Absorbance vs wavelength plot of degraded dye solution after different time intervals with TiO_2-SiO_2

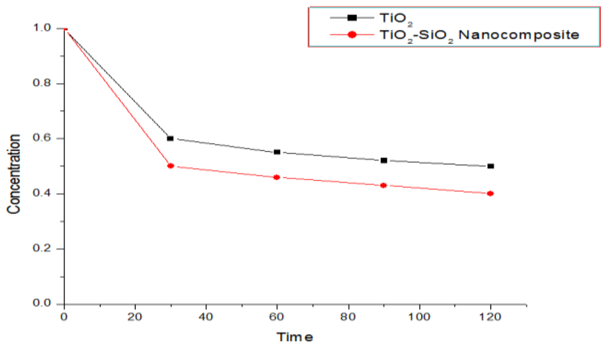

Fig -2 (c): Concentration vs Time plot of degraded dye solution with TiO_2 and TiO_2-SiO_2

3.3 XRD Studies

Fig. 3 (a) and 3 (b) presents the x-ray diffractograms of TiO_2 and TiO_2-SiO_2 respectively. The assignment of different peaks were done by comparison with JCPDS data (21-1272 , 21-1276 and 7631-86-9) and calculation of lattice constants which showed agreement with the reported values. The corresponding data for the two cases are presented in tables 1 and 2. It can be observed from the data that in the case of TiO_2, the maximum intensity corresponds to the (004) plane of TiO_2 anatase. (101) and (105) planes of anatase phase have also been observed. Also,diffraction lines of TiO_2 rutile corresponding to (101), (200) and (210) are seen. These results suggest that the TiO_2 nanostructure is basically a mixed phase. In the case of TiO_2-SiO_2, predominant orientation is towards the (105) plane of TiO_2 anatase. The structure of the nanocomposite dominantly corresponds to TiO_2 anatase along with (101) plane of TiO_{2s} rutile and (201) plane of SiO_2 with low intensity.

