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Preparation and characterization of anatase phase TiO_2 nanoparticles at low temperature

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ABSTRACT: Titanium dioxide nanoparticles have been prepared by hydrolysis of titanium tetra isopropoxide as precursor. The prepared titanium dioxide nanoparticles were analyzed before calcination treatment and after calcination treatment. Synthesized particle is characterized by X-ray diffraction (XRD), Field Emission Scanning Electron Microscope (FESEM), Energy Dispersive X- Ray Analysis (EDAX) and Ultra Violet Visible Spectrum Analysis(UV). Experimental results have shown that the as-prepared particles have entirely consisted with anatase crystalline phase. It is commonly accepted that high temperature calcination of at least 400 ^{0} C is required to obtain anatase phase of TiO_2. However, we have synthesized nano crystalline TiO_2 particles having anatase phase at 100^0 C itself.

KEYWORDS: Hydrolysis; Titanium tetra isopropoxide; TiO_2 Nanoparticles

I. INTRODUCTION

Nanotechnology is science, engineering, and technology deals with objects at the nanoscale (1 to 100 nanometers). Nanotechnology offers the potential of novel nanomaterials for the treatment of surface water, groundwater, waste water, and other environmental materials contaminated by toxic metal ions, organic and inorganic solutes, and microorganisms. Due to their unique activity towards unmanageable contaminants, many nanomaterials are under active research and development for use in the treatment of water. The recent advances in the development of metal oxide nanoparticles (TiO_2, Fe_3O4/Fe_2O_3, MnO_2, CeO_2, MgO and Al_2O_3) and the related processes had paved the way for the treatment of various water resources which have been contaminated by organic solutes, inorganic anions, radionuclides, bacteria and viruses. TiO_2 offers great potential for detoxification or remediation of waste water due to several factors.TiO_2 is a promising versatile material due to its photo–stability, chemical structure, physical, optical, electrical properties, low cost and excellent degradation of organic pollutants. One of the distinctive characteristic of TiO_2 is its disinfective activity upon stimulation by incident light. Its photocatalytic properties have been employed in various environmental applications to eradicate contaminants from both water and air. Over the last two decades, photocatalysis with TiO_2 nanoparticles has been useful for the degradation of waste water pollutants.Titanium dioxide has three crystalline phases: brookite (orthorhombic), anatase (tetragonal), and rutile (tetragonal). Rutile is the stable phase, whereas anatase and brookite are both metastable at all temperatures. The energy band gaps are equal to 3.2 and 3.0 eV for anatase and rutile phases, respectively, which means that ultraviolet (UV) region of the solar spectrum is the proper region for the photo excitation of TiO_2 nanoparticles to produce electron/holes and generation of OH radicals. In this research work, we have tried to prepare TiO_2 nanoparticles and analyzed the sample at two different stages: before calcination and after calcination. In the present work an attempt has been made to prepare anatase phase TiO_2 nanoparticles at very low temperature.

II. EXPERIMENTAL METHOD

In the present work 5ml of analytical grade titanium tetra isopropoxide used as a precursor and was mixed with 15 ml of iso propanol solution. A mixture of 50 ml de-ionised water with 0.01 molar NaOH solution was used as the hydrolysis catalyst. The pH of the catalyst was maintained at 8.86. The precipitation process started when both solutions were mixed together under vigorous stirring. A yellow powder obtained from hydrolysis of titanium tetra isopropoxide heated at 67^0 C for 21 hrs. The prepared powder is washed and filtered with ethanol for four times and dried at 100 ^{0} C for 3 hrs. in vacuum state. Finally the prepared powder was calcinated at a temperature of 800^0 C for 1 hr.The characterization of titanium dioxide before

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calcination treatment and after calcinations treatment were done by using X-ray diffraction (XRD), Field Emission Scanning Electron Microscope (FESEM), Transmission Electron Microscope (TEM), Energy Dispersive X- Ray Analysis (EDAX) and Ultra Violet Visible Spectrum Analysis (UV).Flow chart depicting the synthesis of Nano crystalline TiO2 is shown in figure 2.1

