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RESEARCH PAPER

Preparation and Characterization of Ag Doping TiO_2 Nanomaterial for the Photocatalytic Degradation of Malachite Green as a Model Cationic Dye

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ABSTRACT

Titanium dioxide (TiO_2) NPs were prepared using the modified hydrothermal process. Silver(Ag) doped Titanium dioxide (TiO_2), were prepared photo reduction method, characterization Ag / TiO_2 nanoparticles by x- ray diffraction(XRD), thermos gravimetric analysis (TGA), and UV-Vis Diffuse Reflectance Measurements(BG), FESEM , and TEM .Malachite green (MB) dye was used as a model pollutant to study the photocatalytic activity of Ag /TiO_2 nanoparticles under UV light irradiation. The influence of the amount of Ag /TiO_2 nanoparticles catalyst as well as of initial concentration of MG dye was investigated. When the increase in catalyst amount up to 0.3 g/L increase the number of active site, but beyond 0.3g/L there is little increase in % degradation. Thus best catalyst 0.3g/L used to removal MG dye. Photo catalytic activity rise was observed for Ag / TiO_2 nanoparticles which is about 95.67%. Photocatalytic degradation efficiency (PDE %) increases as the dye concentration decreases from (98.67% - 40.76%), and the photo degradation capacity rise when light intensity increase. It probably deduced, the rise of light intensity caused to excited particles of Ag /TiO_2 nanoparticles. The photo catalytic degradation efficiency was 83.9%, 78.8 %, and 68.5% during 3 cycle compared to standard solution (fresh) was 90.9%. The best reactive oxygen species (ROS) when used hydrogen peroxide (H_2O_2) comparative with methanol, thus the hydrogen peroxide important in enhancing the performance of photo catalytic degradation.

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INTRODUCTION

The past years have witnessed the fast develop of worldwide economy, which brings lot of ecological problems simultaneously. Among them, water pollutant which include inorganic, organic pollutant, drug, and dyes. A lack of clean water can lead to serious problem and diseases poisonous toxic [1,2]. Dye contaminants, found in the wastewater from several chemical industries like paint, food, cosmetic,paper printing and textile. Azo dyes are the utmost utilized dyes and account for more than 60% of total dyes [3-6]. Malachite green(MG)a Cationic dye, it is used in textile manufacture for dyeing silk, wool, also has common utilize in the leather, paper, and pharmaceutical industries,MG is very toxic and soluble in aqueous solution [7-11] Due to the complex aromatic structure and stability, these dyes can stay in the water for a long time This is why different physical ways like coagulation, adsorption, biodegradation,

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and different chemical ways like ozonation, and chlorination [12-15] but physical and biological methods do not remove pollutant ;they only transform them into another phase, as for chemical method using strong oxidants which are themselves pollutants [16, 17]. The most suitable way to eliminate this waste is their degradation by photo catalytic, titanium dioxide (TiO_2) is one of the most effective photo catalysts currently is use due to its strong oxidizing power, nontoxicity and long-term physical and chemical stability[18-20]. TiO_2 has a wide band gab 3.2eV requires only UV light for excitation to form photo activity species of holes (h^+) and electrons (e^-). The (h^+) acts as strong oxidizing agent, meanwhile the (e^-) works as reducing agent [21]. The recombination of h^+ and e^- in the TiO_2 photo catalyst is fast, causing ineffective photo catalytic process. To overcome the photo catalytic limitation TiO_2 loading noble metal such as Au, Pd, Pt and Ag. Among them[22], Ag is the most used in doping TiO_2 which enhanced electron – hole separation and interfacial charge transfer ability, as well as increase of the visible light excitation of TiO_2. The method has been reported to prepare Ag/ TiO_2 by photo reduction [23, 24].

MATERIALS AND METHODS

Materials

Titanium(IV) bis (ammonium lactato) di hydroxide (TALH, 50% aqueous solution), aqueous ammonia solution (25% NH3),Methanol, Ethanol 99.9% and Malachite green dye(MG). All chemicals used directly without further purification.

Preparation Titanium dioxide by Hydrothermal

Titanium dioxide TiO_2 NPs were prepared via the hydrothermal method of titanium(IV) bis(ammonium lactate) di hydrxide (TALH). Typically 10 ml of an aqueous titanium(IV) bis(ammonium lactato) di hydroxide solution was mixed with 15ml of an aqueous ammonia solution with 40 ml ethanol, the resulting solution volume was 115 ml, the mixture stirring on magnetic starred for 5 min, the mixture was transferred into Teflon cup was sealed in an autoclave and placed into an electric furnace held at 180 ˚C for 24h. Finally the autoclave was naturally cooled in air,The resulting TiO_2 nanoparticles,washed several times with deionized water and dried overnight in an oven at 60 ˚C after this step the product powder calcination at temperature 500 ˚C for 2h as in digital furnace as appear in Fig. 1.

