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EDITORIAL TEAM

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Study the Effect of the ultrasonic in the preparation of binary composites ZnO:AgO nanomaterials and its application in removal tetracycline : Studi tentang Pengaruh Gelombang Ultrasonik dalam Pembuatan Komposit Biner Nanomaterial ZnO:AgO dan Penerapannya dalam Penghilangan Tetrasiklin

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Abstract

General Background Nanostructured metal oxides have gained increasing attention due to their versatile applications in environmental remediation. **Specific Background** Zinc oxide-based nanomaterials, particularly when modified with silver, have been widely investigated for their photocatalytic properties under light irradiation. **Knowledge Gap** However, limitations remain in achieving efficient charge separation and improving photocatalytic performance under practical conditions. **Aims** This study aims to synthesize Ag-doped ZnO nanostructures and evaluate their structural, optical, and photocatalytic properties for dye degradation applications. **Results** The synthesized nanocomposites exhibit improved crystallinity, reduced particle size, and enhanced light absorption, leading to higher photocatalytic degradation efficiency compared to pure ZnO. **Novelty** The incorporation of silver into ZnO nanostructures provides a synergistic effect that optimizes photocatalytic activity through improved electron–hole separation mechanisms. **Implications** These findings contribute to the development of advanced nanomaterials for sustainable wastewater treatment and offer potential for broader environmental applications.

Keywords: ZnO Nanostructures, Silver Doping, Photocatalysis, Dye Degradation, Nanocomposites

Key Findings Highlights

1. Ag incorporation modifies structural and optical behavior of ZnO materials
2. Nanocomposites demonstrate superior degradation performance under light exposure
3. Improved charge carrier dynamics observed in modified semiconductor systems

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Introduction:

Inorganic materials are common importance in the fields of manufacturing and construction due to their abilities to be modified with new properties in addition to strength, low-cost, and the specific ability to recycled many times [1]. Huge attentions were spent to redesigning materials and resynthesized at the molecular level, which are not visible in their conventional or bulk counterparts due to their tiny size and new physio-chemical features. There are attentions to increase pressure on global in all fields of live to synthesized nanomaterials with new design of nanostructures the economically viable technological for new solutions with more efficient [2]. The researcher in the fields of nanomaterials always looking for quality with quantity material lead to unique optical, with electrical, and magnetic behaviors able to influenced positively in the reaction of materials at the nanoscale [1-3].

Many inorganic materials were used for aims which mentions previously such as oxide TiO2 , WO3, ZnO, OR pristine materials such Au, Ag, Pt, and many ethers of materials in organic behaviors[3]. There were many techniques depend to enhance the phyio-chemical properties of inorganic materials and that include many strategies such as methodology of preparing or impregnated with other material by specific hybridization [4]. The hyper strategy of inorganic materials was used to create new properties, thus this work deal with synthesized best coordination between ZnO and AgO by change the process of mixing to achieve new properties.

hydrothermal method was used to prepared ZnO materials before modified with varying ratios of Ag using agent materials glucose, a biologically derived, to produce Ag/ZnO materials and applied in photocatalytic reaction to removed Methylene blue [5]. microwave at low temperature was used to prepared Ag/ZnO/graphene in solution and tested the activity for the photocatalytic performance to degradation of methyl orange (MO) solution under UV irradiation. The method was shown real influence for rapid microwave-assisted strategy to scalable and synthesized other metal/semiconductor for different applications in different fields [6]. Some of attentions were used Plant extracts as a reagent to make best hybrid for new design of inorganic materials with ZnO [7-8].

In this work ZnO was impregnated with AgO by two ways, the first used magnetic stirrer while the other was used ultrasonic water bath to make the mixed between the reactant. The X-ray diffraction and measuring surface area were depend to make analysis and compare for the nature of the two surface and make understand for the change the activity towards adsorption and photo degradation of tetracycline.

