July - Sep © 2016 Transactions; 3(11): 487-493

International Journal of Research and Applications

ISSN (online): 2349-0020 ISSN (print): 2394-4544 http://www.ijraonline.com/

Research Article

Synthesis and characterization of Titanium dioxide nanoparticles and nanocomposites with CdS

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DOI:
http://dx.doi.org/
10.17812/IJRA.3.11(81)2016

Manuscript:
Received: 19th July, 2016
Accepted: 16th Aug, 2016
Published: 30th Sep, 2016

Publisher:
Global Science Publishing Group, USA
http://www.globalsciencepg.org/

ABSTRACT

Transition metal oxides possess attracting fundamental and technological properties due to the influence of many factors such as the presence of d-electrons, crystalline structure, oxygen defects and doped impurities. In view of this work, abundant efforts have been attempted to improve charge separation by modifying the surface or bulk properties of TiO_2, such as doping, deposition of metals, size reduction and coupling of two semiconductors and thereby improving the photo catalytic activity. Nanocrystal line TiO_2 has unique physico-chemical, optical and electronic properties, excellent pigmentary properties, high ultraviolet absorption and high stability which allow it to be used in biomedical coatings, electro ceramics, self-cleaning surface coating and building materials. Titanium dioxide nanoparticles and nanocomposites elemental studies were carried out using Fourier Transform Infrared Spectroscopy (FTIR) and Energy Dispersive X-ray Spectroscopy (or EDS). Diffraction studies were carried out using X-Ray Diffraction (XRD) Spectroscopy. Microscopic studies were performed by Scanning Electron Microscope and Transmission Electron Microscope. The TGA curve revealed high thermal stability of the synthesized nanoparticles, absence of any impurity or intermediate complex and high melting point.

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IJRA - Year of 2016 Transactions:
Month: July-September
Volume – 3, Issue – 11, Page No's:487-493
Subject Stream: Chemistry

Paper Communication: Author Direct

Paper Reference Id: IJRA-2016: 3(11)487-493

DOI: 10.17812/IJRA.3.11(81)2016

International Journal of Research and Applications
July - Sep © 2016 Transactions 3(11): 487-493

eISSN : 2349 – 0020
pISSN : 2394 – 4544
www.ijraonline.com

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Synthesis and characterization of Titanium dioxide nanoparticles and nanocomposites with CdS

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ABSTRACT

Transition metal oxides possess attracting fundamental and technological properties due to the influence of many factors such as the presence of d-electrons, crystalline structure, oxygen defects and doped impurities. In view of this work, abundant efforts have been attempted to improve charge separation by modifying the surface or bulk properties of TiO_2, such as doping, deposition of metals, size reduction and coupling of two semiconductors and thereby improving the photo catalytic activity. Nanocrystalline TiO_2 has unique physico-chemical, optical and electronic properties, excellent pigmentary properties, high ultraviolet absorption and high stability which allow it to be used in biomedical coatings, electro ceramics, self-cleaning surface coating and building materials. Titanium dioxide nanoparticles and nanocomposites elemental studies were carried out using Fourier Transform Infrared Spectroscopy (FTIR) and Energy Dispersive X-ray Spectroscopy (or EDS). Diffraction studies were carried out using X-Ray Diffraction (XRD) Spectroscopy. Microscopic studies were performed by Scanning Electron Microscope and Transmission Electron Microscope. The TGA curve revealed high thermal stability of the synthesized nanoparticles, absence of any impurity or intermediate complex and high melting point.

Keywords: Titanium dioxide, Nano composites, FTIR, EDS, XRD and TGA.

1. INTRODUCTION

Titanium dioxide (TiO_2) belongs to one of these prototypes. Titanium dioxide itself has no d-electron, but the number of d-electron can be controlled by doping[1]. In addition to the wide bandgap, TiO_2 exhibits many other interesting properties, such as transparency to visible light, high refractive index, and low absorption coefficient[2]. It is inexpensive, chemically stable and non-toxic. TiO_2 has been used since ancient times as white pigments. It possesses white color because it has no absorption in the visible region[3]. The importance of TiO_2 as photocatalyst for splitting of water was first highlighted by Honda and Fujishima[4,5], since then due to its unique electronic, optoelectronic, Photo catalytic Water Decomposition, Cleaning Building Materials and modified Sol–Gel Process [6-11].TiO_2, is an important IV-VI group semiconductor (SC [12], and can crystallize in three polymorphic forms with Photo catalytic Property of Bicrystalline TiO_2/Rectorite Composites. [13, 14]. It gives a representative diagram of structures of different TiO_2 phases. In its pure form TiO_2 is an n-type semiconductor with indirect band gaps of 3.2eV (387nm) for anatase, 3.02eV (410nm) for rutile and 3.25eV (381nm) for brookite[15], between the filled oxygen 2p valence band (VB) and titanium 3d states at the bottom of the conduction band (CB). All these crystalline structures of TiO_2, consist of deformed TiO_6 octahedron, connected differently by corners and edges. Among these forms, anatase and rutile are most common whereas brookite, is uncommon and unstable[16]. Rutile is the most stable form, whereas anatase and brookite are metastable forms and can readily transform to rutile on heating[17]. The temperature at which anatase to rutile transformation takes place varies from 400°C to 1000°C depending on concentration of

