





## ZnO-Ag Synthesis Nanoparticles: Liquid Laser Ablation for **Photocatalyst Degradation of Textile Industrial Waste Dyes**

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Information	Abstract			
Article history:				
Received: 22 June 2022	The highest absorption optimization occurred in the photocatalyst degradation test under UV light.			
Accepted: 31 December 2022 Published: 31 August 2023	The highest percentage of photocatalyst degradation was 72.14% in the ZnO/Ag 5 sample. It was adjusted according to the FESEM results for the morphology of the ZnO/Ag nanoparticles and the crystal size on XRD. The synthesis process was carried out using a two-step method in laser			
<b>Keywords:</b> Nanoparticles ZnO/Ag PLAL Photocatalysis Waste-dyes	ablation in liquid (PLAL). The laser method supports photocatalytic performance to degrade textile dye waste. The lowest percentage of degradation was obtained in the UV-Visible t-test of 12.37% as a comparison test. The dark test was carried out at an interval of 0 and 8 hours and observed using a UV spectrometer. The degradation resistance in the dark is 7.61% on ZnO/Ag at 8 hours.			
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## **1. INTRODUCTION**

The textile industry is water and energy intensive and produces high volumes of polluted water including various toxic chemicals [1] and identified as one of the most polluting industries based on the process and type of textile materials used [2]. Textile wastewater contains various chemicals such as oil, caustic soda (NaOH), ammonia, sulfides, lead, heavy metals, textile dyes other toxic substances release large amounts of wastewater as discharge to the environment [3]. Before wastewater with released into the ecosystem, it is important to remove contaminants due to considerable restrictions by the government's pollution control agency [4]. Industry releases various chemical compounds through wastewater disposal and seriously damages natural ecosystems are difficult to degrade [5].

Efficient industry to maintain the quality of wastewater textiles before being discharged into the environment. One of the discoveries of photoelectrochemical water separation provided the impetus for photocatalysts as one of the advanced oxidation processes. Photocatalyst degradation is one of the newest technologies where photocatalysts play a role in the breakdown of organic pollutants [6]. Photocatalyst degradation has been widely used to remove textile reactive dyes in wastewater. Among these methods, photocatalyst is more effective than other conventional methods, due to the generation of highly reactive hydroxy radicals (OH-) and superoxide anions (O2-) under light illumination [7].

Heterocatalysts hold great promise in this regard. Various metal oxides, and metal sulfide heterojunction photocatalysts (SnO2, Bi2WO6, CuO2, TiO2, Cds) have been used for color degradation [8]. Among various photocatalysts, semiconductor photocatalyst is one of the best choices due to its good stability and adjustable band gap. In recent decades, ZnO has been widely developed as an active photocatalyst material because of its strong oxidation properties, nontoxicity ease of availability compared to other materials [9]. ZnO has a band gap of 3.2 eV, has been extensively studied in photocatalysis, and exhibits high photooxidation capabilities [10].

However, the photocatalytic ZnO is limited by the high band gap in the ZnO nanostructure. ZnO requires UV light to absorb high-energy photons with wavelengths below 387 nm and fast charge carrier recombination ZnO photocatalysts also have disadvantages such as low utilization of visible light which limits their application for wastewater treatment [12].

Therefore, it is important to extend the optical absorption of ZnO from UV to the visible light region and block photogenerated electron-hole pair recombination to increase the photocatalytic efficiency of ZnO. The nanostructure of ZnO the modified by Ag, Al, Sn, but the ZnO-Ag pair was chosen because Ag (Silver) can interact with visible light through free-electron resonance in the particle [13]. Agmodified ZnO NPs have shown a significant increase in photocatalytic efficiency for the degradation of both organic and textile dyes [14].

In this research, modified ZnO or Ag nanoparticles will be made as supporting additives to improve the photocatalytic performance of ZnO by synthesizing ZnO/Ag using the laser in liquid (PLAL) ablation technique. The PLAL method has proven to be one of the most promising and applied methods for synthesizing various nanoscale forms based on the resulting material which can be either a single structure or a composite without being harmful to the environment because this process is carried out in a liquid medium and allows the production of high purity materials [15]. According to Jung et.al 2018, ZnO/Ag nanoparticles synthesized using PLAL showed an increase in photocatalytic activity after 94 minutes of irradiation [16].

