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BIOREDUCTION AND CHARACTERIZATION OF SILVER NANOPARTICLES FROM OIL PALM EMPTY FRUIT BUNCH

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ABSTRACT

BIOREDUCTION AND CHARACTERIZATION OF SILVER NANOPARTICLES FROM OIL PALM EMPTY FRUIT BUNCH (OPEFB). The synthesis of silver nanoparticles was successfully carried out by extracting oil palm empty fruit bunch. The precursor used was silver nitrate ($AgNO_3$) with a concentration of $9x10^{-4}$ M and 5 wt% of the oil palm empty fruit bunch extract. OPEFB acted as a capping agent in the synthesis of silver nanoparticles. The bioreduction method Ag^+ to Ag^0 produced a silver nanoparticle colloid in brown color. The results of the UV-Vis spectrophotometer showed the silver nanoparticles colloids spectrum at a wavelength of 420 nm with an absorbance value of 0.5. FTIR shows the reduction and shift of absorption peak in the hydroxyl functional group (-OH) at wavenumbers of 3323 cm-1 and the presence of absorption peaks at 560 cm^{-1}. While, XRD pattern showed the specific crystallinity peaks of silver nanoparticles at 2θ: 33.24°; 39.98°; 61.23°; dan 79.13° respectively with the face-centered cubic crystal structure (FCC) and crystallite size of 15 nm. PSA analysis showed two specific peaks with an average size distribution silver nanoparticles of 43.5 nm and a PDI value of 0.4. Analysis of TEM shows the average particle size of 20 nm with a spherical particle shape.

Keywords: Bioreduction, Silver Nanoparticles, Oil Palm Empty Fruit Bunch

ABSTRAK

BIOREDUKSI DAN KARAKTERISASI NANOPARTIKEL PERAK MENGGUNAKAN EKSTRAK TANDAN KOSONG KELAPA SAWIT. Sintesis nanopartikel perak berhasil dilakukan dengan memanfaatkan ekstrak tandan kosong kelapa sawit (TKKS). Prekursor yang digunakan yaitu perak nitrat ($AgNO_3$) dengan konsentrasi $9x10^{-4}$ M dan 5% sampel ekstrak TKKS. Tujuan penggunaan ekstrak tandan kosong kelapa sawit adalah sebagai media sekaligus capping agent dalam sintesis nanopartikel perak. Metode bioreduksi Ag^+ menjadi Ag^0 menghasilkan warna koloid nanopartikel perak berwarna coklat. Hasil spektrofotometer UV-Vis menunjukkan spektrum pada panjang gelombang 420 nm dengan nilai absorbansi 0,5. FTIR menunjukkan pengurangan dan pergeseran serapan pada gugus fungsi hidroksil (-OH) pada bilangan gelombang 3323 cm^{-1} dan hadirnya puncak serapan pada 560 cm^{-1}. XRD menunjukkan puncak kristalinitas yang spesifik nanopartikel perak pada sudut 2θ: 33,24°; 39,98°; 61,23°; dan 79,13° dengan struktur kristal kubus berpusat muka (FCC) dengan ukuran kristalit sebesar 15 nm. Analisis PSA memperlihatkan distribusi ukuran partikel perak sebesar 43,5 nm dan nilai PDI 0,4. Analisis TEM menunjukkan ukuran nanopartikel perak sebesar 20 nm dengan bentuk partikel bulat.

Kata kunci: Bioreduksi, Nanopartikel Perak, Tandan Kosong Kelapa Sawit

INTRODUCTION

Nano-sized material is new, unique, and superior physical and chemical properties compared to its bulk structure due to the increase of the ratio of surface area per volume of the material or particle [1]. In the latest decade, metal nanoparticles are the most widely studied because it is easy to synthesize. Moreover, these materials have a wide range of applications such as catalysts, detectors, surface, coating agents, and antibacterial. Metallic nanoparticles which mostly studied are silver (Ag) [2-3], gold (Au) [4], platinum (Pt) [5], and palladium (Pd) [6]. Colloids of silver nanoparticles were successfully synthesized using silver nitrate precursors with pH and temperature variations. The success of reducing Ag^+ to Ag^0 ions is marked by a brown discoloration [3].

Ag nanoparticle is an interesting material to be studied because it has a variety of particle sizes and good particle stability so that it can be applied in the industry [7]. Applications of silver nanoparticles are widely used as efficiency enhancement of dye-sensitized solar cells [8], photothermal treatment of ovarian cancer [9], antibacterial activity [10], biological activities [11], and antibacterial additive [12]. Ag nanoparticles can be synthesized with several methods, including chemical reduction. This method is often used due to its more comfortable and economical [13]. The synthesis of silver nanoparticles requires a reducing and capping agent. The capping agent used to stabilize a particle size of silver to prevent agglomeration [14]. The edible polymer is typical material widely used as a capping agents [15].

