

Synthesis TiO₂ Nanoparticles and Apply in Photo Catalysis Application (Water treatment)

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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. The obtained products have been characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results indicate that: the crystallite size average 51.88 nm for (TiO₂) by XRD, and 53.5 nm by SEM. The XRD analysis exhibits a retille tetragonal phase of the (TiO₂) Nano crystals. The effects of TiO₂ different concentration on the Escherichia E- coli water samples after 4 week decrease pH-meter for all samples according to increase the concentration and dyes , also decrease of the number of viable cells when concentration of TiO₂ increase and also decrease by week increase .

Keywords: Titanium Dioxide, Photo Catalysis, X-ray diffraction (XRD), Scanning Electron Microscopy (SEM).

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 for 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 [10-13].

This work Prepare Synthesis TiO₂ nanomaterial and use it to treat water, same work done on the chemistry department and biology department from Al-Neenlen University- Faculty of Science and Technology. Investigate the comparative photo catalytic activity of TiO₂ (visible light active) nanoparticles synthesized by acid catalyzed sol-gel technique.

Material & Method

Synthesis titanium dioxide nanoparticles TiO₂ by sol gel method used 50 ml of Ti Cl₄ 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 claimed at 600°C in the muffle furnace for 2 hours [10]. Then used XRD and ESM spectroscopy to calculated nanoparticles. And apply the sample in bio application to treatment water.

Results

After preparing TiO₂ sample and study the morphology properties by used XRD technique and ESM, apply the sample in bio application to treatment water.

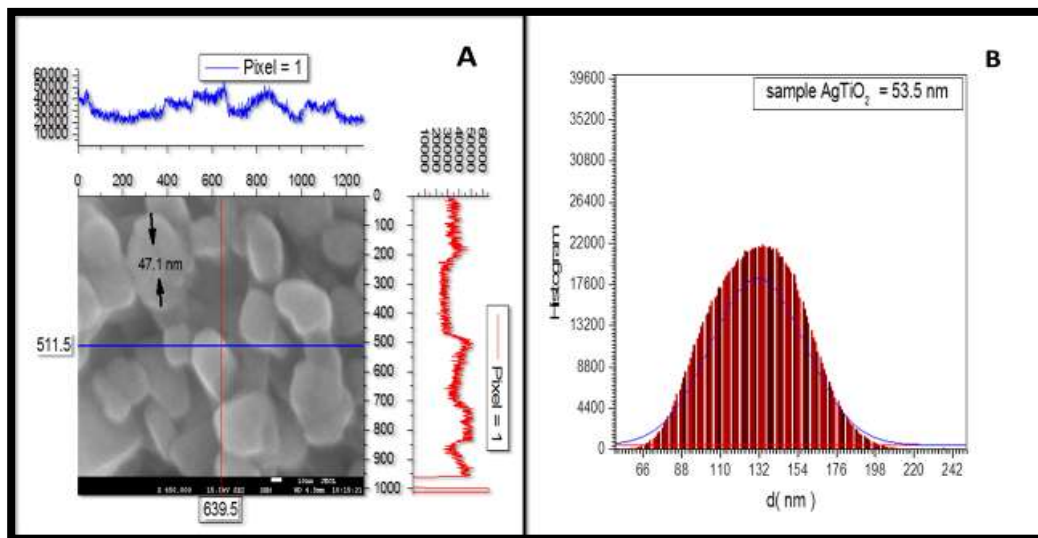


Fig (1) (A) SEM image of TiO₂, (B) Histogram SEM image of TiO₂

The SEM micrographs of the Titanium dioxide (TiO₂) sample, was shown from fig (1-A) and fig (1-B). The images indicate that the particles have non-uniform size with high degree of agglomeration with wide size distribution for the (TiO₂) sample, the morphology of the specimen surface changes obviously with increasing the average particles size and more agglomeration occurs among particles. 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.

