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Synthesis and Characterization of Titanium Dioxide TiO_2

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Abstract— The pure TiO_2 nano particles have been prepared by sol gel method using surfactant. The anatase phase of TiO_2 has been confirmed using XRD. The typical value of the crystallite size was calculated for the TiO_2 powder from XRD pattern is ~12nm.Transmission Electron Microscopy has been used to the nano particle size of the TiO_2 nano particles.The Scanning Electron Microscope reveals the morphology of the pure TiO_2 nano particles.

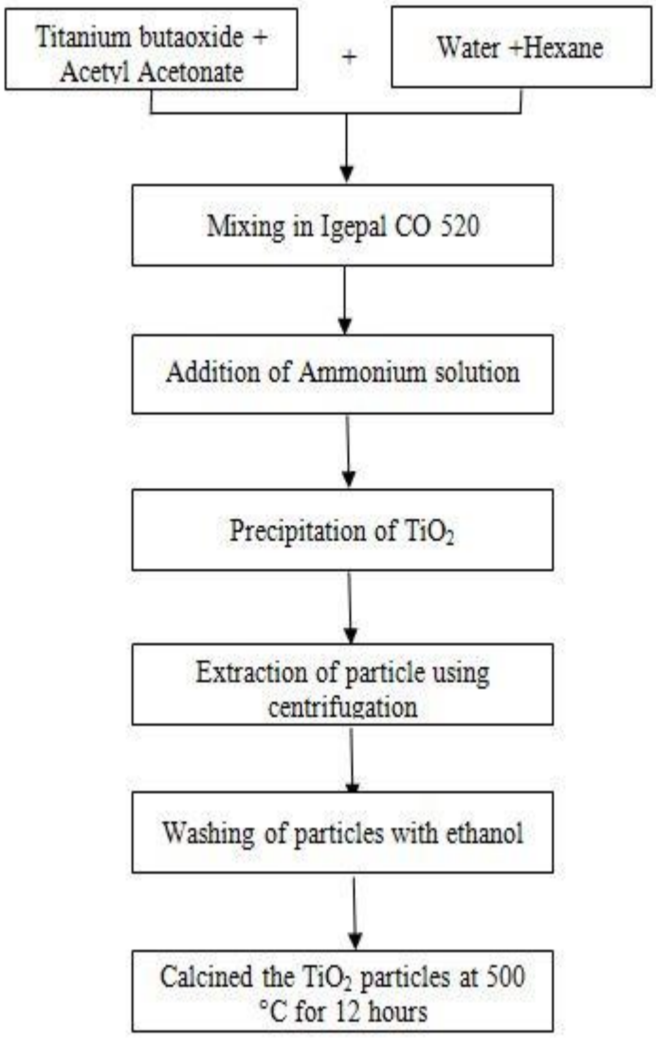
Keywords— *TiO_2, SEM, Characterization, Synthesis*

I. INTRODUCTION

In the last decade, the photovoltaic devices have been fabricated using conventional semiconductor materials such as silicon (Si) [1]. Dye-sensitized solar cells (DSSCs) are attractive alternative to Si-based solar cells as they can be inexpensive, portable, thermally stable, light in weight and flexible [2-4]. In general, there are three major components of DSSCs: an n-type semiconductor, a sensitizer (i.e. dye), and a redox electrolyte [5-6]. TiO_2 is the one of the most commonly used n-type semiconductor with an energy band gap of 3.2 e V [7]. Because of its high brightness, it is commonly used as pigments for paints, coatings, papers and plastics. The dielectric constant and refractive index of TiO_2 is very high which makes it an excellent optical coating or dopant for dielectric materials. In these days, nanosized TiO_2 has attracted much attention because of its wide applications as a base material for dye-sensitized solar cells, photo catalyst and sensors [8-9]. Different nanostructured TiO_2 have been utilized as photo anodes to fabricate DSSCs, including one dimensional (1D) nano-rods, two dimensional (2D) nanotubes and three dimensional (3D) materials. Furthermore, TiO_2 nano crystals are non-toxic materials and commonly used in biological applications [10]. To enhance the chemical and physical properties of TiO_2, some additives or modifiers are applied as coatings on the pure nanosized TiO_2. These additives generate more active photo catalytic sites in TiO_2 and increase the thermal stability of nanosized TiO_2 through the realization of high specific area, enhanced interface quality, and fast electron transport [11-12]. These applications of nanosized TiO_2 are basically determined by its physico-chemical properties such as structure of crystal, grain size, surface to volume ratio, porosity and thermal stability. Furthermore these physic-chemical properties depending upon the different synthesize techniques [13]. Sol gel process is a most commonly used technique to synthesize nano particles of TiO_2. This is very simple means of synthesizing the nano particles at room temperature under atmosphere pressure. Surface modification of TiO_2 semiconductor particles has attracted potential because of the application of these materials in electro chromic and photochromic devices and dye-sensitized solar cells [14-15].

