Synthesis and Characterizations of Silver Titanium Nanoparticles and Their Application as Photocatalysts for Water Treatment

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Abstract. In the area of water treatment, nanotechnology offers materials that can be used effectively for the detection and removal chemical and biological contaminants from water. In this study silver titanium nanoparticles ($AgTiO_2$) were synthesized using liquid impregnation method for use in waste water treatment. The crystal structure was studied via XRD (X-ray diffraction), the surface morphology and particle size distribution of obtained nanoparticles investigated by SEM (scanning electron microscope) and UV-VIS Spectrometer was used to study some optical properties of prepared nanoparticles. X-ray results show that the $AgTiO_2$ crystals have tetragonal crystal system with average crystallite sizes equal 56.3 nm, SEM image display that the surface of $AgTiO_2$ nanoparticles was ununiform with

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surface roughness equal 22.91 pm particles size equal 53.28 nm. The absorption of $AgTiO_2$ nanoparticles value equal 3.386 (a.u) at wavelength (332) nm While the reflection equal (0.204%) at wavelength (330) nm, both absorbance and reflection in UV region. The synthesized $AgTiO_2$ nanoparticles were added to water contaminated with Escherichia coli at different concentration (0.0, 0.1, 0.3, 0.5 and 0.7) ppm during four weeks, the number of Escherichia coli and pH level in contaminated water decreased when $AgTiO_2$ nanoparticles increased, the water becomes pH neutral when adding $AgTiO_2$ (0.5ppm) at fourth week.

Keywords: Water treatment, Escherichia coli, sliver titanium nanoparticles and pH

1 Introduction

Environmental contamination is one of the significant risks to environment and all forms of life [1]. In the industry area, and according to development during the last few decades, there has been a rapid increase in pollution of all kinds such as water, soil or air which may include organic and inorganic contaminants, heavy metals, in addition to other types of pollutants [2.3]. Wastewater is a water that contains many pollutants as a result of various human activities such as industry, commercial, agricultural, and sewage inflow [4,5]. In wastewater Treatment, there are many different technologies which can be used such as electrodialysis, membrane filtration, precipitation, adsorption, Photocatalytic degradation, and electrodeionization [6-11]. Photocatalysis is an innovative method providing a wide domine of applications, involving chemical and degradation of dye, antimicrobial activity, and fuel generation via water splitting and carbon dioxide reduction [12]. Many studies have been found that most inorganic semiconductors can be used as photocatalysts [13]. In the photodegradation technique, semiconductors act as catalysis to convert natural products into CO_2, water, in addition to mineral acids [14-15]. Understanding the relationship between the physicochemical characteristics of photocatalytic materials and their performances, as well as the fundamentals in catalytic processes, is essential for designing and synthesizing photocatalytic materials [16]. Titanium dioxide (TiO_2) is one of the most semiconductors material used in photocatalysis, and it has three crystalline forms: Anatase, Rutile and Brookite. Anatase is the most widely used in photocatalyst because of its energy band gap (3.2 eV) [17-24]. TiO_2 particles can also have an antibacterial effect when activated by UV light. Silver oxide (AgO) is a material that has a cubic crystal structure and energy band gap of 2.5 eV [23]. Silver nanoparticles (AgNPs) can be used in biomedicine application such as antibacterial and therapeutic for their broad-spectrum and highly efficient antimicrobial and anticancer activities [24]. In this study, silver titanium nanoparticles were added water contaminated with *Escherichia coli* (*E. coli*) for the purpose of treatment.