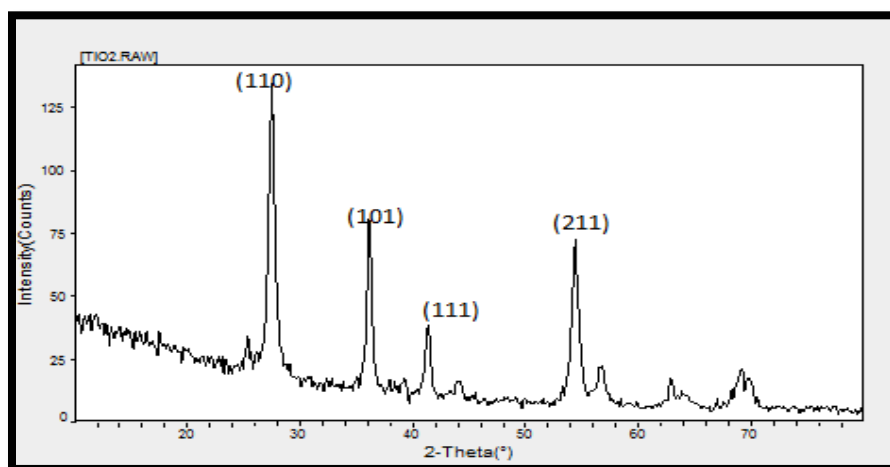


Fig (2) XRD spectrum of Nano TiO₂ sample

Table (1) Calculate Lattice Constants from Peak Locations and Miller Indices [Tetragonal] of TiO₂ sample

2 θ	d (nm)	h k l	X _S (nm)
27.494	3.2458	1 1 0	45.3
36.125	2.4855	1 0 1	48.3
41.353	2.1839	1 1 1	53.6
54.393	1.6805	2 1 1	60.3

Average Lattice Constants = 4.8585

a= b = 4.585 c = 2.964

$\alpha = \beta = \gamma = 90^\circ$

Density = 4.2592 mg.cm⁻³

Crystal Form: Tetragonal – Primitive - Space Group: P42/mm (136)

The crystal structure of all samples characterized at room temperature using a Philips PW1700 X-ray diffract meter (operated at 40 kV and current of 30 mA) and samples were scanned between 10⁰ and 80⁰ at a scanning speed of 0.06 °C/s using Cu K α radiation with $\lambda = 1.5418\text{\AA}$. The representative XRD charts of TiO₂ sample as show in fig (2). Miller indices provided in the figure and all peaks determine transformation of dried powder to TiO₂ crystallites with tetragonal rutile crystal structure. Table (1) shows the XRD parameters of TiO₂ Nano powder at various crystalline orientations. The existence of the (110), (101), (111,) and (211) major lattice planes in the XRD patterns confirms the formation of spinel Tetragonal – Primitive , Miller indices provided in the fig (2) and all peaks determine transformation of dried TiO₂ crystallites with Tetragonal rutile crystal structure. The density of sample equal 4.2592 mg.cm⁻³ and average Lattice Constants equal 4.8585. For the XRD results used the cheyer equation to calculate the particles size, and the average particles size equal 51.88 nm is the approximately matching with that find by SEM.

Escherichia coli Water and TiO2 Results

After Synthesis titanium dioxide nanoparticles TiO₂ by sol gel method, and apply the sample in bio application to treatment water as showing in fig(3) blow and determine the pH-meter of all samples as showing in table (3)



Fig (3) effect of TiO₂ nanoparticles different concentration from 0.1 ppm to 0.8 ppm on the survival of Number of viable cells Escherichia coli (CFU/ml) for 4 Weeks on the water

Table (2) the effect of TiO₂ different concentration from 0.1 ppm to 0.8 ppm on the survival of Number of viable cells Escherichia E- coli (CFU/ml) after 4 Weeks on the water

	First Week	Second Week	Thread Week	Fourths Week
Sample 0	4.75x10 ²	4.21x10 ²	3.83x10 ²	2.95x10 ²
Sample 1	4.64x10 ²	2.63x10 ²	2.24x10 ²	1.95x10 ²
Sample 2	4.52x10 ²	2.41x10 ²	2.12x10 ²	1.83x10 ²
Sample 3	4.48x10 ²	2.21x10 ²	2.05x10 ²	1.73x10 ²
Sample 4	4.29x10 ²	1.93x10 ²	1.81x10 ²	1.52x10 ²

Sample 5	4.13×10^2	1.72×10^2	1.69×10^2	1.46×10^2
Sample 6	4.05×10^2	1.54×10^2	1.34×10^2	1.23×10^2
Sample 7	4.02×10^2	1.36×10^2	1.24×10^2	0.95×10^2