Meanwhile, there are many reducing agents which used to reduce Ag^+ to Ag^0 ions on silver nitrate and silver chloride precursors such as NaOH [16], ammonia [17], sodium citrate and NaCl [18], and polyvinyl alcohol [19]. However, it is hazardous and not environmentally friendly. Therefore, we would like to replace hazardous materials that have been used in previous studies by using natural ingredients. Currently, this method is called a green synthesis method to produce metallic nanoparticles. The use of eco-friendly reducing agents such as leaf, flower, seed, and root extracts in plants is one of the methods on the synthesis of silver nanoparticles.

Natural reducing used on the synthesis of silver nanoparticles such as, *Terminalia arjuna bark* extract [20], *Triticum aestivum* [21], aqueous extract of *P. peruviana* [22], herbal plant of *Solanum trilobatum* [23], *Euphorbia amygdaloides* [10], *Citrullus lanatus* fruit rind extract [24], Honey [25], *Annona muricata* [26], *Fenugreek* seed extract [27], *Murraya koeniigi L* [28], Orange peel extract [29], Sugar [30], *Trigonella foenum-graecum* [31], *Impatiens balsamina and Lantana camara* [7], *Pelargonium sidoides DC* [32], *Aspergillus fumigatus* [33], *Moringa oleifera oil* [34], *Rumex hymenosepalus* [35], *Crinum latifolium* [36] *Chlorophytum borivilianum L* [37], and *Ligustrum lucidum* leaves [38], successfully used as a medium in the formation of silver nanoparticles.

Based on the facts mentioned above, in this research, the green synthesis of Ag nanoparticles was carried out using OPEFB because of abundant resources in Indonesia especially in the sanggau area (West Kalimantan). Oil palm plants produce 23 wt% of OPEFB. OPEFB containing 45 wt% of cellulose and 16.5 wt% of lignin. Phenolic compounds can be produced from lignin extraction in the OPEFB [39].

Recently, the synthesis of silver nanoparticles with a green chemistry approach has been successfully carried out. The use of eco-friendly reducing agents such as leaf, flower, seed, and root extracts in plants is one of the methods on the synthesis of silver nanoparticles. In this research, silver nanoparticles will be synthesized using oil palm empty fruit bunch (OPEFB) extract. Hydroxyl groups (-OH) in phenol on OPEFB predicted can be used as a source of electrons in reducing Ag^+ to Ag^0 ions. In this study, oil palm empty fruit bunch (OPEFB)'s waste extracted using methanol, n-hexane, ethyl-acetate, and water. The water fraction results of oil palm empty fruit bunch extraction served as a medium for synthesizing of silver nanoparticles. The bioreduction reaction of Ag^+ to Ag^0 took place at room temperature.

EXPERIMENTAL METHOD

Materials and Instruments

Oil palm empty fruit bunch (*Elaeis guineensis J)* (OPEFB) was collected from Sanggau area, (West Kalimantan, Indonesia). Silver nitrate (99,98%) was purchased from Merck (Darmstad, Germany). Methanol (95%), n-hexane (99%), ethyl-acetate (99%) purchased from Sigma-Aldrich (Missouri, United States).

The colloid of silver nanoparticles was characterized using UV-Vis Spectrophotometer (Shimadzu 2600) at 200-800 nm wavelength, FTIR Spectroscopy (Shimadzu Prestige 21) at 4000-400 cm^{-1} wavenumber, X-ray diffraction (XRD Shimadzu 610) at diffraction angles (2θ) 20^0-80^0, Particle Size Analyzer (PSA Malvern ZEN 1600) with dynamic light scattering system, Transmission Electron Microscopy (TEM JEM 1400) using 350 keV of electron beam energy.

Method and Procedure

To obtain phenolic compounds in the OPEFB extract, a maceration process was carried out. Amount of 100 g of OPEFB was macerated with 200 mL methanol for 7 days at room temperature. Then, the extract of OPEFB methanol fraction was partitioned using n-hexane, ethyl-acetate, and distilled water to obtain a sample of OPEFB extract [40].

Synthesis of silver nanoparticles was conducted by adding 1 mL of OPEFB extract to 10 mL of silver nitrate solution ($1x10^{-4}$ M). As a comparison, the absorption peak was measured on solution precursor pure of silver nitrate ($1x10^{-4}$ M) and OPEFB extract of water fraction. The reaction of bioreduction Ag^+ to Ag^0 was carried out at room temperature for 1 hour. Discoloration from clear to brown indicated that colloid of silver nanoparticles had been formed [38].

