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Original Research Article

Enhanced Photocatalytic Performance of ZnO NPs in the Presence of Solar Light and H_2O_2 for Degradation of Tetracycline from Wastewater: Optimal Conditions for Green Products

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ABSTRACT

In recent years, there has been an increase in the consumption of pharmaceuticals, especially antibiotics. Tetracycline (TC) is one of the widely used antibiotics, as it causes resistance to all microorganisms in ecosystems. In addition, it is classified as a controlled antibiotic. In this work, the efficiency of TC drug degradation via photolysis and hetero-generous photo catalytic methods is studied under effect of several volumes of H_2O_2 (0-4 mL) as an oxidizing agent. Characterization of the sample was carried out via UV-visible spectroscopy, XRD, FESEM, EDX, and TEM. The reveal results the photocatalytic degradation of TC drug is 92.67 %, at the best conditions weight of catalyst ZnO NPs 0.3 g, volume of hydrogen peroxide (3 mL), and concentration of TC drug 50 mg/L, pH=6, at irradiation time 60 min. H_2O_2 doses (0 and 4 mL) appear to have important variances in the first 10 min of the reaction for 4 mL of H_20_2 comparative with the solution without H_2O_2 giving the best PDE% (98.44%). The (PDE%) rises as concentration of TC drugs decreases from (92.67%-29.76%), but light intensity increases. Regeneration/recycling of ZnO NPs the photo catalytic degradation efficiency was 88.9 %, 82.8 %, and 77.5% through 4 cycles compared to standard solution(fresh) was 92.67 %.

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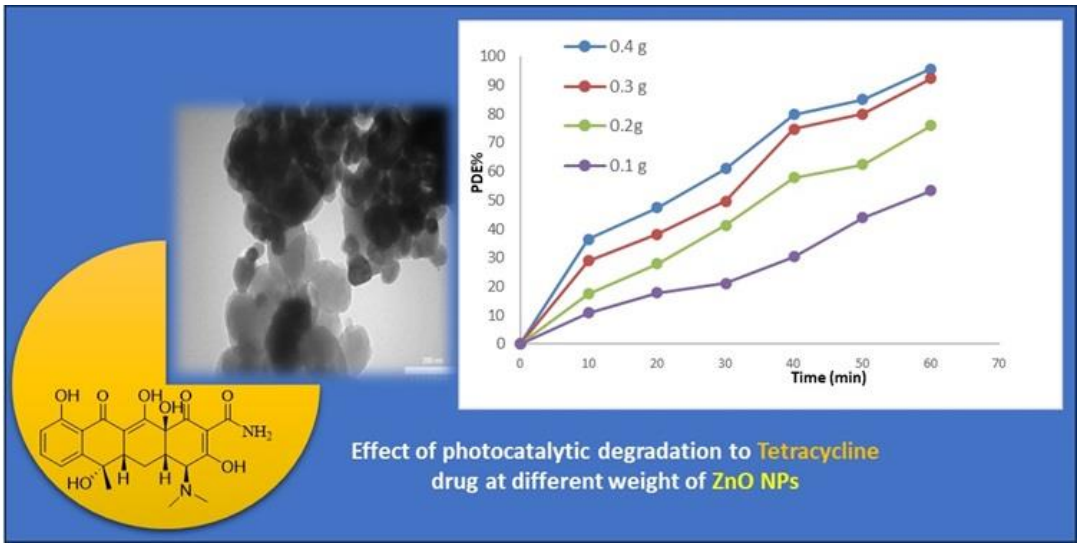
GRAPHICAL ABSTRACT



Introduction

The term "advanced oxidation processes" (AOPs) describes a group of chemical treatment techniques used to oxidize organic (and occasionally inorganic) compounds in water and wastewater. These reactions take place in the presence of hydroxyl radicals. These oxidation techniques may effectively eliminate all contaminants, even those with minimal reactivity, and mineralize them thoroughly. Heterogeneous photocatalytic is one of the advanced oxidation processes used for removal of pharmaceutical and organic pollutant, in this type, the reactant and semiconductor will be found in different phase [1,2].