Preparation of Silver (Ag) doping TiO_2 *nanoparticles*

Silver (Ag) doping TiO_2 nanoparticles was prepared via suspending 1g of TiO_2 NPs powder in 100 ml of distilled water and 2.5 ml AgNO3(0.01M) by sonication for 5min, followed by exposing the surface of the mixture to nitrogen gas for 10 min. Then added 1.5 ml of methanol, after which the mixture was irradiation with light intensity(1.71mW/cm^2) (using UVA LED Lamp) over night, with continuous stirring, The resulting powder was washed several times with deionized water and dried 24 h in an oven at 60 ˚C, to obtain the nanoparticles. Fig. 2 shows Real image of the deposition of silver onto TiO_2, (A) pugulate the mixed surface to nitrogen gas (B) Irradiate the mixture with lamp LED.

Preparation standard solution of Malachite green (MG) dye

Preparation of stock solution (1000mg/L) of

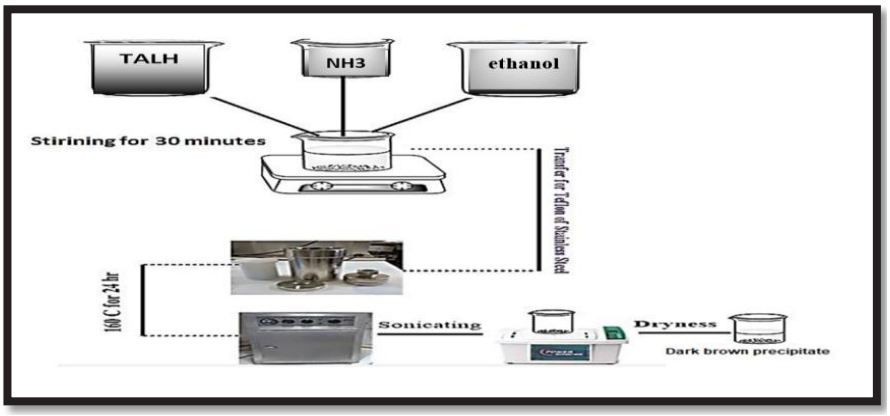

Fig. 1. Precreation Titanium dioxide TiO_2 nanoparticles.

aqueous solution to malachite green (MG) dye by dissolving 1g of MG in distilled water and complete the solution was made up to 1000 mL with distilled water. Where the maximum wavelength of the solution was estimated from its highest absorption in the UV-Vis spectrum found at the wavelength λ_{max} MG= 624 nm.

Characterization

Crystalline phases present in the photo catalysts were determine by x-ray diffraction method. The pattern scanned from 10-80 ˚C. Thermal gravimetric analysis (TGA) behavior of the as-prepared samples under air atmosphere in the temperature range 25-800 ˚C and with heating rate of 5 C/min. FESEM, TEM and UV -Vis Diffuse Reflectance Measurements.

Photo Catalysis Experiment

The photocatalytic of the Ag/ TiO_2 nanoparticles catalyst was estimated via the degradation of Malachite Green (MG) dye. All experiments were carried out in a photo- reaction vessel, the beaker was put under the UV light maintaining the

Fig. 2. Real image for the photo deposition of silver doped TiO_2 (A) pugulate the mixed surface to nitrogen gas (B) Irradiate the mixture with lamp LED.

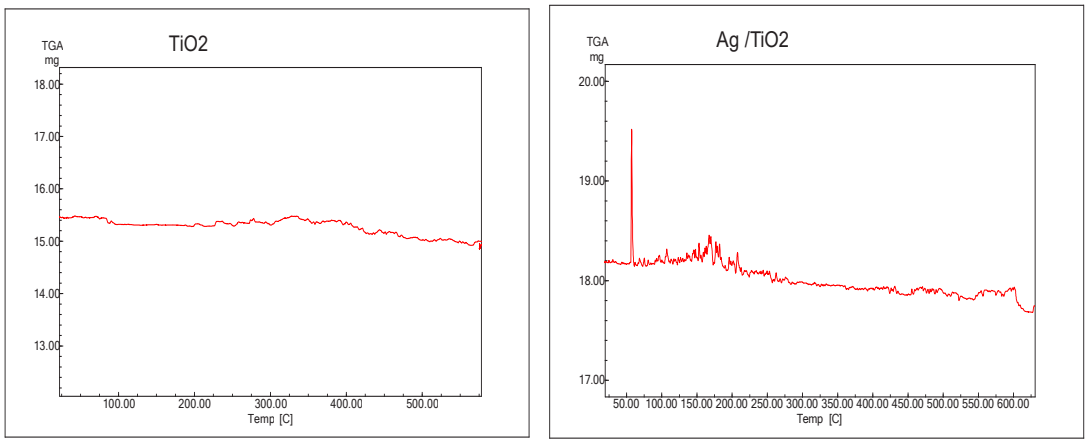

Fig. 3. TGA curve of a) TiO_2 NPs and b) Ag/TiO_2 nanocomposite.

