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Green Synthesis AgNPs Immobilized to Whatman Paper Using *Chromolaena odorata* Extract and Its Application as Photocatalyst

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Abstract
Synthesis and immobilization of silver nanoparticles (AgNPs) to Whatman paper carried out using *Cromolaena odorata* extract irradiated by sunlight. The UV-Vis shows silver nanoparticles successfully formed from $AgNO_3$ 1 mM, 2 mM, and 3 mM with the absorption peaks at 455 nm, 452 nm, and 451 nm, respectively. The SEM shows AgNPs are spherical with an average particle size are 60.45 nm, 63.19 nm, and 68.42 nm, respectively. The EDX spectrum shows the composition of silver immobilized to Whatman paper increase with increasing concentration of 10.14Wt%, 64.48Wt%, and 70.48 Wt%, respectively. AgNPs/Whatman paper has a cubic crystal structure, space group Fm-3m, lattice parameter (*a*) 4.0862 Å and crystal size of 42.94 nm. FTIR reach peaks at 520.78 cm^{-1} and 1,059 cm^{-1} explains the vibration of the Ag-O bonds indicated the formation of AgNPs. Furthermore, the photocatalyst ability for dye degradation was evaluated using methylene blue 10 ppm under sunlight for 6 hours. The result shows a changed colour to fade and decreases the absorbance ability of methylene blue. Therefore, it can be concluded that green synthesis and immobilization of AgNPs/Whatman paper have a potential to be applied as photocatalyst materials for dye degradation.
Keywords: Green synthesis, immobilization, AgNPs, photocatalyst.

1. Introduction

Silver nanoparticles (AgNPs) as photocatalysts are reported to have an effective, efficient, and very stable for dye degradation [1]. It is required to produce a material that can be adjusted as a photocatalyst medium to optimize its utilization as photocatalysts. Immobilization of AgNPs into a solid matrix has the potential to be applied as a catalyst, antimicrobial, and electronic material [2]. Based on its characteristics, the function of paper-based materials has become an interesting topic because of its porosity and high absorbance, biodegradable, inert, mesoporous structure, and environmentally friendly [3], [4]. Several studies have successfully demonstrated the immobilization of AgNPs in the Whatman paper [5]–[7]. The research involved using chemicals such as NaOH, $NaBH_4$, and NH_4OH within 2 to 18 hours to synthesize and immobilize AgNPs. In addition, no studies have reported its ability as a photocatalyst for dye degradation. Therefore, the utilization of green synthesis in this study is an alternative method to replace the use of chemical compounds.

Green synthesis is a synthesis method of nanoparticles by utilizing a natural material, such as plant extracts or microorganisms [8]. This method is simple and environmentally friendly because it does not use chemicals, low cost, easy to handle, and can be used on a large scale. Plant extracts are widely used because of the presence of phytochemical components that can act as reducing agents as well as stabilizing agents and capping agents [1], [9], [10]. This can be evidenced by several studies that have successfully demonstrated the photocatalyst ability of AgNPs to be synthesized from *ndaliman* fruit extracts [1], basil leaves [11], *Angelica gigas* [12], *Hibiscus sabdariff* [9], and *Gmelina*

arborea fruit [10]. AgNPs were synthesized using *Cordia dichotoma* leaf extract showed that methylene blue degradated after irradiated under sunlight for 6 hours. This study successfully demonstrated the potential of AgNPs as photocatalysts to degrade methylene blue under sunlight [13].

Cromolaena odorata contains main components such as phenols and flavonoids [14], which can reduce Ag ions. Previous research has shown that *Cromolaena odorata* leaf extract can reduce Ag ions to form AgNPs with an average particle size of 27.86 nm for 120 minutes by hydrothermal and its application as antibacterial [15]. Another study explains that irradiation by the sun during the synthesis process can accelerate the formation of AgNPs and produce nanoparticles with a size of 5–63 nm [16]. In this study, synthesis and immobilization of AgNPs on Whatman paper will be carried out using *Cromolaena odorata* leaf extract irradiated by the sun for 2 hours. AgNPs that have been successfully immobilized on Whatman paper aim to be applied as a photocatalyst material for dye degradation.

2. Method

2.1. Materials

Silver nanoparticles (AgNPs) were synthesized from precursor silver nitrate (AgNO3). Other materials include *Cromolaena odorata* leaves, Whatman filter paper (3 × 3 cm), distilled water, and methylene blue.