The photo catalysis is a process in which semiconductor absorbs light energy to form radicals in the solution, these radicals are capable of oxidizing or reducing destroying the target contaminants. Zinc oxide (ZnO) is a wide band gap (E_g= 3.37 eV at room temperature, near-UV spectral region) semiconductor with high exaction binding energy (60 meV) enabling persistence of excitonic emission processes at or above room temperature. It has great potential for various applications, such as UV light emitters, photo catalysts, surface acoustic wave devices, piezoelectric transducers, and optical waveguides [3-7] . Pharmaceutical are found in different types of water, for example wastewater, groundwater, surface water, and drinking water[8]. Pharmaceutical preparations are present in very small concentrations and quantities, causing various problems for living organisms. Aquatic organisms, such as fish, are affected by these pollutants, which it generates changes such as feminization or masculinization resulting from hormones and the environment, and it is difficult to get rid of them [9-11]. Since both birds and fish are important mediators in the food chain, the ingestion of these organisms affected by these pollutants leads to the accumulation of these pollutants in humans, and causes resistance to antibiotics, and tissue disorders [5,12-13].

Pathogenicity, a serious endocrine problem, and also serious problems for newborns. Antibiotics are now utilized in several settings counting livestock operations and aquaculture for therapeutic and treatment purposes [14-17]. Traces of antibiotics are created in several natural environments like aquatic ecosystems. Of these, those belonging to the family of antibiotics

β-lactam are the ones that look utmost frequently, like cephalosporin , penicillin G, amoxicillin, and tetracycline which found in several concentrations ng/L to mg/L, both in surface waters and in waste water [18].

Tetracycline, have different brand names, and named as a four ("tetra-") hydrocarbon rings ("-cycl-") derivation ("-ine"). is an antibiotic in the tetracycline's family of drugs, utilized to treat a number of infections, like plague, cholera, acne, brucellosis, malaria, and syphilis. Its chemical formula is $C_{22}H_{24}N_2O_8$, Molar mass is 444.440 $g.mol^{-1}$, and chemical structure of a drug is shown in Figure 1 including side effects have diarrhea, poor tooth, sun burning, vomiting, kidney problems rash, and loss of appetite [19-22].

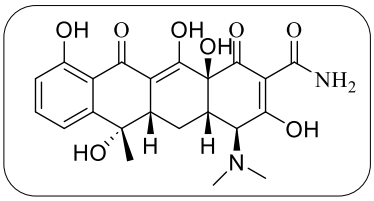


Figure 1. Chemical structure of tetracycline drug.

Experimental Part

Materials

The zinc acetate, Oxalic acid, and hydrogen peroxide were purchased via supported from (Sigma-Aldrich, Germany). Tetracycline TC drug was purchased from factory samara-Iraq. All chemicals used in the research that degree analytical and used directly without purification.

Preparation of ZnO NPs

Hydrothermal process was utilized to prepare ZnO NPs. Initially, in a beaker dissolving zinc acetate 5 g in 50 mL distilled water, and dissolving Oxalic acid in 8g in 50 mL distilled water, and then mixing two beakers to formation of a ZnO colloid. The solution was transfered in Teflon cup (the autoclave) in an oven at to 140 ˚C for 24 hours, as explained in details for another groups [23].

Photocatalytic experiment

The photo degradation of TC drug as a pharmaceutical pollutant model was studied by means of photocatalysts. 200 mL solution TC drug with a 50 mg/L concentration with photocatalyst was stirred for 10 min in the dark (adsorption) with 0.3 g of ZnO NPs. The solution was irradiated via (UVA-meter) with 1.21 mw/cm^2 as visible light sources for 60 min, and sampled every 10 min. During the photocatalytic degradation, at 25 °C. The absorbance of TC drug solution was recorded by a spectrophotometer (λ_{max}= 335 nm), and photocatalytic degradation efficiency (%PDE) of drug was obtained using Equation (1):

$$PDE\ (\%) = (C_0 - C_t)/C_0 \times 100 \quad (1)$$

Result and discussion

Characterization

The surface morphology of the synthesized nanomaterials can be examined via employing SEM analysis. The morphology of the nanoparticles differs from small spherical particles to big polyhedral nanoparticles. According to the image SEM, ZnO NPs had spherical-like morphology with some agglomeration in cluster form. From image SEM it can be noticed that the particle sizes are clearly. The scanning electron microscopy is an ideal analytical technique for characterizing and visualizing the elemental composition of a specimen [5,24]. The transmission electron microscopy gives a more accurate picture of the structure of prepared samples; the crystalline, distribution and the particles size of nanoparticle, as shown in Figure 2a.

