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Green Synthesis of Silver Nanoparticles using Ketapang Leaf Extract (*Terminalia Catappa* L.) Assisted by Ultrasound for Photodegradation of Methylene Blue

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Abstract

Green synthesis of silver nanoparticles using plants has been interesting in recent years. In the present study, the silver nanoparticles were synthesized using a bioreductor from ketapang leaf extract (*Terminalia catappa L.)* assisted by sonochemical methods. This green synthesis provides an economic, eco-friendly, and clean synthesis route for silver nanoparticles. Different concentrations of $AgNO_3$ precursors (0.5; 1.0 and 1.5 mM) were initially reacted with ketapang leaf extract with PVA 1% solution as stabilizers that were sonicated for 30 minutes. Silver nanoparticle colloidal solutions were characterized using UV-Vis spectrophotometer, Particle Size Analyzers (PSA), and Transmission Electron Microscopes (TEM). Maximum absorption of silver nanoparticles was obtained at wavelength 420-450 nm. Based on UV data, the silver nanoparticles showed stability for up to 3 weeks. The XRD peaks indicated that the (111) crystallographic plane was more predominant than other planes. The average size of the silver nanoparticles was 79.7 nm from the PSA result. TEM imaging depicted that the nanoparticles were spherical. Finally, the result proved that the silver nanoparticles effectively removed the methylene blue up to 76.43% within optimum conditions (3 ppm of methylene blue, 15 minutes contact time, and 8% nanoparticle concentration).

INTRODUCTION

Nowadays, nanotechnology is undergoing rapid and remarkable development, playing a crucial role in a multitude of scientific fields (Canama et al., 2023). Silver nanoparticles have become as a subject of significant interest (Patabang et al., 2019). Nanoparticles, solid or colloidal particles with diameters falling within the 1 to 100-nanometer range, have exhibited paramount versatility and the potential for numerous applications. Silver nanoparticles, in particular, have captivated the scientific community due to their unique properties, including size-dependent properties which is the ability to adjust optical, electrical, and magnetic characteristics based on their size and shape (Maarebia et al., 2019). Silver nanoparticles are emerged as promising materials for textile dye degradation (Rajkumar & Sundar, 2022a), photocatalytic activities (Aryan et al., 2021), antibacterial (Ontong et al., 2019), anti-fungal, antioxidant activities (Rajkumar & Sundar, 2022b), etc.

Several methods developed in synthesizing silver nanoparticles are physical or chemical methods, obtaining nanoparticles in the desired shape, size, and composition. However, these approaches suffer from inherent drawbacks, including high production costs and adverse environmental impacts. To address these

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limitations, environmentally friendly and non-toxic alternative methods for silver nanoparticle synthesis are imperative. One approach, known as the "green synthesis method" offers diverse advantages such as eco-friendly, low-cost, and minimal energy requirement. In this method, plants are used as bioreductants for the synthesis of silver nanoparticles due to the presence of secondary metabolites within them, which function as reducing agents (Karimah et al., 2020). Ketapang leave is a well-known and abundant plant in East Kalimantan. The use of ketapang leaf extract can be an alternative reductant of natural materials that are environmentally friendly and economical. Additionally, this technique was merged with sonochemistry. Sonochemistry utilizes powerful ultrasonic waves to generate acoustic cavitation in a liquid, thereby enhancing or facilitating specific chemical reactions (Mekaru, 2023). The utilization of sonochemical method aimed to minimize synthesis duration and achieve nanoparticles with reduced size and improved dispersion.

Meanwhile, the prevalence of organic pollutants stemming from industrial sources like plastic, paint, textile, and paper has emerged as a significant concern in the realm of environmental degradation (Lü et al., 2024). The obstruction caused by organic dye pollution, a byproduct of industrial waste, has far-reaching implications. Moreover, synthetic dyes present in these pollutants pose a severe threat to human health. The chemicals inherent in these dyes can trigger various health hazards upon exposure, highlighting the urgency of addressing this issue comprehensively. Thankfully, innovative methodologies have been devised to mitigate these challenges. Techniques such as catalytic degradation, where pollutants are broken down using catalysts, and fabric filtration, which involves using specialized materials to sieve out contaminants, have shown promise in tackling organic dye pollution (Jothi Ramalingam et al., 2021).

