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# Hydrothermal Synthesis of Nanocrystals of Titania from TTIP and Powder $TiO_2$

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ABSTRACT

This paper addressing the synthesis (hydrothermal route) and comparative study of $TiO_2$ nanocrystals from two different sources TTIP (titanium tetra isopropoxide) and $TiO_2$ powder. $TiO_2$ nano crystals having excellent properties and can be used in various applications including glass window self-cleaning, purification of air, water and antimicrobial coating by tapping the photo catalytic properties of nano particles. The synthesized titania was characterized by SEM, XRD, FTIR and UV-Vis spectroscopy. The particles size calculation was done by applying Scherrer equation. The resulted size of titania particles was vary from 20-100 micrometers. The crystal size of particles are vary from 10-47 nanometers.

## 1. Introduction

Titanium dioxide is a silvery-white transition metal powder that resists corrosion by forming a shielding layer which prevent water, air, acids and alkalis attacks. To give a shape or a form to make a useful alloy (titanium with other elements), it requires to heat it up which remove its hardness and brittleness [1]. $TiO_2$ is a wide band gap semiconductor that has been used extensively to make products like paper, plastics, lipstick, toothpaste, and pharmaceutical tablets [2]. Moreover, $TiO_2$ nano particles in the 10-50 nm range has excellent properties and can be used for glass window self-cleaning, purification of air, water and antimicrobial coating by tapping the photo catalytic characteristics of nanoparticles. Scientists have used them for nitrogen oxides removal from exhausts and also looking forward at the ways to utilize these environmental catalysts to treat diesel automobile secretions. $TiO_2$ is a semiconductor whose band gap is wide and researchers are looking at it as a substitute for silicon to make solar power cells, as well as battery storage media [3]. Titanium dioxide is chemically stable and not dissolved in water; although some other semi conductive photocatalytic compounds are dissolved when irradiated in water. $TiO_2$ does not soluble in all types of organic, acidic and basic solvents because it is highly stable (chemically) and useful for some cosmetic product manufacturing. Photo catalyst titanium dioxide is stated as harmless and ecofriendly material which is used as a catalyst for some chemical reactions because it is available in fine oxidative form. $TiO_2$ coated ceramic tiles are considered to be very effective against organic and inorganic material, as well as against bacteria so $TiO_2$ coated tiles help to reduce infections [4-6]. The present work focus on the comparative studies on titania, synthesized from two different precursors by hydrothermal route.

## 2. Experimental Methods

### *2.1 Materials*

$TiO_2$ powder (99.5% pure) procured from Sigma Aldrich. Titanium tetra isopropoxide (97% pure) procured from Sigma Aldrich. Hydrochloric acid and acetic acid glacial (99.85% pure) obtained from RFCL Ltd. Delhi. Sodium hydroxide pallets procured from Thermo Fisher scientific India Pvt. Ltd. Mumbai.

### *2.2 Synthesis of Nano $Tio_2$ from $TiO_2$ Powder*

About 24.0 gram pure $TiO_2$ powder was mixed with 60 mL of 10 M NaOH aqueous solution. The NaOH solution mix with this solution and transferred to a teflon based autoclave and gave heat treatment at 120 °C for 20 hour. The filtered precipitate were dried at 80 °C and gave alkali treatment to the powdered $TiO_2$ which required washing with distilled water and further with 0.1 M HCl solution followed by distilled water until pH reached around 7. The obtained white sample was dried at 80 °C for 5 hours and the nanoparticles were collected [7].

### *2.3 Synthesis of $TiO_2$ Nanoparticles from TTIP*

The TTIP (titanium tetra isopropoxide) was mixed with distilled water to make colloidal solution. 1 M of TTIP was mixed together with 4 M of acetic acid. 1 M TTIP solution was prepared by 15.26 mL of TTIP in 50 mL of Distilled water. The solution acetic acid mixed with 10 mL distilled water followed by continuous stirring of one hour. The mixture solution was placed for aging (24 hours), 25 mL of this solution was transferred to stain less steel autoclave and placed in oven at 180 °C for 12 hours. The solution was dried at 100 °C and crushed into fine powders [8].

