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XRD analysis of undoped and Fe doped TiO_2 nanoparticles by Williamson Hall Method

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Abstract: Undoped and Fe doped titanium dioxide (TiO_2) nanoparticles were synthesized by sol-gel method at room temperature. The synthesized samples were annealed at 500^{o}C. For structural analysis, the prepared samples were characterized by X-ray diffraction (XRD). The crystallite size of TiO_2 and Fe doped TiO_2 nanoparticles were calculated by Scherer's formula, and was found to be 15 nm and 11 nm, respectively. Reduction in crystallite size of TiO_2 with Fe doping was observed. The anatase phase of Fe-doped TiO_2 nanoparticles was also confirmed by X-ray diffraction. By using Williamson-Hall method, lattice strain and crystallite size were also calculated. Williamson–Hall plot indicates the presence of compressive strain for TiO_2 and tensile strain for Fe-TiO_2 nanoparticles annealed at 500^{o}C.

Keywords: nanostructured materials, chemical synthesis, X – ray diffraction, W – H analysis.

INTRODUCTION

Nano crystalline transition metal oxides have attracted considerable attention due to their potential application. Among them TiO_2 attracted precise regard on account of its exceptional properties such as low cost, high stability, high chemical inertness, biocompatibility, non toxicity etc. [1-3]. TiO_2 has wide range of application including air pollution, water treatment, antibacterial and self-cleaning due to its effective photocatalytic activity.The applications of TiO_2 strongly depend on the high homogeneity and definite phase composition, particle size, high surface area and porosity. Moreover, in the evaluation of thermal stability and mechanical properties of nanocrystalline TiO_2, grain size, surface free energy, activation energy of phase transformation and initial strain are very important factors. TiO_2 is a wide band gap semiconductor (3.2ev), it can only absorb UV light (6387 nm), which accounts for only a small part (3–5%) of solar energy. Many attempts have been made to improve the photocatalytic activity of TiO_2 under visible-light irradiation such as doping with transition metal (Fe, Co, Ni, Cr, Mn etc). Iron has been considered an appropriate candidate, owing to the radius of Fe^{3+} (0.64 Å) being similar to that of Ti^{4+} (0.68Å). Therefore, it can be inferred that Fe ions might easily be incorporated with the crystal lattice of TiO_2. Deviations from perfect crystallinity extend infinitely in all directions, leads to broadening of the diffraction peaks. Size and lattice strain are the two main properties which could be extracted from the peak width analysis. W-H analysis is an integral breadth method; using this method in this present study we have calculated strain and crystallite size of $TiO_2$and Fe-TiO_2 nanoparticles.Various methods have been reported in literature to synthesize nanocrystalline TiO_2. In this paper we have adopted sol gel method for the formation of TiO_2 nanoparticles. The advantages of sol gel approach in the formation of nanoparticles fabrication are the simple processing steps, flexibility of solution chemistry, low temperature treatment etc.

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EXPERIMENTAL SECTION

TiO_2 nanoparticles were prepared by sol-gel method using titanium isopropoxide [$Ti(OC_3H_7)_4$] as a precursor and ethanol (C_2H_5OH) as a solvent at room temperature. 6.5 ml of $Ti(OC_3H_7)_4$ were dissolved in 60 ml of C_2H_5OH. The pH of the above solution was adjusted by adding NH_4 which was stirred for 30 min and after that 3 ml of distilled water was added in the above solution with continuous stirring for 1 hour. In proceeding method, a gel was formed which was dried at 80^{o}C.

The final powder was annealed at 500^{o}C for 1 hour. Fe doped TiO_2 nanoparticles were also formed following the stated method by adding 0.05%of iron chloride ($FeCl_3$).

RESULTS AND DISCUSSION

The XRD spectra gives information about structure, crystallite size and lattice strain. Fig. 1 shows the XRD spectra of TiO_2 and Fe-TiO_2 nanoparticles.

