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Synthesis and Characterization of Ag/TiO_2 Nanoparticles using *Mirabilis jalapa* Plant Extract

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Abstract

Green synthesized nanomaterials have been widely developed because of their less toxicity, low energy process, environmentally friendly, effective, cheap, and pollution-free. Green synthesis of silver doped titanium dioxide nanoparticles (Ag/TiO_2 NPs) was carried out using *Mirabilis jalapa* plant extract. The plant extract was used as a reducing agent. The functional groups, morphology, and crystalline structure of as-synthesized Ag/TiO_2 NPs were investigated by FT-IR, FESEM, and XRD. Analysis by FESEM confirmed that the morphology of Ag/TiO_2 NPs is spherical with an average size of ~ 400 nm. Crystallite size for the Ag/TiO_2 NPs was calculated by the Scherrer formula dan the average size found to be in the range of 15.72 nm. The result of XRD analysis showing the fcc structure for metallic silver and TiO_2 particles in the anatase and rutile phases.

Keywords: Green synthesis, nanoparticles Ag/TiO_2, *Mirabilis jalapa*

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1. INTRODUCTION

Nanoparticles (NPs) have been widely developed because of their unique properties compared to their bulk material. Nanoparticles have unique characteristics due to the size, morphology, shape, and distribution (Amaliyah et al., 2020). Nanoparticles with green synthesis methods have been developed in the last decade. Green synthesis is a facile synthesis method that has the advantage of being an approach environmentally friendly, less toxic, low energy process, effective, inexpensive, and pollution-free. Plants, microorganisms, algae, or extracts plants as reducing agents can be used to control the shape and size of nanoparticles (Jegadeeswaran et al., 2016). The green synthesis nanoparticles show good morphology and stability (Rao et al., 2019).

Titanium dioxide, zinc oxide, silver, gold, copper, graphene oxide nanoparticles have been synthesized by green chemistry and an environmentally friendly approach (Jegadeeswaran et al., 2016). Titanium dioxide (TiO_2) nanoparticles with unique surface chemistry, morphologies, good stability, non-toxicity, and photocatalytic oxidation ability have been used in several applications. To increase the photocatalytic activity of TiO_2, various modifications have been made with noble metals (Au, Ag) (Liu et al., 2019). The energy bandgap of TiO_2 NPs manipulated by a doped of metals. Ag/TiO_2 NPs have been synthesized due to non-toxic, high availability, and low-cost material (Rao et al., 2019).

Recently, Ag/TiO_2 NPs have been synthesized from various plants including *Padina tetrastromatica* (seaweed), *Acacia nilotica*, *Euphorbia heterophylla* (Atarod et al., 2016; Jegadeeswaran et al., 2016; Rao et al., 2019). *Padina tetrastromatica* (seaweed) plant extract has been successfully used in the synthesis of Ag/TiO_2 nanocomposites. Seaweed extract acted as a capping agent and reducing agent in the formation of nanocomposites. The addition of plant extracts controlled the shape and size of the nanocomposites. The particle size of Ag/TiO_2 nanocomposite synthesized with seaweed was 25 nm (Jegadeeswaran et al., 2016).

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However, the green synthesis of Ag/TiO_2 NPs using plant extracts as a reducing agent is still rarely investigated. Phytochemical tests of *Mirabilis jalapa* shown that the plant contains flavonoids, saponins, tannins, and terpenoids (Oktaviana, 2018). These compounds have the potential as a capping agent and reducing agent for the synthesis of NPs. *Mirabilis jalapa* has been used for the synthesis of Ag/ZnO nanoparticles. This plant is used as a reducing agent for the bioreduction of zinc acetate and $AgNO_3$ in the formation of Ag/ZnO nanoparticles (Sumbal et al., 2019). In this study, we reported *Mirabilis jalapa* plant extract which can be used to synthesizing Ag/TiO_2 NPs. Ag/TiO_2 NPs were characterized using UV-Vis, FTIR, XRD, and FESEM.

2. MATERIALS AND METHODS

Material

The materials used in this study were titanium (IV) isopropoxide (TTIP) (Sigma Aldrich 98%), silver nitrate ($AgNO_3$) (Merck), ethanol, and *Mirabilis jalapa* plant extract.