Experimental:

Preparation of AgO nanoparticles and binary composites ZnO:AgO

The chemicals that used in this work was: Tetracycline (TCN), silver nitrate, zinc oxide were purchased from Sigma-Aldrich without any further purifications. AgO nanoparticles were prepared by two methodologies as follows: the first was using magnetic stirrer at 200 rpm, and that include dropping 15 m L of (40%H2O2) with rate of addition (5 m L/min.) into 50 m L of (2 g AgNO3) which mixing for 1 h as mentions previously. The second AgO nanoparticles were prepared by using the same quantities and qualities of reactant the different represent by using ultrasonic water bath to make mixing process for the reactants under wave 40 Hz for 30 min. by ultra-sonicated a Q500 sonicator (750 W, 40 kHz at room temperature and pH 6.0 for all AgNO3 solutions.

Figure 1 with the two parts refers to process of preparing all pristine and binary composites. The binary inorganic composites ZnO:AgO with magnetic stirring and by ultrasonic water bath nixing was prepared with the same conditions after dispersion 2 g of ZnO with AgNO3 solutions to achieve 1: 0.33 ratios for (ZnO:AgO).

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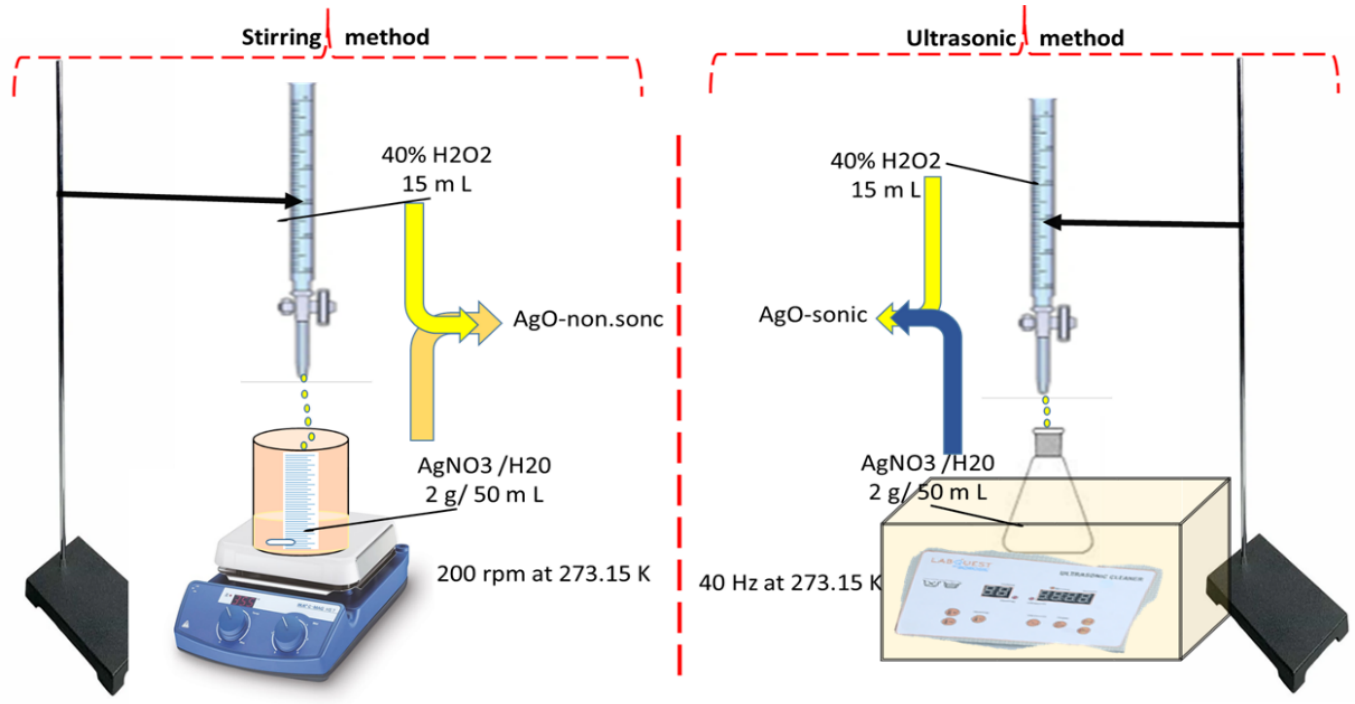

Figure 1. Figure 1: The skim for preparation AgO by two methods at the left stirring at 200 rpm and at the right by ultrasonic water bath by 40 Hz.