Several methods can be used to synthesize nanoparticles such as chemical reduction, sonochemistry, photochemistry and so on. Nanoparticle synthesis using sonochemical techniques uses ultrasonic tools to break down metal solids into nano-sized particles (Lu, et, al., 2008). Herein, silver nanoparticles (AgNPs) have been synthesized through sonochemical methods by applying ultrasonic wave radiation (20 KHz to 10 MHz) or using an ultrasonic bath (sonicator) in which AgNPs of different sizes are produced. Physical phenomena in sonochemistry involve cavitation and nebulization. In sonication, cavitation involves the formation, growth, and bursting of bubbles in creating suitable conditions for the synthesis of nanoparticles (Kithokoi et al., 2019). This study explores the application of ultrasonic-assisted green synthesis employing ketapang leaf extract as a natural bioreductant for the production of AgNPs.

EXPERIMENTAL DESIGN

Materials

Materials and solvents were purchased without further purification. $AgNO_3$ and polyvinyl alcohol (PVA) were purchased from Sigma Aldrich. Methylene blue was purchased from Merck, Fresh Ketapang leaves were collected from Lempake Village Samarinda, East Kalimantan, Indonesia.

Extract Preparation of Ketapang Leaf

Fresh Ketapang leaves, 100 g, were harvested, washed, and cut into small pieces. Then, the leaves were air-dried at room temperature. The dried leaves were ground into a fine powder. For the preparation of the Ketapang leaf extract, 10 g of the leaf powder was mixed with 150 mL of distilled water and then stirred at 60°C for 1 hour. Afterward, the resulting solution was filtered through filter paper to remove any solid particulates. The resulting extract was stored at 4°C and remained stable for up to two weeks, suitable for the next step.

Synthesis of AgNPs Using Ketapang Leaf Extract with Ultrasound Assistance

For this synthesis step, 20 mL of $AgNO_3$ with a concentration of 0.5; 1.0, and 1.5 mM respectively was put into a beaker glass, then added ketapang leaf extract (5% v/v) to the volume of $AgNO_3$. It was put into a Beaker glass flask, and then 1 mL of 1% PVA solution was added. The flask was immersed in an ultrasonic bath for 30 minutes, until the color changed from yellowish to brown, indicating that the nanoparticles had been formed.

Characterization of AgNPs

UV-Vis was used to monitor the formation of AgNPs. The peaks of colloidal nanoparticles range between 400-450 nm. The absorbance of AgNPs was measured continuously for 7 days.

AgNPs with the highest stability were characterized using PSA and TEM. PSA aims to determine the size distribution of nanometer-sized particles. The use of TEM for the characterization process aims to determine the surface morphology information of the synthesized AgNP*s* that are displayed in the form of images.

Photodegradation Activity

Degradation of methylene blue dye using AgNPs with varying photocatalytic contact time

8% of the most stable AgNPs were added into a 25 mL solution containing 5 ppm of methylene blue. Then, the resulting mixture was transferred into a reactor, irradiated with a UV lamp, and stirred with a magnetic stirrer with a variation time of 5, 10, 15, 30, 45, 60, and 90 minutes. Afterwards, the suspension was centrifuged for 10 minutes and 2 mL filtrate was taken. Then the absorbance value was measured using a UV-Vis spectrophotometer, enabling the calculation of the percentage degradation of methylene blue.

Degradation of methylene blue with varying AgNPs Catalyst Volume

25 mL of methylene blue solution with a concentration of 5 ppm was put into a glass beaker and 4; 8; 12 and 16% AgNPs were added respectively. The mixtures were transferred into a reactor irradiated with a UV lamp and stirred with a magnetic stirrer for the optimum contact time (15 minutes). The resulting mixtures were centrifuged for 10 minutes, then 2 mL filtrate was collected. The absorbance value was measured using a UV-Vis spectrophotometer and the % degradation was calculated.

Degradation of methylene Blue Using AgNPs with Varying Methylene Blue Concentrations

AgNPs with the highest stability at 8% concentration were placed into three 50 mL Beaker glasses, to which 25 mL of methylene blue, 3; 5; and 7 ppm respectively, was added. Subsequently, these mixtures were transferred to a reactor and exposed to UV light for 15 minutes. Following this, the resulting mixtures were centrifuged. The filtrate (2 mL) was collected. The absorbance values were then measured using a UV-Vis spectrophotometer, and the degradation percentage was calculated.