distance among the light source and the surface of the solution controlled via utilizing UVA-meter. Experimental tests were performed, 0.3 g Ag/ TiO_2 photo catalyst was added into 200 mL solution MG dye. The mixture maintained in the dark for 10 min under stirring to reach adsorption equilibrium, and was then irradiated at several time intervals at 1 hr and separation the sample by centrifuged at 3500 tr/min for 10 min. The concentration of MG dye was estimated via measuring the absorption

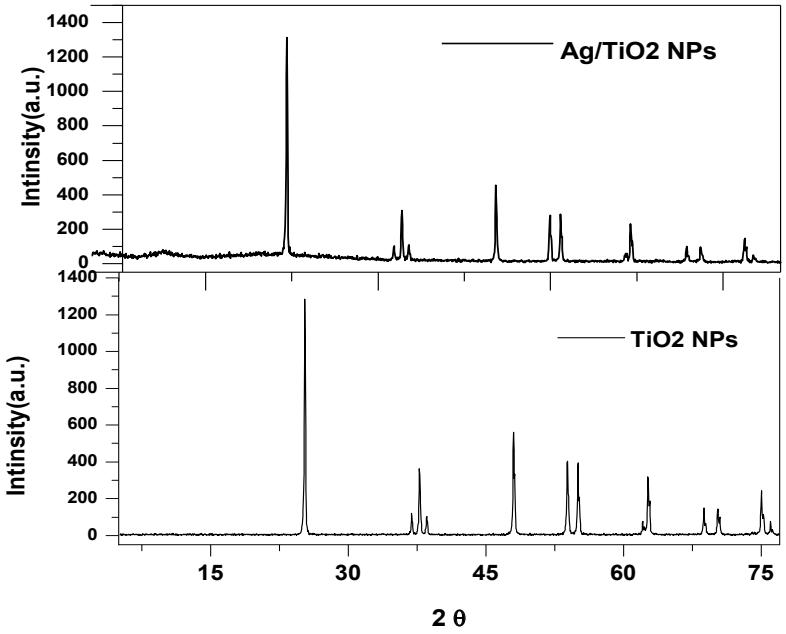

Fig. 4. XRD diffraction of nanocomposite.

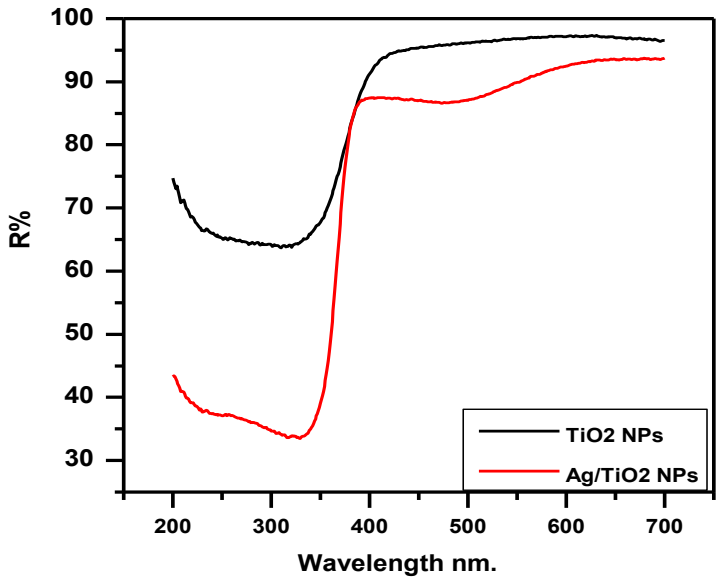

Fig. 5. Effect of energy band gap of nanocomposite.

at its maximum absorbance wavelength of MG dye =625 nm, via utilizing a UV-Vis spectrophotometer. Photo degradation efficiency PDE% of MG dye was measured by calculate Eq. 1:

$$\%PDE = (C_o - C_t)/ C_o \times 100 \quad (1)$$

Where C_o is the initial MG dye concentration in the solution, and C_t is MG dye concentration in the solution at time t.

RESULT AND DISCUSSION

Characterization of nanocomposite

Thermogravimetric analysis (TGA)technique in which the mass of a sample is monitored against temperature or time, to study the thermal stability of nanocomposite and indicate the purity of nanocomposites, the TGA curve of a) TiO_2 NPs and b) Ag/TiO_2 nanocomposite in the temperature range 20- 600 °C Fig. 3. The TGA curve of TiO_2 NPs appear straight line due to thermal stability, and very low weight loss in the one step of the Ag/TiO_2 nanocomposite in the range 300–600 °C was due to the thermal dissociation of Ag NPs doping of the TiO_2 Nps. thus Ag/TiO_2 nanocomposite have higher resistance to thermal degradation comparative with other nanocomposite [25-27].

