

Synthesis Structure Characterization of TiO₂ Doped by Silver Ion

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Abstract

Titanium dioxide (TiO₂), Nano crystalline was synthesized by TiCl₄ solution was slowly added, it is distilled water in an ice bath, and silver ion (Ag⁺) doped on TiO₂ by liquid impregnation method. The obtained products have been characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results indicate that: the crystallite size increases from 17.6 nm for (TiO₂), to 40.7 nm as the silver ion (Ag⁺) doped on TiO₂. The XRD analysis exhibits a retille tetragonal phase of the (TiO₂) Nano crystals but silver ion (Ag⁺) doped on TiO₂ is Orthorhombic. The crystalline is developed due to a decrease of lattice distortion ratio and dislocation density as the silver ion (Ag⁺) doped on TiO₂ is increases. Surface area and d – spacing of (TiO₂) and silver ion (Ag⁺) doped on TiO₂ nanoparticles were measured.

Keywords: Titanium Dioxide, Nano crystalline, silver ion (Ag⁺) doped, XRD, retille, density.

Introduction

Titanium dioxide (TiO₂), a metal oxide semiconductor, has been found to be one of the most effective photo catalysts due to its high efficiency and stability [1]. The strong oxidation and reduction power of photo excited titanium dioxide was realized from the discovery of the Honda-Fujishima effect. In 1972, Fujishima and Honda reported photo induced decomposition of water on TiO₂ electrodes. Since Frank and Bard first examined the possibilities of using TiO₂ to decompose cyanide in water, there has been an increasing interest in environmental applications. Based on these works, TiO₂ photo catalysts are widely used for air purification, deodorization, sterilization, anti-fouling, and mist removal [2, 3]

Although TiO₂ has the advantage of good chemical stability, high activity, absence of toxicity and relative low price [4] however, its band gap is so large ($E_g = 3.20$ eV) that it can only be excited by ultraviolet light with a Wavelength no longer than 387.5 nm, which accounts for only 5% of the incoming solar energy. Thus, it is significant to develop a visible-light driven photo catalyst with high photo catalytic activity or this purpose, an initial approach of doping TiO₂ with transition metals was extensively investigated [5,6] However, the photo catalytic activity of metal doping is impaired by thermal instability and an increase in carrier recombination facilities. Therefore, many researchers have started to use anionic nonmetal Dopants to extend the photo catalytic activity into the visible-light region because the related impurity states are near the valence band edge and do not act as charge carriers. TiO₂ is white, inexpensive, and nontoxic [7]. It is one of the most widely used photo catalysts for disinfection [6, 8]. Since the discovery of the photo catalytic splitting of water on a TiO₂ electrode under ultraviolet (UV) light [9], a great deal of research efforts have been made on semiconductor-based photo catalysts on both energy conversion and environmental applications.

This work Prepare and characterize TiO_2 nanomaterial and use it to treat water, same work done by Kiran Gupta from Department of Chemistry. Investigate the comparative photo catalytic activity of TiO_2 and Ag-doped TiO_2 (visible light active) nanoparticles synthesized by acid-catalyzed sol–gel technique, This work reports the structural and optical properties and comparative photo catalytic activity of TiO_2 and Ag-doped TiO_2 nanoparticles against different bacterial strains under visible-light irradiation ,then found from XRD pattern revealed that the annealed sample of TiO_2 has both anatase and rutil phases while only an anatase phase was found in Ag-doped TiO_2 nanoparticles. The decreased band-gap energy of Ag-doped TiO_2 nanoparticles in comparison to TiO_2 nanoparticles was investigated by UV is spectroscopy. The rate of recombination and transfer behavior of the photo excited electron–hole pairs in the semiconductors was recorded by photoluminescence, in this work we found indicate that the crystallite size increases from 17.6 nm for (TiO_2), to 40.7 nm as the silver ion (Ag^+) doped on TiO_2 . The XRD analysis exhibits a rutil tetragonal phase of the (TiO_2) Nano crystals but silver ion (Ag^+) doped on TiO_2 is Orthorhombic. The crystalline is developed due to a decrease of lattice distortion ratio and dislocation density as the silver ion (Ag^+) doped on TiO_2 is increases. Surface area and d –spacing of (TiO_2) and silver ion (Ag^+) doped on TiO_2 nanoparticles are measured.