The TEM image clearly shows that the ZnO NPs consist of spherical and hexagonal like shapes and mostly with particle sizes smaller than 200

nm. In addition, the TEM images show significant agglomerations of nanoparticles which may be due to the annealing of the sample at an almost high temperature. It is reported previously that the surface energy and specific surface area of nanoparticles are significantly higher than those of larger particles. Hence, the nanoparticles tend to agglomerate in liquid media at high temperature [5, 25-26], as depicted in Figure 2b.

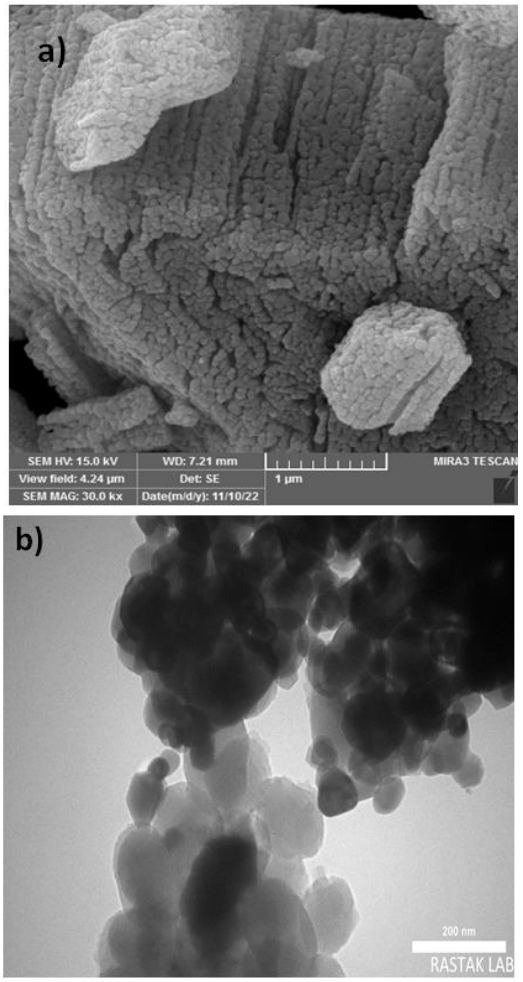


Figure 2. Effect of a) SEM of ZnO NPs and b) TEM of ZnO NPs.

X-ray diffraction is a highly effective non-destructive way for crystalline material characterization. Figure 3 shows the XRD patterns of pure ZnO NPs. The calcined ZnO NPs at 500 °C XRD patterns**.** After calcination at 500 °C, impurities are removed and crystallinity is improved. At 500 °C, all ZnO NPs peaks were discovered to be sharper, confirming that crystal formation occurs at higher calcination temperatures. The existence of ZnO NPs crystalline peaks at (31.6º, 34.4º, 36.2 º, 47.4 º, 56.6º, 62.8 º, 67.8º, and 69º). All the diffraction peaks can also be well indicating to the hexagonal Wurtzite structure of ZnO NPs. Pure phase with highly crystalline was produced due to very sharp and intense peaks [27,28].

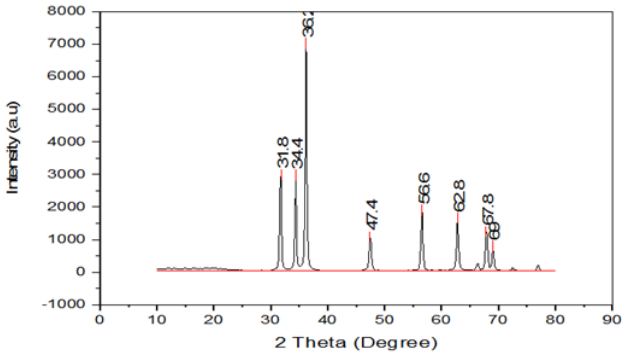


Figure 3. X-ray diffraction of ZnO NPs.