Titanium tetra isopropoxide(TTIP) (5 ml) + ISO PROPANOL (15ml) +
DISTILLED WATER (50ml) + NaOH (0.01 molar)

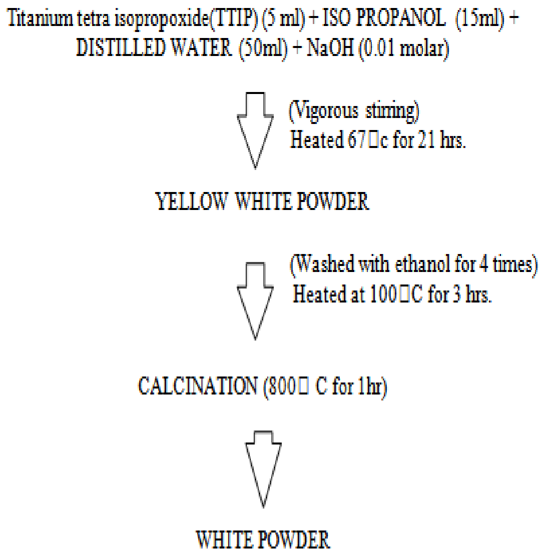

Fig 2.1 Flow chart for the synthesis of TiO_2 Nanoparticles

III. RESULT AND DISCUSSION

X-Ray Diffraction Analysis:

XRD is a powerful non-destructive technique for characterizing crystalline materials. For this reason, in order to investigate the phase formation and micro structure studies, the XRD analysis was carried out on the prepared TiO_2 nanoparticles. The XRD pattern of Titanium dioxide nano particle before calcination treatment is as shown in figure (3.1). The average crystalline size was calculated by using the data from X-ray diffraction pattern with the Debye-Scherrer formula.

The Debye-Scherrer equation is,

$$D = k\lambda / \beta \cos\theta$$

Where,

D = crystallite size

λ = wave length of the filament used in XRD machine (1.5418 A^0)

β = Full Width Half Maximum (FWHM)

θ = angle of the corresponding peak

k = shape factor(0.94)

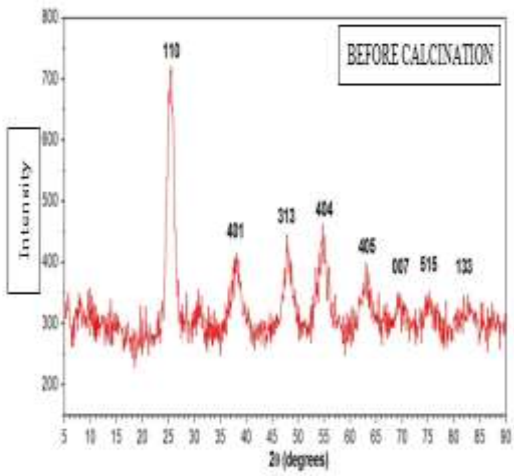

Fig 3.1 XRD pattern of TiO_2 nano particles before calcination.

The XRD spectrum showed peaks around 25.4, 38, 48, 54.7, 62.8, 69.4, 75 and 82.9. The most intense peaks observed around 2θ = 25.4, 54.7, 48 respectively corresponds to (110), (404) and (313) orientation. The TiO_2 possessed evident diffraction peaks characteristics of anatase, which indicates that the TiO_2 nanoparticles prepared are predominantly of the anatase crystal phase. The diffraction data were in good agreement with JCPDS card no 21- 1272. It is commonly accepted that high temperature calcinations of at least 400^0 C is required to obtain anatase TiO_2 nano particles. However, we prepared anatase crystalline particles at 100^0 C.

The XRD pattern of Titanium dioxide nano particle calcinated at 800^0C is shown in figure (3.2).