Procedure

In the Ag/TiO_2 synthesis, the Ag nanoparticles were synthesized separately first. A total of 50 ml of extract from *Mirabilis jalapa* was added to a 0.003 M $AgNO_3$ solution. Then the solution was stirred at 100 ^{o}C until the color changed. The solution formed is diluted using aquadest.

The Ag/TiO_2 synthesis was carried out by adding 50 ml of ethanol to 5 ml of titanium (IV) isopropoxide solution and stirred for 1 hour. The solution was added with $AgNO_3$ solution and *Mirabilis Jalapa* extract solution and continued by stirring for 1 hour. The solution was calcined at 450 ^{o}C for 2 hours.

The characterization of the material was carried out using UV-Vis, FT-IR, XRD and SEM.

3. RESULTS AND DISCUSSION

In this study, Ag/TiO_2 nanoparticles were synthesized using a solution of *Mirabilis jalapa* plant extract as a solvent. Green synthesis of Ag/TiO_2 using *Mirabilis jalapa* plant extract has been investigated which is a facile, cost-effective, non-toxic, environmentally friendly, and efficient method for utilization of *Mirabilis jalapa* plant extract. The plant extract acted both as a reducing agent and stabilizer in the synthesis of nanoparticles.

The addition of *Mirabilis jalapa* plant in the aqueous extract to $AgNO_3$ solution generated a color change from colorless to dark brown after stirring 1 h of reaction. The color changes indicated that AgNPs have been successfully formed (Sumbal et al., 2019). The color changes depending on the concentration of $AgNO_3$. The **s**ynthesis of AgNPs with various concentration variations can be seen in **Figure 1.** AgNPs were synthesized by $AgNO_3$ 5 mM which showed the optimum concentration of $AgNO_3$.

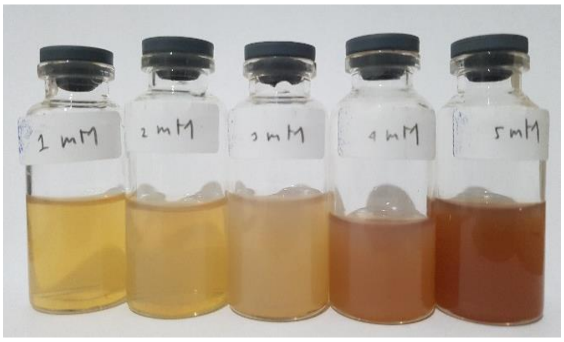

Figure 1. AgNPs solution with various concentrations of $AgNO_3$ in aqueous solvent.

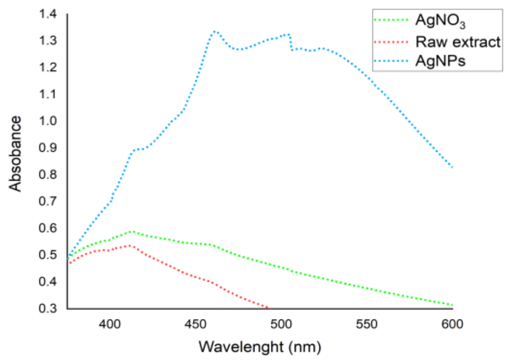

Figure 2. UV-Vis spectra of AgNPs with SPR band at 460 nm

Figure 2 showed that a sharp absorption band at 460 nm indicates the particles had dispersed. There was a shift in wavelength due to the addition of *Mirabilis Jalapa* extract which indicated that there was oxidation due to the addition of *Mirabilis Jalapa* (Fatimah & Mutiara, 2016). The surface plasmon resonance (SPR) band of AgNPs was exhibited at 390 – 500 nm which showed the characteristic of the absorption band of silver nanoparticles. Round-shaped nanoparticles were confirmed at 300 to 700 nm which indicated a single surface plasmon resonance (Yousaf et al., 2020). The broadening of the absorbance affected the shape and size of the NPs (Gopalakrishnan *et al.*, 2019).