The percentage degradation of methylene blue by AgNPs was calculated using Eq. (1).

$$\%Degradation = \frac{C_0 - C_e}{C_0} \times 100\% \qquad (1)$$

C_o represents the initial concentration of the methylene blue solution, while C_e signifies the concentration of the methylene blue solution after degradation

RESULTS AND DISCUSSION

Optimization of AgNPs Synthesis

The reduction method involved the utilization of a bioreductant combined with varying concentrations of $AgNO_3$ (0.5, 1.0, and 1.5 mM), followed by the addition of 1% PVA as a stabilizing agent. Subsequently, the synthesis of AgNPs was facilitated through ultrasonic waves (sonication) for 30 minutes. During the synthesis process employing ketapang leaf extract as the bioreductant, the silver ions (Ag^{+}) in $AgNO_3$ were reduced to elemental silver (Ag^0), resulting in the formation of a brownish solution, as illustrated in Figure 1.

Figure 1. Synthesis of AgNPs with various concentrations of $AgNO_3$: (a) 0.5 mM; (b) 1.0 mM and (c) 1.5 mM.

The resulting mixtures (Figure 1) appeared in the form of a brownish solution which indicates the formation of AgNPs. There were differences in the colour of the solutions formed. This indicates that the number of nanoparticles formed varies. The more intense the colour, the greater the concentration of nanoparticles, the greater the number of nanoparticles. However, this requires reconfirmation with other characterisations. The indication was also strengthened by the UV-Vis spectrophotometer data. UV-Vis absorption spectra of synthesized AgNPs at various concentrations of $AgNO_3$ (0.5; 1.0 and 1.5 mM) are shown in Figure 2. At a concentration of 0.5 mM, a maximum absorption peak occurred at a wavelength of 430 nm. Upon increasing the concentration to 1.0 mM, an absorption peak appeared at a wavelength of 420 nm and was still prominent at a concentration of 1.5 mM. The data implies that the higher the

absorbance value corresponded the greater the number of AgNPs formed.

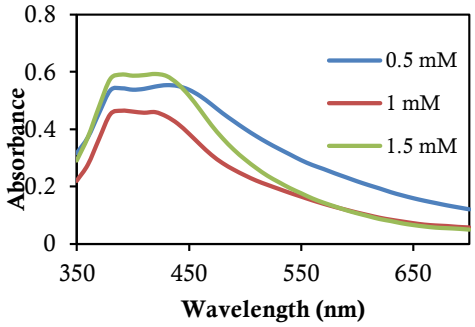
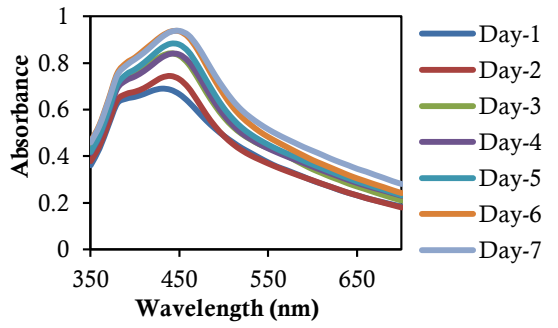
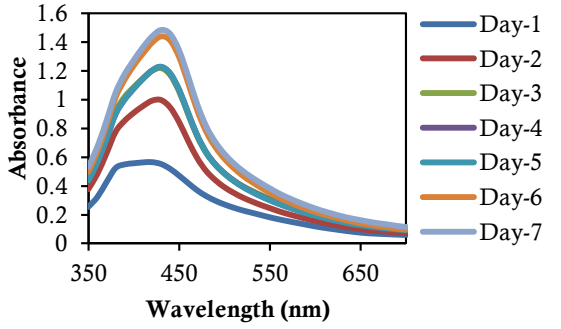


Figure 2. UV-Vis spectra on the formation of AgNPs based on the variation of $AgNO_3$ concentration 0.5; 1.0 and 1.5 mM.