II. EXPERIMENT PROCEDURE

The TiO_2 nano particles has been prepared using sol gel method. The flow chart of the synthesis is as follows.



III. RESULTS AND DISCUSSIONS

The X-ray diffraction is an important tool to analyze the crystal structure of the materials. The X-Ray diffraction pattern of TiO_2 is shown in fig 1.1. The XRD pattern clearly reveals the crystalline nature of the sample. All the peaks in TiO_2 nanoparticles are indexed according to the Anatase phase of TiO_2.

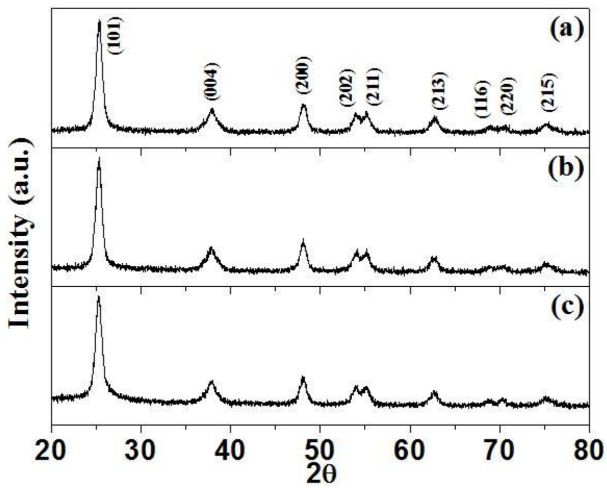


Fig. 1: X-Ray diffraction patterns for the pure TiO_2 nano particles, spinach dye coated nano particles and marry gold flower TiO_2 nano particles using Cu kα radiations

No secondary phase has been detected in the XRD pattern. The crystal structure of the sample is tetragonal with large "c/a" ratio. All the peeks of the above diffraction pattern were indexed using space group $I4_1/amd$ (space group number 141). The crystallite size of the TiO_2 powder has been calculated using Scherer equation.

$$\tau = K\lambda/\beta \cos\theta$$

Where τ is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the grain size. K is a dimensionless **shape factor**, with a value close to unity. The shape factor has a typical value of about 0.9, but varies with the actual shape of the crystallite. λ is the X-ray wavelength. β is the line broadening at half the maximum intensity (FWHM), after subtracting the instrumental line broadening, in radians. θ is the Bragg angle. The typical value of the crystallite size was calculated for the TiO_2 powder from XRD pattern is ~12nm.

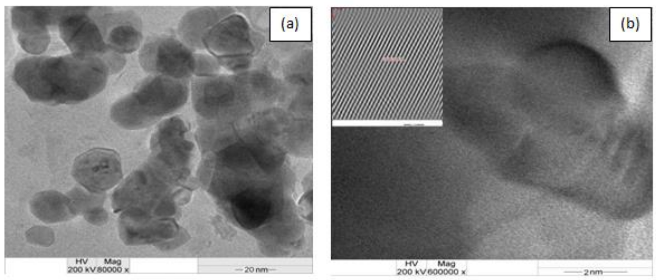


Fig.2: (a) TEM image of the TiO_2 nano particles. The average size of the particle is found to be ~17 nm. (b) HRTEM image of the TiO_2 particle clearly evident the crystal planes. The FFT image of the crystal planes (inset).

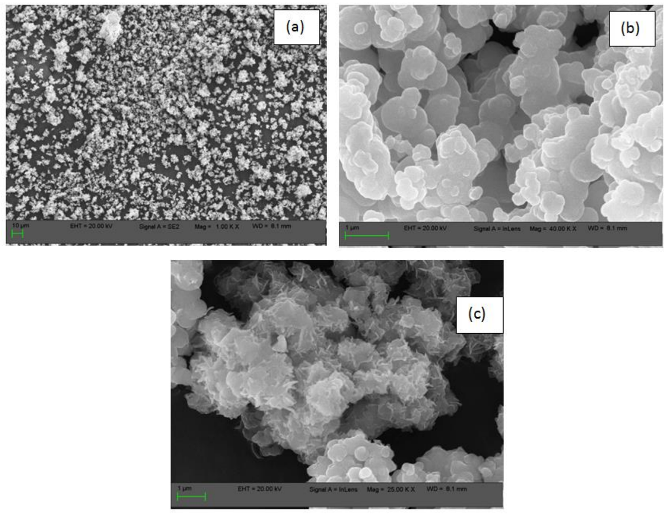


Fig. 3: The SEM micrographs for the pure TiO2 powder at various magnifications.