Fig. 4 show the XRD patterns at of prepared TiO_2 and Ag- TiO_2. The XRD of TiO_2 there are no other summit indicate the presence of the impurity due to effect of calcination (500 ˚C), the diffraction peaks were observed at 25.3°, 38.0°, 48.2°, 54.5 2°, 55.3°,62.02°and 69.10°, which correspond to the (101), (004), (200), (105), (211), (204)

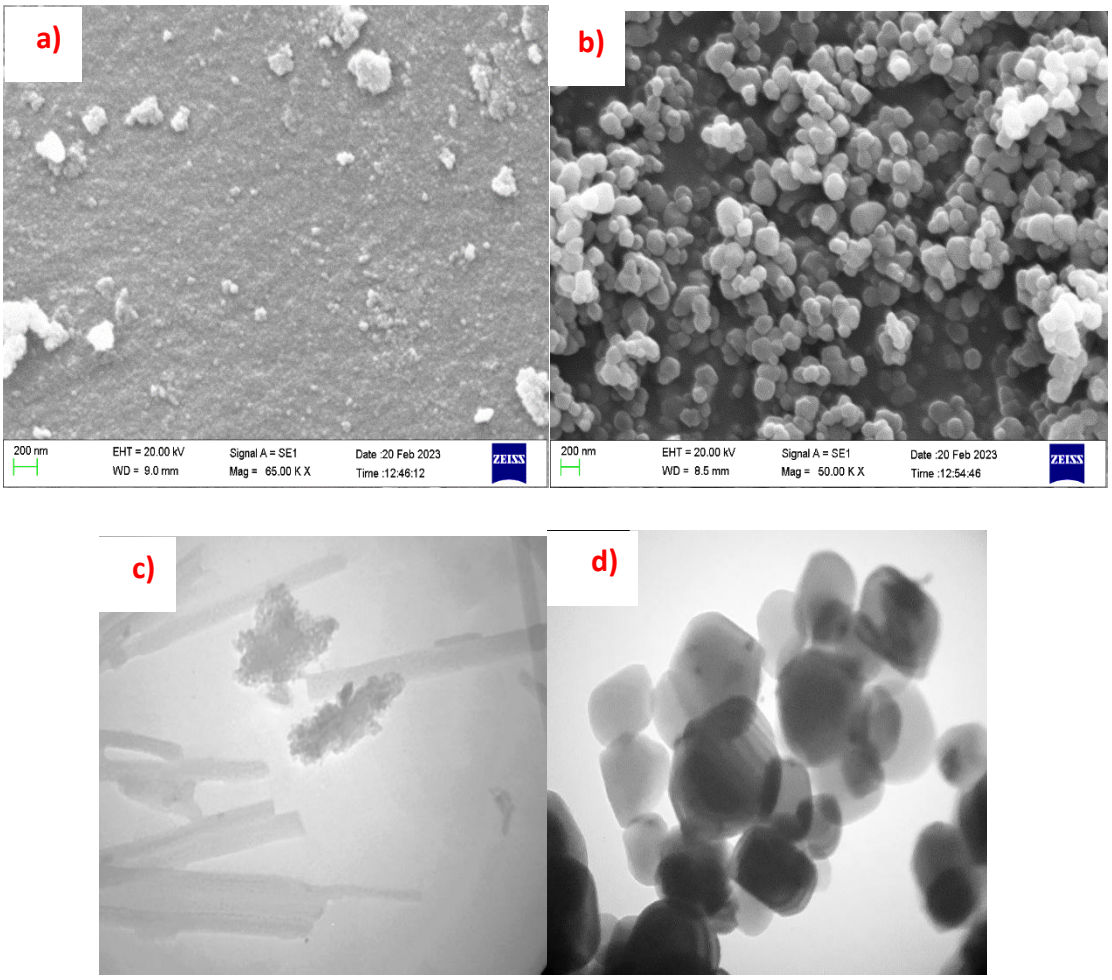

Fig. 6. FESEM a) Ag/ TiO_2 nanocomposite, b) TiO_2 NPS , TEM c) TiO_2 NPs, d) Ag /TiO_2 NPs.

and (220),reflection planes, respectively. All the diffraction peaks can also be well indicate to the anatase structure of TiO_2 (JCPDS card no. 21-1272) [28, 29] and pure phase with highly crystalline produced due to very sharp and intense peaks. In Fig. 3 Ag/ TiO_2 NPs no crystalline phase involving Ag could be observed. This indicates that the Ag is too dispersed in the TiO_2 lattice is not sufficient for