2.2. Preparation of Cromolaena Odorata Leaf Extract

Preparation of *Cromolaena odorata* leaves from the local area of Sumbawa, Indonesia, shown in Figure 1. The leaves were washed and dried under sunlight and then mashed. The 2.5 grams of *Cromolaena odorata* powder were dissolved in 100 mL distilled water and stirred using a hotplate stirrer for 15 minutes at 60 °C. Then, the mixtures were filtered to obtain a *Cromolaena odorata* extract [15].

2.3. Synthesis and Immobilization of Silver Nanoparticles on Whatman Paper

Silver nanoparticles are synthesized from $AgNO_3$ solution with various concentrations, which 1 mM, 2 mM, and 3 mM in 50 mL. Whatman paper (3 × 3 cm) is soaked in $AgNO_3$ solution and then added 5 mL of *Cromolaena odorata* extract with the ratio of leaf extract to precursor solution being 1 : 9. The mixtures are irradiated by the sun for 2 hours [16]. Then, AgNPs/Whatman paper is washed using distilled water and dried using the oven at 100 °C.

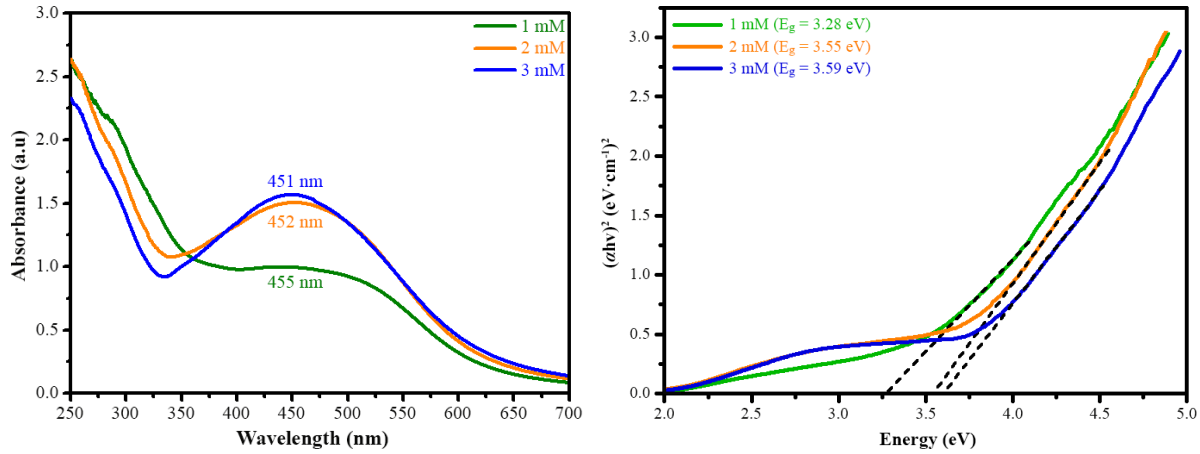
2.4. Photocatalyst Evaluation

AgNPs immobilized on Whatman paper are tested for it photocatalyst ability using methylene blue. AgNPs/Whatman paper is put into 50 mL methylene blue 10 ppm, then irradiated with sunlight for 6 hours [13]. As a comparison, the same process was carried out in the Whatman paper.

2.5. Materials Characterization

To identify the silver nanoparticles formed and its photocatalyst activity, a UV-Vis spectrophotometer is observed at a wavelength of 200–800 nm. The crystal size and structure, as well as the lattice parameters formed on AgNPs, are analyzed using X-Ray Diffraction (XRD) patterns in the range of 2θ = (10°–90°) on an X'Pert PRO diffractometer operating 40 kV with CuKα (λ = 1.5406 Å) radiation. The surface morphologies and compositions are observed by Scanning Electron Microscopy (SEM) operating at 20 kV and Energy Dispersive X-Ray (EDX) spectroscopy. Particle size is computed by using the ImageJ application. The functional groups formed from the synthesis AgNPs were determined using Fourier Transform Infrared (FTIR) in 400–4000 cm^{-1}.

Figure 1. Preparation of *Cromolaena odorata* extract.



(a) (b)

Figure 2. (a) UV-Vis absorption of AgNPs and (b) Tauc-Plot's of band gap energy (*Eg*) for 1 mM, 2 mM, and 3 mM.