impurities in the crystals or annealing atmosphere[18].

White pigment in paints, food coloring, toothpastes, cosmetics[19-27], in environmental friendly method for air purification[28], flexible dye-sensitized solar cells[29], humidity sensors[30], electro chromic devices, photovoltaic fuel cells, and above all as photo catalyst for air and water purification and also for water splitting to produce H_2.Diffraction (XRD) Spectroscopy, Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). TiO_2 NP absorbs light at <385nm leading to excitation of electrons (e^-) from the valence band to the conduction band, leaving behind a positively charged vacancy called a hole (h^+). The hole by itself is a very powerful oxidizing agent and may lead to the generation of hydroxyl radicals in presence of water and molecular oxygen or can directly oxidize adsorbed species on NP surface. TiO_2 has an advantage of having a capability to perform function using sunlight as an energy source. These photo induced charges have very short lifetime due to charge recombination, releasing the absorbed light energy as heat, with no chemical effect. Therefore, it is important to prevent electron-hole recombination in order to improve the efficiency and efficacy of TiO_2. Also TiO_2 can only effectively absorb in UV region of the solar spectrum which makes up only about 5% of the total solar energy that falls on the earth's surface, thus limiting its use.

2. MATERIALS AND METHODS

Synthetic technique for TiO_2 nanoparticles involved controlled precipitation of nanoparticles from precursors dissolved in a solution. Here, a simple method was proposed for the synthesis of TiO_2 nanoparticles from the reductive hydrolysis of Titanium Tetra Isopropoxide (TTIP) in methanol at ambient temperature and pressure without calcinations. A series of TiO_2 NP (**Tma**, **Tmb**, **Tmc**, **Tmd** and **Tme**) was synthesized by varying the concentration of TTIP and keeping the amount of methanol (24.44M) constant at 100mL. The concentration of TTIP varies as; for **Tma**=0.25M, **Tmb**=0.20M, **Tmc**=0.15M, **Tmd**=0.1M and **Tme**=0.05M. Thereactions proceeded as; 100mL methanol (24.44M) was taken in a conical flask and TTIP was added to it drop wise (20 drops per minute) while vigorous stirring, which was continued for additional 5 hours. White precipitates were obtained, which were washed with water and acetone several times and then air dried.In a reaction vessel 100mL aqueous $Cd(NO_3)_2$(0.085M) was taken and50mL methanol (24.44M) was added drop wise with continuous stirring. The reaction was then carried out in H_2S atmosphere for 1 minute with vigorous stirring, which was continued for 2 hours. The solution turned transparent to yellow. To this solution 3.53mL TTIP (0.1M) was added drop-wise (20 drops per minute). Stirring was continued to additional 5hours. The solution turned to light yellow in color. The synthesized TiO_2 NP and CdS-TiO_2 NC were characterized by various techniques.

3. RESULTS AND DISCUSSION

The white colored fine powders were obtained as a result of reductive hydrolysis of TTIP suggesting the formation of TiO_2 nanoparticles. **Figure 4.2** shows the as-synthesized TiO_2 nanoparticles in suspension form, and **Figure 4.3** shows them in dried form. The synthesized TiO_2 having a mixture of anatase, brookite and rutile crystal structure was expected. For CdS-TiO_2 the color of the sample was yellow.