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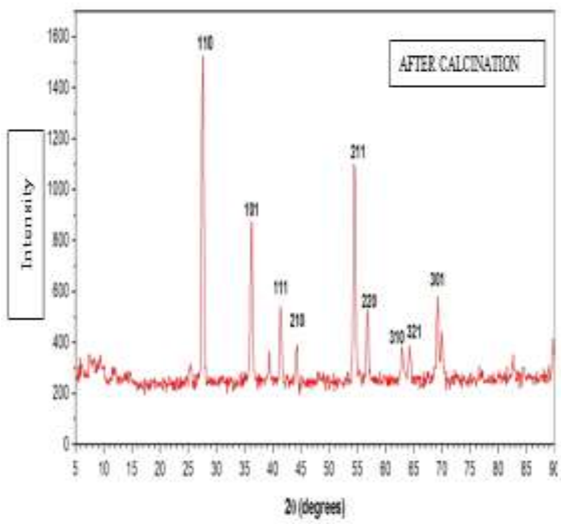

Fig 3.2 XRD pattern of TiO_2 nano particles calcinated at 800^0C

The XRD spectrum showed sharp peak around 2θ = 27.5, 36, 41, 44, 54.4, 56.7, 62.8, 64 and 69. The intense peaks are around 27.5, 36 and 54.4 are respectively correspond to (110), (101) and (211) orientations. The values of 2θ and d are in good agreement with the standard JCPDS card number [21-1272]. The average crystalline size was found to be in the ranges of 4 to 20 nm. The minimum crystalline structure is observed for before calcination and the maximum crystalline size is observed for after calcination. The crystallite size increased after calcination.

The peak details of the TiO_2 nanoparticles before and after calcination are listed below in the table 3.1.

Table 3.1 – The peak details of the TiO_2 nanoparticles before calcination and after calcination

sample	2θ(degree)	Phase and orientation (hkl)	Crystallite size(nm)	d_{hkl}
TiO_2 Before calcination	25.4	(110)	4.99	3.49
	54.7	(404)	4.50	1.67
	48.0	(313)	4.79	1.89
TiO_2 After calcination	27.5	(110)	18.55	3.23
	54.4	(211)	19.84	1.68
	36.1	(101)	19.99	2.48

Field Emission Scanning Electron Microscope (FESEM):

The surface morphology of titanium dioxide nanoparticles subjected to calcination (800^0 C) treatment has been studied by FESEM. The magnified image of titanium dioxide nanoparticle is shown in figure (3.3).The FESEM image showed agglomeration of spherical cluster.

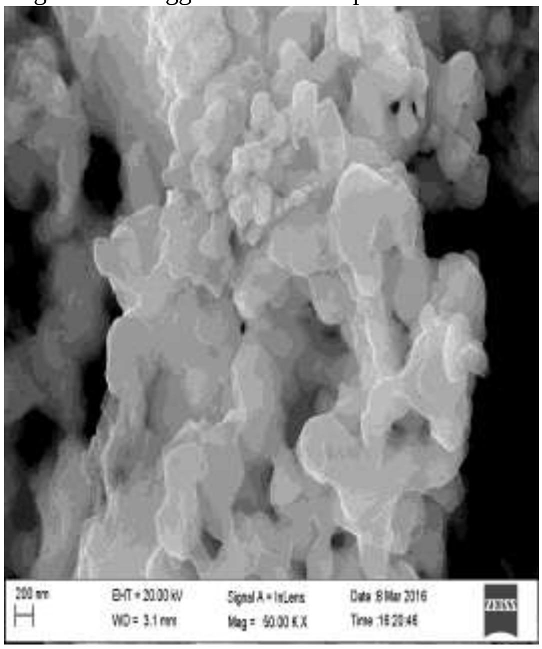

Fig 3.3 FESEM image of TiO_2 nanoparticle

Energy Dispersive X-Ray Analysis:

The EDAX spectrums of Titanium dioxide nanoparticle before calcinations treatment and after calcination are shown in figure (3.4) and (3.5) respectively. The EDAX spectrum revealed the presence of titanium and oxygen in the prepared samples. It also indicated the absence of any impurities in the prepared sample.

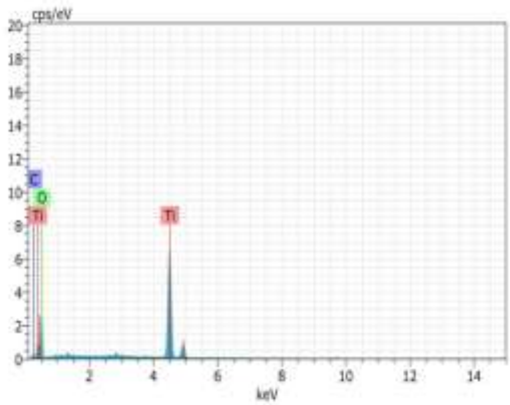

Fig 3.4 EDAX spectrum of TiO_2 nanoparticle before subjected to calcination temperature.