RESULT AND DISCUSSION

Silver nanoparticles were successfully synthesized using natural bioreductor from the extract of OPEFB. The results of the extraction of oil palm empty fruit bunch (OPEFB) is 5 wt% of the extracted concentration of a stock solution. Figure 1 shows the bioreduction reaction of silver nanoparticles using the extract of oil palm empty fruit bunch (OPEFB). Visually, the formation of colloid of silver nanoparticles can be observed by the discoloration of the solution from clear to brown [38]. The mechanism is equation (1):

$$Ag^+ \longrightarrow Ag^0 \quad .... \quad (1)$$

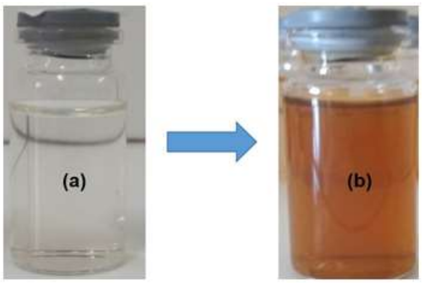

Figure 1. (a) Before and (b) After Bioreduction

Functional groups of hydroxyl (-OH) in these phenol compounds are predicted as ions sources of reducing Ag^+ to Ag^0 [41].

The colloid of silver nanoparticles has a maximum absorption peak (λ_{max}) at a wavelength of 420 nm with an absorbance value of 0.5, as shown in Figure 2(c). Figure 2 shows the surface plasmon resonance (SPR) phenomena of silver nanoparticles characterized using a UV-Vis spectrophotometer.

Phenomena of surface plasmon resonance (SPR) is only owned by the colloid of metal [2]. Both $AgNO_3$ precursor and OPEFB which showed on Figures 2(a) and 2(b) respectively have not a maximum absorption peak.

Based on the spectrum of FTIR spectroscopy, OPEFB extract has four absorption peaks at wavenumbers: 3323 cm^{-1} (%OH stretching); 1760 cm^{-1} (C=O ketone); 1650 cm^{-1} (C=C alkene); and 1350 cm^{-1} (C%H alkane) [42].

Differences of functional groups at OPEFB extract and colloid of silver nanoparticles are seen in Table 1.

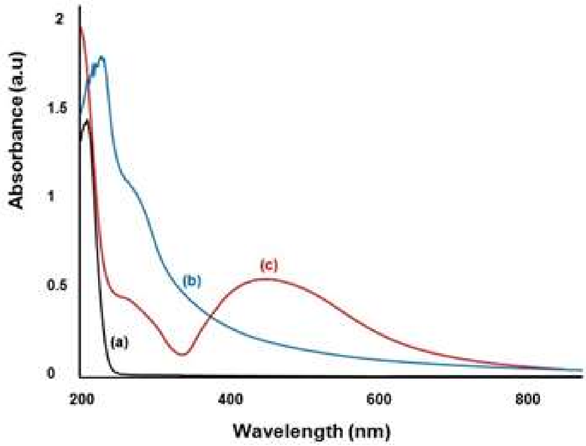

Figure 2. UV-Vis Spectra (a) $AgNO_3$ precursor, (b) Oil palm empty fruit bunch (OPEFB), and (c) Colloid of silver nanoparticles.

Table 1. Changes of the absorption peak between OPEFB extract and colloid of silver nanoparticles.

Functional Groups	OPEFB (cm^{-1})	Silver Nanoparticles (cm^{-1})
-OH	3323	3375
C=O	1760	1620
C=C	1650	1388
C-H	1350	1080

Figure 3 shows the FTIR spectrum of silver nanoparticles and OPEFB extract. Figure 3c shows the decrease of absorption peak in the hydroxyl group (-OH) at wavenumbers of 3323 cm^{-1}. This result indicated that the bioreduction of Ag^+ to Ag^0 has been successful. The formation of silver nanoparticles is characterized by the presence of absorption peaks at wavenumbers of 560 cm^{-1}[23].