Fourier Transform Infrared Spectroscopy (FTIR) Spectroscopy:

The FTIR spectra confirmed the purity and composition of the samples as several peaks related to TiO_2 were observed in all samples without any other ele mental impurity. The bands for Ti-O andTi-O-Ti bonds were present in the 800-40 $0cm^{-1}$ region. The FTIR spectra of TiO_2 might be in the form of broad band centered a t 400-$800cm^{-1}$ attributed to the vibration of Ti–O bonds in the TiO_2 lattice or as peaks centered at $760cm^{-1}$, $680cm^{-1}$, $600cm^{-1}$, $560cm^{-1}$, $500cm^{-1}$, $468cm^{-1}$, 41 $0cm^{-1}$, $385cm^{-1}$ and $350cm^{-1}$ assigned to the (Ti-O-Ti) stretching vibration. **Figure 4.4** shows the FTIR spectra of the synthesized TiO_2 nanoparticles and its nanocomposites with CdS (**CdS-TiO_2**). In the present case the FTIR spectra of TiO_2 NPs and **CdS-TiO_2** NC was in the form of broad peak in the region 400-$800cm^{-1}$ with several small peaks in it. The b road peak appearing at 3100–$3600cm^{-1}$ was assigned to vibrations of hydroxyl groups of water

adsorbed by the samples. The weak absorption band at 1620-1630c m^{-1} was attributed to CO_2 adsorbed on the surface of the particles. As it is known that adsorption of water and CO_2 are common for all powder samples exposed to atmosphere and are even more pronounced for nanosized particles with high surface area. In case of **CdS-TiO₂**, the formation of CdS was confirmed by the appearance of peak at 405cm^{-1} assigned for Cd-S bond, and the presence of broad band centered at 400-800cm^{-1} with several small peaks in it confirms the presence of TiO_2 in the nanocomposite. **Figure 4.5** contains the EDS spectra of the synthesized nanoparticles. The EDSspectra showed the presence of Ti and O peaks confirming the formation of pure TiO_2 with no other elemental impurity for the samples **Tma**, **Tmb**, **Tmc**, **Tmd** and **Tme**. Other peaks in this figure corresponded to oxygen, carbon and silicate were due to sputter coating of glass substrate on the EDS stage and were not considered. The EDS spectra of **CdS-TiO₂** showed the presence of peak corresponded to Cd and S along with the peaks of Ti and O, thus confirming the formation of CdS-TiO_2nanocomposite.

X-Ray Diffraction (XRD) Spectroscopy:

X-ray diffraction patterns for TiO_2 NPs were of rather poor quality due to the physical nature of the samples. However, the XRD pattern (**Figure 4.6**) of the synthesized TiO_2 NPs revealed the presence of mixtures of anatase, brookite and rutile. The **Figure 4.6**, shows the peaks corresponded to anatase at 2 =25.56˚ (101), 37.8˚ (103), 48.07˚ (200), 54.18˚ (105), 62.42˚ (204) and 75.2˚ (215), rutile at 2 =27.01˚ (110), 36.14˚ (101), 42.121˚ (111), 54.89˚ (211) and 68.72˚ (301) andbrookite at 2 =30.9˚ (121) thus confirming the presence of mixed crystal phase. The estimated rystallite size for TiO_2 samples (**Tma**, **Tmb**, **Tmc**, **Tmd** and **Tme**) based on the X-ray diffraction peak was not possible due to the presence of mixed peaks of anatase, rutile and brookite TiO_2.

Figure 4.7 shows the XRD pattern of **CdS-TiO₂**nanocomposites, the pattern revealedthe formation of cubic CdS and anatase TiO_2 nanoparticles. The presence of peaks corresponded only to anatase TiO_2 at 2 =25.56˚ (101), 37.282˚ (103), 48.07˚ (200), 54.18˚ (105), 62.42˚ (204), was related to the formation of anatase TiO_2 in **CdS-TiO₂**, while the formation of cubic CdS was confirmed by the presence ofpeaks corresponded only to cubic CdS at 2 =26.719˚ (111), 29.900˚ (200), 43.000˚ (220), 51.061˚ (311).

Scanning Electron Microscopy (SEM):

The SEM micrographs showed the formation of well-defined spherical mesoporous TiO_2 nanoclusters which were attributed to the high surface energy of nanosized TiO2 particles. **Figure 4.8 (a-e)**, shows the SEM images of the synthesized TiO_2 NP above 7000 times magnification (7kx). **Figure 4.8 (f)** shows the SEM images of the synthesized CdS-TiO_2 NC at 9000 times magnification (9kx). X-ray diffraction peak was not possible due to the presence of mixed peaks of cubic CdS and anatase TiO_2.

Transmission Electron Microscopy (TEM):

The TEM micrograph (**Figure 4.9 (a-f)**) of TiO_2 NP (**Tma**, **Tmb**, **Tmc**, **Tmd**and**Tme**) showed the formation of spherical nanoclusters comprised of very small sized**TiO_2**NPs (1-2nm). The images showed a decrease in size of the nanocluster from>500nm (**Tma**) to <50nm (**Tme**). But the sizes of the individual TiO_2 nanoparticles were less than 2nm in all the cases. For **CdS-TiO₂** the TEM image (**Figure 4.10**) confirmed the presence of both CdS and TiO_2 nanoparticles. This was in good agreement with the EDS and XRD results.