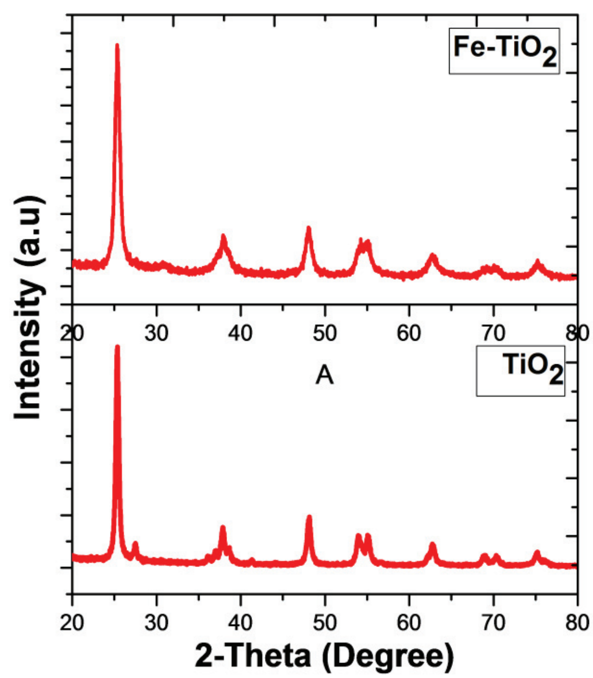

FIGURE 1. XRD Spectra of TiO_2 and Fe doped TiO_2 nanoparticles

All the diffraction peaks are assigned to tetragonal anatase crystalline phase ofTiO_2. The crystalline size (D) of TiO_2 and Fe doped TiO_2 nanoparticles were calculated from the broadening of the diffraction line using Debye Scherrer's formula i.e. $D = 0.9\lambda/ \beta\cos\theta$, where λ is the wavelength of the X-rays, β is the full width at the half maximum intensity (FWHM) [4].The calculated crystallite size for TiO_2 and Fe doped TiO_2 were 15 nm and 11nm, respectively as shown in Table 1. Due to very low Fe content, any crystalline phase containing Fe could not be observed by XRD in Fe doped TiO_2. Also, the similar ionic radii, Fe^{3+}can easily substitute Ti^{4+}into TiO_2 lattice. These results support that the doping of Fe shows uniform distribution of the dopant, forming stable solid solution within TiO_2. The diffraction pattern also confirms that the materials prepared are in the form of small particles, as the peaks are broad.

TABLE1. Structural parameter for TiO_2 and Fe-Doped TiO_2 nanoparticles

Sample name	Scherrer's method				Williamson Hall Method	
	D (nm)	**d(Å)**	**MI**	**δ ($\times 10^{-9} m^{-2}$)**	**D(nm)**	**ε**
TiO_2	15	3.515	0.005	0.004	16	-0.19
Fe-TiO_2	11	3.516	0.005	0.008	14	0.13

It can also be concluded from the Schererr's equation that doping iron ion with proper contentdecreases the crystal size. These doping modifications may prevent particles agglomeration, forming well defined Nano crystalline powder with high surface area. It demonstrates that the addition of iron ions will restrains the growth of TiO2 crystallites.

Williamson–Hall plot provide information about lattice strain and effective crystallite size. Using Williamson–Hall plot, we have calculated the lattice strain and effective particle size through the following relation:

$$\beta\cos\theta = (k\lambda/D) + 4\varepsilon \sin\theta \qquad (1)$$

Fig. 2 shows the Williamson–Hall plot for TiO_2 and Fe-TiO_2

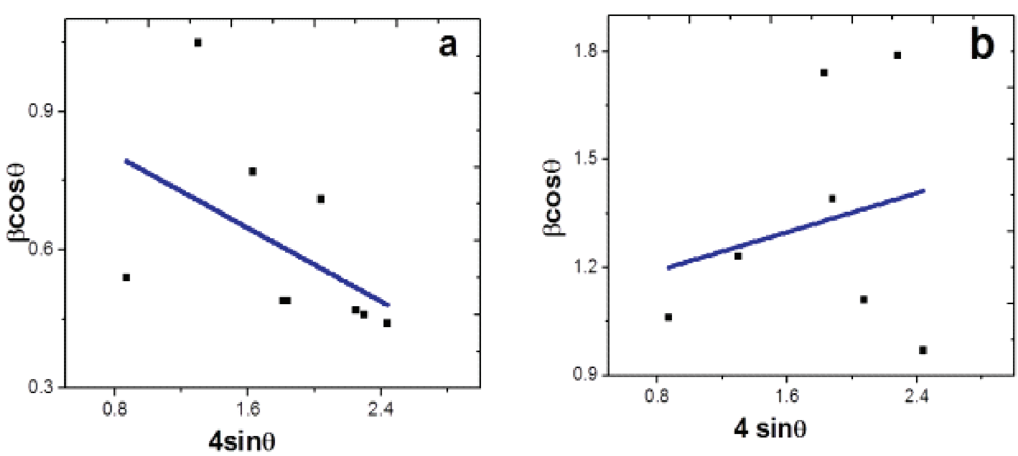

FIGURE2. Williamson-Hall Plot (a) TiO_2 (b) Fe-TiO_2

It is plotted with $\sin\theta$ on the x-axis and $\beta\cos\theta$ on the y-axis. A linear fit is got for the data and form it; particles size and strain are extracted from y intercept and slope respectively. It has been reported in literature that a negative slope in the plot indicate the presence of compressive strain [5], whereas the appearance of positive slope indicate the possibility of tensile strain [6]. It has been observed from the above data that negative slope for TiO_2 reveals the presence of compressive strain and positive slope reveals the presence of tensile strain as shown in Table 1.

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CONCLUSION

Undoped and Fe-TiO_2 nanoparticles were synthesized by sol gel method. The crystallite size of prepared sample was calculated from the Scherrer's formula i.e 15 nm and 11 nm for TiO_2 and Fe doped TiO_2. The results showed that the Fe^{3+} doping did not change the crystal structure of TiO_2. The size and strain contributions to the line broadening were analyzed by Williamson Hall method. The results show thepresence of compressive strain for TiO_2 and tensile strain for Fe-TiO_2 nanoparticles annealed at 500°C.

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