Characterization of Mirabilis jalapa mediated Ag/TiO_2 nanoparticles

The FTIR analysis of Ag/TiO_2 nanoparticles shown in **Figure 3.** FTIR analysis is to determine the functional groups present in the synthesized Ag/TiO_2 nanoparticles. The peak at 1350 cm^{-1} is characteristic of the Ti–ligand bond (Alsharaeh et al., 2017; Jegadeeswaran et al., 2016). The peaks shown at 1639 and 3495 cm^{-1} indicated the presence of –OH bending and –OH stretching, respectively. The band located at 460 cm^{-1} was generated due to the oxide structure formation (Jegadeeswaran *et al.*, 2016). Furthermore, the peak at 2347 cm^{-1} indicated the transition metal carbonyls group (Rao *et al.*, 2019). The peaks at 1084 cm^{-1} and 1526 cm^{-1} represent the C=C aromatic ring and C – OH stretching vibration, respectively. The presence of the functional groups from polyphenolics indicated the phenolics compound in the plant extract. The reduction of metal ions and formation of nanocomposite could be probably due to the presence of a phenolic compound (Atarod *et al.*, 2016).

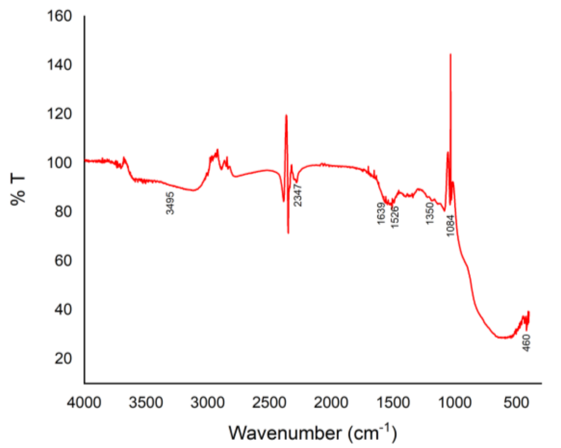

Figure 3. FTIR analysis of green synthesized Ag/TiO_2 nanoparticles.

Figure 4 shows the XRD pattern of Ag/TiO_2 NPs. The result showed that there were eleven peaks of the diffractogram, consisting four peaks of titanium substrate, five peaks of anatase TiO_2 crystals and 2 peaks of Ag crystals. The peaks in the synthesized Ag/TiO_2 nanoparticles were compared with the JCPDS data No. 1272 for TiO_2 anatase ; 1294 for TiO_2 rutile and 040783 for Ag. All peaks at 23.374°, 37.008°, 48.068° and 55.000° corresponds to the crystal planes of (101), (103), (200) and (211) respectively, indicating the formation of TiO_2 NPs in anatase phase. The peak at 62.743°, 70.457°, 74.276° and 76.175° corresponds and marked by their indices crystal planes of (110), (103), (200) and (112) respectively, which proves the formation of TiO_2 NPs in rutile phase had successfully synthesized (Purnomo et al., 2018). The peak of 37.994° and 45.000° respectively, indicating the present of Ag. Crystallite size for the Ag/TiO_2 nanoparticles was calculated by the Debye-Scherrer equation and the average size was found to be around 15.72 nm.

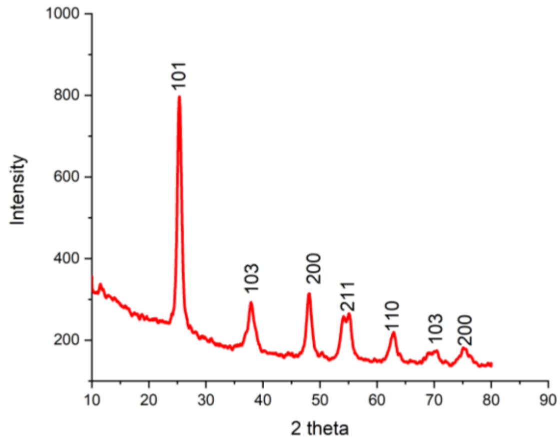

Figure 4. XRD analysis of green synthesized Ag/TiO_2 nanoparticles.

To determine the morphology and size of Ag/TiO_2 nanoparticles, characterization was carried out by using FESEM. The result of the FESEM analysis is shown in **Fig. 5.** The average size of the Ag/TiO_2 nanoparticles was ± 160 nm according to the ImageJ software. The morphology of the synthesized Ag/TiO_2 nanoparticles is spherical-shaped. This finding similar to the results reported by Sumbal et al., 2019 and Yousaf et al., 2020 that the Ag nanoparticles with extract plant in aqueous solution produce a spherical shape. The morphology variation of nanoparticles depend on the type of metal, conditions of the reaction, concentration of salt, and the composition of plant extracts (Sumbal *et al.*, 2019).