(a)



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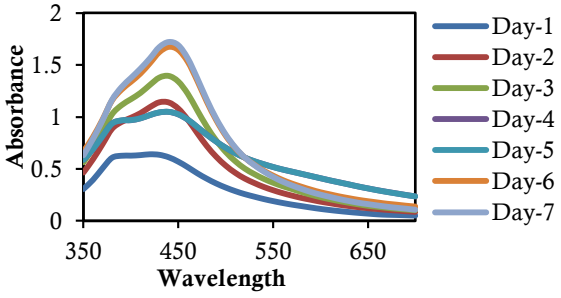


(c)

Figure 3. The stability of silver nanoparticles with bioreductant for 7 days (a) $AgNO_3$ 0.5 mM, (b) $AgNO_3$ 1.0 mM, and (c) $AgNO_3$ 1.5 mM.

After AgNPs were formed, the stability of the them was assessed for 7 days using a UV-Vis spectrophotometer with a concentration variation of 0.5 mM $AgNO_3$; 1.0 mM, and 1.5 mM. A noteworthy observation in this study was a redshift or shift towards longer wavelengths in the absorption peak which signified reduced stability of the colloidal AgNPs solution, likely due to agglomeration. The data obtained from the investigation is presented in Figure 3.

Stability data at a concentration of 0.5 mM and 1.5 mM indicated an unstable state as shown by absorption peaks at a wavelength of 430-450 nm and 420-440 respectively. The shift in the wavelength peak was attributed to the agglomeration, resulting in the formation of dark color suspension. In contrast, a concentration of 1.0 mM showed stability due to the shift of the absorption peak at a wavelength of 420 nm to 430 nm. Therefore, the optimum concentration of $AgNO_3$ for the synthesis of AgNPs was 1.0 mM. In addition, the optimization was also performed with $AgNO_3$ concentration of 1.0 mM and varying bioreductant concentrations of 2.5%; 5%, and 7.5% (v/v). Figure 4 depicts the color change of the AgNPs solution formed based on variations in the volume of the bioreductant. In this process, a brownish-yellow solution was an indication of the formation of AgNPs. The absorption data of the resulting mixture was analyzed using a UV-Vis spectrophotometer and the data were shown in Figure 5.

(a) (b) (c)

Figure 4. The results of the synthesis of 1.0 mM silver nanoparticles using a bioreductant of Ketapang leaf extract with a volume variation of (a) 2.5%; (b) 5%, (c) 7.5% (v/v)

The UV-Vis data, Figure 5, above confirmed a significant influence of bioreductants quantity on nanoparticle formation. When synthesizing AgNPs with a 2.5% bioreductant concentration, a wavelength of 430 nm was achieved, accompanied by an absorbance value of 0.537. Similarly, using a 5% bioreductant

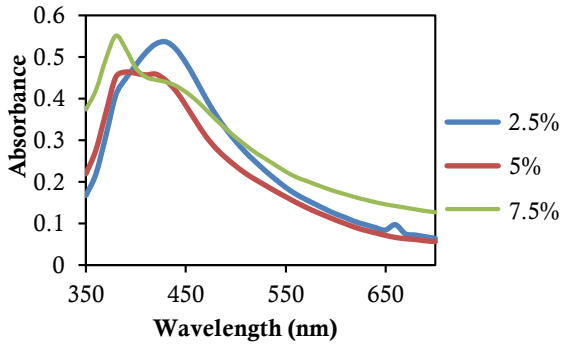


Figure 5. UV-Vis spectra on the formation of AgNPs based on variations in the number of bioreductors 2.5; 5 and 7.5%.

concentration yielded a wavelength of 420 nm with an absorbance value of 0.459. However, when a 7.5% bioreductant concentration was employed, the resulting wavelength was 380 nm, with an absorbance value of 0.551. From the results, 2.5% and 5% of bioreductants were confirmed to facilitate the formation of AgNPs with desired maximum UV-Vis absorption at 400-500 nm (Popov et al., 2015). Flavonoid, alkaloid, and phenolic compounds containing aldehyde functional groups in plants were able to function as reducing silver ions in the synthesis of AgNPs (Prasetyaningtyas et al., 2020). Functional groups in secondary metabolites donated electrons to Ag^{+} ions to produce AgNPs.