3. Result and Discussion

3.1. UV-Visible

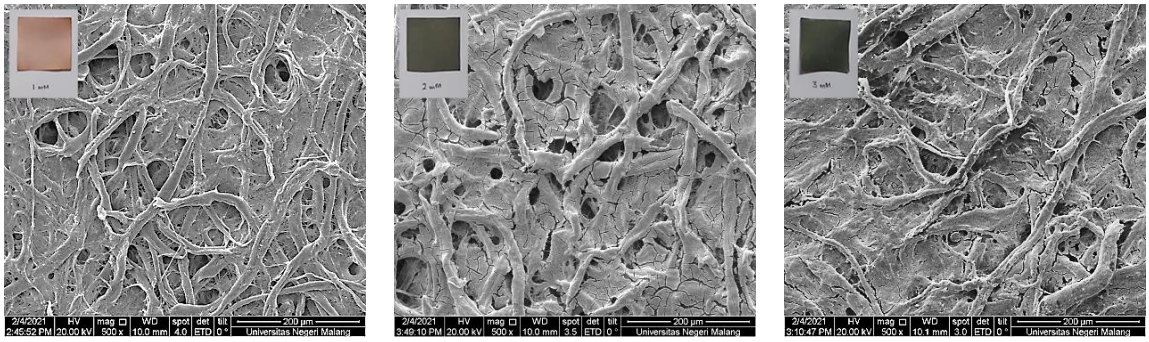
Figure 2a shows lower absorption peaks at 455 nm and higher at 452 nm and 451 nm. It explains that the bioreduction process takes longer at lower concentrations and affects the number of AgNPs. Meanwhile, a higher absorption peak indicates the fast progress of bioreduction and increases the number of AgNPs. The previous studies show the same result, whereas the higher absorption peak explained the significant progress of bioreduction due to increasing the concentration [17].

Figure 2b is the bandgap energy (*Eg*) for samples 1 mM, 2 mM, and 3 mM are 3.28 eV, 3.55 eV, and 3.59 eV, respectively. Previous studies have suggested that the increase in silver concentration boosts the bandgap energy. The change of absorption peak from UV-Vis spectra explains the progressive metallization process [18]. It is in line with a previous statement where the highest absorption peak indicated progressive bioreduction. As a result, the metallization process or coating of silver metal on the surface of Whatman paper is higher. The bandgap energy is related to the application as photocatalysts. A photocatalyst material can absorb light in the UV light (3.26–3.94 eV) and visible light (1.6–3.26 eV) ranges [19]. Based on the bandgap energy obtained in this study, it can be seen that silver nanoparticles can only absorb UV light for application as a photocatalyst.

3.2. Scanning Electron Microscope–Energy Dispersive X-Ray (SEM-EDX)

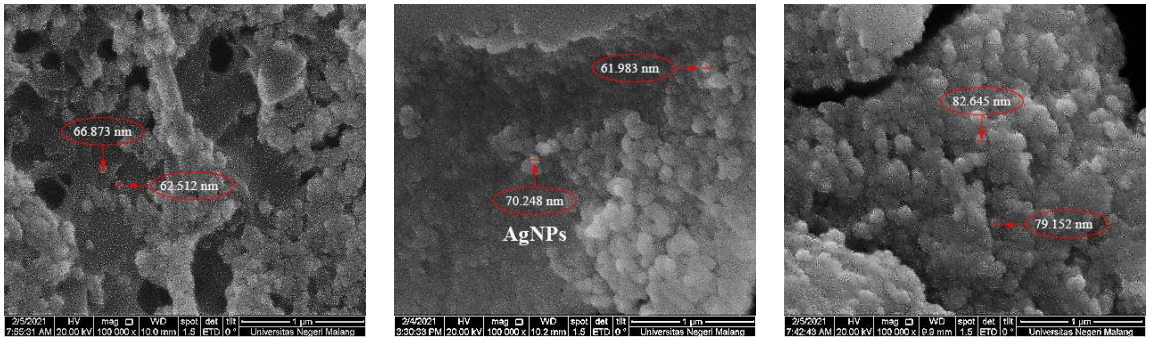
Representative SEM for magnification 500× in Figure 3 shows the distribution of silver nanoparticles immobilized on the surface of Whatman paper is evenly distributed as increasing concentration. It is indicated by the pores that form between the paper fibre networks getting closed as increasing concentration. For a magnification of 100,000× in Figure 4, it can be observed that there are spherical particles attached to the long fibres of Whatman paper [3]. The average size of AgNPs for 1 mM, 2 mM, and 3 mM are 60.45 nm, 63.19 nm, and 68.42 nm, respectively. Ghosh *et al.* explained that the higher the concentration indicates an increase in the size of the AgNPs [20].

