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Synthesis and Characterization of TIO_2 Nano Powder Using SOL-GEL Method

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Abstract: The titanium dioxide (TiO_2) powder was prepared by Sol-Gel method and characterized with different temperature. TiO_2 powder were prepared using $TiCl_4$ solution added in deionized water in very low temperature around 10^oC and C_2H_5OH was added with vigorous stirring for a hour at 30^oC temperature. At 150^oC the procured gel solution was dried using oven at for 6 hours. Then, the dried gel was processed at different calcinations temperature using furnace for 5 hours each. The synthesized TiO_2 nano powders were characterized by XRD, Transmission electron microscope (TEM), Energy dispersive spectroscopy (EDS).XRD pattern and TEM result shows TiO_2 nano powders are in high crystalline structure with high purity. The particles size of the synthesized TiO_2 nano powders were calculated from XRD result. The EDS results show the concentration of titanium and chlorine were decreased due to increasing the processing temperature. But the concentration of oxygen were increased which is qualitatively confirmed with energy dispersive x-ray spectroscopy results.
Keywords: Titanium dioxide (TiO_2), SolGel Synthesis, X-ray Diffraction, Spectroscopy.

I. INTRODUCTION

More attenuation has been given by the researcher on nano materials due the unusual chemical, mechanical, optical, electrical and magnetic properties compared with traditional materials [1].Nano materials have a wide range of applications. Nano materials add incredibly role in semiconductors world. Most nano products produced on an industrial scale are nano materials, although they also arise as by products in the manufacture of medicine [2].Titanium dioxide nano particles has attracted great attention in the fields of environmental purification, solar energy cells, photocatalysts, gas sensors, photo electrodes and electronic devices due to their unique technological properties and applications [3].

Titanium dioxide exhibits both crystalline and amorphous structure and mainly exposes the three crystalline polymorphos structure are namely Anatase, Rutile and Brookite. Brookite shows orthorhombic structure and Anatase and Rutile shows tetragonal structure [4].

There are many methods for preparing nano particles of TiO_2 sol-gel method [5], ball milling [6], chemical vapour deposition [7] and microemulsion [8]. A number of methods are reported for preparation of nano crystallite titania, most of the methods are belonging to wet chemical method. A. Muramastu et al discussed the benefits of the wet chemical method [9]. In wet chemical methods hydrothermal [10, 11] and sol–gel [12] are the best methods. For synthesizing TiO_2 nano particles in Sol–gel method is economical, easy and most often used method. In sol–gel method TiO_2 nano particles are synthesis with different morphologies like sheets, tubes, particles, wires, rods.

The modern technology needs very small presided particles for develop microwave devices and components. The researchers took lot of effect to fabrication and characterization of nano-sized dielectric materials [13]. In particular sensing devices like gas and temperature [14], photo-catalytic devices [15] and photoelectric devices [16] TiO_2commonly utilized. For the fabrication of capacitors in microelectronic devices TiO_2 gotenticed attention because of its unusual dielectric constant [17], due to difficult fabrication process its acquired less attraction. The good dielectric permittivity and very low loss factors give a wide frequency range to develop electronic memories optical filters and capacitors the modified dielectric properties were utilized [18].

Synthesis of TiO_2 sol-gel method is an efficient and easy method [19]. Since in this method the solution, tailoring of certain structural characteristics such as homogeneity, grain size and morphology and porosityis are possible. The uniform distributions of particles are important for optimal control of grain size and micro structure to maintain reliability [20].

This paper reports the effect of temperature on the properties of TiO_2 nano materials, synthesis and to characterize the prepared samples using techniques like powder X-Ray Diffraction (XRD) and Transmission Electron Microscope (TEM) to study percentage yield of element, the crystal structure, chemical analysis and grain size properties.

II. METHODS

A. Chemicals

$TiCl_4$ (MW 200 g/mol, 99.9%), Ethanol, C_2H_5OH (MW 65 g/mol, 99.8%), $AgNO_3$ (MW 170 g/mol, 98.4%), Ammonium hydroxide, NH_4OH(MW, 20 g/mol, 25%) chemicals were used for this study.