Characterization:

the powdered samples (AgO , and ZnO:AgO with and without sonication, in addition to ZnO were characterized by X-ray diffraction, using Philips X'Pert MPD X-ray diffractometer provided radiation of Cu Kα at λ = 1.54056 Å with 2θ ranges from 10° to 80°, 0.02° s−1 of scanning rate. Generally, Figure 1 and 2 reported the patterns of samples, however showed a multiphase material including pristine Ag oxide and Zn oxide and binary inorganic composites ZnO:AgO. According to figure 1 and 2, The XRD technique can estimate the influence of ultra-sonication process on the nature of crystallinity and properties of structure with distribution active site of pristine AgO and modifying with ZnO particles.

Figure1, reported the characteristic peaks of ZnO with seven primary [[8] identifications peaks starting from 2Θ=18.7˚ until 2Θ=65.1˚. The XRD peaks at 18.7˚, 26.5˚, 32.91°, 36.30°, 47.10°, 58.6°, and 65.1°, were identified as (1 0 0), (2 0 1), (1 0 1), (1 0 2), (1 1 0), (1 0 3), and (2 0 0), reflections, respectively. The ZnO nanoparticles had crystalline structure which evaluated by the sharper with stronger diffraction peaks. The crystal size (d) average of ZnO was evaluated by Scherrer's equation was found to be 36 nm. also stated the similar diffraction peaks at 1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0).

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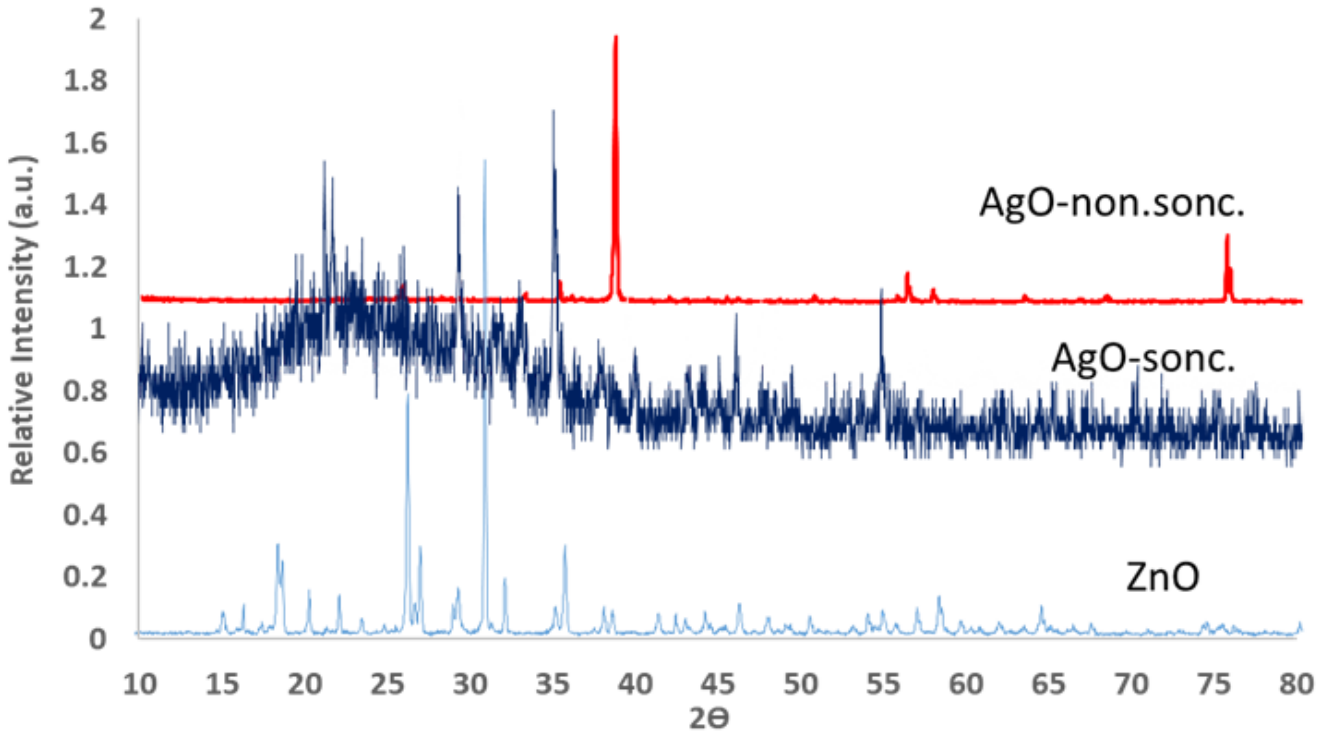