EDX spectrum proves the presence of silver on the surface of Whatman paper (Figure 5). Increasing the precursor concentration creates a higher peak of silver in the EDX spectrum. It refers to a silver composition immobilized on the surface of the Whatman paper. Figure 6 shows the silver composition (wt%) immobilized on Whatman paper for various concentrations of 1 mM, 2 mM, and 3 mM are 10.14%, 64.48%, and 70.48%, respectively. In addition, the silver composition also explains the dark colour changed from the Whatman paper, and the pores of Whatman paper are increasingly closed due to the large number of silver atoms that have successfully immobilized on Whatman paper. According to research by Ghosh *et al.*, the increasing amount of AgNPs causes an increase in the concentration of Ag ions [20]



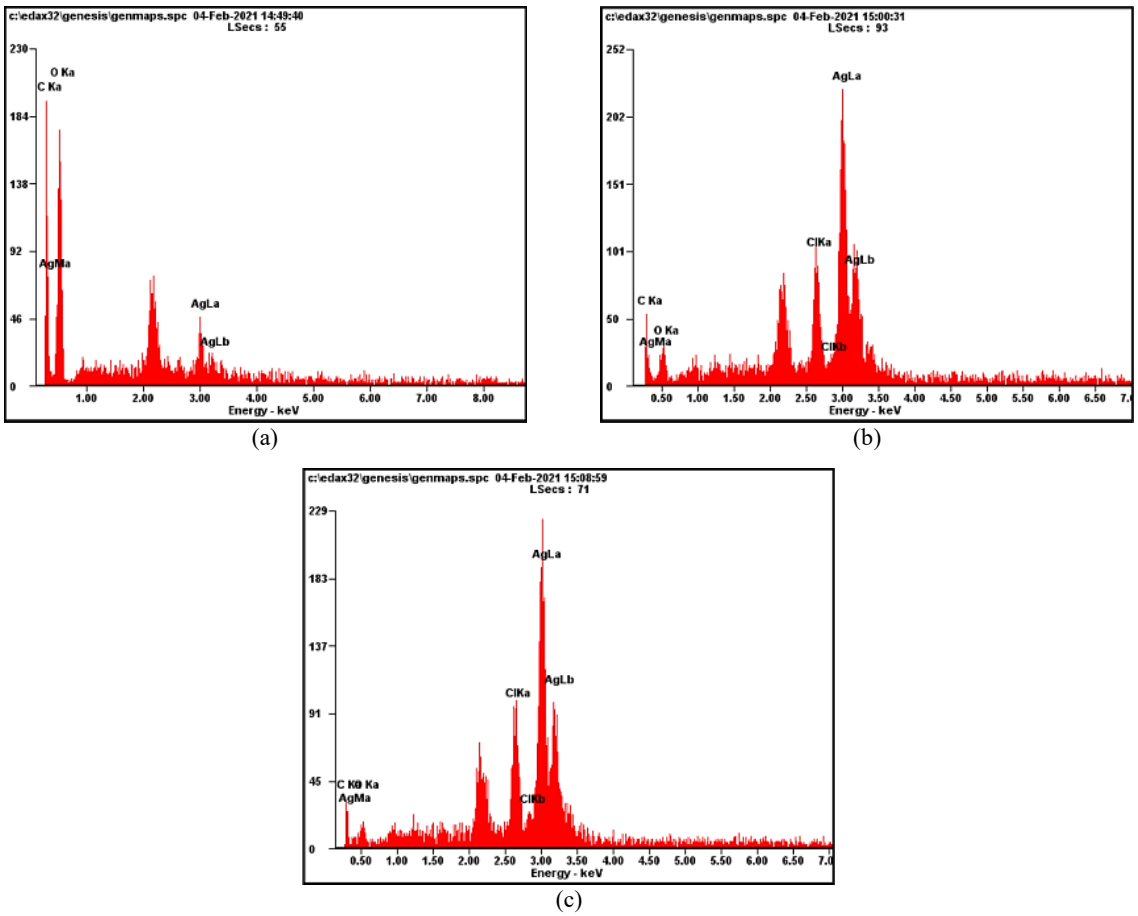
(a) (b) (c)

Figure 3. Representative of SEM for magnification 500× for (a) 1 mM, (b) 2 mM, and (c) 3mM.



(a) (b) (c)

Figure 4. Representative of SEM for magnification 1,000× for (a) 1 mM, (b) 2 mM, and (c) 3mM.



(a) (b)

(c)

Figure 5. Representative of EDX spectra for (a) 1 mM, (b) 2 mM, and (c) 3mM.