After AgNPs were formed, the stability of AgNPs was determined for 7 days using a UV-Vis spectrophotometer with a concentration of 1.0 mM $AgNO_3$ bioreductant volume variation of 2.5%; 5% and 7.5% (v/v), the resulting data can be seen in Figure 6.

Based on Figure 6, AgNPs with a 2.5% bioreductant indicated stability as a minor shift occurred at a wavelength of 430 nm to 440 nm. On AgNPs with a bioreductant variation of 5%, it shows that the graph formed is quite stable, because shift of the absorption peak at a wavelength of 420 to 430. Then AgNPs with a volume variation of 7.5% bioreductant tend to be unstable. Due to the shift in the absorption peak at the wavelength of 380-440. This is presumably because the formed AgNPs have undergone agglomeration, namely clumping or accumulation of particles into one. According to Prasetyaningtyas et al., (2020) the large surface tension causes the cohesion between the particles to be greater so that interactions between AgNPs are more likely to occur. This causes the size of AgNPs to tend to be larger

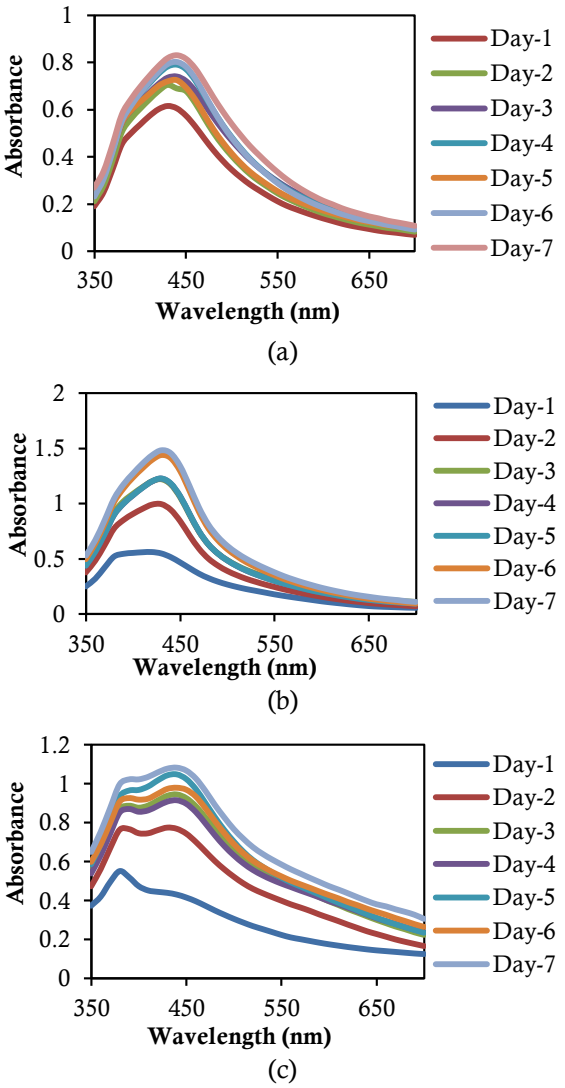


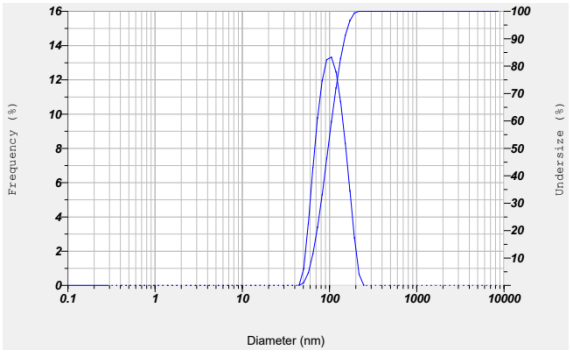
Figure 6. The stability of silver nanoparticles with bioreductors for 7 days with variations in the amount of bioreductors (a) 2.5% (b) 5% and (c) 7.5%.

because the clusters formation. Additionally, several factors such as reducing agents and reaction time can contribute to the nanoparticle size. Excessive bioreductants interfere with nucleation formation due to their high abundance in the medium, resulting in larger nanoparticle clusters. This causes the size of AgNPs to tend to be larger because the clusters formation. Furthermore, the large nanoparticle size may be influenced by several factors such as reducing agents and reaction time. To sum up, the volume of the bioreductant that produces the most stable AgNPs was 5% (v/v).