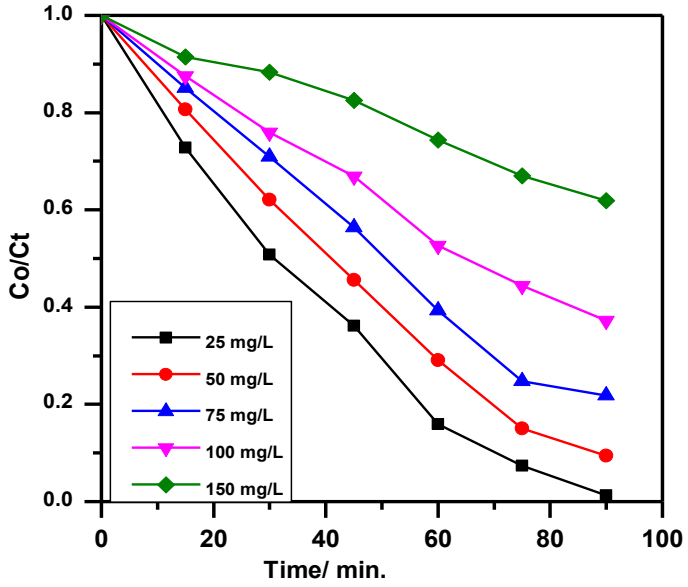

Fig. 7. Effect Photocatalytic degradation by (Ag/TiO_2) NPS at several concentration of MG dye.

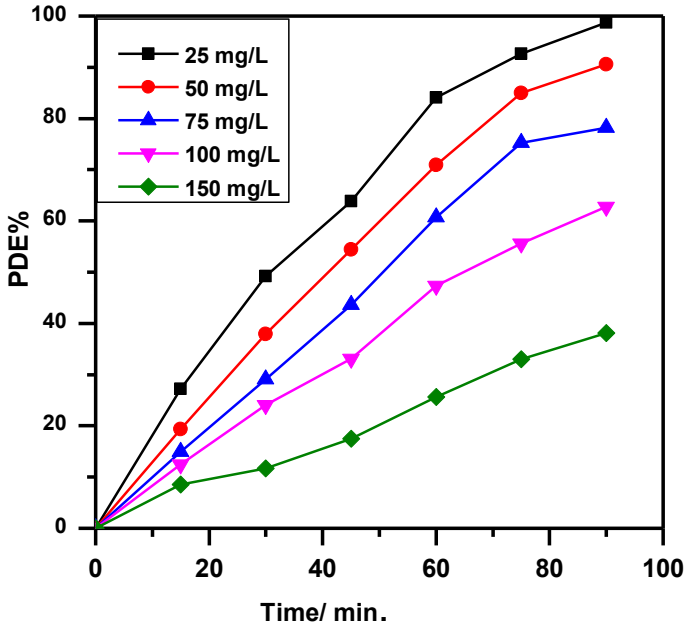

Fig. 8. Effect concentration of MG dye on the PDE%.

clear crystal formation or the Ag content is below the detection limit. Interestingly, no Ag crystal phase was detected. Thus, at low Ag content, higher dispersion of smaller Ag nanoparticles is on the sur face and pores are clearly achieved [30].

Absorption spectroscopy is a powerful tool for determining the optical properties of nanoparticles. The absorption spectra of TiO_2

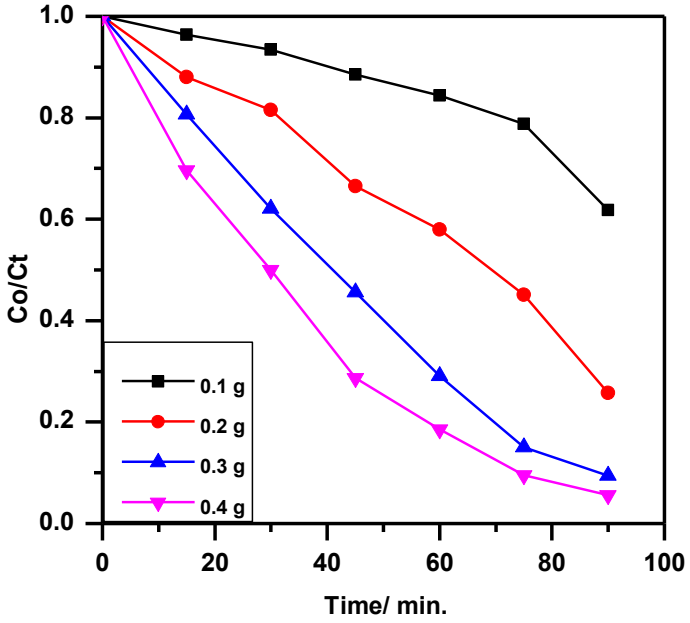

Fig. 9. Photocatalytic degradation of MG dye at several mass (Ag/TiO_2) NPS.