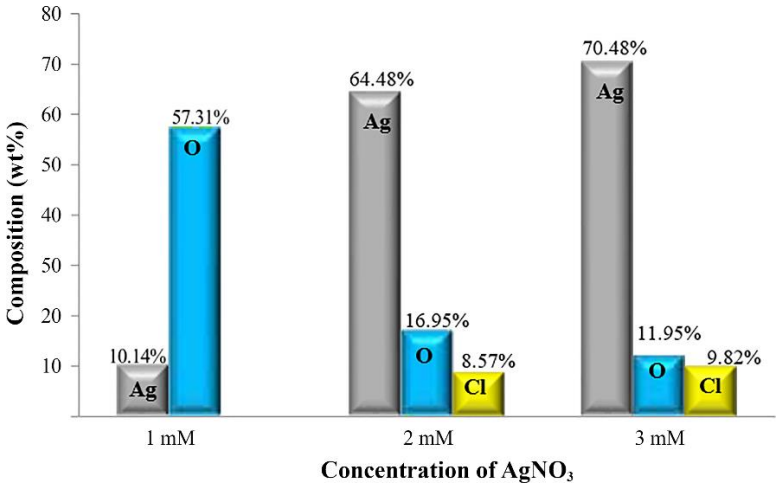


Figure 6. Compositions of AgNPs/Whatman paper based on EDX spectra.

The presence of other elements were identified based on the EDX spectrum, such as O and Cl. The O and Cl elements found from biomolecules in organic compounds play a role in forming Ag and AgCl [21], [22]. The decreasing oxygen composition with an increase in the Ag concentration indicates less oxygen is produced from the ester, ether, carboxyl, and hydroxyl compounds. This result is according to Atta and Abumelha research in which the lower oxygen composition is caused by an increased amount of AgNPs that have successfully formed [23]. In addition, the Ag concentration also affects the Cl composition. Research by Kubasheva *et al.* has shown that a lower concentration of Ag ions causes a limited of silver nanoparticles formed by binding to Cl ions [24]. As a result, there is no chlorine (Cl) from AgNPs immobilized on Whatman paper for a concentration of 1 mM. However, the presence of Cl for concentrations 2 mM and 3 mM plays a role in the formation of AgCl as evidenced by the XRD result.

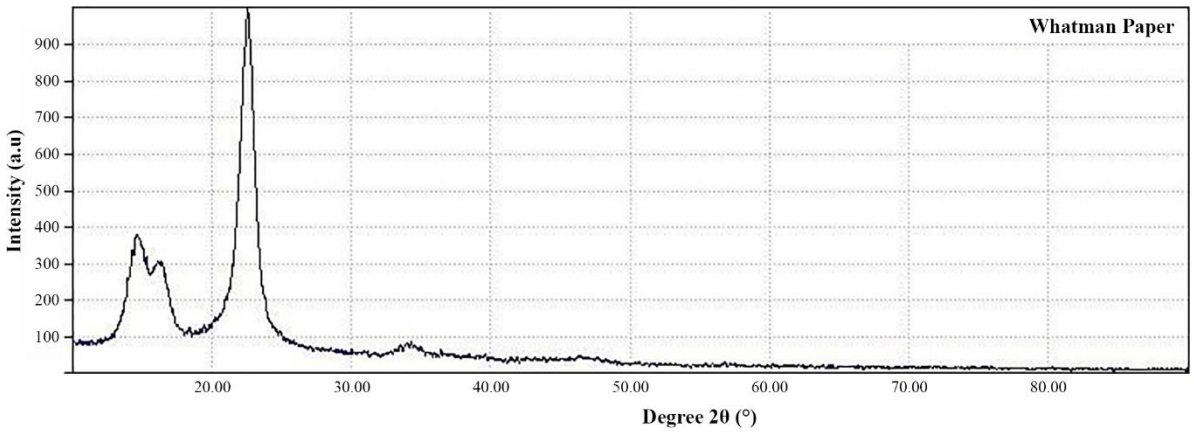
3.3. X-Ray Difraction (XRD)

Figure 7a and 7b shows the XRD pattern of Whatman paper and AgNPs/Whatman paper, respectively. It can be observed that different diffraction peaks appear after the immobilization of silver nanoparticles. Silver nanoparticles immobilized on Whatman paper has a diffraction peak at 38.25°, 44.65°, 64.53°, and 77.89° with lattices index 111, 200, 202, 311, and 222, respectively (Figure 7b). It showed a cubic crystal structure with a crystal size of 42.94 nm. Furthermore, the diffraction peaks of Ag from AgCl at 27.79°, 32.2°, 46.32°, and 54.83° have a lattices index of 111, 200, 202, and 311 respectvely, with a cubic crystal structure and crystal size of 17.33 nm. These results are in accordance with the diffraction peaks of silver-based on JCPDS 04-0783 and AgCl based on JCPDS 32-1238 immobilized on cellulose paper [3].