Material & Method

Distilled water was used as solvent in the process. The water employed in all preparations was purified by a distilled, Titanium tetrachloride, Silver.

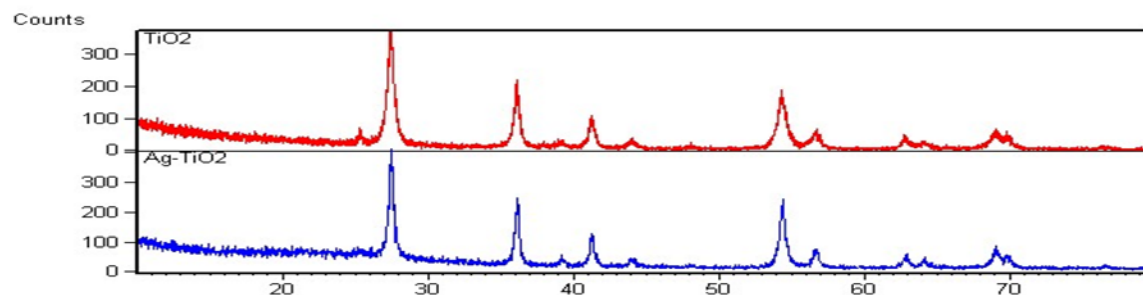
Synthesis of titanium dioxide nanoparticles TiO_2

50 ml of TiCl_4 solution were slowly added to 200 ml of distilled water in an ice bath. After the addition completed, the mixture was stirred for 30 minutes at room temperature. The solution was heated in water bath for 90 minutes under refluxing. Then, it was filtered using vacuum pump and calcined at 600°C in the muffle furnace for 2 hours [10].

Synthesis of Ag- TiO_2 Nanoparticles

Liquid Impregnation Method. In the liquid impregnation method [11] silver ion (Ag^+) doped on TiO_2 was prepared according to the following steps. We prepare 80 g of TiO_2 -Ag nanoparticles; 79.2 g of TiO_2 was added to 500ml of deionizer water. Preparation of silver-doped TiO_2 nanoparticles, 1.7 g of AgNO_3 for doping was added to TiO_2 suspension; the silver concentration was of 1% (mole ratio) versus TiO_2 . The slurry was stirred well for 6 hours and allowed to rest for 24 h and then dried in an air oven at 100°C for 12 h. The dried solids were crushed to fine powder in an agate mortar and calcined at 400°C for 6h in a muffle furnace. In this method the metal gets deposited on the surface of the photo catalyst [12].

Results



Fig(1) the XRD charts of Titanium dioxide (TiO_2) and silver ion (Ag^+) doped on TiO_2 samples

Table (1) the crystallite lattice parameter of Titanium dioxide (TiO₂) and silver ion (Ag⁺) doped on TiO₂ samples

	Titanium dioxide (TiO ₂),	silver ion (Ag ⁺) doped on TiO ₂
2-Theta	27.494	27.362
	36.125	36.027
	41.353	41.191
	54.393	56.546
d-spacing (nm)	0.32414	0.32568
	0.24844	0.24909
	0.21815	0.21897
	0.16854	0.16262
Intensity	108	127
	65	62
	26	29
	60	17
Miller indices (hkl)	1 1 0	1 1 0
	1 0 1	1 0 1
	1 1 1	1 1 1
	2 1 1	7 1 1
Crystallite size (nm)	15.7	21.5
	19.6	27.3
	19.0	23.9
	16.1	40.7
Unit Cell (a b c)	(4.585,4.585,2.964)	(12.869,10.49,3.6638)
(α - β - γ)	α = β = γ = 90 ⁰	α = β = γ = 90 ⁰
density(g/m ²)	4.2592	7.0835
Phase group	Tetragonal	orthorhombic