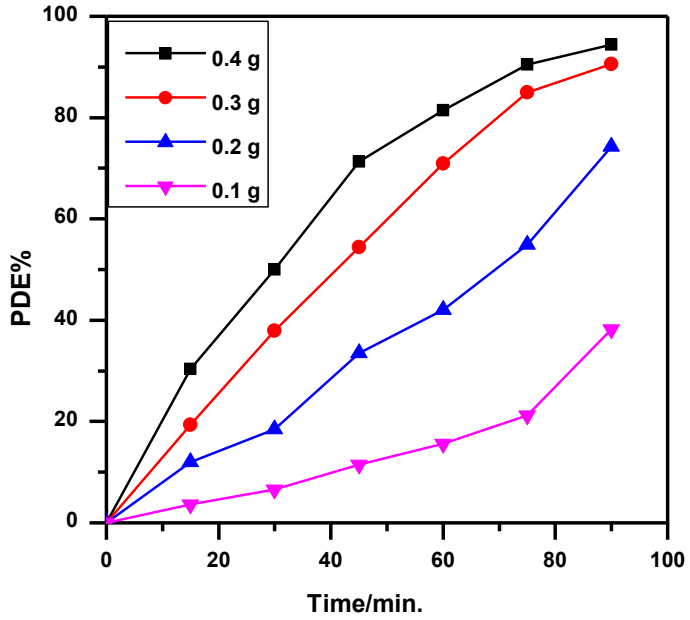

Fig. 10. Effect weight Ag/TiO_2NPs onto photocatalytic degradation MG dye.

NPs in the UV and visible ranges at various thermal treatment temperatures are presented. All spectra showed absorption edges between 370 and 390 nm, which is consistent with zinc oxide intrinsic band gap absorpat generated by electron-transitions as of valince for conduction

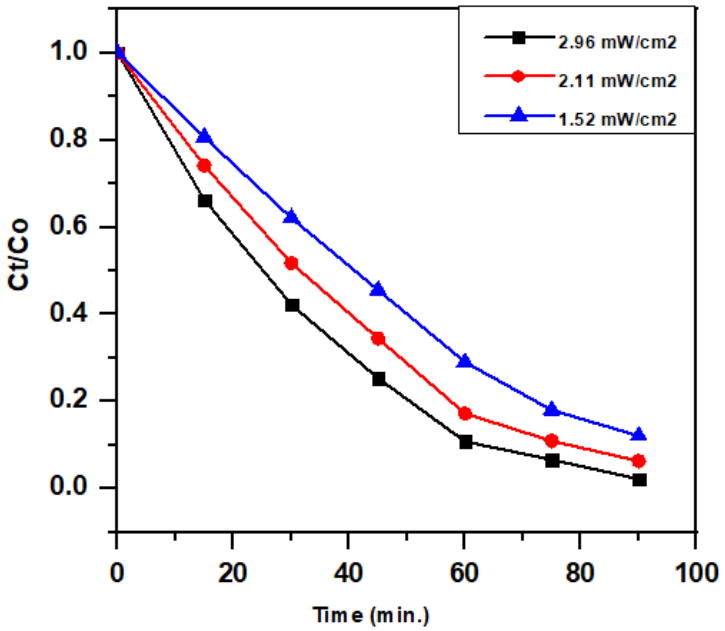

Fig. 11. Effect Photocatalytic degradation by (Ag/TiO_2) NPS at several light intensity.

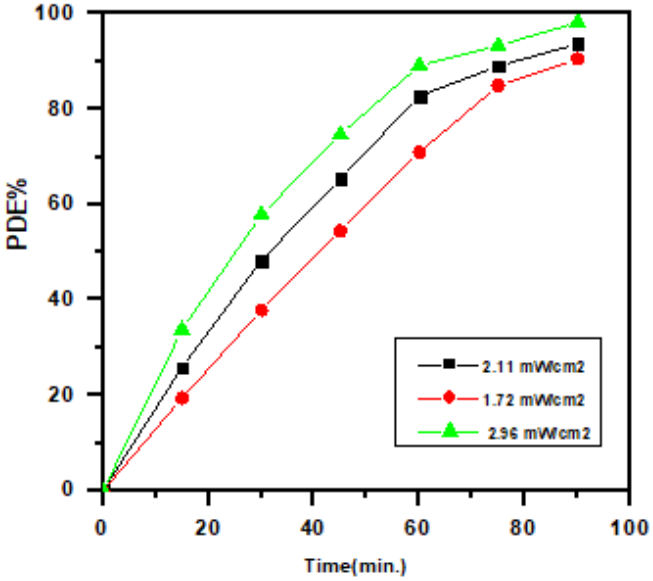

Fig. 12. Effect light intensity onto photocatalytic degradation MG dye

bands (O 2p → TiO_2 3d). Fig. 5, Demonstrates that experimental results imply that TiO_2 NPs have a larger band gap than larger spherical crystals. This can be explained by the nanoscale properties of materials [31].