2 Materials and Method

Sol gel method was used to prepeare silver titanium nanoparticles ($AgTiO_2$), 80 g of TiO_2 powder (Xilong Scientific Co., Ltd., 99.8%) was added to deionized water (500 mL), 1.7 g of $AgNO_3$ (Pheonix, > 99%) in presence of ethanol (C_2H_5OH) (Hayman Ltd, 99.7%), was added to TiO_2 suspension. The solution was stirred for 6 hours and left at room temperature for 24 h, after that dried in an air oven at 100°C for 24 hours to get dried solids. The agate mortar was used to crushed the dried solids to fine powder and calcined at 400°C for 6h in a muffle furnace [25]. The obtained particles were analyzed using X-ray diffraction spectroscopy (XRD Holland Philips X-ray powder diffractometer (Cu Kα, λ= 1.5406 Å)) to study their crystal structure, scanning electron microscope (JEOL, JSM-IT200) using to study their morphology and UV-VIS Spectrometer (Shimadzu mini 1240 spectrophotometer) using to investigate some of their optical properties. The synthesized $AgTiO_2$ nanoparticles were added to water contaminated with Escherichia coli during four weeks under sun light as photocatalytic reagent. To achieve that, (0.002, 0.006,0.010 and 0.014) g of silver titanium nanoparticles were added to four petri dishes (every dish contains 20 ml of water contaminated with Escherichia coli) to obtain (0.0, 0.1, 0.3, 0.5 and 0.7) ppm concentrations respectively as shown in fig (1-A). The experimental setup for photocatalytic treatment displayed in fig (1-B), the number of Escherichia coli in each dish was counted under light microscope (BEBANG Monocular microscope 40X-2000X, WX-C03-620) and pH level was measured using pH meter (Extech PH220-C).

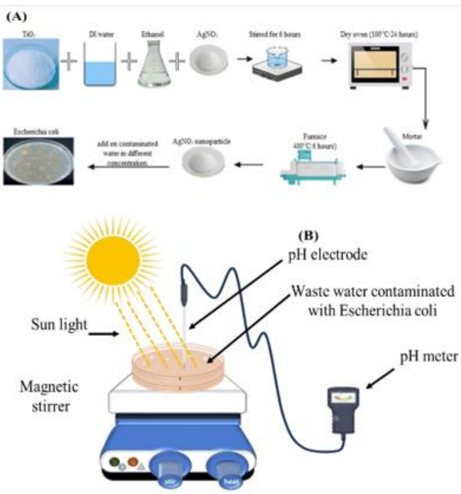

Fig.1. (A) schematic diagram of synthesis of silver titanium nanoparticles ($AgTiO_2$) method and applied in water contaminated by Escherichia coli treatment, (B) experimental setup for the photocatalytic treatment

3 Results and Discussion

Fig.2. represented the XRD spectrum of $AgTiO_2$ nanoparticles and their crystal structures which show that the $AgTiO_2$ crystals have tetragonal crystal system, the strong peaks at 27.45° and 36.5° refer to anatase phase (space group 14/mmm (139), a=b= 3.82510 Å and c= 28.884 Å, volume 422.6131 Å^3), this result is almost close to the result obtained by (Xiao, J and et al-2016) [3] and (Fatma Ezzahra Benmohamed and et al -2024),this is a result consistent with increasing the effectiveness of alight catalyst to improve water treatment process light as, stated in the study of (Guangmin Ren and et al - 2021) [2], (Xiao - 2016)[3], (Wang and et al J2023)[13] and (S.A. Ansari and et al -2016)[18] . The average crystallite sizes can be estimated using Scherrer's formula

$$D = k\lambda/\beta cos\theta \qquad (1)$$

Where D is the average crystallite sizes, λ is the X-ray radiation wavelength (Cu–Kα=1.5406 Å), K is a constant (0.89), β full width at half maximum height (FWHM in radian) and θ is the Brage angle, just as stated in the study (Xu, W-2023 and et al-2023) [25], the calculated average crystallite sizes for $AgTiO_2$ particles equal 56.3 nm. From XRD results, it is clear that $AgTiO_2$ nanoparticles were suitable for photocatalytic applications due to enhancement of light absorption and increased surface area- to- volume ratio (Sun, J and et al-2021) [26]. SEM Image of $AgTiO_2$ and its Morphology were displayed in fig.3., fig.3.A is SEM image of $AgTiO_2$ nanoparticles which display that irregularity in their spherical shape. The particle size distribution histogram was used to investigate the average size of the $AgTiO_2$ particle for each sample which can be displayed as:

$$f(D) = \left(\frac{1}{\sqrt{2\,\pi\sigma_D}}\right)\exp\left[-\frac{ln^2\left(\frac{D}{D_o}\right)}{2\sigma^2}\right] \qquad (2)$$

where D is the average particle size and σ_D is the standard deviation.[27].