Figure 2. **Figure 1:The skim of XRD patterns for ZnO, and AgO with two different conditions of preparation with sonication process(AgO-sonc.) and without sonication process (AgO-non.sonc.)**

According to figure 1, two samples of silver-oxide was prepared at the same amount of reactant with variance in methodology of reaction, thus the two samples were shown different behavior in XRD patterns. The first sample AgO-non.sonc. which was prepared by magnetic stirrer as way to mixing the reactants and that was shown the peaks: (36.2˚) AgO (111), (38.1˚) AgO (111), (54.8˚)AgO (200), (56.1˚) Ag (311), and (77.4˚) Ag (311) respectively. The mixing process were likely to result in the formation of silver nanoparticles [9] in addition to AgO.

The other reaction which used ultrasonic water bath were used as source for mixed the reactants witnessed shown two phases Ag and AgO nanoparticles with higher ratios compare with the last AgO. Figure 1 was reported higher ratios for silver in pristine and oxide form as shown in (17.2˚) AgO (121), (26.0˚) AgO (200), (30.4˚) AgO (121) ,(32.4˚) AgO(200), (36.7˚) Ag (111), (37.8˚) Ag (111), (55.4˚)Ag (200), (45.6˚) Ag (200), and (76.9˚) Ag (311) respectively[10-12].

One of the distinctive features revealed by the ultra-sonication method is the emergence of the fine structures of nanomaterials, along with the presence of amorphous structures, as shown with noises and broad peaks. The estimation of average particle size by Scherrer's equation were shown 10.3 nm and 19.7 nm for AgO-sonc. and AgO-non.sonc. respectively.

After impregnated with ZnO , the behavior of particles AgO were shown influence in the nature of particle size and morphology and nature of orientation active site in binary. The effect of methodology was shown in distribution of nanoparticles AgO as shown in figure 2. The orientations may be shown more similarities but that did not prevent shown the variance between the two structure and that represent by: firstly, presence the amorphous structure due to ultrasonic method. Second the dispersion of AgO in the surface of ZnO was more ratios and more broad and systematic as compare with the binary composites ZnO:AgO-non.sonc. third the particle size also shown change and different between the two binary composites which was 47 nm and 59 nm for ZnO:AgO-sonc and ZnO:AgO-non.sonc [13-14].

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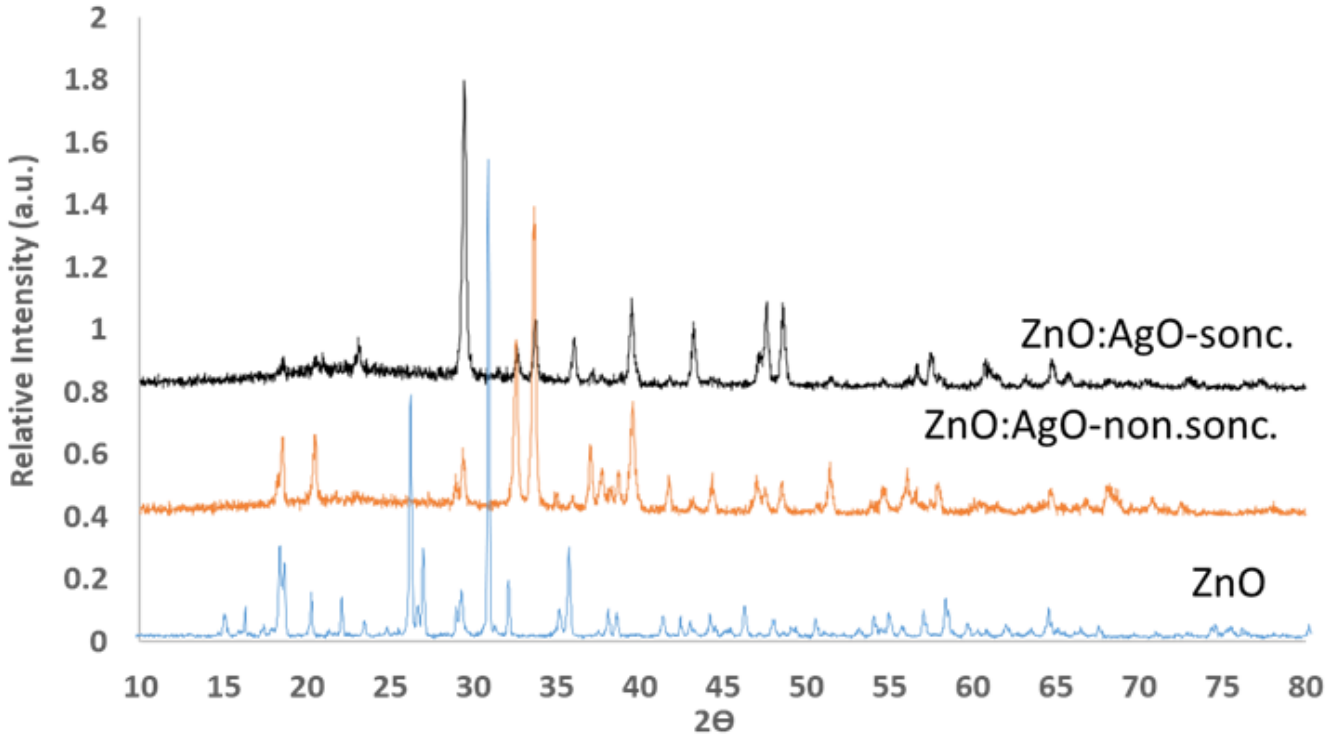