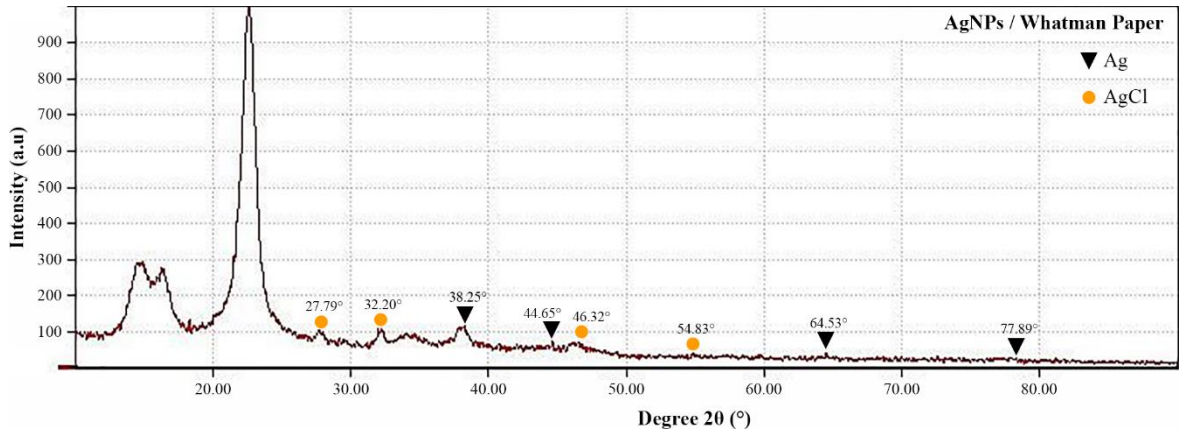
Figure 7c shows the XRD pattern of silver nanoparticles powder. It can be observed that diffraction peak of silver at 38.18°, 44.36°, 64.59°, 77.5°, and 81.66° have lattices index 111, 200, 202, 311, and 222, respectively. These peaks indicate silver has a cubic structure, space group Fm-3m and lattices parameter (*a*) 4.0862 Å. The crystal size based on the Scherrer equation is 35.59 nm. Then the silver peaks of AgCl were found at 27.89°, 32.3°, 46.3°, 55°, and 57.8° with lattices index 111, 200, 202, 311, and 222, respectively. These peaks indicate that AgCl has a cubic structure, space group Fm-3m and lattice parameter (*a*) 5.546 Å. The crystal size based on the Scherrer equation is 19.45 nm. The diffraction peaks in this result are in accordance with the previous studies where the presence of Cl that forms AgCl is explained from the compound bonds of the extract [26], [27]. The EDX spectrum also proved the presence of Cl in AgNPs/Whatman paper (Figure 6).