Thermal Gravimetric Analysis (TGA):

The synthesized TiO_2 NPs (**Tma**, **Tmb**, **Tmc**, **Tmd** and **Tme**) and **CdS-TiO₂** NC were found to be thermally stable upto temperature as high as 1000°C with a small weight loss at around 100°C, which was probably due to the presence of moisture and other volatile solvents. **Figure 4.11** shows the TGA results of synthesized TiO_2 nanoparticles (**Tma**, **Tmb**, **Tmc**, **Tmd** and **Tme**) and **CdS-TiO₂**nanocomposite.

4. CONCLUSION

A series of Titanium Dioxide nanoparticles (**Tma**, **Tmb**, **Tmc**, **Tmd** and **Tme**) were successfully synthesized via single pot chemical precipitation method under ambient conditions. The synthesized TiO_2 NP clearly showed that as the concentration of the Ti precursor decreased the size of the TiO_2 nanocluster also decreased. The

size of the individual TiO_2 particle was not much affected. The synthesized NP showed good elemental purity without any contamination and good thermal stability. A mixed crystalline phase was observed in all TiO_2 samples. The micrographic studies revealed the formation of spherical clusters whose sizes decreased dramatically as the concentration of the Ti precursor decreased.

Figure 4.1 The synthesized TiO_2nanoparticles (**Tma**,**Tmb**,**Tmc**,**Tmd**and**Tme**) obtained as such in suspension form.

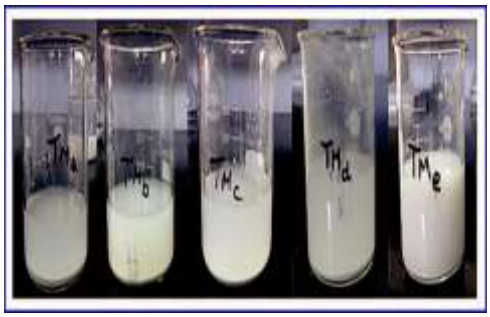

Figure 4.2 The synthesized TiO_2nanoparticles (**Tma**,**Tmb**,**Tmc**,**Tmd**and**Tme**) obtained after washing and drying.

Figure 4.3 The FTIR Spectra of the synthesized TiO_2nanoparticles (**Tma**,**Tmb**, **Tmc**,**Tmd**and**Tme**) and its nanocomposites with CdS (**CdS-TiO_2**).

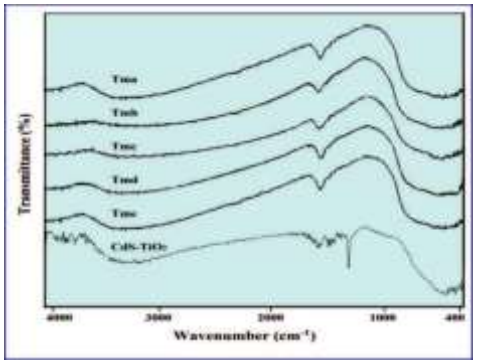

Figure 4.4 EDS of the synthesized TiO_2nanoparticles (**Tma**,**Tmb**,**Tmc**,**Tmd**and **Tme**) and its nanocomposites with CdS (**CdS-TiO_2**).

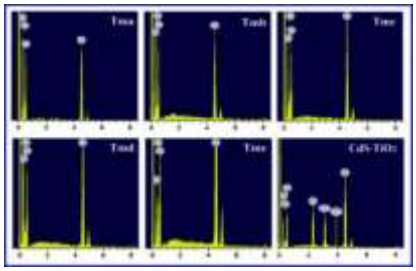

Figure 4.6 XRD spectra of synthesized TiO_2nanoparticles (**Tm a**, **Tm b**, **Tm c**, **Tm d** and **Tm e**).

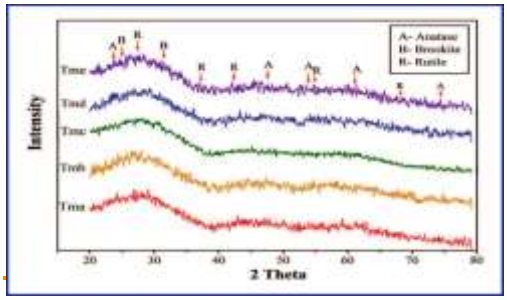

Figure 4.7 XRD spectra of **CdS-TiO_2** nanocomposites in comparison to pure cubicCdS and Degussa P-25 TiO_2

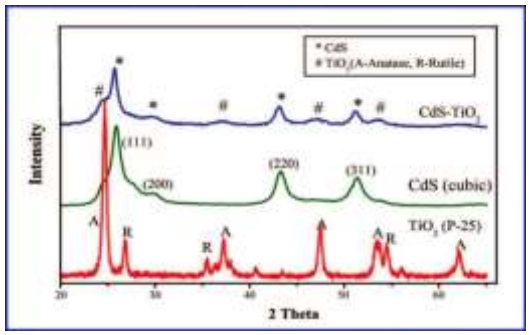

Figure 4.8 SEM images (a) **Tm a**, (b) **Tm b**, (c) **Tm c**, (d) **Tm d**, (e) **Tm e** and (f) **CdS-TiO_2**.