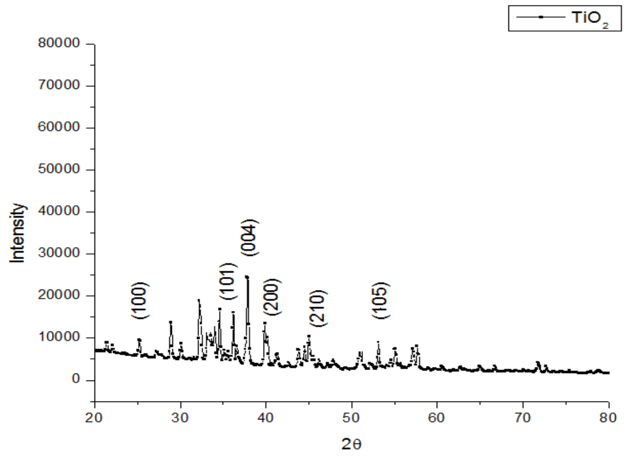

Fig.3(a) X-Ray Diffractogram of TiO_2

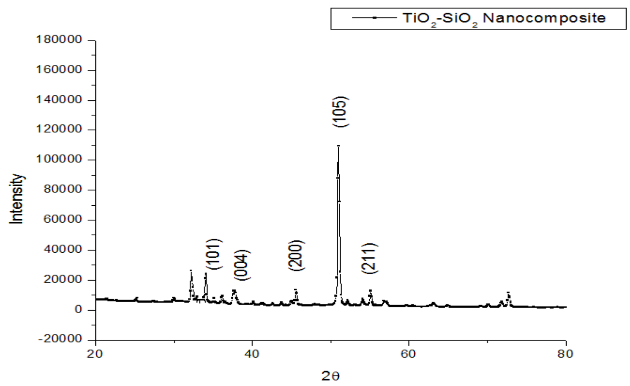

Fig.3 (b) X-Ray Diffractogram of TiO2-SiO2 Nanocomposite

Table 1: XRD data of TiO_2 Prepared by Sonochemical Method

d Value (A^0)		Relative Intensity		hkl	Lattice Constant (A^0)	
(Obs)	(Rep)	(Obs)	(Rep)		(Obs)	(Rep)
3.5334	3.5200	38	100	$(101)_A$	a=3.978 c=9.391	a=3.785 c=9.513
2.4785	2.870	65	50	$(101)_R$	a=4.501 c=2.969	a=4.593 c=2.959
2.3787	2.3780	100	20	$(004)_A$	a=3.978 c=9.347	a=3.785 c=9.513
2.2582	2.2870	54	8	$(200)_R$	a=4.501	a=4.593 c=2.959
2.0133	2.0540	45	10	$(210)_R$	a=4.501	a=4.593 c=2.959
1.7226	1.6999	36	20	$(105)_A$	a=3.978 c=9.391	a=3.785 c=9.513

Table 2: XRD data of TiO_2-SiO_2 Prepared by Sonochemical Method

d Value (A^0)		Relative Intensity		hkl	Lattice Constant (A^0)	
(Obs)	(Rep)	(Obs)	(Rep)		(Obs)	(Rep)
2.4820	2.4870	4	50	$(101)_R$ TiO_2	a=4.501 c=2.975	a=4.593 c=2.959
2.3755	2.3780	12	20	$(004)_A$ TiO_2	a=3.978 c=9.502	a=3.785 c=9.513
1.9893	1.9792	12	4	(201) SiO_2	a=3.978	a=3.785 c=9.513
1.7884	1.699	100	20	$(105)_A$ TiO_2	a=3.978 c=10.010	a=3.785 c=9.513
1.6663	1.6665	11	20	$(211)_A$ TiO_2	a=3.978 c=9.492	a=3.785 c=9.513

3.4 FESEM Studies:

Figures 4 (a) and 4(b) present the FESEM micrographs of TiO_2 and TiO_2-SiO_2 respectively at a magnification of 50K. The FESEM image of TiO_2 reveals the presence of irregular spherical particles of average diameter 100nm. The presence of some cylindrical rod like structures is also observed. It can be observed from Fig.b that, thin rod like structure is formed in the case of TiO_2-SiO_2 with some spherical particles having a cloudy appearance. This may be due to the presence of amorphous SiO_2 around the anatase TiO_2.

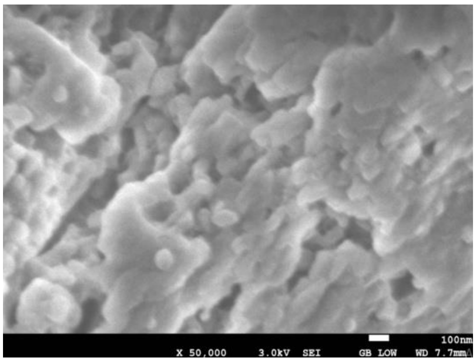

Fig.4(a) FESEM image of TiO_2 nanoparticle

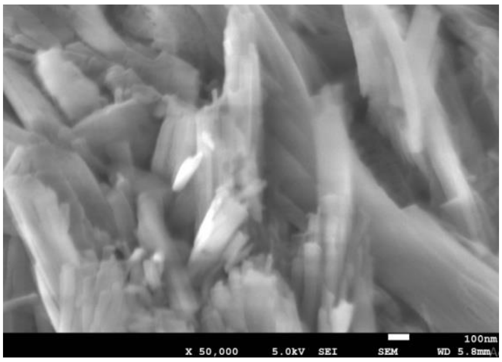

Fig.4(b) FESEM image of TiO_2-SiO_2 nanoparticle

4. CONCLUSION-

TiO_2 nanoparticle and TiO_2-SiO_2 nanocomposites were synthesized using the simple sonochemical technique and their photocatalytic properties were studied using the Rh B dye solution at different time intervals and a good improvement in the photocatalytic property was observed in the case of nanocomposite when compared to TiO_2. TiO_2-SiO_2 as composite was found to be more efficient in photcatalytic applications. XRD studies suggest the presence of mixture of anatase and rutile phase in TiO_2 and as composite it shows highly anatase TiO_2 sturucture with some low intensity peaks of rutile and SiO_2. FESEM results show that the structure was rod like along with some spherical formations giving it a cloudy appearance.

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