3.4. Fourier Transform Infrared (FTIR)

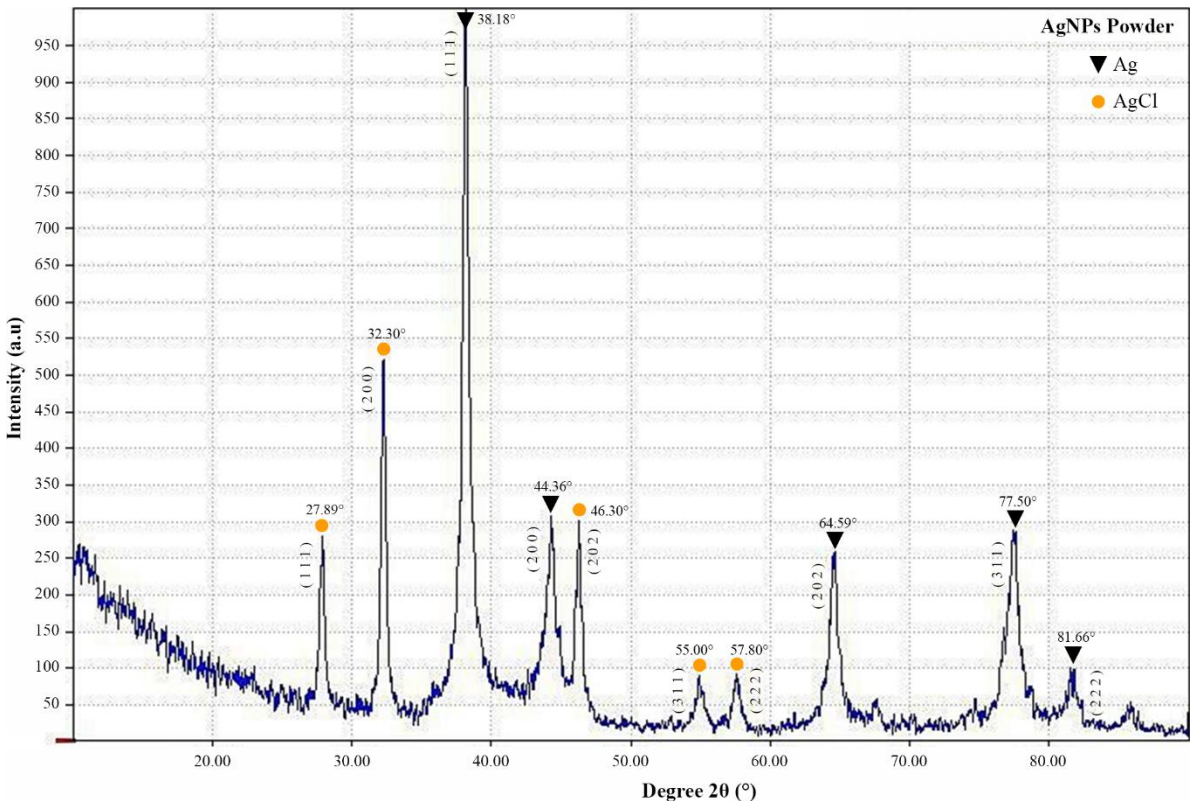
Figure 8 shows the FTIR spectra of AgNPs/Whatman paper. Peaks at 1,159 and 1,035 cm^{-1} are identified as a C-O bond of alcohols, carboxylic acids, esters, ether, and amine group stretches. Compounds derived from the bonds of these biomolecules, such as alkaloids and flavonoids, operate as a capping agent [16], [27]. The peaks at 1,651 and 1,425 cm^{-1} indicate the presence of the C=C alkene bond and



(a)



(b)



(c)

Figure 7. XRD pattern for (a) Whatman paper, (b) AgNPs/Whatman paper, and (c) AgNPs powder.