The particle size and distribution are statistically measured as shown in fig.3.B, the particle size distribution histograms show that $AgTiO_2$ particles size equal 53.28 nm with standard deviation 1.166 nm, these value approximately in agreement with the particle size calculated through XRD. Surface roughness have significant impact in efficiency and effectiveness of photocatalytic treatment because it is increased surface area which result from increased of the area available for photocatalyst reactions, in addition to that, surface roughness can scatter light more effectively which increase the photocatalysis process by increasing the chance of interacting between light and photocatalyst ,as everyone has achieved (Sanjeet Kumar Paswan and et al -2021), (Naik, D and et al - 2023) [28,29] , fig.3.C display surface plot which describe $AgTiO_2$ particles roughness, surface roughness of sample equal 22.91 pm.

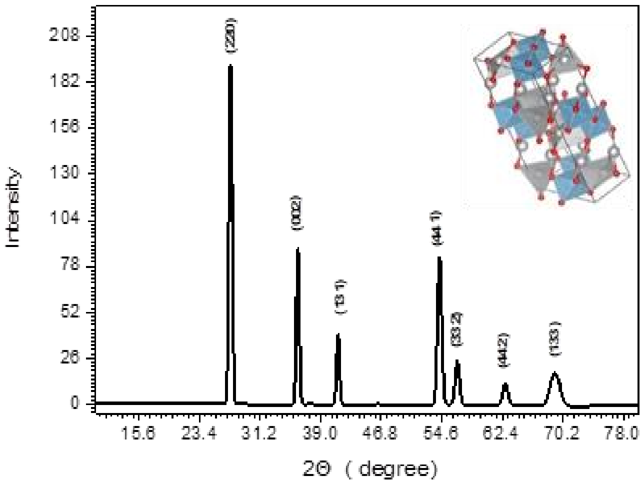

Fig .2. XRD pattern of $AgTiO_2$ nanoparticles and their crystal structures

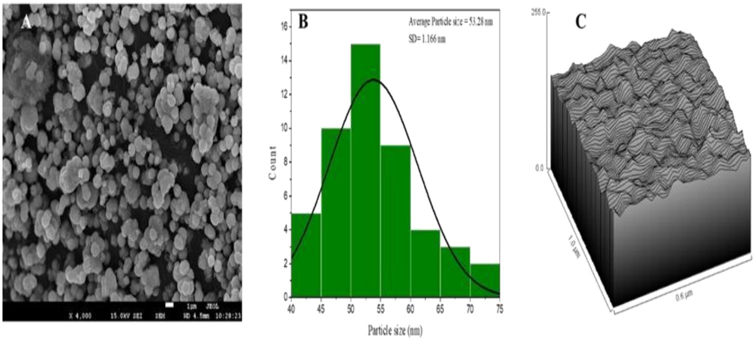

Fig.3. Morphology of $AgTiO_2$ (A) SEM image, (B) surface plot and (C) particle size distribution

The absorbance spectrum of $AgTiO_2$ nanoparticle was captured by UV-VIS Spectrometer. Fig.4.A shows the absorbance of $AgTiO_2$ nanoparticles as function in wavelength (λ) in range of (200-400) nm. The maximum absorption value equal 3.386 (a.u) at wavelength 232nm corresponding to photon energy 5.34 eV in UV region and optical band gab equal 3.557 eV which is suitable to bio-optical applications (photochemical reaction), as a study found (Zheng, L and et al -2024) [33]. In fig.4.B show the transmission spectrum of $AgTiO_2$ nanoparticles, transmission is opposite process to absorption, meaning that the highest absorption corresponding to lowest transmission and vice versa. The reflection of $AgTiO_2$ nanoparticles was shown in fig.4.C, it was noticed that the reflectivity increased to reach a certain value (0.204%) at wavelength (330) nm in the ultraviolet region and then it decreased. Due to figs.4.A, 4.B and 4.C, maximum value of transmission before wavelength 370nm, maximum value of reflection at wavelength 330nm while maximum absorption value at wavelength 232nm this means that absorption value was100% in this region and it is suitable to photocatalytic water treatments, Refractive index is quantity that expresses the ratio between speed of light in vacuum to it is speed in medium, it gives information about light behavior in media, as shown in fig.4.D $AgTiO_2$ nanoparticles have maximum refractive index equal 2.171 at wavelength 330 nm (Zheng, L and et al -2024)[33].