The EDAX analysis confirmed the formation of titanium dioxide nanoparticle. After calcination the amount of titanium and oxygen seems to have increased. The percentage of titanium has doubled after calcination.

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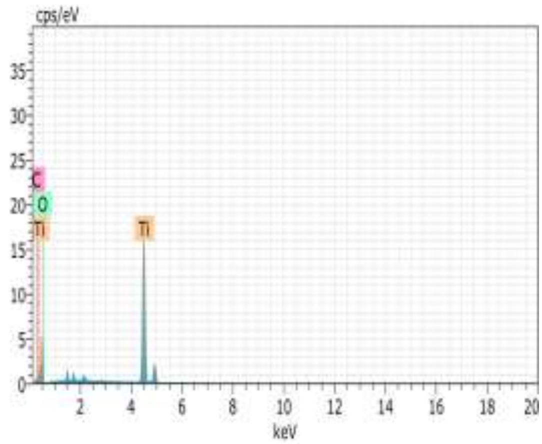

Fig 3.5 EDAX spectrum of TiO_2 nanoparticles calcinated at 800^0C

UV - Visible Spectroscopy:

The energy band gap value was determined based on the mathematical derivative of the optical absorption coefficient. The fundamental absorption method corresponds to band to band transitions by using energy relation. The energy band gap was found using the relation,

$$Eg = 1240/\lambda g$$

The UV-VIS spectra of TiO_2 nanoparticles before subject to calcination temperature are respectively shown in fig (3.6) and fig. (3.7).

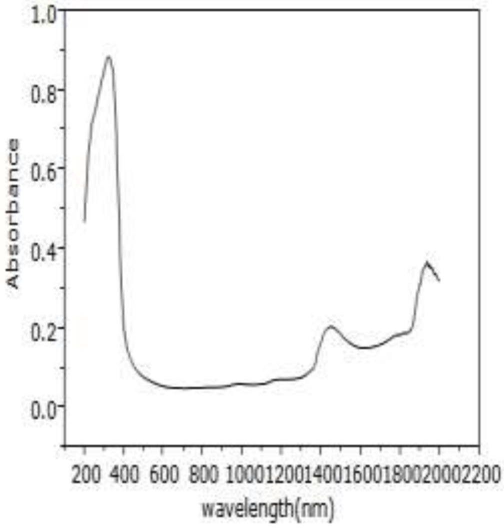

Fig 3.6 Absorbance spectrum of TiO_2 nanoparticles before subjected to calcination temperature.

It is observed that maximum absorbance takes place at 370 nm for samples before subjected to calcination treatment. Using the relation, the energy band gap value was found to be 3.35eV.

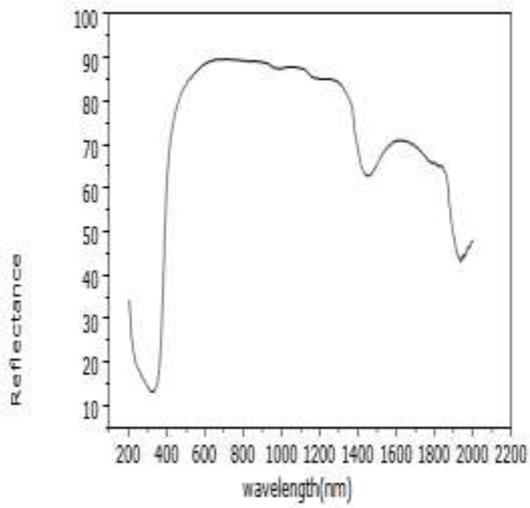

Fig 3.7 – Reflectance spectrum of TiO2 nanoparticles before subjected to calcination temperature

After calcination treatment the peak is absorbed around 360 nm, which is the characteristic band for the TiO2. No other peak was observed indicating the purity of sample synthesized. And the band gap energy of the calcinated sample is obtained as 3.44 eV.