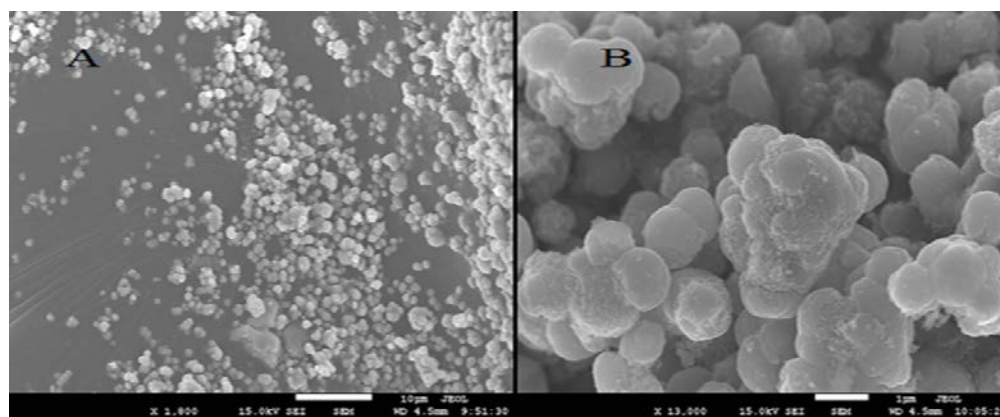


Fig (2) SEM image of TiO₂ (A) at 10 μm and (B) at 1 μm

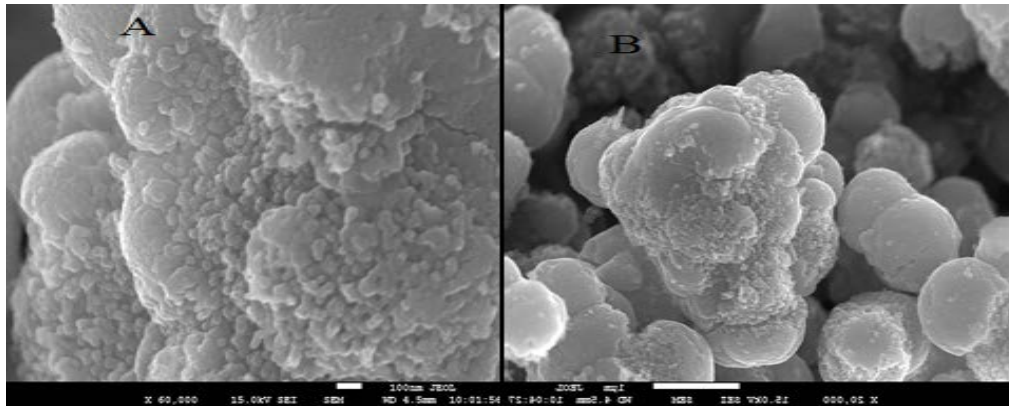


Fig (3) SEM image of TiO₂ (A) at 100 nm and (B) at 1μm

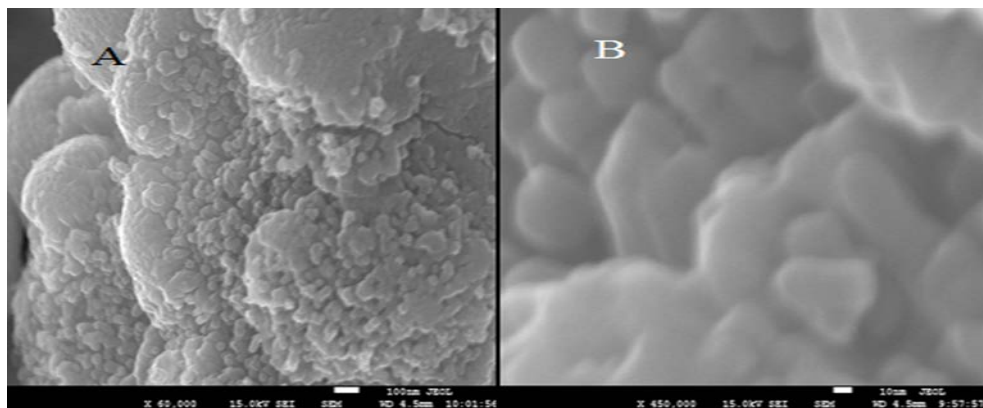


Fig (4) SEM image of TiO₂ (A) at 100 nm and (B) at 10 nm

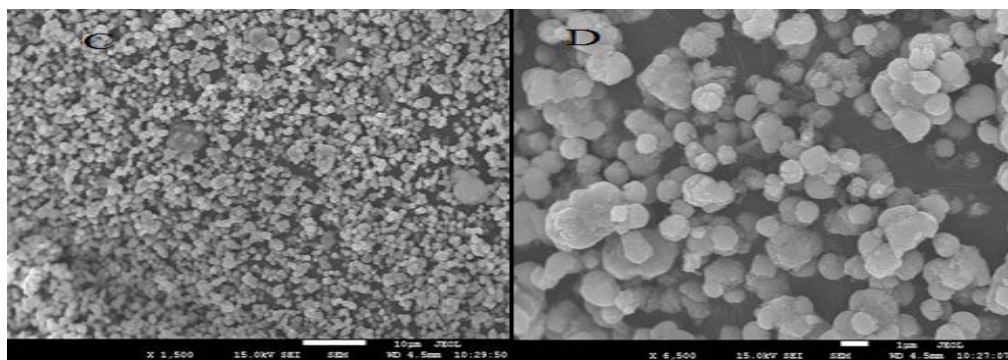


Fig (5) SEM image of Ag TiO₂ (C) at 10 μm and (D) at 1 μm

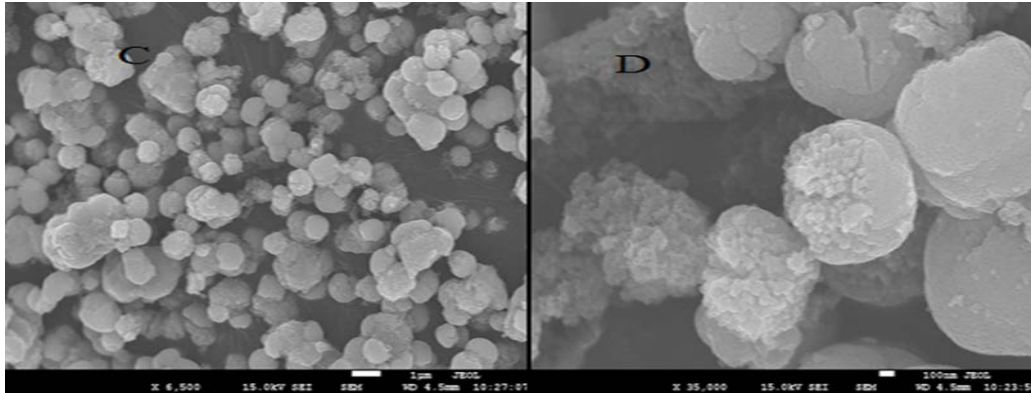


Fig (6) SEM image of Ag TiO₂ (C) at 1 μm and (D) at 100nm

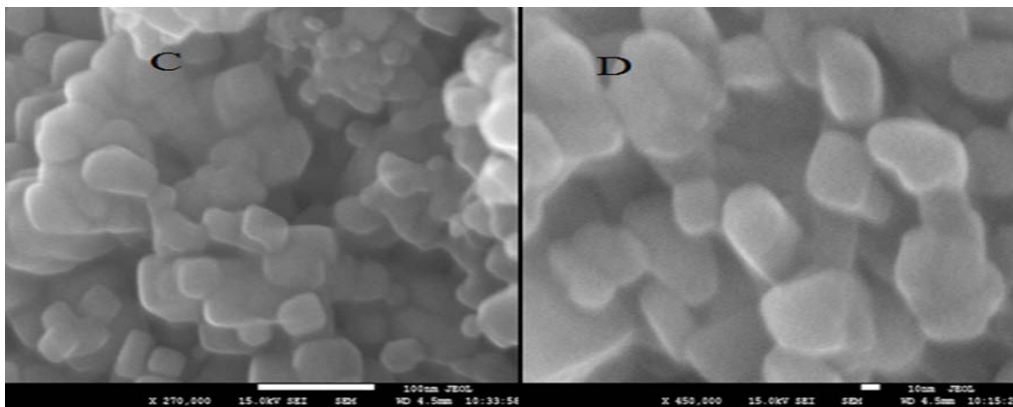


Fig (7) SEM image of Ag TiO₂ (C) at 100nm and (D) at 10nm

Conclusion

The crystal structure of all samples characterized at room temperature using X-ray diffraction (XRD; PANalytical Xpert Pro.) pattern measured with Cu-K α Radiation ($\lambda=1.54178\text{\AA}$) used to examine the surface morphology of particles. Dependence of crystallite size, crystal lattice distortion and dislocation density on the silver ion (Ag⁺) doped: The analysis of the structure and size determination of (TiO₂) and (AgTiO₂) Nano crystals were carried out by using modified Scherer equation in the form [13]

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