Thus, the stage of this work is the preparation of dispersion of ZnO/Ag nanoparticles in a liquid by laser ablation technique; obtained colloidal ZnO with the addition of Ag to produce ZnO/Ag nanoparticles and investigation of the mechanism of action of ZnO/Ag photocatalyst for textile dye degradation. This research was divided into three stages, namely: the process of synthesizing ZnO and ZnO-Ag the process of diluting textile dyes as a waste material to be degraded, characterizing ZnO/Ag nanoparticles using UV-Vis and XRD spectrophotometers, and testing of textile dyes degraded by UV-Vis, visible light and dark. For FESEM testing to determine the morphology of ZnO/Ag nanoparticles.

The process of making ZnO and ZnO-Ag colloids was carried out using the pulsed laser ablation in the liquid technique. The mechanism for making the colloid, with a thickness of 1.6x1.6 mm silver plate (Ag). A total of 10 ml of distilled water was coated with a silver plate by a pulsed laser by focusing on variations in Ag ablation of 1 minute, 3 minutes, and 5 minutes, then 6 ml of colloidal Ag volume was coated again with a ZnO disk synthesized for 10 minutes by a laser wavelength of 1064 nm using laser energy. 50 and 100 mJ to produce ZnO/Ag. nanoparticles. Furthermore, it was evaluated using a UV-Vis spectrometer from a wavelength of 500-800 nm. This process can be seen in the illustration of Figure 1 and Figure 2 are colloids synthesized by ZnO/Ag by laser ablation method.

### 2. EXPERIMENT

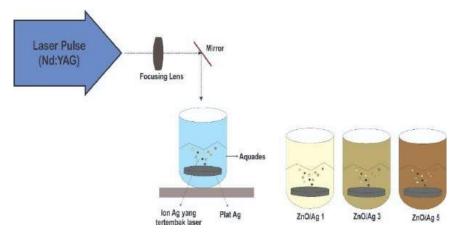


Figure 1. Proccess Sythesis nanoparticles ZnO/Ag



Figure 2. (a) Colloid ZnO, (b) ZnO/Ag 1 min, (c) ZnO/Ag 3 min, (d) ZnO/Ag

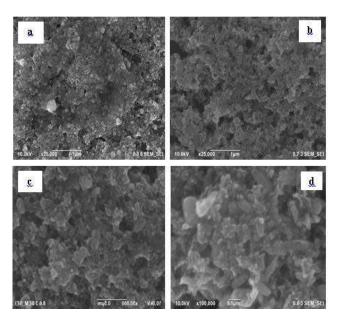
## 4. RESULTS AND DISCUSSION

ZnO-Ag nanoparticles were synthesized by a pulsed laser aimed at characterizing ZnO-Ag nanoparticles and the colloidal color physical properties of ZnO/Ag in Figure 2. The mechanism NP formation during PLAL is known to be a complex nonequilibrium process, depending on the laser and target parameters and the liquid composition as well as including various reactions in high temperature and highpressure plasma. The PLAL process is a cavitation bubble formed from particles and liquid components that are released after plasma formation as a result of the distribution of plasma energy into the ambient liquid [17].

The results of the synthesis are shown in Figure 2. The color of the ZnO colloid is clear, then after being synthesized with the addition of Ag the color begins to change to grayish gray (ZnO/Ag 1). It is clear that the longer the duration of ablation the color of the resulting colloid changes in ZnO/Ag 3 minutes the color of the colloid becomes grayish yellow, and ZnO/Ag 5 min more yellow.

#### 3.1 FESEM morphology of ZnO, ZnO/Ag nanoparticles

The surface morphology of the prepared pure ZnO sample appears as an irregular sphere as shown in Figure 3a which is enlarged at 1µm in size. Pure ZnO showed irregular spherical morphology [18] and irregular spherical shape and nonuniform grain size were evenly distributed. Pure ZnO was clean and smooth [19] ZnO-Ag 1 (Figure 3b) and Figure 3c had a grain size of 0.5 m and ZnO/Ag 5 min (Figure 3d), showing more individual nanomaterial nanomaterials with two different morphologies (nanorods, nanosheets) with magnification ~100 nm [20].