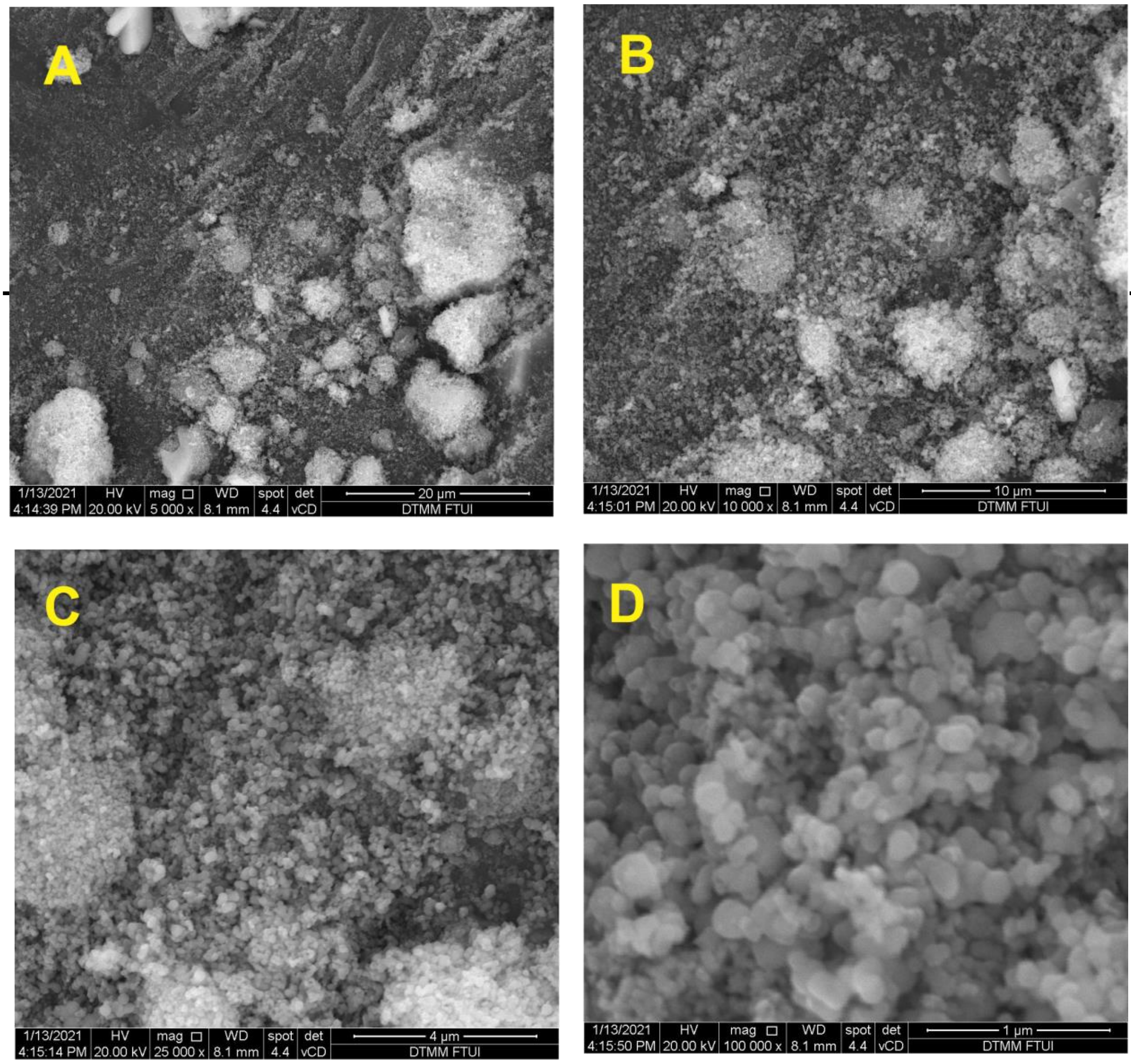

Fig. 5. FESEM analysis of green synthesis Ag/TiO_2 nanoparticles a) 5.000 magnification; b) 10.000 magnification; c) 25.000 magnification and d) 100.000 magnification