where β is defined as $\beta = (\beta^2_{\text{observed}} - \beta^2_{\text{instrumental}})^{1/2}$ (in radians), k is the shape factor equals to 0.89, λ is the X-ray wave length for Cu $k\alpha$ radiation ($\lambda = 1.54178 \text{ \AA}$), θ is the Bragg diffraction angle (in degrees) and β is the full width at half maximum (FWHM) of the observed peak which is corrected by considering the instrumental broadening of the standard (TiO₂). The representative XRD charts, Fig1, of dried Titanium dioxide (TiO₂) and silver ion (Ag⁺) doped on TiO₂ samples. Miller indices provided in the figure and all peaks determine transformation of dried powder to (TiO₂) and (AgTiO₂) crystallites with tetragonal retille crystal structure but for (AgTiO₂) was Orthorhombic. In addition, the number of reflection increases with (AgTiO₂) but the degree of crystalline changes at 56.546° for Ag (TiO₂) indicating fully crystalline (TiO₂)

Nano powders. Table (1) shows the XRD parameters of (TiO₂) and (AgTiO₂) Nano powder at various crystalline orientations. The SEM micrographs of the Titanium dioxide (TiO₂) and silver ion (Ag⁺) doped on TiO₂ samples, are shown from fig (2) to fig(7). All images indicate that the particles have non-uniform size with high degree of agglomeration. The as-dried powder fig (2), fig (3) and fig (4) has a non-uniformed size of particles with wide size distribution for the (TiO₂) sample. After silver ion (Ag⁺) doped, fig (5), fig(6) and fig(7) the morphology of the specimen surface changes obviously with increasing the average particles size and more agglomeration occurs among particles as XRD results. On the other hand, all particles exhibit a spherical shape with a high degree of agglomeration among fine particles. Therefore, one cannot measure the particles size. From the study of Titanium dioxide (TiO₂) and silver ion (Ag⁺) doped on TiO₂ samples nanoparticles the following conclusions can be drawn: The crystallite size is silver ion (Ag⁺) doped dependent. The crystallite size of the samples increased from 17.6 nm for (TiO₂), to 40.7 nm as the silver ion (Ag⁺) doped on TiO₂ respectively. The crystalline of TiO₂ nanoparticles might be improved by decreasing the lattice distortion and dislocation density with increasing silver ion (Ag⁺) doped. The SEM examinations reveal that when the calcinations temperature increases the agglomeration of non-uniform particles increases.

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