Figure 3. **Figure 2 :The binary composites skim of XRD for ZnO: AgO -sonc and ZnO: AgO non.sonc. with pristine ZnO.**

The surface area S_{BET} for the materials were 36 m2/g , 7.7 m2/g , 16.2 m2/g , 44.6 m2/g and 59.8 m2/g , for ZnO , AgO-non.sonc., AgO-sonc., ZnO:AgO- AgO-non.sonc. and ZnO: AgO-sonc. respectively. The results of S_{BET}maybe give indication for the increase in active sites due to enhance the probability for emergence of several active groups This phenomenon is also observed and is in good agreement with the previous reports [15-16]. When the AgO after addition by ultrasonic [16-17] formed reduced particle size to less size, the crystallinity tends to be reduced due to uniform nature of molecular structure which formed. In addition, these crystalline binary inorganic composites arrange all of it in ideal orientation via oxide groups and silver groups towards best molecular inorganic charges interaction and that was proved in XRD analysis [18].

Removal reaction of Tetracycline TCN :

The removal process of 15 ppm Tetracycline (TCN) was include two sections dark reaction (adsorption) and (photo degradation) by UV-Light (λ300 nm) at 298.15 K . The two process were done at 25˚C using a sealed batch glass with photo-reactor with the capacity of 400 m L, equipped with a 450 W ultraviolet (UV-lamp) and without light. The typical experiments include solutions 100mL of TCN solution (15 ppm), and 0. 200 mg of the synthesized inorganic composites ZnO:AgO and pristine ZnO and to ensure homogeneous solution, a magnetic stirrer was used at 200 rpm . all the solutions under testing were reached adsorption-desorption equilibrium by stirred in the dark for 60 minutes. after equilibrium time of adsorption, the UV-light was light one to start irradiation and a 3 mL of aliquots samples were collected at certain time interval, which centrifuged for the removal of synthesized catalysts before measured absorbance at 359 nm

Results:

Adsorption process:

The first test for the change and modification of binary composites was the reaction at the dark conditions which is adsorption reaction as reported in Figure 3. The effect of adsorption was estimated by The total adsorption of TCN after 1 h using the expression {% adsorption = (A0 − A□□)/ A0 × 100 1) Where, A0 is the initial TCN absorbance and A□□ is the absorbance of TCN at given time, t. the results shown efficiency of removal by adsorption 11%. 15.6%, and 20.7% for ZnO, ZnO:AgO-non.sonc. and ZnO:AgO-sonc. respectively with equilibrium adsorption/desorption time reach to 45 min. for the all materials.