**Figure 3.** FESEM (a)Pure ZnO, (b) ZnO/Ag 1, (c) ZnO/Ag 3, (d) ZnO/Ag 5

### 3.2 XRD (X-Ray Diffraction)

XRD for all samples depicted in Figure 4. confirmed the formation of wurtzite hexagonal ZnO following the standard diffraction pattern of ZnO on the lattice (100, 002, 101, 110, and 102)  $2\Theta$ = 34.37°, 36.21° 56.53°, and 47.48° as crystalline. with peak Ag (200) 44.14° indexed to a cubic structure and constant values similar to ICSD no: 00-002-1098 and ICSD 01-076-0277 confirming ZnO/Ag nanoparticles.

The sharp and intense peak detection proved that the ZnO microspheres were crystalline. These peaks are still clearly visible in the ZnO/Ag XRD pattern, where the diffraction peaks arising in the crystallized plane of the silver (Ag) structure are further detected and there is no significant shift in position due to the ZnO diffraction peaks indicating the presence of Ag ions as additions. does not particularly affect

the ZnO lattice [21]. The following is the calculation of the average crystal size value using Debye Scherrer [22]:

$$D = \frac{\beta \cdot \beta \lambda}{\beta \cos \theta}$$
(1)

where is  $\lambda$  the X-ray wavelength, is the Bragg diffraction angle, and is the half-maximum width of the XRD peak that occurs at the diffraction angle.

Furthermore, in detail the crystal size of ZnO/Ag nanoparticles has been presented in Table 1 as follows:

**Table 1.** The crystal size of ZnO/Ag nanoparticles

Sample	D (average crystal size) nm		
ZnO/Ag 1	18.43		
ZnO/Ag 3	15.33		
ZnO/Ag 5	12.83		

The difference in crystal size of each sample changes the position of the diffraction peaks by the hexagonal wurtizite ZnO crystal plane which indicates a strain effect with increasing laser ablation time. The shift towards a lower angle position shows the value of the strain getting smaller as shown in Figure 4. Strain is one of the important information that can be obtained from the analysis of peak shift and peak broadening of lattice dislocations.

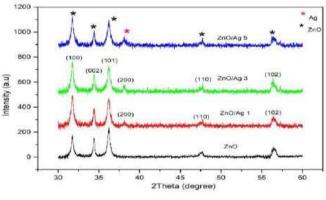


Figure 4. Spectrum XRD ZnO, ZnO/Ag

## 3.3 Absorbance and energy gap of ZnO/Ag nanoparticles

The UV absorption spectrum for pure ZnO is shown in Figure 5. The increase in pure ZnO with modifications to the Ag surface can be explained by the addition of Ag to ZnO which leads to increased absorption of the samples observed and analyzed using UV spectra. [23]. Pure ZnO nanoparticles shows a broad spectrum from 300 to 400 nm. The absorption spectrum of nanoparticles on pure ZnO shows the UV absorption edge at 345 nm and pure Ag at 435 nm The addition of silver to ZnO shifts from the absorption edge towards the major wavelength because the Zn metal is laser ablated. The resulting nanoparticles will come out and spread towards (aqua bikes) and bond with oxygen [24]. The band gap for ZnO is 3.31 eV, ZnO/Ag for 1 minute of ablation is 3.30 eV, ZnO/Ag for 3 minutes is 3.29 eV, and ZnO/Ag for 5 minutes is 3.28 eV. The decrease in gap energy with different ablation times in each sample can affect the quality of colloidal nanoparticles. The increase in ablation time causes the colloid to turn yellow because the Ag molecules involved affect the agglomeration process. The energy gap represents the movement of electrons across the valence band to the conduction band. The small energy band gap value shows that electrons passing through the excitation region can affect the absorbance intensity of nanoparticles.