The optical absorbance and reflectance of TiO2 nanoparticles calcinated at 8000C are respectively shown in fig (3.8) and fig. (3.9).

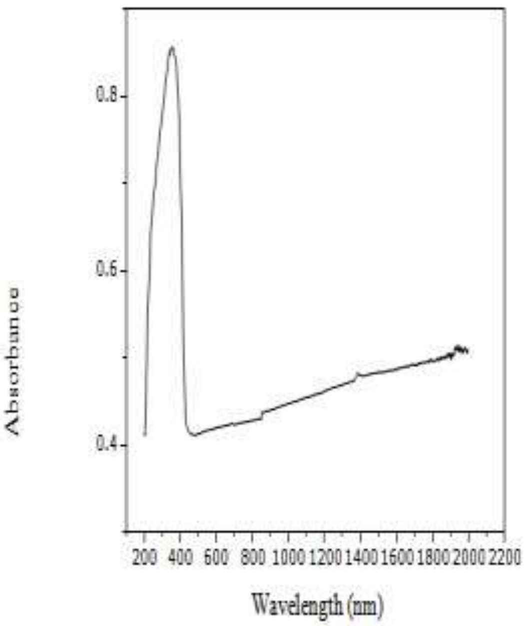

Fig **3.8** Absorbance spectrum of TiO_2 nanoparticles after subjected to calcination temperature

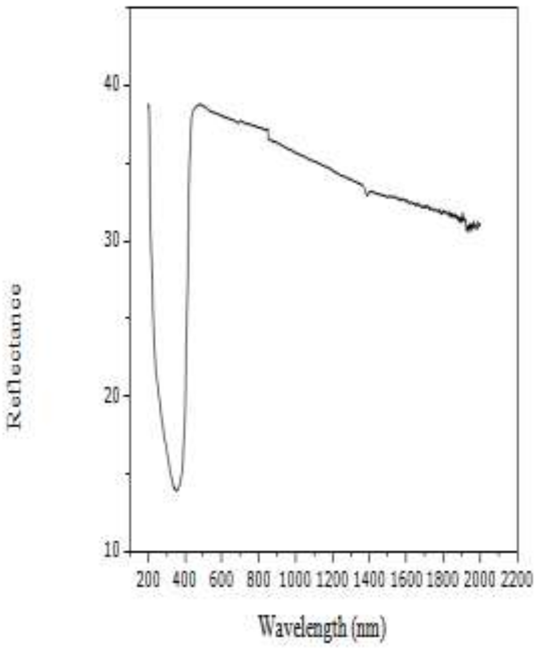

Fig 3.9– Reflectance spectrum of TiO_2 nanoparticles after subjected to calcination temperature

Table3.1: Wavelength of Absorption and band gap

Condition	Wavelength	Band gap	% A
Before Calcination	370	3.35	0.9
After Calcination	360	3.44	0.9

IV. CONCLUSION

Titanium dioxide nanoparticles were prepared using a simple and cost effective hydrolysis method. The prepared nanoparticles were analysed before and after calcination treatment. The X-ray diffraction studies revealed that the prepared TiO_2 nano particle were in anatase phase. The crystallite size ranges from 4nm to 20 nm. From the result of FESEM, the prepared TiO_2 nanoparticles showed spherical cluster like structure and shows agglomeration. The EDAX spectrum reveals that there is no impurity present in the sample and also an increase in the concentration of Ti and O after calcination is observed.

It is commonly accepted that high temperature calcination, at least at 400 ^{0} C is required to obtain anatase phase of TiO_2. However, we have synthesized nano crystalline TiO_2 nanoparticles having anatase phase at a lower temperature around 100^0C. Among the three mentioned phases of titanium dioxide, TiO_2 anatase owing to its higher photocatalytic activity is commonly used for photo catalysis.

The UV analysis pattern measurement reveals that the band gap energy of the sample has increased when compared to the bulk band gap 3.2eV. The absorption peak observed around 360nm indicated that the prepared nanoparticles could be used for UV ray protection.

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