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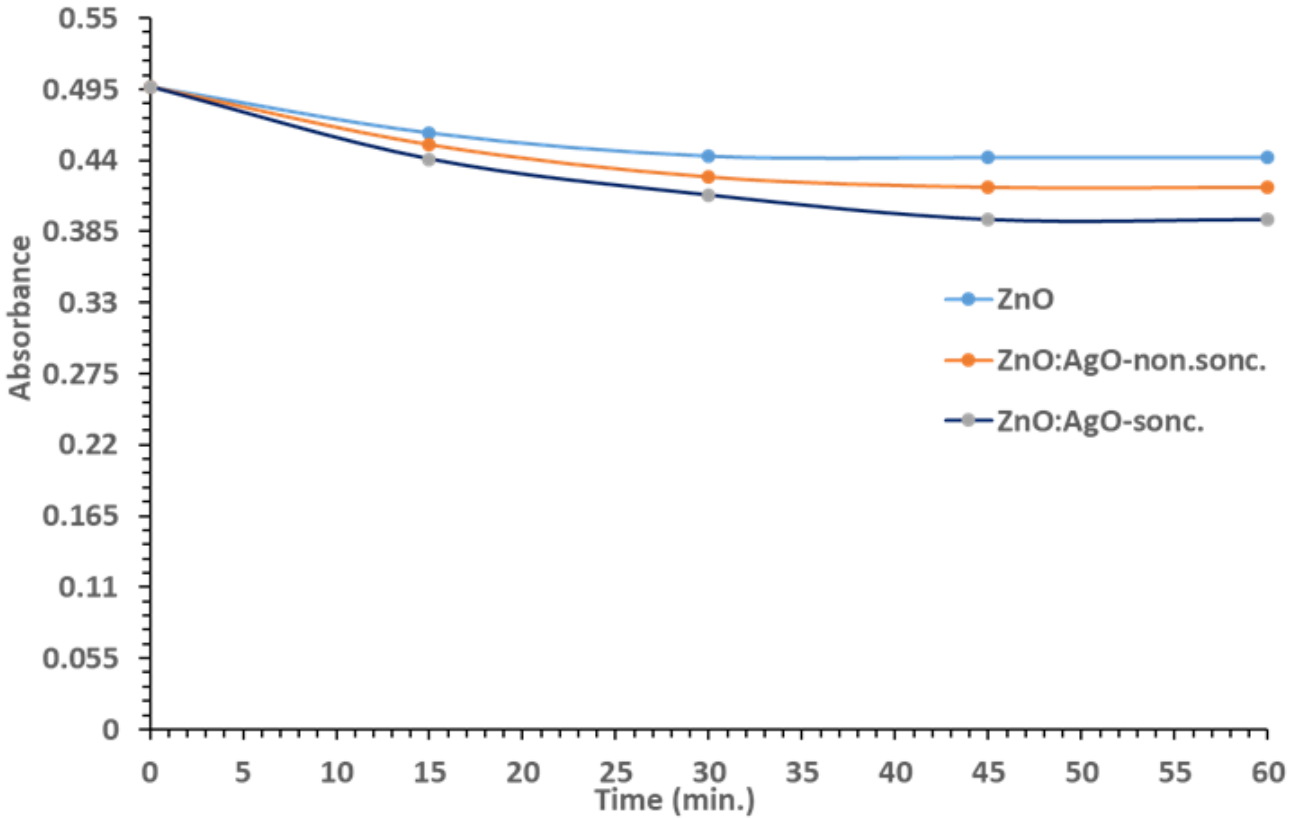

Figure 4. Figure 4: The skim for adsorption process for 15 ppm of TCN by pristine ZnO and ZnO:AgO-non.son. and ZnO:AgO-sonc. at 298.15K.

Figure 4, reported the activity of inorganic [19-20] nanomaterials with uv-light, which shown the binary ZnO:AgO-sonc. overlay were demonstrated by its superior removal performance compared to other ZnO, and ZnO:AgO-non.sonc. the percent of removal due to UV-light / catalyst was 32.7%, 51.5%, and 65.7% for ZnO, ZnO:AgO-non.sonc. and ZnO:AgO-sonc. respectively. The accumulation ratios for removal was shown that ZnO:AgO-sonc the best efficiency towards removal by adsorption and photocatalytic reaction.