Energy dispersive spectroscopy EDX verified the ZnO NPs existence. Strong and precise diffraction peaks show that the nanocrystal line ZnO NPs good crystallinities EDX spectra show that only Zn, C, and O can be found in pure ZnO NPs A semi-quantitative evaluation of the atomic concentration (atom%) is demonstrated in Figure 4 which shows that the products' Zinc (Zn), Oxygen (O) and, Carbon (C) elements content (70.5 %, 25 .0%, and 4.5 %), respectively [28].

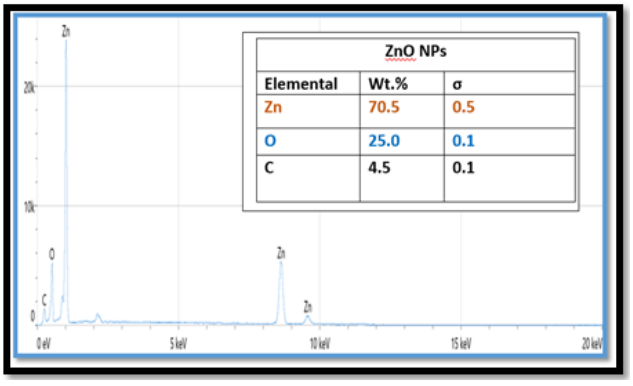


Figure 4. Energy dispersive spectroscopy EDX of ZnO NPs.

Effect of weight ZnO NPs

The impact of the weight of ZnO NPs (0.1-0.4 g) on the photocatalytic degradation of TC drug was investigated at 25 ˚C, 50 mg/L, flow rate of O_2 (5 mL/min) light intensity (1.2 mW/cm²), and solution pH 6.1 when the weight of ZnO NPs increase the photocatalytic degradation increase because the number of active sites increase, as illustrated in Figure 5. This shows that the present degradation of modified catalyst increase with increase in the amount of catalyst from 0.1-0.3 g/L and photocatalytic degradation increase (53.77% to 92.99 %) but above this limit is few changes or retained without notice change of photocatalytic degradation. This indicate that the active site provided for the adsorption of substrate on the catalyst surface is limited to catalyst amount of 0.3 g/L and after that low change in the degradation [17,29].

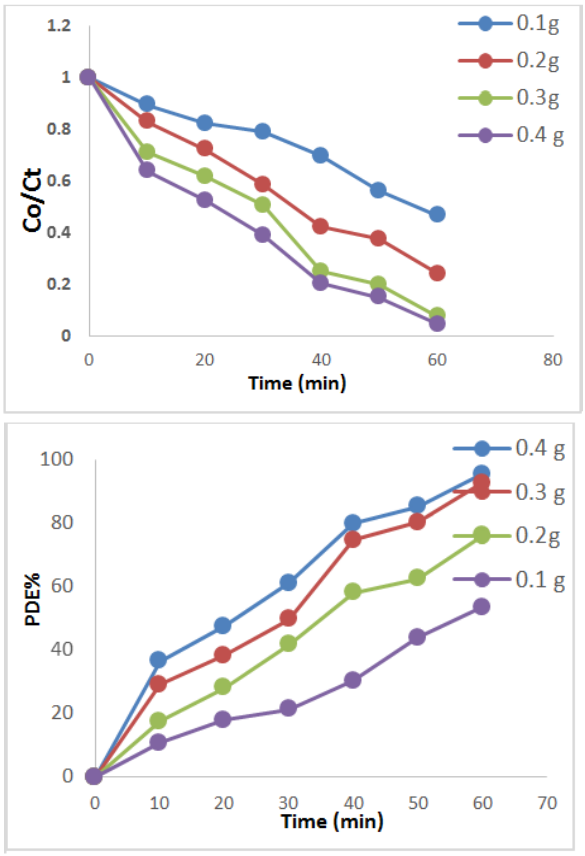


Figure 5. Effect of photocatalytic degradation to TC drug at different weight of ZnO NPs.