The transmission electron micrograph of the TiO_2 particles has been observed (Fig 2 (a)). It is clearly evident from the image that the uniform particles has been formed by this synthesis method. The average particle size is found to be ~ 17 nm. HRTEM image of the particle has been taken at very high magnification ~ 6,00,000 X. The fringes are clearly seen from the figure2 (b).

The FF transform of the fringes were done to find out the distance between the fringes. The distance of one black fringe is ~0.099nm. Which is in good agreement with the XRD data. The morphology of the powder samples was studied using Scanning electron Microscope (Carl Zeiss, Germany). The micrographs were taken by spreading the powder particles on the carbon tape. The micrographs was taken at various magnifications and at different locations to analyze the same extensively. Fig. 3 shows various micrographs. Fig. 3 (a) shows the morphology of the samples at low magnification ~1000 X. The overview of the sample shows that the morphology is same and uniform. As the magnification was increased two different kind of morphologies are observed. The figures 3(b) and (c) show both type of morphologies. In the sample the morphology which is shown in fig.3(c) is predominant in the sample as compared to the fig. 3(b). So it can be concluded that there may be mixing of rutile and anatase phase of the TiO_2 in the sample. Due to very less amount of the rutile phase the Anatase phase is clearly evident in the XRD whereas some cluster of the particles show rutile phase formation on SEM micrographs.

IV. CONCLUSION

The pure TiO_2 nano particles are synthesized by Sol gel method. The pure TiO_2 nano particles are found of the size of ~11nm - 20 nm. The XRD pattern clearly reveals the crystalline nature of the sample. All the peaks in TiO_2 nanoparticles are indexed according to the Anatase phase of TiO_2.

V. REFERENCES

[1] Green MA, Emery K, Hisikawa Y, Warta W, Solar cell efficiency tables (version 30). Prog Photovolt Res Appl Prog Photovolt 15:425-430 (2007).

[2] Huynh WU, Dittmer JJ, Alivisatos AP, Hybrid nanorod-polymer solar cells. Science 295:2425-2427 (2002).

[3] O'Regan B, Gratzel M, A low-cost, high efficiency solar cell based on dye-sensitized colloidal TiO2 films, Nature 353:737-740 (1991).

[4] Kim Y, Cook S, Tuladhar SM, Choulis SA, Nelson J, Durrant JR, Wang R, Hashimoto K, Fujishima A, Chikuni M, Kojima E, Kitamura A, Yoshida M, Prasad PN, Sol gel processed SiO2/TiO2/poly(vinylpyrrolidone) composite materials for optical wavegiudes. Chem Mater 8:235-241 (1996).

[5] Xin X, Scheiner M, Ye M, Lin Z, Surface –Treated TiO2 Nanoparticles for Dye-Sensitized Solar Cells with Remarkably Enhanced Performance, Langmuir 27: 14594-14598 (2001).

[6] Xin X, He M, Han W, Jang J, Lin Z, Angew, Chem., Int.Ed., doi:10.1002/anie.201104786 (2001)

[7] Gratzel M, Nature 414:338-344 (2001).

[8] Chau JLH, Lin YM, Li AK, Su WF, Chang KS, Hsu SLC, Li TL, Transparent high refractive index nanocomposite thin films. Mater Lett 61:2908-2910 (2007).

[9] Duncan WR, Prezdho OV, Theoretical studies of photo induced electron transfer in dye-sensitized TiO2, Annu Rev Phys Chem 58:143-184 (2007).

[10] Zhang J,Wang X, Zheng WT, Kong XG, Sun YJ, Wang X, Mater Lett 61:1658 (2007).

[11] Krebs FC, Biancardo M, Solar Energy Mater. Solar Cells 90:142-165 (2006).

[12] Yan KY, Qiu YC, Chen W, Zhang M, Yang SH, Energy Environ. Sci. 4:2168-2176 (2011).

[13] Zhang WF, He YL, Zhang MS, Yin Z, Chen Q, J Phys Chem B 110:927 (2000).

[14] Venkatachalam N, Palanicharmy M, Arabindoo B, Murugesan V, Mater Chem Phys 104: 454 (2007).

[15] Gouadec G, Colomban P, Prog Cryst Growth Char Mater 53:1 (2007).