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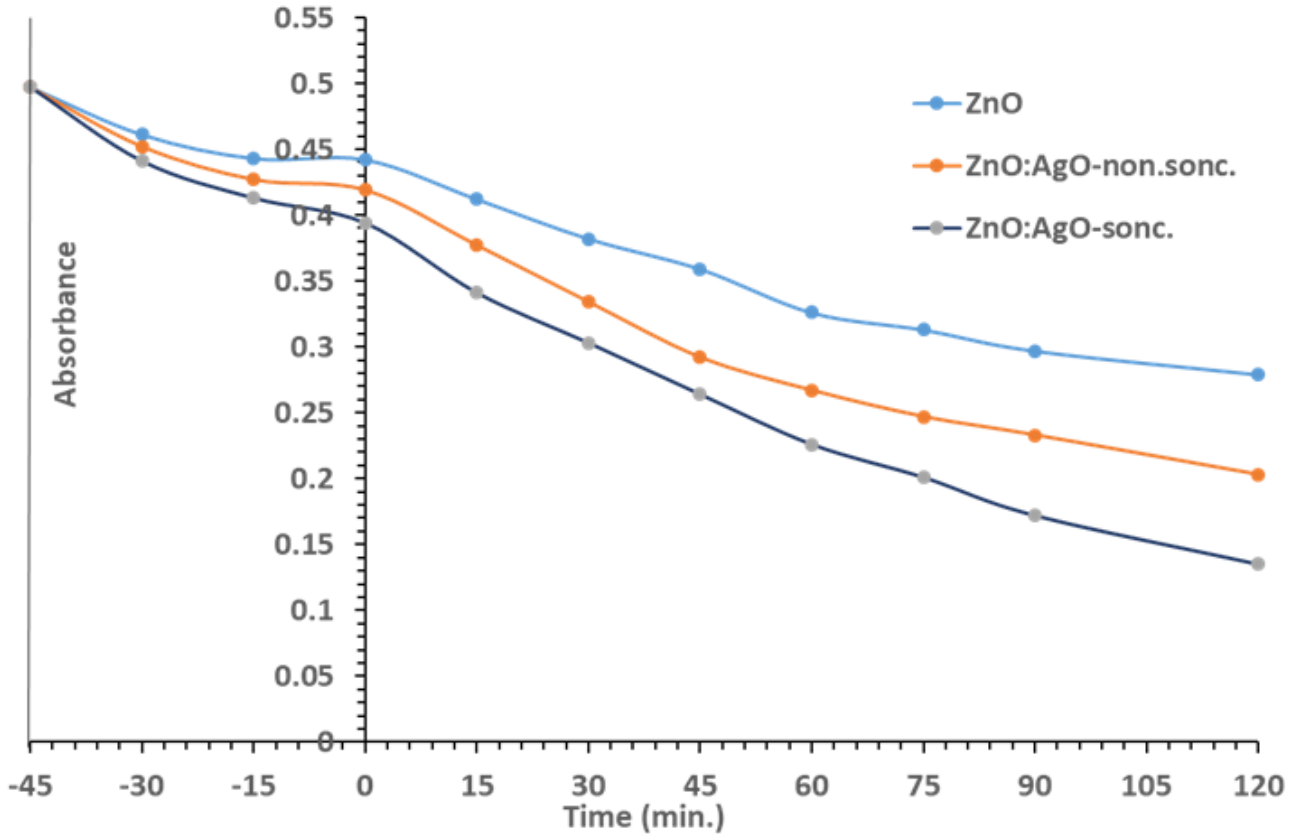

Figure 5. Figure 4: the skim for removel by UV-Light for 15 ppm of TCN by pristine ZnO and ZnO:AgO-non.son. and ZnO:AgO-sonc. at 298.15K.

Many reported literatures shown high agreements with this results in spite of variance in the methodology of prepared nanomaterials.