B. Method of Preparation of TiO2 Nano powder

Titanium tetra chloride ($TiCl_4$) of 5 ml was added to 100 ml deionized water in ice bath and the process was done under fume hood followed by the addition of 35 ml of ethanol with vigorous stirring for 60 min at 30^{o}C. Slowly drops of ammonium hydroxide were added into solution to neutralize it and precipitate was obtained. After stirring vigorously, the solution was made to settle for a day. The processed precipitates were centrifuged. To remove the chloride ions the obtained precipitate were washed with deionized water until and was centrifugally separated. The precipitate was dried at 150°C using electrical oven to remove part of the absorbed water for 6 hours and lastly amorphous TiO_2 was obtained. Finally the TiO_2 nano powder were obtained due to calcinated processes at different temperatures of 150°C, 350°C, and 550°C for 5 hours step by step [8, 21].

III. RESULTS AND DISCUSSION

A. Powder Preparation of TiO_2 Nanomaterials

Exothermic reactions were observed when 5 ml of $TiCl_4$ solution was added to 100 ml of deionized water under fume hood. While adding ammonium hydroxide a white precipitate was observed and the yellow gel rose obtained. The formation of $Ti(OH)_4$were indicated when gel being yellow in colour. The gel was stirred using magnetic stirrer for an hour and allowed to settle for a day precipitation were formed and the precipitate was centrifuged. The centrifuged precipitations were dried at 150^{o} C for 6 hours, which leads to form white amorphous $TiO_2$when cooling to room temperature. The product was placed in oven at 150°C, 350°C and 550°C for 5 hours to calcinate. Due to calcination process white TiO_2were formed. The products were cooled to room temperature and crushed using agate mortar. Finally, physical grinding method is adopted in this method to reduce the size of TiO_2 powder. The product material was taken into a grinding machine which having rotator with high rpm and it grinded and crushed well for 30 minutes with precaution to avoid any contamination. Thus the powdered materials subjected to different calcination temperatures of 150°C, 350°C and 550°C respectively.

B. Powder X-Ray Diffraction (XRD) Characterization of TiO_2 Nanomaterials

The particles size, crystal structure crystal orientation and lattice parameter are identified using Powder X-Ray Diffraction (XRD). The XRD analysis of the processed samples of TiO_2 nano powder were characterized using a Bruker make diffractometer, Cu-Kα X-rays of wavelength (λ)=1.5406 Å and data was taken for the 2θ range of 10° to 70° with a step of 0.1972°. The results confirmed the nano sized powder TiO_2.

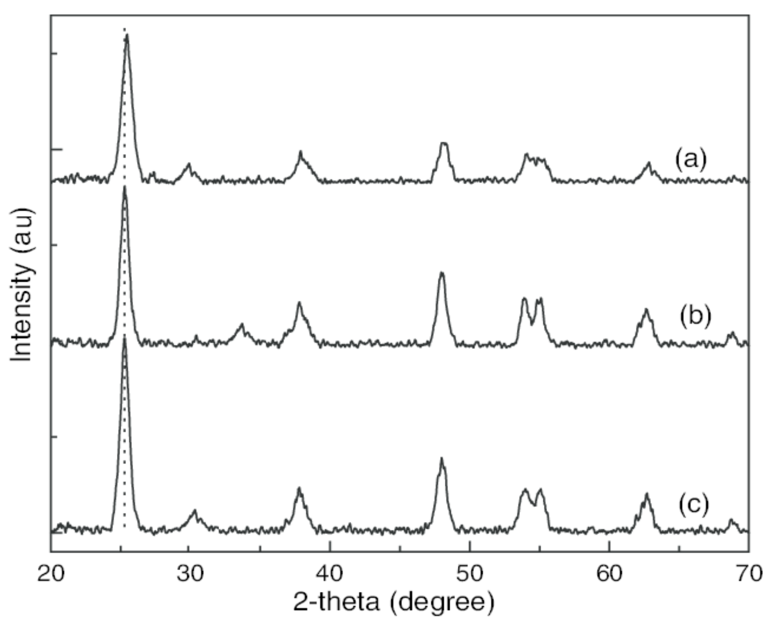

Figure 1. The XRD pattern of TiO2 nano powders calcinations temperatures at (a) 150°C, (b) 350°C and (c) 550°C