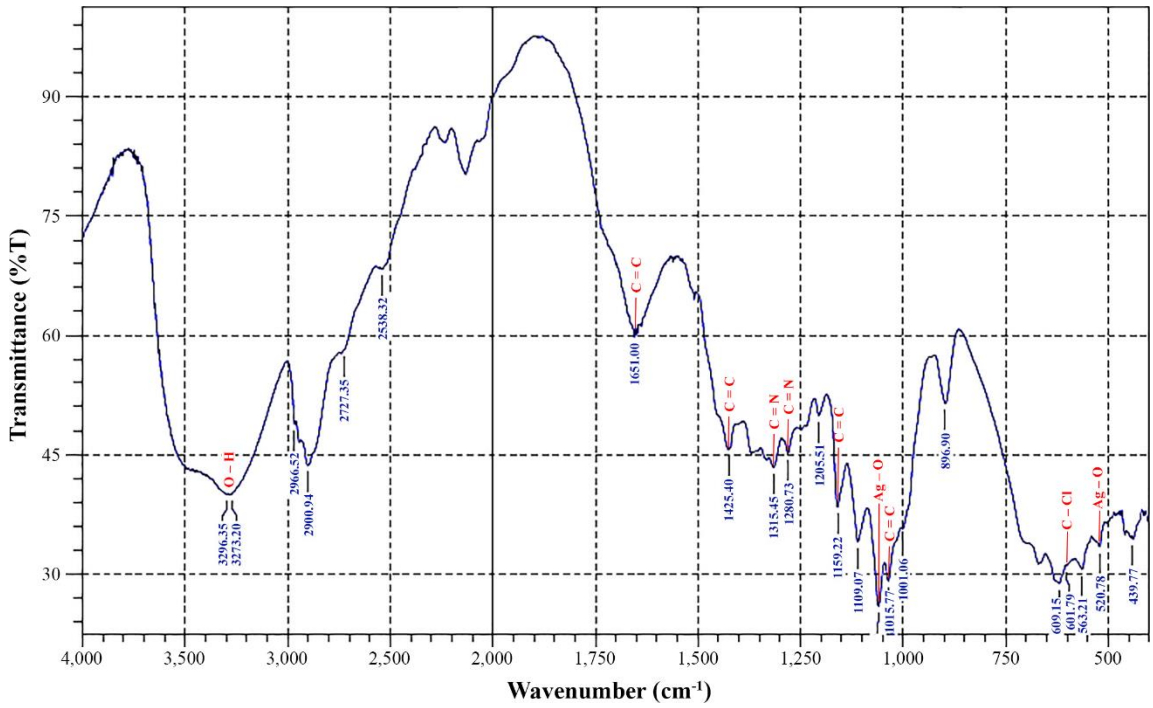


Figure 8. FTIR spectra of AgNPs/Whatman paper.

the vibration of protein bonds, which play a strong role as a capping and anti-agglomeration [28]. The width peak at 3,200–3,600 cm^{-1} indicates the O-H bond of phenol. Based on FTIR spectrum analysis, it can be concluded that the biomolecules and functional groups identified prove that *Cromolaena odorata* extract acts as a reducing agent and a stabilizer for the synthesis of AgNPs.

Peaks at 1,280 and 1,315 cm^{-1} indicate an interaction between C-N from the amine/amide group and Ag ion due to the complex formation of AgNPs [29], [30]. Peaks at 520.78 cm^{-1} and 1,059 cm^{-1} indicate a stretching of the Ag-O bonds. The peak that appears at 896 cm^{-1} explained the growth of AgNPs particles related to the larger size of AgNPs [31]. Peaks at 601 cm^{-1} indicate the presence C-Cl bond stretching of alkyl chloride explains the formation of AgCl. Nguyen also found the same result in the presence of AgCl from the green synthesis of AgNPs using *Cromolaena odorata* extract without adding chemicals containing Cl [27]. Moreover, the same results were found in several previous studies. The presence of Cl from halide compounds in plant extracts plays a significant role in forming AgCl [32], [33]. Therefore, it can be concluded that AgNPs were successfully formed and immobilized on the Whatman paper.

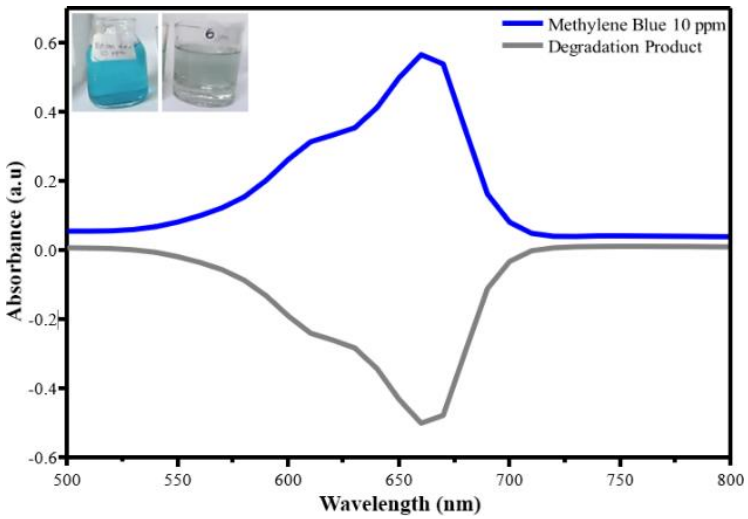


Figure 8. FTIR spectra of AgNPs/Whatman paper.

3.5. Photocatalysts Activity

Figure 8 shows the absorbance of methylene blue decreases after being irradiated by the sun for 6 hours. Kumari *et al.*, which also showed absorbance decreased, explains the degradation of the methylene blue structure [13]. Furthermore, the solution changes from blue to less colour after being irradiated by the sun for 6 hours. Therefore, the AgNPs/Whatman paper synthesized using *Cromolaena odorata* extract can be applied as a photocatalyst to degrade methylene blue.

4. Conclusion

Green synthesis and immobilization of AgNPs on Whatman paper using *Cromolaena odorata* extract were successful. Immobilized AgNPs on Whatman paper has spherical particles. Average particle sizes increased with increased precursor concentrations, which are 60.45 nm, 63.19 nm, and 68.42 nm, respectively. AgNPs have a cubic crystal structure and a crystal size of 42.94 nm. The photocatalyst activity of AgNPs/Whatman paper showed that methylene blue was successfully degraded. Therefore, immobilized AgNPs on Whatman paper has the potential to be applied as a photocatalyst material.

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