Effect of concentration of TC drug

The effect of initial concentration of TC drug (25-100 mg/L) on photocatalytic degradation method of TC drug was studied utilizing 0.3 g/L. The light intensity (1.2 mW/cm²) at 25 ˚C. It has been observed that of photo catalytic degradation gradually decreases with increasing of initial concentration of TC drug, this behavior could be explained the concentration 50 mg/L was the optimum concentration to over largest area of the ZnO NPs. Therefore, the photocatalytic degradation was decreased, but the TC drug concentration 50 mg/L gives the best optimum photo catalytic degradation efficiency which is 92.99 %, as shown in Figure 6 [30,31].

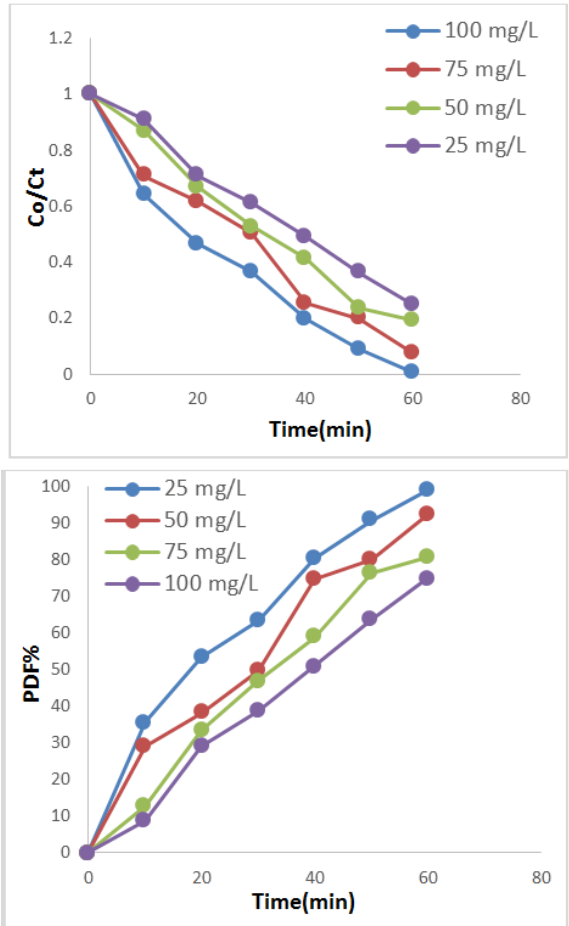


Figure 6. Effect of photocatalytic degradation to TC drug at different concentrations of TC drug.

Roles of reactive oxygen species (ROS)

To distinguish the contribution of the surface reaction with ($OH^{\bullet}$, $O_2^{-\bullet}$, and H_2O_2) species, several ROS were employed for check their impact on the relative photonic efficiencies of TC drug. The reaction pathway of drug 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. $OH^{\bullet}$ 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 electrons and holes is an important in enhancing the performance of photo catalytic degradation [32], while when methanol was added, the photo catalytic degradation changed and reduced indicating that $OH^{\bullet}$ can be important in the photo catalytic, and shows that degradation efficiency between H_2O_2 and Methanol, as demonstrated in Figure 7.

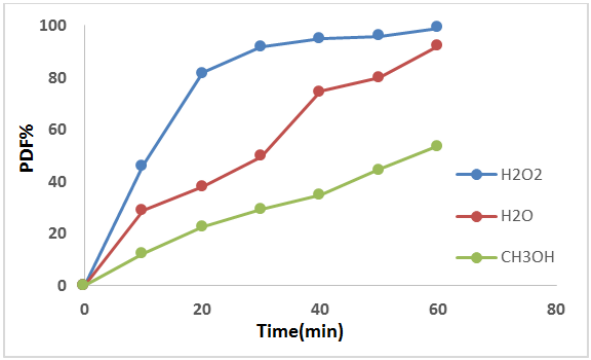


Figure 7. Effect H_2O_2 on photocatalytic. Experimental conditions: 0.3 g, TC drug concentration 50 mg/L, and light intensity 1.2 mW/cm^2.