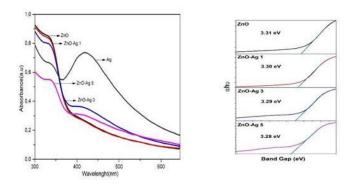


Figure 5. (a) UV-Vis absorption spectra of pure ZnO, ZnO doped Ag and (b) Ag Band gap of ZnO/Ag nanoparticles

In the case of the optical band gap transition and the absorption coefficient are correlated, with the equation  $\alpha hv [A(hv-Eg)]$  which can be defined, where the absorption coefficient can be calculated using the Urbach formula:

$$\alpha(\Omega) = \alpha_0 \operatorname{Exp}\left(\frac{\hbar\Omega}{\Delta E}\right) \tag{2}$$

where  $\alpha_0 = \text{constant } \alpha(0)$  is the absorbent coefficient and energy [25].

## 3.4 Study of UV photocatalyst degradation of ZnO/Ag nanoparticles

Initially, the photocatalytic degradation of textile dyes on UV irradiation has previously been studied. Pure aqua bikes and ZnO under UV irradiation for different times were plotted and shown in Figure 6. A small decrease in the intensity of the emission band. However, the decrease in emission and absorption in pure ZnO began to show an increase with the length of irradiation time, and the loss of characteristics in the absorption band at wavelength (620 nm) after 8 hours of irradiation showed that it was degraded around 22.93%. The highest absorption bands consecutively showed a significant increase from the ZnO/Ag 1, ZnO/Ag 3, and ZnO/Ag samples and occurred at 8 hours of irradiation (Figure 7). So, it can be said that the lower the absorbance value, the lower the concentration of the solution. and it also affects the addition of Ag [26].

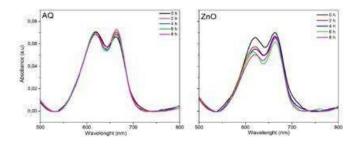


Figure 6. Photocatalyst absorbance of ZnO nanoparticles and Aqua bikes UV light

According to the results of a study conducted by Kumar et.al [27] UV irradiation for MB in various variations of irradiation time. MB degradation increased with the addition of Ag compared to pure ZnO and the absorption band was at a wavelength of 664 nm for MB and was associated with the degradation of ZnO/Ag photocatalyst for textile dyes in the range of 620 nm. In addition, another supporting factor is the result of a report by, Koo et.al [28] that laser ablation can also affect the crystallographic and microstructural characteristics.

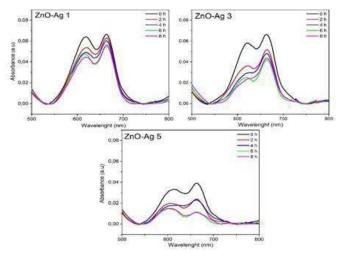


Figure 7. Absorbance UV ZnO/Ag 1, ZnO/Ag 3 dan ZnO/Ag 5

#### 3.5 Activity degradation photocatalyst

The presentation of the degradation can be seen in Figure 8. The absorption peak is at 620 nm. However, the substantial increase in the photocatalytic activity of ZnO/Ag which was observed showed the effect of different operational parameters, by varying the laser ablation time on the ZnO/Ag sample [29]. The degradation performance of textile dyes can be obtained by the following

equation:

$$\% \text{Deg} = \frac{\text{Cawal-Ct}}{\text{Cawal}} \times 100\% \quad (3).$$

The value of degradation at the time of ultraviolet (UV) irradiation for 8 hours was obtained at the highest value of 72.14% in the ZnO/Ag sample 5 as shown in Table 2.

Table 2. Degradation value under UV irradiation

		·			
No	Sample	2 hours	4 hours	6 hours	8 hours
1	Aquades	1.16 %	2.52 %	3.54 %	3.87 %
2	ZnO	12.57%	15.54%	20.16%	22.93%
3	ZnO/Ag 1	15.90%	23.28%	26.13%	30.47%
4	ZnO/Ag 3	37.17%	48.97%	56.76%	58.95%
5	ZnO/Ag 5	38.5%	41.06%	69.22%	72.14%

For this reason, the order of photocatalytic efficiency of Aqubides, pure ZnO, ZnO/Ag 1, ZnO/Ag 3, and ZnO/Ag 5 where at 5 minutes ZnO/Ag laser ablation is higher than ZnO under UV light are shown in Figure 9. Overall performance photocatalytic is better and more competitive [30]. Charge transfer between ZnO and Ag under UV light excitation,

serves as a donor and reduces the band gap in ZnO, and is supported by the morphology of ZnO/Ag nanoparticles.