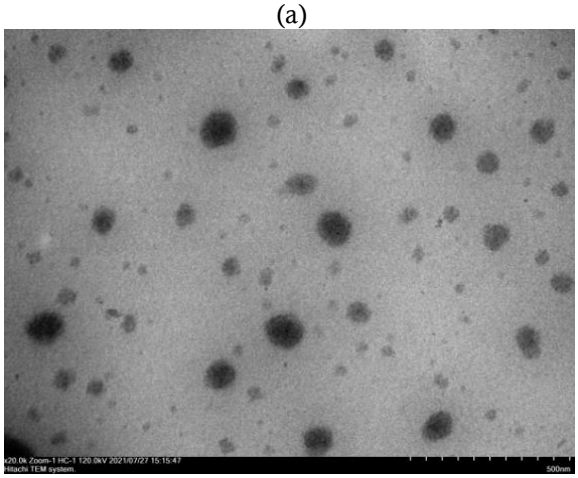
Characterization of AgNPs

Based on PSA data (Figure 7), the particle size distribution of AgNPs from Ketapang leaf

extract ranged from 50.53 to 193.48 nm with an average of 79.7 nm with a polydispersity index (PI) value of 0.268. The average size distribution of AgNPs was below 100 nm. It can be concluded that ultrasonic-assisted synthesis of AgNPs may offer control over their size (Sumaiti, et al., 2018). Based on the results of the TEM characterization above, as shown in Figure 7, the morphology of AgNPs was spherical but tended to be less uniform. This is due to the aggregation of AgNPs influenced by the concentration of $AgNO_3$ precursors and the purity of the reducer (Dony & Aziz, 2013).



(a)



(b)

Figure 7. (a) PSA Images (b) TEM Images of silver nanoparticles.

Degradation of Methylene Blue Dye Using AgNPs

Variation of Contact Time on Methylene Blue Degradation

Variations in contact time were carried out to determine the optimum contact time for using 8% (v/v) AgNPs catalyst in 25 mL of 5 ppm methylene blue. The data were presented graphically as the relationship between percent degradation and contact time, is illustrated in Figure 8.

The results from varying the catalyst contact time with methylene blue indicated 71.54% degradation in 5 minutes. This lower result can be attributed to the short contact time of the catalyst with methylene blue that caused the degradation process to be not optimal. In the 10, 15, 30, 45, 60 and 90 min, the degradation percentage increased to 74.24; 75.4; 75.74; 76.22; 77.1 and 78.08 (%) respectively. The data revealed that the longer the catalyst contact time with *methylene blue,* the higher the percentage of degradation and at 90 min the degradation still ongoing, this trend was same according to Hindryawati et al. (2020) and Widihati et al., (2011). Moreover, the radiation contact time bolstered the photodegradation process which resulted in a higher amount of dye degraded (Suprihatin et al., 2022). However, 15 minutes was determined as the optimum contact time because the increase in degradation percentage from 30-90 minutes did not exhibit a significant difference.

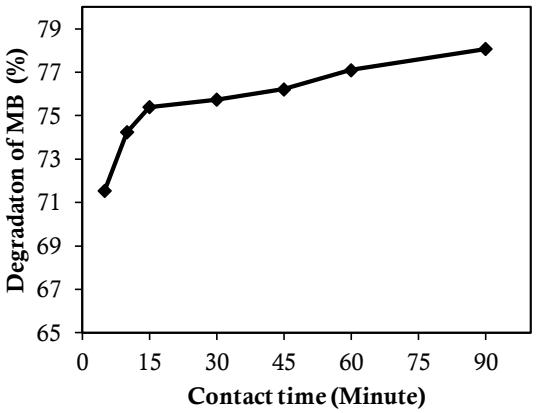


Figure 8. Variation of contact time on % degradation (8% (v/v) number of nanoparticles in 5 ppm methylene blue concentration).

Catalyst Loading Variation of AgNPs Catalyst in Degradation of Methylene Blue

Varying the volume of the AgNPs catalyst was carried out to determine the optimum amount of AgNPs in the photocatalytic process when contacted with 5 ppm methylene blue. The use of AgNPs catalyst in the methylene blue photodegradation reaction began with irradiation with UV light. The influence of the volume of AgNPs on the % degradation of methylene blue was illustrated graphically as the relationship between percent degradation and percent volume of AgNPs shown in Figure 9.