From the Figure 1 the XRD peaks in the angle range of $20^o \leq 2\theta \leq 70^o$ determined that peaks from 150°C are 25.4°, 31.6°, 37.9°, 38.52°, 48.01°, 53.89°, 55.03°, 63.64°, 69.89°, the peaks for 350°C are 25.4°, 34.56°, 38.52°, 48.01°, 53.89°, 55.03°, 63.64°, 68.7°, 69.89° and the peaks for 550°C are 25.4°, 31.6°, 37.9°, 38.52°, 48.01°, 53.89°, 55.03°, 63.64°, 69.89° were observed. Among the XRD peaks, the width of 25.4° and 48.01° are useful peak since it has high intensity which is used to determine the crystals size. The two peak values confirm its anatase phase. The highest intensity was achieved for 25.4°at temperature of 550°C and lowest intensity of 25.4°was achieved at a temperature of 150°C.

Average particle size and Inter-planar spacing between atoms (d-spacing) was calculated using Debye-Scherer formula[22] and Bragg's Law and listed in Table 1.

$$D = \frac{0.9\,\lambda}{\beta \cos\theta} \text{-------------- (1)}$$

$$2d \sin\theta = n\lambda \text{----------- (2)}$$

Where, λ is wave length of X-Ray (0.1540 nm)

β is FWHM (full width at half maximum)

θ is diffraction angle

D is particle diameter size.

d is d-spacing

Table 1. XRD Data of TiO_2 Nano powder calcinations temperature at 550°C

2θ	θ	Cos θ	Sin θ	FWHM (°)	FWHM Radian	β cos θ	Size nm	d-spacing
25.4	12.7	0.97553	0.21986	0.109	0.0019024	0.001856	75	3.50223
31.6	15.8	0.96221	0.27228	0.105	0.0018326	0.001763	79	2.82797
37.9	18.95	0.94580	0.32474	0.121	0.0021118	0.001997	69	2.37113
38.52	19.26	0.94403	0.32985	0.128	0.0022340	0.002109	66	2.33439
48.01	24.005	0.91350	0.40681	0.107	0.0018675	0.001706	81	1.89278
53.89	26.945	0.89144	0.44846	0.122	0.0021293	0.001898	73	1.71699
55.03	27.515	0.88688	0.46198	0.114	0.0019897	0.001765	79	1.66674
63.64	31.82	0.84970	0.52725	0.141	0.0024609	0.002091	66	1.46041
69.89	34.945	0.81970	0.57278	0.136	0.0023736	0.001946	71	1.34432

Table 2. Crystallite size (d) of the TiO2 nano powder calcination temperatures at 250°C, 400°C and 600°C

Calcinations temperature of samples °C	2θ degree	β degree	Crystalline size nm
150	25.4	0.224943	36
350	25.4	0.167647	49
550	25.4	0.109	75

From the Figure 1 one can understand the intensity of the diffraction signal increases with increasing the calcination temperature. This indicates that the concentration of the particles increases. The Table 2 and Figure 1shows increasing particles size has direct relation to calcination temperature and inverse relation to FWHM. The sharp peaks in XRD pattern show the crystallinity and purity of TiO_2nanomaterial.

C. Transmission Electron Microscope (TEM)

Transmission electron microscope (TEM) was used to study the crystal structure, morphology, shapes and particle size. For TEM imaging with a JEM-1200EX TEM, the TiO_2 particles prepared by calcining were dispersed in anhydrous ethanol by an ultrasonic process and a drop of the suspension was placed onto a carbon coated copper grid. The excess liquid was removed using a paper wick and the deposit was dried in air prior to imaging. The size and morphology of TiO_2 particles were obtained from the TEM micrograph.