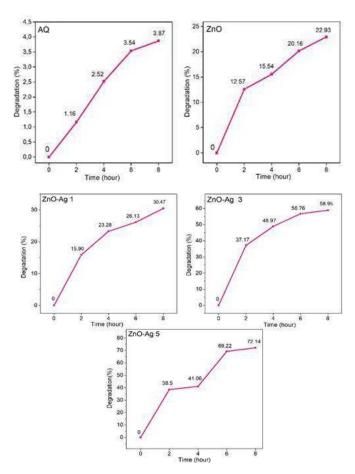


Figure 8. Presentation of UV light ZnO/Ag photocatalyst degradation of textile dyes

The relevant calculation results are in Figure 9, where C0 and Ct are the maximum absorption intensity of textile dyes and the photodegradation rate kinetics through the following equation  $\ln (Ct/C0) = kt$ . Based on the results of the degradation rate kinetics, it was identified that the ability of photocatalyst activity was also supported by a decrease in the band gap caused by the addition of Ag and it can be said that silver can shorten the valence bandwidth with the conduction band thereby increasing the amount of degraded product.

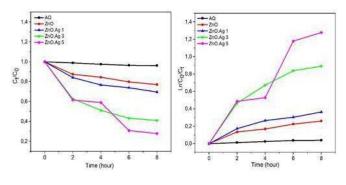


Figure 9. Irradiation time terhadap degradasi fotokatalis ZnO/Ag UV light.

3.6 Study of UV-Visble light photocatalyst degradation

In the degradation test of ZnO/Ag photocatalyst under visible light, the absorbance was very low, it was due to the use of low laser energy in the synthesis process and a low-temperature state, causing Ag ions to be ablated at different time intervals of 1, 3, and 5 minutes also affects the success of ZnO-Ag nanoparticles. In this case, researchers have synthesized ZnO/Ag nanoparticles in a low-temperature state, the results obtained are not so significant that it requires an adequate room temperature so that the photodegradation results are maximal enough for visible light. in visible light conditions. Ag (silver) did not significantly change the value of the ZnO band gap (figure 10a). It was observed that the absorption of ZnO/Ag in the region where there was an increase compared to pure ZnO nanoparticles was highly reflective in the 585 nm region. Increased absorption of visible light is usually associated with surface plasmon resonance of silver nanostructures [31]. The results are shown in Figure 10b. The absorbance of the photocatalyst ZnO/Ag (1,3, and 5) absorption region was at 600-630 nm with a degradation percentage of 12.37% at 8 hours of ZnO/Ag 5 irradiation. .al [32] on the degradation of MB photocatalyst under UV-Visible light irradiation for 8 hours was observed.

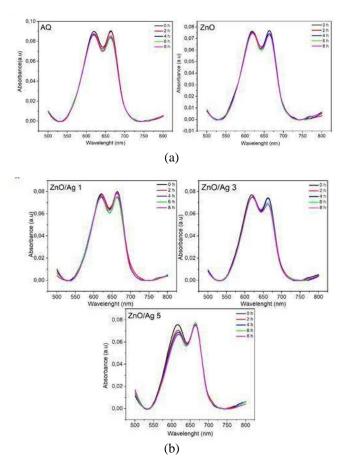


Figure 10. (a) Absorbance aqua bikes, ZnO UV-Visible light (b) Absorbance ZnO/Ag UV-Visible light

# 3.7 Photocatalyst activation of ZnO/Ag UV-Visble light degradation

Figure 11. The photodegradation of textile dyes is considered to be pseudo-first order then, the photocatalytic reaction kinetics can be expressed as follows ln' (C0/Ct) = k+a where t is the irradiation time and k is a first-order constant.