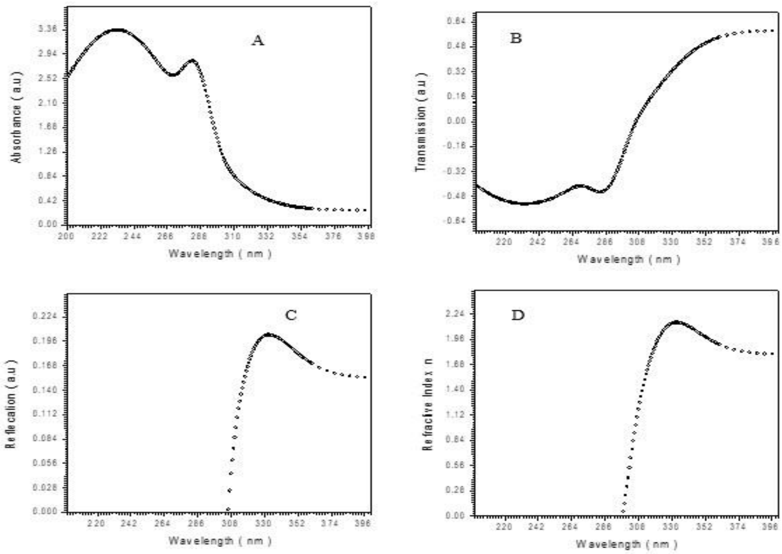

Fig.4. Some optical properties of $AgTiO_2$ nanoparticles sample (A) absorbance (B) transmission (C) reflection (D) refractive index

Photocatalysis is a process happen when the light energy, typically from UV light activates a catalysis, often a semiconductor material, to foster chemical reactions like study of (Wu, H. et al-2023) [30]. After synthesized and characterized $AgTiO_2$ nanoparticle, it was added to water contaminated by Escherichia coli (CFU/ml) at different concentration (0, 0.1, 0.3, 0.5 and 0.7) ppm and observed it is effect during four weeks by counting the number of Escherichia coli (CFU/ml) for every concentration as shown in table.1. and fig.6., the pH was measured corresponding to each sample as shown in table.2. and fig.7., when silver titanium nanoparticles exposed to light, the energy from the light excited electrons and stimulates the transfer of the electrons in the TiO_2 from the valance band to the conduction band generates pairs of electrons and holes, while silver works as an electron trap, capturing the photogenerated electrons which reduces the reconstruction rate of electron-hole pairs leading to availability of reactive species. Also, the photogenerated electrons and holes participate in redox reactions at the surface of $AgTiO_2$, producing reactive oxygen species like hydroxyl radicals which are highly oxidative and can break down organic and inorganic contaminates in water through oxidation, leading to the mineralization of organic compounds into harmless products such as CO_2 and H_2O and mineral acids. From another hand, the hydroxyl radicals and the reduction of oxygen can lead to formation and consumption of protons (H^+) in the solution leading to neutralize, this is consistent with the following studies (water Wu, H and et al-2023), (Sharma, P and et al-2023), (. Zhang, Y and et al-2024), (Zheng, L and et al-2024) and (Liu, Y. and et al2024) [30-34].