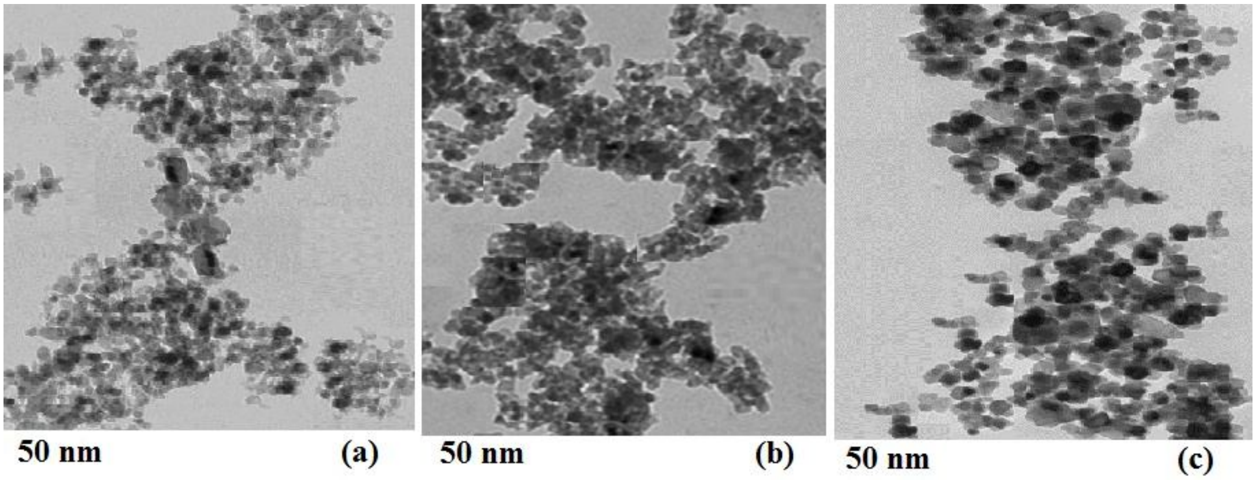

Figure 2. The TEM images of the TiO_2 nano powders calcinations temperatures at (a) 150°C, (b) 350°C and (c) 550°C

Figure 2 clearly showed that the calcinations temperatures influence the particle size, polygonal structure and shapes of TiO_2 nano powders. While increasing the calcination temperatures the shapes of the TiO_2 nano materials leads to spherical. As observed from figure 2 (a) of TEM TiO_2 nano materials calcination at temperature of 150^{o}C, consists of particles with unfortunate fusion and aggregation. Again, observed from figure 2 (a) and 2 (b) when the calcinations temperature of 150^{o}C the size of the nanoparticles uniform compare with calcination temperatures of 350^{o}C. As the calcination temperature increased to 550^{o}C, the sizes of particle are no quite uniform compared with calcinations temperatures of 150^{o}C and 350^{o}C as observed from figure 2 (c). It was learnt from figure 2 that, an increase in calcination temperatures leads to increase particle size, particles fusion and aggregation.

IV. CONCLUSION

TiO2 nano powders were synthesized using the convent way of synthesizing method known as sol gel method. In This method have various advantages over other methods, such as co-precipitation, molecular scale mixing, and high purity of the precursors and homogeneity of solgel products with high purity physical, morphological and chemical properties. XRD results showed that when the calcinations temperatures increase, the FWHM decrease and the particles size increase for the three selected temperatures the interesting peak value (2θ) are the same (25.4^o). As observed from the result of TEM and XRD, in this work, pure anatase titanium dioxide was also obtained. The synthesized TiO_2 nano powder average particle sizes obtained from TEM micrographs with calcinations temperatures at 150^{o}C, 350^{o}C, and 550^{o}C were found to be 36 nm, 49 nm, and 75 nm respectively. In general, it has been observed from XRD and TEM, when the calcinations temperature increases, the sizeof TiO_2 nano powder increases.