Additionally, FESEM shows the morphology of the TiO_2 NPs and Ag/TiO_2 Nanocomposite, TiO_2 NPs The surface appears smooth with small white clusters indicating the preparation of titanium oxide with a high surface area. Ag/TiO_2 nanocomposite exhibits architecture structure and clearly demonstrates particle aggregation. It is thought that the aggregation of particles (or the production of larger particles) occurred as a result of the large specific surfaces aria and higher surfaces energy to TiO_2 NPs. as show in Fig. 6 (a, b), Also Fig. 6(c, d) shows image of TEM of TiO_2 NPs and TiO_2/ Ag NPs appears in the form of clusters inside the surface in addition to needle shapes [32, 33].

Effect of several Factor of Photo Catalysis

Effect concentration of Malachite green (MG) dye

The influence concentration of MG dye under UV light used was in the range 25-150 mg/L in the presence of 0.3 g of Ag/ TiO_2 NPS, the light intensity equal to (1.27mW/cm2). The results appear in Fig. 7. Dye MG concentration is a limiting parameter in utmost of the removal of dyes, especially in photo degradation, As the concentration of Mg dye increases, it prevents the penetration of light irradiation into the medium, so that MG dye photo degradation would be reduced remarkably, it means, the higher the concentration, the lower the dye photo degradation[34].

Shown Fig. 8, it is observed that the dye photocatalytic degradation efficiency (PDE%) increases as the dye concentration decreases, the PDE% increased from (98.67% - 40.76%), and this occurs either by reducing holes or (OH) because the active sites will complete the coverage with the MG, or A rise in the dye concentration caused an increased adsorption of the dye on the (Ag/ TiO_2) NPS, which leads to reduced (OH) radical generation, because that is low availability of the free active site on the surface.

Effect of Ag/ TiO_2 NPS dosage

The effect of Ag/ TiO_2 NPs dosage at (0.1-0.4g/ L) on the photo catalytic degradation of MG dye at an initial MG dye concentration of 50 mg /L, light intensity (1.27 mW/cm^2) and pH 6.7. When the increase in catalyst amount up to 0.3 g/L increase the number of active site and therefore causing the increase in hydroxyl radicals which consecutively increase the degradation of the MG dye. But beyond 0.3g/L there is little increase in % degradation of MG dye. Thus chosen 0.3g/L of all experimental as show in Fig. 9 [35-37].

Fig. 10 represents the PDE% of MG dye degradation against the multiple amounts of Ag/ TiO_2 NPs. The Fig. 6 shows that the percentage of

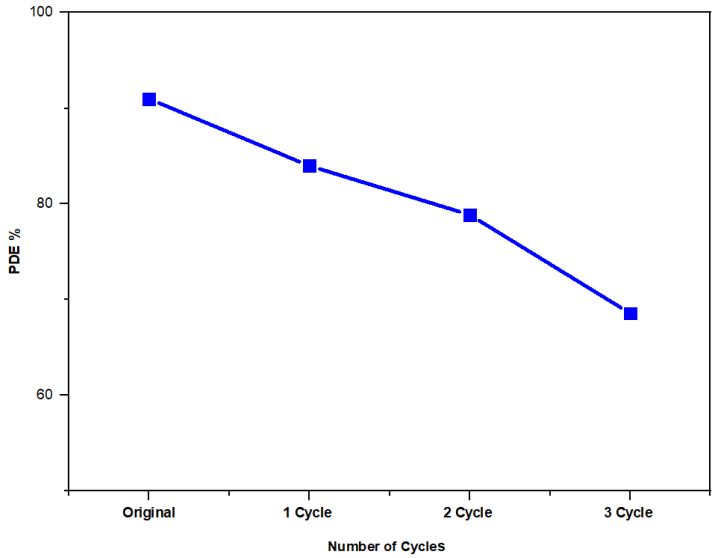

Fig. 13. Effect of Recycling of catalyst Ag/TiO_2 NPs.

degradation of the modified Ag/ TiO_2 NPs increases with increasing surface from 0.1-0.4 g/200 ml. This indicates that the active site provided for substrate adsorption on the surface is limited to the amount of Ag/ TiO_2 NPs 0.3g/200ml. because the vacant sites are consumed by intermediate products present during reactions that delay further degradation of the substrate.[38].

Light intensity (L.I.)