The effects of $AgTiO_2$ in different concentration on number of Escherichia Coli cells were decreased as the concentration of $AgTiO_2$ and number of days increased [table (1)], compared with the first week, the number of Escherichia E-coli in water on the second week decreased by 10.4 % for $AgTiO_2$ (0.0 ppm), $AgTiO_2$ (0.1 ppm) decreased by 42.5 %, $AgTiO_2$ (0.3 ppm) decreased by 51.1 %, $AgTiO_2$ (0.5 ppm) decreased by 59.6 % and $AgTiO_2$ (0.7 ppm) decreased by 65.8%. on third decreased by 18.75 % for $AgTiO_2$ (0.0 ppm), $AgTiO_2$ (0.1 ppm) decreased by 51.1 %, $AgTiO_2$ (0.3 ppm) decreased by 53.3 %, $AgTiO_2$ (0.5 ppm) decreased by 59.5 % and $AgTiO_2$ (0.7 ppm) decreased by 68.2%, at last week decreased by 35.4 % for $AgTiO_2$ (0.0 ppm), $AgTiO_2$ (0.1 ppm) decreased by 57.4 %, $AgTiO_2$ (0.3 ppm) decreased by 60.0 %, $AgTiO_2$ (0.5 ppm) decreased by 64.3 % and $AgTiO_2$ (0.7 ppm) decreased by 76.6%.

In fig (7) pH of contaminated water samples with Escherichia E-coli were treated by $AgTiO_2$ nanoparticles in different concentration measuring during 4 weeks, the measured results shown in table (2) and the statistical representation of these results displayed in fig (7). All of $AgTiO_2$ nanoparticles concentrations were decreased the pH level of water samples, compared with first week, the decrease in pH level on the second week 2.57 pH for $AgTiO_2$ (0.0 ppm), $AgTiO_2$ (0.1 ppm) decreased by 3.06 pH, $AgTiO_2$ (0.3 ppm) decreased by 4.35 pH, $AgTiO_2$ (0.5 ppm) decreased by 4.47 PH and $AgTiO_2$ (0.7 ppm) decreased by 5.73 pH ,on third week pH level decrease by 2.95 pH for $AgTiO_2$ (0.0 ppm), $AgTiO_2$ (0.1 ppm) decreased by 4.46 pH, $AgTiO_2$ (0.3 ppm) decreased by 5.51 pH, $AgTiO_2$ (0.5 ppm) decreased by 5.51 Ph and $AgTiO_2$ (0.7 ppm) decreased by 6.87 pH ,at last week pH level decrease by 3.86 pH for $AgTiO_2$ (0.0 ppm), $AgTiO_2$ (0.1 ppm) decreased by 4.77 pH, $AgTiO_2$ (0.3 ppm) decreased by 5.83 pH, $AgTiO_2$ (0.5 ppm) decreased by 6.67 PH and $AgTiO_2$ (0.7 ppm) decreased by 7.16 pH. These results agree with Hima Bindu Mantravadi's study (Mantravadi, H. B and et al-2017) [35] and super passed Mohammad Reza Amiri and et al's study (Amiri, M. R. and et al-2022) [36] by demonstrating that the inclusion of silver showed significant enhancement in Photocatalytic reaction.

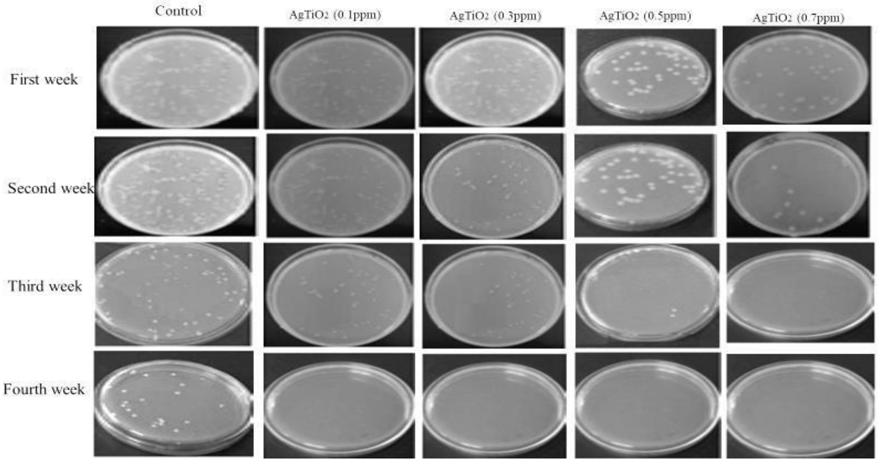

Fig. 5. Effect of different concentration $AgTiO_2$ nanoparticles (0, 0.1, 0.3, 0.5 and 0.7) ppm on the survival of viable cells of Escherichia coli (CFU/ml) during 4 Weeks on the water

Table 1. The number of Escherichia coli (CFU/ml) cells during 4 Weeks on water after adding $AgTiO_2$.