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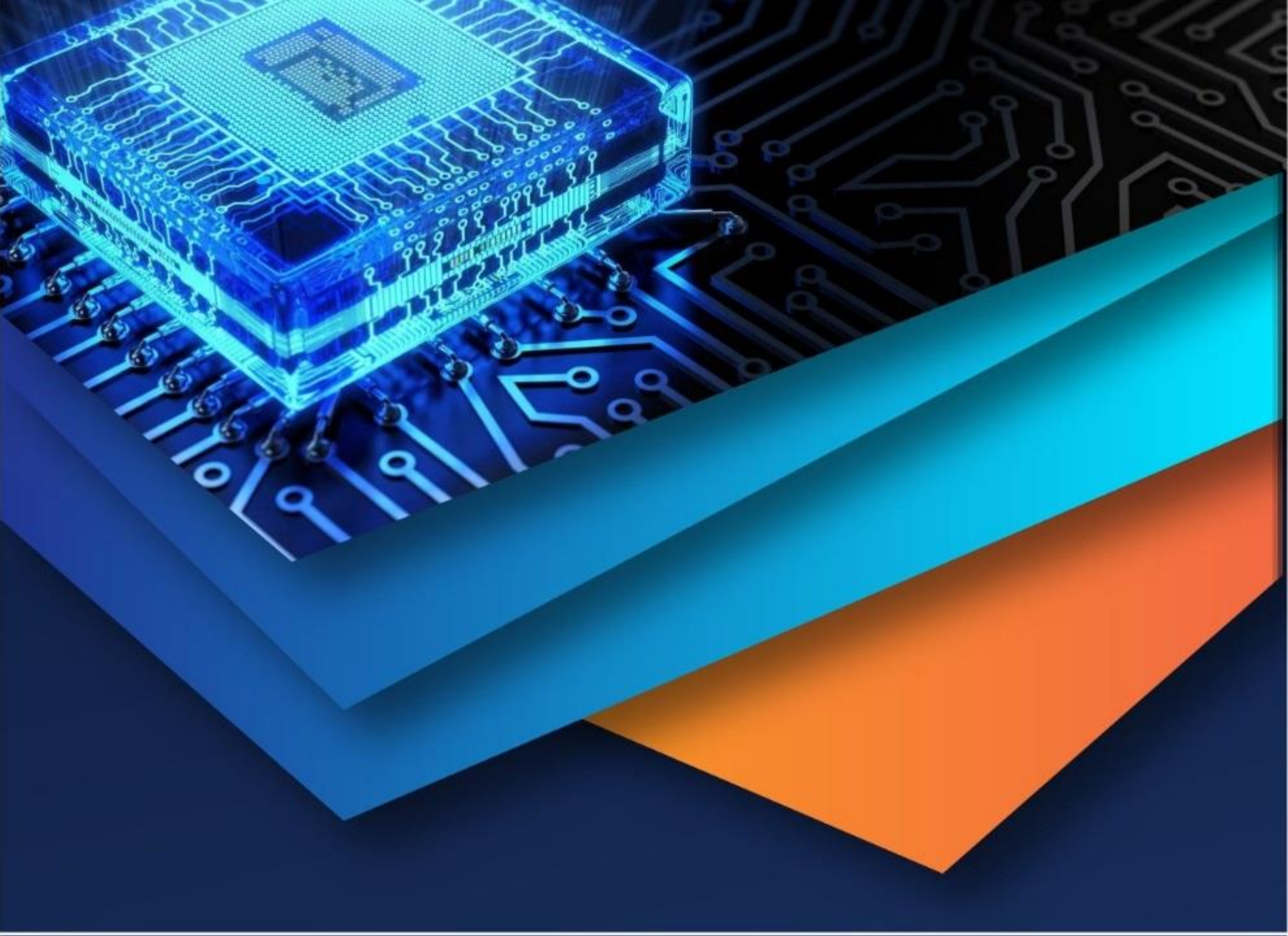

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