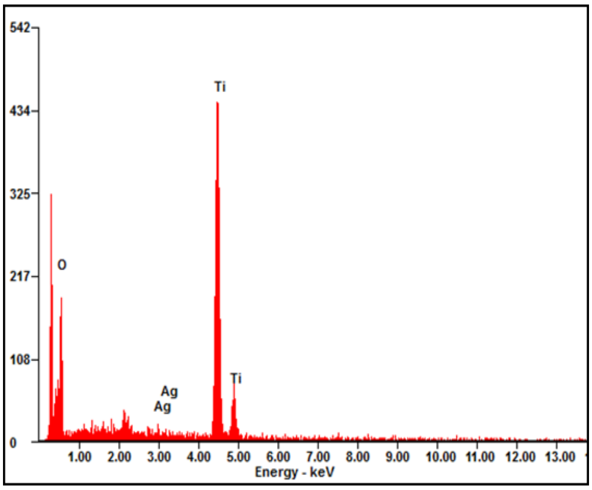

Fig 6. EDS analysis of Ag/TiO_2

Photophysical properties of Ag/TiO_2 were characterized using UV – Vis DRS. **Fig. 7** shown that the presence of Ag caused the decrease reflectance of TiO_2 nanoparticle. Decreasing of the reflectance value (% R) was observed in the UV and visible light region, which is around 350 – 800 nm. TiO_2 strongly absorbs light in 400 nm, while Ag/TiO_2 absorption occurs in the visible region and edge redshift at a wavelength of 450 nm. A broadened absorption of Ag/TiO_2 was formed due to the SPR effect of AgNPs. Band gap of TiO_2 and Ag/TiO_2 calculated by Kubelka – Munk and Tauc equation. Tauc equation of TiO_2 and Ag/TiO_2 shown in **Fig. 8.** Band gap of TiO_2 and Ag/TiO_2 is 3,26 eV and 3,02 eV, respectively.

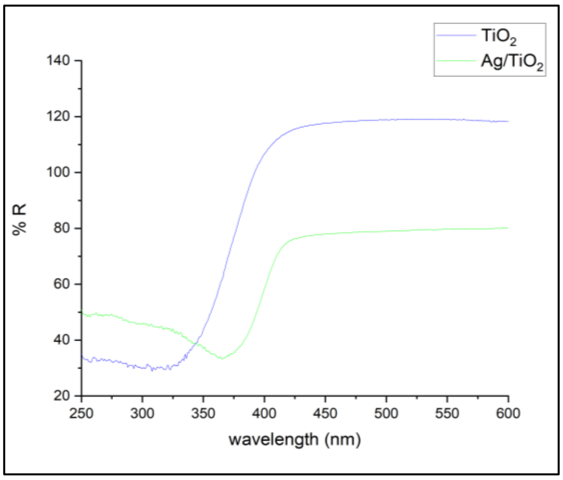

Figure 7. UV–Vis DRS analysis of TiO_2 and Ag/TiO_2

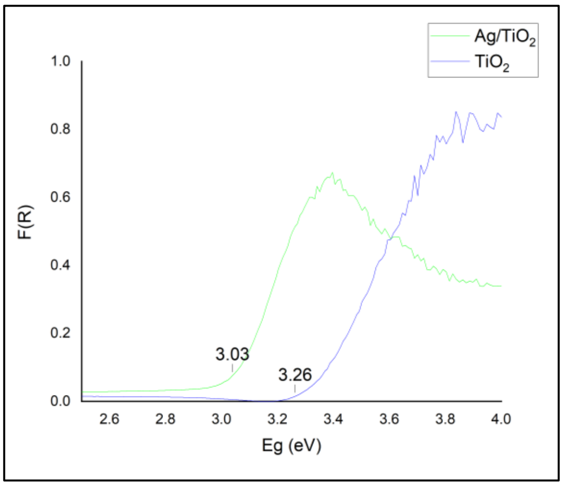

Figure 8. Band gap of TiO_2 and Ag/TiO_2 by Tauc Plot equation

4. CONCLUSION

We have successfully synthesized Ag/TiO_2 NPs from *Mirabilis jalapa* plant extract, as an eco-friendly, cost-effective, and non-toxic method. Ag/TiO_2 NPs were synthesized with the addition of Mirabilis jalapa plant extract as a reducing and stabilizing agent. The average size of the Ag/TiO_2 nanoparticles was ± 160 nm.

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