## 3. Results and Discussion

### *3.1 XRD Analysis*

Identification of $TiO_2$ phase carried out using X-ray diffraction technique. These examples had run with the typical peaks of $TiO_2$ polycrystalline anatase nanoparticle with no recognizable dopant-related peaks. The powder $TiO_2$ samples were documented by using Cu-Ka (1.5406 Å) radiation at room temperature in between 20° to 80° range at the $2\theta$ scale. The XRD pattern as shown in Fig. 1 of $TiO_2$ nanoparticles prepared from two different precursors (TTIP and commercial $TiO_2$ powder).

From the Fig. 1, it is clear that the anatase phase of the $TiO_2$ nanoparticles is obtained by using TTIP precursor, while using $TiO_2$ powder precursor, the rutile phase is obtained. The anatase phase formation is confirmed by using JCPDS 21-1272 as well as rutile phase by JCPDS 21-1276.

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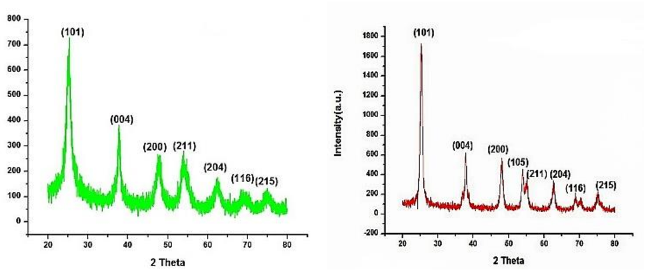



**Fig. 1** XRD pattern of $TiO_2$nanoparticles prepared using TTIP and $TiO_2$

### *3.2 Particle Size Calculation*

The Scherrer condition, in X-ray diffraction is a recipe that relates the span of sub-micrometer particles, or crystallites, in a strong to the widening of a peak in a diffraction design. It is utilized as a part of the assurance of size of particles of crystals in powder.

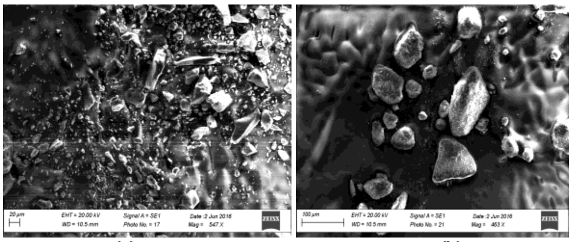
The Scherrer equation can be written as $\tau = K\lambda/\beta\cos\theta$. The size of the particles is calculated from the XRD data by Scherrer equation. The Scherrer equation was used for each of the XRD patterns separately and the calculations were done accordingly (Table 1).

**Table 1** Size of the nanoparticles by Scherrer method

| S. no. | Sample Name | $2\theta$ | FWHM | Crystal Size (nm) |
|---|---|---|---|---|
| 1 | $TiO_2$ hydrothermal ($TiO_2$) | 27.41 | 0.17311 | 47.28 |
| 2 | $TiO_2$Hydrothermal (TTIP) | 25.292 | 0.7813 | 10.43 |

### *3.3 SEM Analysis*

The surface structure and pore sizes of composite sample was viewed by scanning electron microscopy (SEM, JEOL). The surface and fracture section of the samples were coated with Au prior surface detail, homogeneity and elemental composition can be determined in one experiment on the same sample. The Fig. 2 shows the scanning electron microscope image of the nanoparticles prepared by hydrothermal method. The images show that the particles have inhomogeneous distribution and some of them are in agglomerated form. The average size of $TiO_2$ particles synthesized from TTIP and $TiO_2$ powder are 20 and 100 micrometers respectively. These particles are irregular in shape and size.