The results of variations in the volume of the AgNPs catalyst showed that with a 4% volume

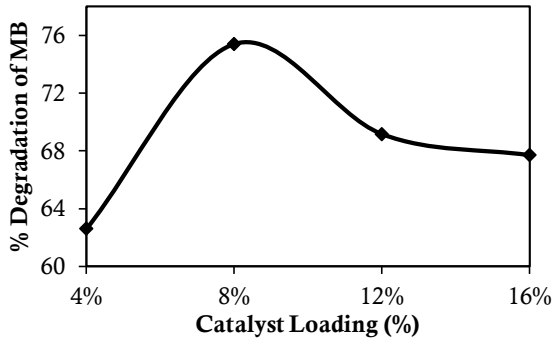


Figure 9. Variation in the amount of AgNPs catalyst (methylene blue concentration 5 ppm within 15 minutes).

of AgNPs, a degradation percentage of 62.6% was obtained. The relatively low percentage of degradation was due to the limited presence of AgNPs catalyst, resulting in reduced interaction with methylene blue. Then, at 8% volume of AgNPs catalyst, the largest percentage of degradation was achieved at 75.4%. This significant improvement in degradation percentage was linked to the increased catalyst volume, leading to a greater presence of hydroxide radicals and superoxide ions. These reactive species play a crucial role in the degradation of methylene blue. However, when the AgNPs catalyst volume was further increased to 12% and 16%, the degradation percentages decreased to 69.16% and 67.7%, respectively. This decline is likely due to an excessive amount of catalyst, which may cause the methylene blue solution to become cloudy and potentially disrupt the degradation process.

Variation of Methylene blue Concentration in Photocatalysis using AgNPs

The aim of varying the concentration of methylene blue was to determine the optimum concentration of methylene blue used in the photocatalytic reaction as it combined with an 8% volume of AgNPs catalyst. The data was obtained in the form of a graph showing the relationship between % degradation and methylene blue concentration.

Based on Figure 10, the degradation of methylene blue achieved 76.43% at a concentration of 3 ppm. However, at concentrations of 5 and 7 ppm, the degradation decreased to 75.4% and 64.83%. The results showed that the degradation rate in the photocatalytic process decreased with increasing methylene blue concentration. This decrease in degradation percentage can be attributed to the reduced effectiveness of the catalyst in generating radicals. The extensive presence of methylene blue molecules tends to obstruct the surface of AgNPs, hindering their ability to absorb photons from UV light optimally. As a result, the photon absorption process becomes less efficient.

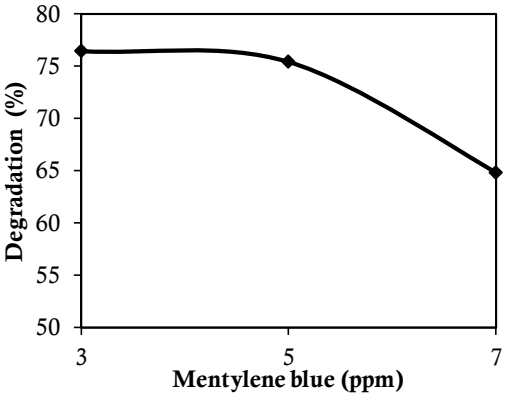


Figure 10. Variations in Methylene Blue Concentration 3; 5 and 7 ppm against AgNPs catalyst (Amount of AgNPs 8% (v/v) in 15 minutes contact time).

CONCLUSION

In this research, AgNPs were efficiently synthesized using ketapang leaf extract as bioreductant and assisted by ultrasound. The nanoparticles exhibited stable properties, with a maximum absorption wavelength of 420-450 nm, and an average size of 79.7 nm. These green-synthesized nanoparticles demonstrated excellent methylene blue removal capabilities, achieving up to 76.43% removal under optimized conditions (3 ppm methylene blue, 15 minutes contact time, and 8% nanoparticle concentration). This method offers an eco-friendly and cost-effective route for AgNPs production with potential photocatalytic applications.

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