Effect of several doses of H_2O_2 (0 and 4 mL) as an oxidizing agent. The reveal results the photocatalytic degradation of TC drug is 92.9 %, at the best conditions weight of catalyst ZnO NPs 0.3 g, volume of H_2O_2 (3 mL), and concentration of TC drug 50 mg/L, pH=6, at irradiation time 60 min. The H_2O_2 doses (0 and 4 mL) appear important variances in the first 10 min of the reaction for 4 mL of H_2O_2 comparative with the solution without H_2O_2 gave the best PDE% (99%), as shown in Figure 8 [2,29].

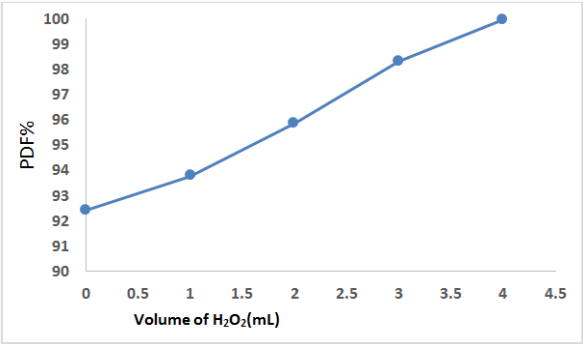


Figure 8. Effect of volume hydrogen peroxide.

Regeneration /Recycling of ZnO NPs

To reduce the economic cost using an environmentally friendly surface that is easily prepared from available, inexpensive materials, it can be used more than once to remove pollutants because of the surface's high efficiency and active sites that can be reactivated. The ZnO NPs regeneration is one of the important steps in assessing the practical useful of photo catalysts and in developing heterogeneous photo activity technology for waste water treatment.

An examination of the photo catalytic activity of the reused of ZnO NPs was carried out onto TC drug. The photo catalytic degradation efficiency were 88.9%, 82.8 %, and 77.5% three cycle compared to standard solution (fresh (1)) was 92.9%, as shown in Figure 9. The result of photo catalysts is effective, and thus the photo catalyst is basically stable and is therefore promising for environmental remediation [33,34].

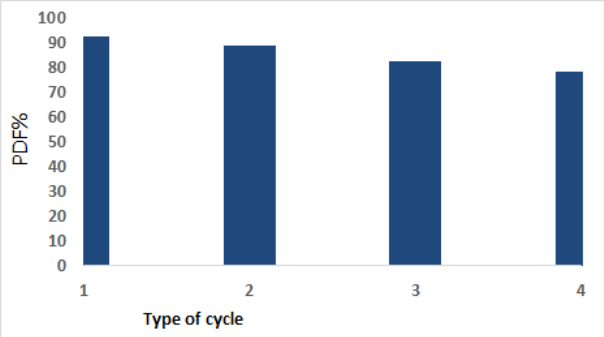


Figure 9. Effect of regeneration/recycling of ZnO NPs.

Conclusion

The aim of the study was to evaluate the efficiency of a catalyst ZnO NPs by assisted photo catalytic method by TC drug, an antibiotic major utilized in veterinary human and medicine. Initially, Photocatalytic degradation of TC drugs necessary to degrade 50 mg /L of TC drug, and 60 min. Thus, it is sufficient to fully degrade 50 mg/L drug and 92.9 % from aqueous solution. The TC drug was completely degraded after 1 hr of illumination; the good removal was obtained at 0.3 g. More than 98% of TC drugs have been oxidized after 1 hour irradiation time at the best condition of photocatalytic degradation. The good reactive oxygen species (ROS) were found when utilized (H_2O_2) comparative with methanol. Thus, the H_2O_2 is significant in improving the performance of photocatalytic degradation.

Disclosure statement

No potential conflict of interest was reported by the authors in this study.

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