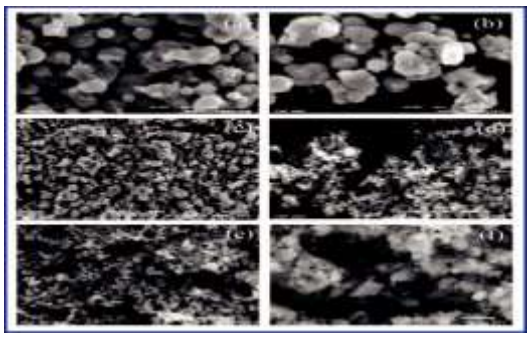

Figure 4.9 TEM images of (a)**Tma**, (b)**Tmb**, (c)**Tmc**, (d) magnified portion of **Tmc**, (e)**Tmd**, (f)**Tme**, and in the inset are the magnified portion of the corresponding images.

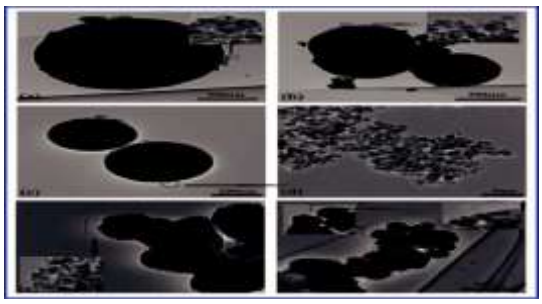

Figure 4.10 (a), shows two different sized nanoparticles, the smaller were TiO_2andthe larger CdS nanoparticles. **Figure 4.10 (b)**, shows the representative diagram of **CdS-TiO_2**. The particle sizes obtained by TEM are listed in **Table 4.2**.

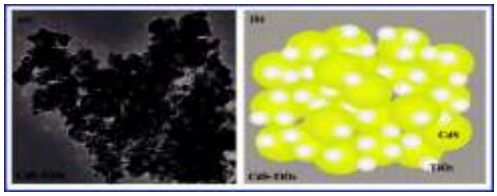

Figure 4.11 TGA results of synthesized TiO_2nanoparticles (**Tm a**, **Tm b**, **Tm c**, **Tm d** and **Tm e**) and **CdS-TiO_2**nanocomposite

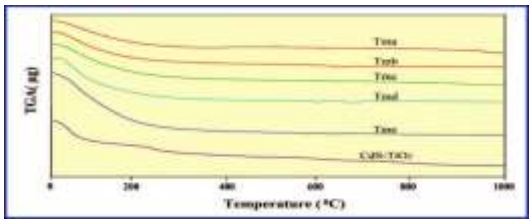

Table 4.1 The explanation of the various peaks obtained by the FTIR spectra of the synthesized TiO_2 nanoparticles (**Tm a**, **Tm b**, **Tm c**, **Tm d** and **Tm e**) and **CdS-TiO_2**.

Peak	Region	Intensity	Significance
A	400-420	Small and weak	Cd-S bond (CdS nanoparticles)
B	400-800	broad band	Ti-O bond vibration (TiO_2 nanoparticles)
C	570-620	Small and weak	S-S bond (crystal S-S bond)
D	1380-1420	Sharp or Broad	C-H bending of CH_3 (Acetone)
E	1620-1740	Small and weak	CO_2 bending or C-H bending (Acetone)
F	3140-3470	Broad	Intermolecular H-bonds (Lattice water)

Table 4.2: Average particle sizes of the synthesized TiO_2NP (**Tm a**, **Tm b**, **Tm c**, **Tm d** and **Tm e**) and **CdS-TiO_2**NC obtained by TEM

Nanoparticles	**Tma**	**Tmb**	**Tmc**	**Tmd**	**Tme**	**CdS-TiO_2**
Particle Size (nm)	1.5-2.0	1.5-1.6	1.2-1.5	1-1.2	0.8-1.0	5-6 (CdS), 1.5-2 (TiO_2)

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