	First Week	Second Week	Third Week	Fourth Week
TiAgO2 (0.0ppm)	480	430	390	310
TiAgO2 (0.1ppm)	470	270	230	200
TiAgO2 (0.3ppm)	450	220	210	180
TiAgO2 (0.5ppm)	420	170	170	150
TiAgO2 (0.7ppm)	410	140	130	96

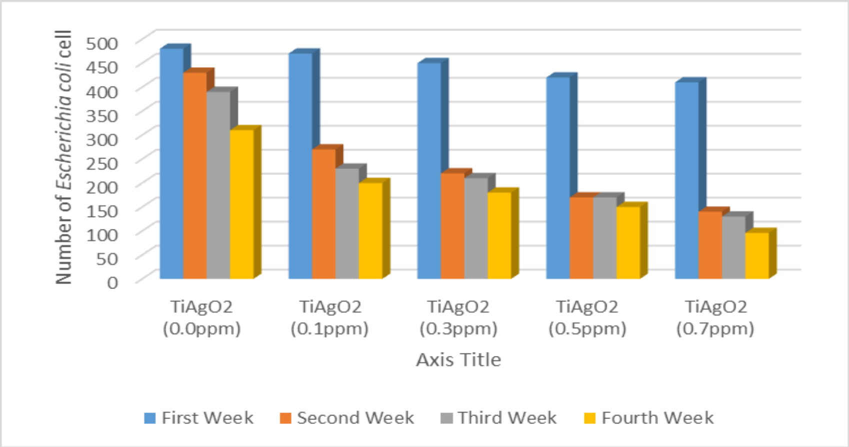

Fig. 6. Statistical representation of the number of Escherichia E-coli (CFU/ml) cells during 4 Weeks on water after adding $AgTiO_2$ at different concentrations (0.1, 0.3, 0.5, and 0.7) ppm.

Table 2. The pH level of water contaminated by of Escherichia Coli (CFU/ml) cells during 4 Weeks on water after adding $AgTiO_2$ at different concentrations (0.1, 0.3, 0.5, and 0.7) ppm.

	First Week pH	Second Week pH	Thread Week pH	Fourth Week pH
$TiAgO_2$ (0.0ppm)	13.92	11.35	10.97	10.06
$TiAgO_2$ (0.1ppm)	13.82	10.76	9.360	9.05
$TiAgO_2$ (0.3ppm)	13.65	9.33	8.14	7.82
$TiAgO_2$ (0.5ppm)	13.29	8.82	7.78	6.53
$TiAgO_2$ (0.7ppm)	13.18	7.45	6.31	6.02

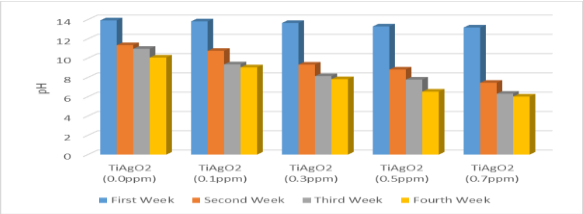

Fig.7. The statistical representation for pH level in water contaminated by of Escherichia Coli (CFU/ml) cells during 4 Weeks on water after adding $AgTiO_2$ at different concentrations (0.1, 0.3, 0.5, and 0.7) ppm

4 Conclusion

This study successfully synthesized silver titanium nanoparticles ($AgTiO_2$) via the sol-gel method and evaluated their potential as photocatalysts for water treatment. The $AgTiO_2$ nanoparticles, characterized by a tetragonal crystal structure and significant UV absorbance, effectively reduced Escherichia coli populations and neutralized water pH. The efficiency increased with higher nanoparticle concentrations. These results suggest that $AgTiO_2$ nanoparticles are a promising solution for improving water quality, offering a practical approach to microbial decontamination and pH adjustment in wastewater treatment applications. Further research should aim to optimize their synthesis and explore their longterm use in various environmental conditions.

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