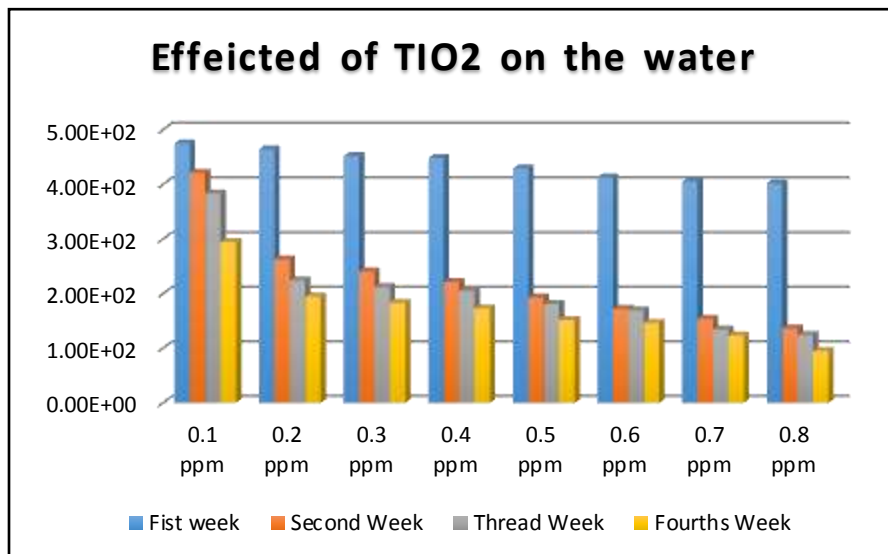


Fig (4) effect of TiO₂ different concentration from 0.1 ppm to 0.8 ppm on the survival of Number of viable cells Escherichia coli (CFU/ml) after 4 Weeks

In this study 8 samples of Escherichia E- coli water effects by different concentration of TiO₂ (0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7 and 0.8) ppm to 4 weeks and measure number of viable cells with 4 week . All results were show in fig (3) and statistical calculations on table (2) and fig (4). All samples of Escherichia E- coli water that effects by different concentration of TiO₂ (0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7 and 0.8) ppm measured was decrease of the number of viable cells when concentration of TiO₂ increase and also decrease by week increase and recording that in table (2) by statically riding of Escherichia E- coli water sample by different concentration of TiO₂ rated 37.9% for sample 1 (0.1 ppm of TiO₂), 57.9% for sample 2 (0.2 ppm of TiO₂), 59.5 % for sample 3 (0.3 ppm of TiO₂), 61.3 % for sample 4 (0.4 ppm of TiO₂), 64.57% for sample 5 (0.5 ppm of TiO₂) , 64.65 % for sample 6 (0.6 ppm of TiO₂), 69.63 % for sample 7 (0.7 ppm of TiO₂) and 76.4 % for sample 8 (0.8 ppm of TiO₂) . The effects of all number of viable cells for Escherichia E- coli water samples that effects by different concentration of TiO₂ after 4 week decrease number of viable cells for all samples as showing in fig (4).

Acid (pH meter) Water and TiO₂ Results

Table (3) effect of TiO₂ on the survival of pH meter (acid) with 4 Weeks on the water condiment by Escherichia coli

	First Week pH	Second Week pH	Thread Week pH	Fourths Week pH
Sample 0	14.75	12.21	11.83	9.95
Sample 1	14.64	11.63	10.24	8.95
Sample 2	14.52	10.41	9.12	8.83
Sample 3	14.48	10.21	8.05	7.73
Sample 4	14.29	9.93	7.81	7.52
Sample 5	14.13	8.72	7.69	6.46
Sample 6	14.05	8.54	7.34	6.23
Sample 7	14.02	7.36^2	6.24	5.95

The acid (pH – meter) of Escherichia E- coli water that effects by different concentration of TiO₂ (0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7 and 0.8) ppm measured was decrease the pH-meter when concentration of TiO₂ increase and also decrease by week increase as showing in table (3), the statically riding of Escherichia E- coli pH- meter on water sample by different concentration of TiO₂ are decreased by rated 37.9% for sample 1 (0.1 ppm of TiO₂), 57.9% for sample 2 (0.2 ppm of TiO₂), 59.5 % for sample 3 (0.3 ppm of TiO₂), 61.3 % for sample 4 (0.4 ppm of TiO₂), 64.57% for sample 5 (0.5 ppm of TiO₂), 64.65 % for sample 6 (0.6 ppm of TiO₂), 69.63 % for sample 7 (0.7 ppm of TiO₂) and 76.4 % for sample 8 (0.8 ppm of TiO₂). The effects of TiO₂ different concentration on the Escherichia E- coli water samples after 4 week decrease pH-meter for all samples according to increase the concentration and dyes.

Conclusion

Synthesis titanium dioxide nanoparticles TiO₂ by sol gel method, then used XRD and ESM spectroscopy to calculated nanoparticles. And apply the sample in bio application to treatment water. The effects of TiO₂ different concentration on the Escherichia E- coli water samples after 4 week decrease acid (pH-meter) and number of viable cells for all samples for all samples according to increase the concentration and dyes.

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