The light intensity effect about (2.3-1.3 mW/ cm2) was observed via variable of distance among source light and exposed in (Ag/TiO_2) NPS surface. Photo degradation of MG dye via the light intensity effect was investigation in the presence of 0.3 g/L of (Ag/TiO_2) NPS, concentration of MG dye 50 mg//L,and pH 6.8. It was that found wholly reactions still follow the first order kinetics as appear in Fig. 11.

The photo degradation PDE% increase when light intensity increase. It probably deduced, the rise of light intensity L.I caused to excited particles of (Ag/TiO_2) NPS to lead hole pair electron. and photo catalysis at low light intensities (1.3 mW.cm-2), decrease because of low light intensity reactions involving formation hole electron are predominant and hole electron re-combination is negligible, as shown in Fig. 12.

Recycling of catalyst Ag/ TiO_2 *NPs*

The recycling of catalyst is one of the key steps in assessing the practical application of photo catalysts and in developing heterogeneous photo catalysis technology for wastewater treatment. An examination of the photo catalytic activity of the recycled Ag/TiO_2 NPs catalyst was carried out on malachite green dye. The photo catalytic degradation efficiency were 83.9%,78.8 %,and 68.5% during 3 cycle compared to standard solution(fresh) was 90.9% as show in Fig. 13. The result reveal of photo catalysts is effective,and thus the photo catalyst is basically stable and is therefore promising for environmental remediation.

Roles of reactive oxygen species (ROS)

In order to distinguish the contribution of the surface reaction with (OH•, O_2-•, H_2O_2) species, different ROS were employed for check their effects on the relative photonic efficiencies of MG dye. The reaction pathway of MG degradation through generation of radicals from photo generated electron-hole pairs (e^- CB; h^+ VB). Hydroxyl radicals and electrons and holes (e^- CB; h^+ VB) have an affected on photo catalytic degradation process. OH • radical considered oxidizing agent which contributing to photo catalytic degradation of organic substrate. Nevertheless e^- can be recombination with h^+ causing decrease in the availability of the photo induced h^+ without electron acceptor, the limitation lead to the recombination of electrons and holes reduce photo catalytic efficiency and cause radiation energy loss,therefore suppressing the recombination of

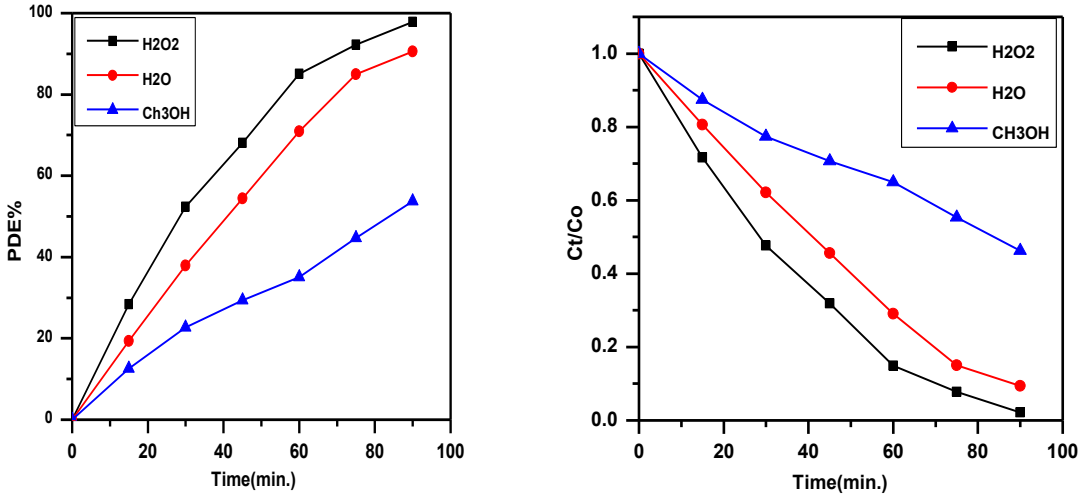

Fig. 14. Effect of Roles of reactive oxygen species (ROS).

electrons and holes is an important in enhancing the performance of photo catalytic degradation [25], while when methanol was added, the photo catalytic degradation changed and reduced indicating that OH• can be important in the photo catalytic, and shows that degradation efficiency between H_2O_2 and Methanol as shown in Fig. 14.

CONCLUSION

Photo catalytic degradation of Malachite green (MG) dye was studied in the aqueous onto (Ag/TiO_2) nanoparticles under UV light, we found photo catalysts was consist of mainly the anatase phase of TiO_2. The photo degradation of the MG dye follows first order kinetic initial] concentration of MG dye effect the photo degradation and the results appear photo degradation PDE% increase when light intensity increase. When the increase in catalyst amount thus causing the increase in hydroxyl radicals which increase the degradation MG dye.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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