Photocatalytic Degradation Mechanism:

The role of free radicals in the photocatalytic process represent the primary and critical causes for the activities of nanomaterials after modifications. May be adding radical scavengers, for the Nano-inorganic materials responsible to shown modified activity with higher value of which starting with positive holes and negative electrons and ending with active site which responsible to shown modifying activities [21]. The best experiment capture represent by such as Ag, AgO, and many ether inorganic metallic which able to to capture the positive radicals h^+, and negative radicals e^- to forming more concentration of superoxide $\bullet O2^-$, and$\bullet$OH, respectively [22]. The analysis by XRD patents and results of measuring surface area [23] was concern and recommended forming many active site with Ag and AgO nanoparticles with higher ratios for the samples which prepared by sonication process as shown in propose mechanism in figure 6. The mechanism in figure 1 also refer to important critical parameters which represent by the ratios and distribution of Ag and AgO, when witnessed systematic and more order distribution when used sonication process and that was proven by XRD analysis. The induction from analysis shown less value of agglomerations as compare with the samples which prepared by mixing with magnetic stirrer and that make reduce in the activities.

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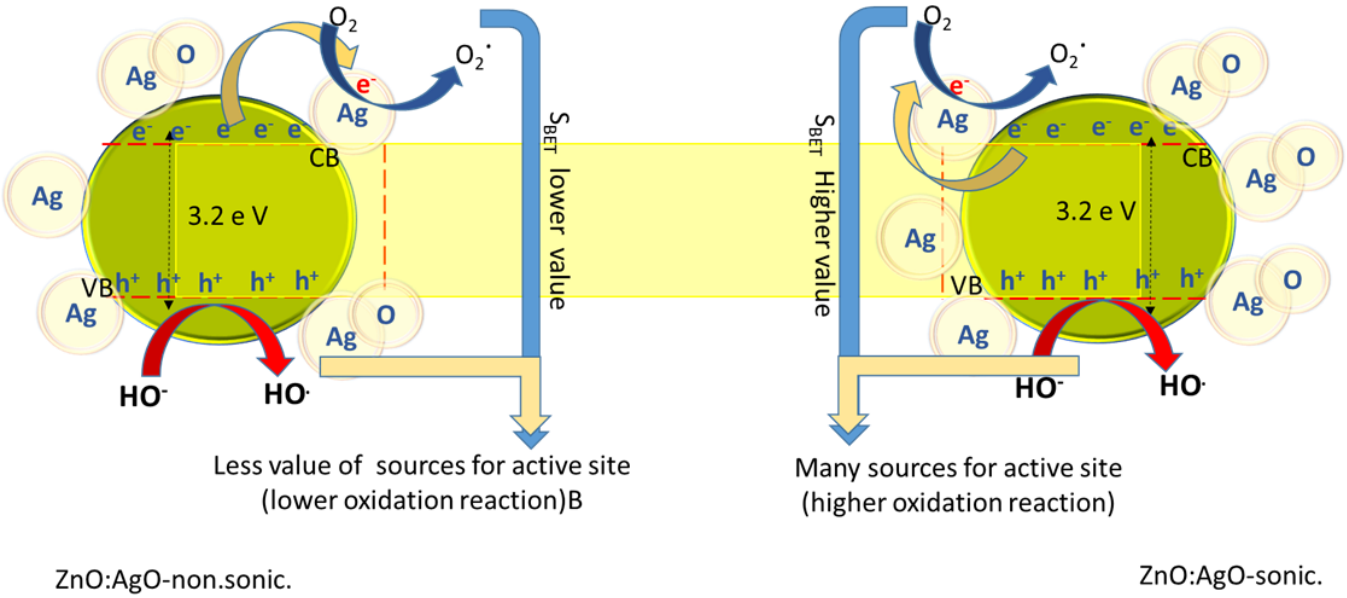

Figure 6. Figure 6: the skim for propose mechanism for ZnO:AgO-non.sonc. and ZnO:AgO-sonc.

Conclusion:

The results of activity for binary composites ZnO:AgO which prepared by mixing with magnetic stirrer or by ultrasonic water bath were shown higher activities as compare with pristine ZnO. The XRD patterns for AgO-sonc and AgO-non.sonc. was shown different in the nature of crystalline and particle size due to the variance in the method of prepaid the two materials. The variance in method give chance to molecules to make less particle size when waves of ultrasonic prevent agglomerations and that give two indicators the first was reduce the particle size and the second was amorphous structure which formed with AgO-sonc. the same effect did not prevent binary composite to shown the same results, inspire of change the surface of precipitation when represent by ZnO , however it was enhance to reduce amorphous structures and reduce practical size with increased the surface area for ZnO:AgO-sonc. which prepared by ultrasonic water bath.

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