(a) (b)

**Fig. 2** SEM image of $TiO_2$ nanoparticles by hydrothermal using (a) TTIP and (b) $TiO_2$ powder

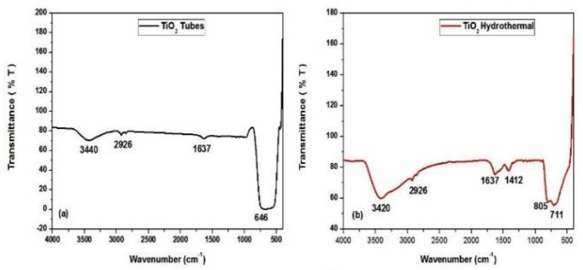



**Fig. 3** FTIR spectroscopic image of $TiO_2$ nanoparticles

### *3.4 FTIR Analysis*

The Perkin Elmer spectrum RX1 spectrometer (FTIR) characterization technique was utilized to analyze synthesized $TiO_2$ samples from 4000 $cm^{-1}$ to 400 $cm^{-1}$ range at a scanning rate of $cm^{-1}$/min. Fig. 3 shows the FTIR spectrum of both samples. There is OH stretching present between 3400 $cm^{-1}$ and 3440 $cm^{-1}$ in both samples. The band present at 2926 $cm^{-1}$ is due to Ti-O stretching vibrations. The strong band located at 700 $cm^{-1}$ attributed to Ti–O and Ti–O–Ti bridging stretching. The peak present in the samples near 1637 $cm^{-1}$ is due to the bending vibrations of adsorbed $H_2O$ molecules. The peak at 1412 $cm^{-1}$ present in $TiO_2$ hydrothermal is due to Ti-O-Ti. The characteristic vibrations present in 600-800 $cm^{-1}$ are due to the inorganic Ti-O stretch. The bands of Ti-O present confirm the formation of $TiO_2$ nanoparticles. After heat treatment to the samples, the water absorption is decreased as shown in the annealed sample FTIR results [9].

### *3.5 UV-Vis Analysis*

The Fig. 4 shows the UV-vis results for the $TiO_2$ nanoparticles prepared from $TiO_2$ powder and TTIP. If the concentration of any precursor will be reduced it cause blue shifted absorption edge. The main 2p orbitals of oxygen consisted in valence band in the ionic lattice of titania with the conforming wave functions substantially localized on the $O^{2-}$ lattice sites. The conduction band consists mostly of excited states of $Ti^{4+}$. The adsorption of UV-Vis rays found at 250 nm and 270 nm for titania. For the rutile phase there is a considerable increment noticed in the absorption in the visible range while for the anatase there is absorption noticed at about 350 nm due to UV radiation absorption and scattering by $TiO_2$ nanoparticles [10].

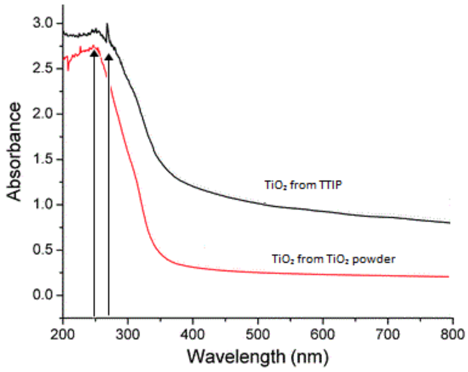



**Fig. 4** UV-Vis spectrum of Titania synthesized from $TiO_2$ powder (a) and TTIP (b)

## 4. Conclusion

The white powder nano crystals of titania were synthesized by hydrothermal method. The resultant powder titania were characterized by SEM, XRD and UV-Vis spectroscopy. The rutile form of particles formed from commercial $TiO_2$ and anatase form produced from TTIP. The nano crystal of 10-47 nanometers were calculated by Scherrer equation. The TTIP precursor produced crystal size of 10.43 nm while $TiO_2$ powder based nanocrystals were of 47.28 nm. The scanning electron microscopy results confirmed the size of synthesized titania. The particles size of TTIP precursor based particles were 20 micrometers and $TiO_2$ powder given 100 micrometers size particles. The TTIP precursor is more prominent as compared to $TiO_2$ powder for the synthesis of titania. The small sized and loosely packed fine crystals were formed by TTIP. The molar ratio of TTIP plays crucial role in the synthesis of fine crystal formation.

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