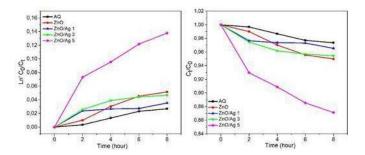


Figure 11. Irradiation time UV-Visible light ZnO/Ag nanoparticles

The photodegradation curve in the form ln (C0/Ct) variation of laser ablation time for Ag of several samples wherein, ZnO/Ag 5 was quite good at visible light irradiation compared to pure ZnO and several other samples. Based on the order of photocatalytic activity for the sample can be summarized as follows: ZnO<ZnO/Ag 1<ZnO/Ag 3<ZnO/Ag 5. In this case, confirming that Ag in sufficiently large concentrations can also improve photocatalytic performance. The data has been confirmed in Figure 12.

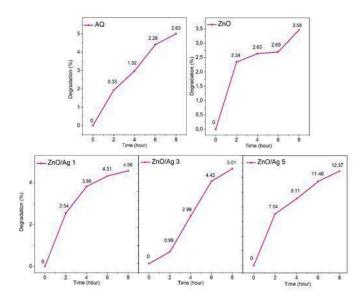


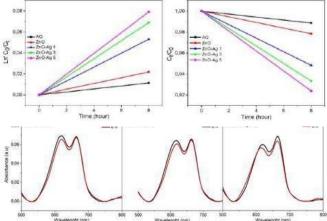
Figure 12. Curve presence degradation ZnO/Ag for UV- Visible light irradiation time

The presentation contained a fairly good ZnO/Ag 5 curve from all samples. In the ZnO/Ag 3 curve, the percentage degradation value was only 5.01% at 8 hours, then ZnO/Ag 1 was at 4.56% and very low in pure ZnO at 3.58%.

### 3.8 Dark-UV Test

The dark test on the sample was carried out as a comparison for photocatalytic activity and degradation intensity in the dark where the sample showed that the degradation ability of textile dyes was also successfully carried out although the results obtained were very low. The absorption absorbance is at a wavelength of 600 nm (Figure 13). The results of the absorption curve against time are shown in Figure 14.

The order of presentation dark test degradation within 8 hours can be summarized as follows: Aqua bikes (1.13%)<ZnO(2.16%)<ZnO/Ag1, (5.17%)<ZnO/Ag3, (6.66%)<ZnO/Ag 5 (7.61%). Observations that were left in



the dark the absorbance of the dye solution did not change [33]. Figure 14 ln' (C0/Ct) shows a good linear relationship to the illumination time according to the Quarsi-first law of kinetics [34].

Figure 13. Dark-UV absorbance test for textile dyes

Figure 14. Irradiation time dark-UV test

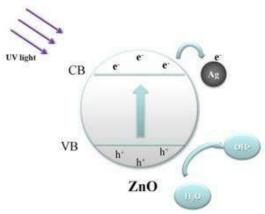


Figure 15. Systematic diagram of photocatalyst

The following is a systematic flow of photocatalyst degradation for textile dye waste.

$$ZnO + hv \rightarrow ZnO (e^{-} + h^{+}) e^{-} + Ag^{+} \rightarrow Ag$$
$$h^{+} + H2O \rightarrow OH^{*} + H^{+}$$
$$OH^{*} + product \rightarrow CO2 + H2O$$
$$e^{-} + h^{+} \rightarrow recombination$$

When light shines on ZnO, it absorbs light at a certain wavelength, then transfers electrons from the valence band to the conduction band so that the hole results in the formation of hydroxyl radicals which then reduces the adsorbed pollutants to the photocatalyst surface. Electrons and holes recombine and separate simultaneously. Furthermore, Ag regulates electron transfer so that it effectively separates electrons and holes which can increase photocatalytic efficiency.

## **5. CONCLUSION**

ZnO, ZnO/Ag nanoparticles were successfully carried out using the Pulsed laser ablation in liquid (PLAL) synthesis method which was applied to degrade textile dyes by observing a UV spectrometer. Samples were characterized using FESEM, XRD, and Degradation testing was carried out in three stages, namely: UV light, UV-Visible light and darkThe best degradation results were obtained from ZnO/Ag (5) 72.14% irradiation time of 8 hours. From all the tests, the increase in the percentage of degradation occurred consistently in all samples.

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