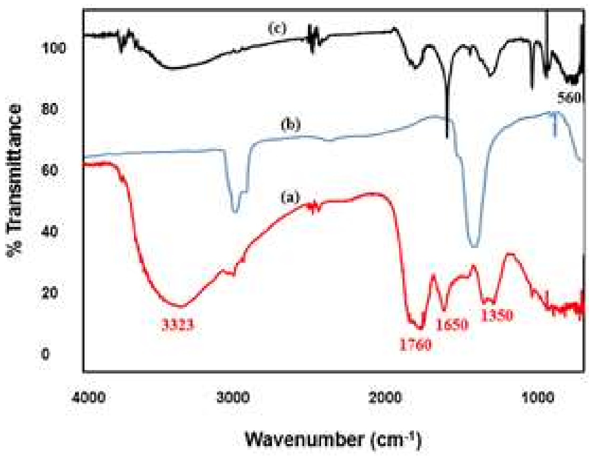

Figure 3. FTIR Spectrum (a) Oil palm empty fruit bunch (OPEFB), (b) $AgNO_3$, and (c) Colloid of silver nanoparticles

Figure 4 shows the crystallinity of silver nanoparticles. Silver nanoparticles have a four specific peaks at 2θ: 33.24°; 39.98°; 61.23°; and 79.13° which

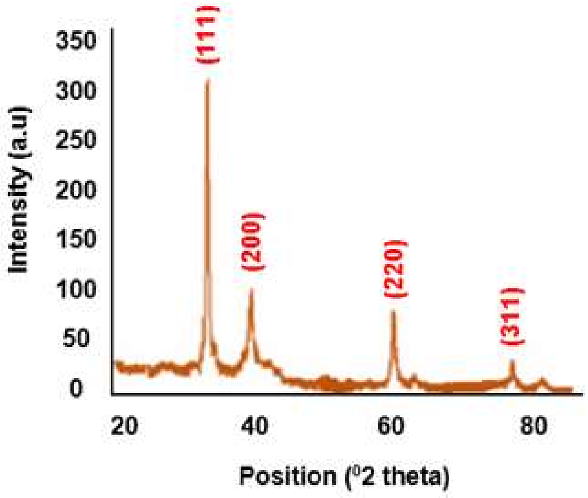

Figure 4. XRD pattern of silver nanoparticles

revealed Bragg's reflection from Ag (111), Ag (200), Ag (220), and Ag (311).

Silver nanoparticles have a face-centered cubic crystal structure (FCC) with a crystallite size of 15 nm. The crystal structure of silver nanoparticles has been published according to JCPDS data (No.04-0783) [43].

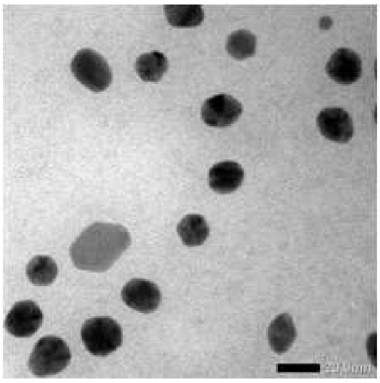

Figure 5. TEM images of silver nanoparticles synthesized by oil palm empty fruit bunch (OPEFB) extract.

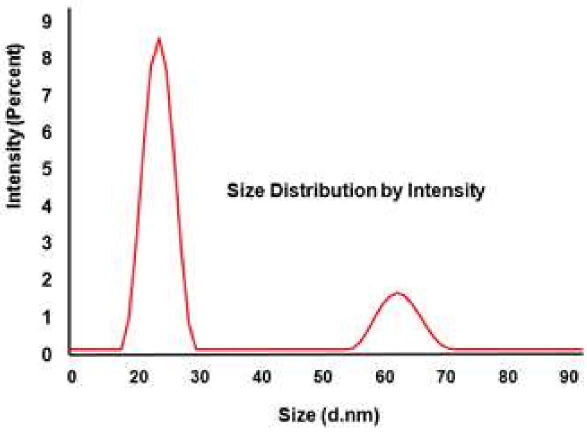

Figure 6. PSA distribution of silver nanoparticles synthesized by oil palm empty fruit bunch (OPEFB) extract.

TEM data shows that silver nanoparticles have a sphere shape with an average particle size of 20 nm [44]. Figure 5 shows the size distribution and shape of the silver nanoparticles.

Based on the data of particle size analyzer which showed in figure 6. Silver nanoparticles have a particle size distribution of 43.5 nm and a polydispersity index (PDI) value of 0.4 [45].

CONCLUSION

Silver nanoparticles were successfully synthesized using an extract of oil palm empty fruit bunch (OPEFB). The functional of secondary metabolite compounds in OPEFB extract as a source of electrons in reduction Ag^+ to Ag^0 ions and a capping agent in the synthesis of silver nanoparticles. The results of UV-Vis showed the maximum absorption peak of silver nanoparticles at a wavelength of 420 nm with an absorbance value of 0.5. Data of FTIR showed a reduction in absorption peaks at wavenumbers 3323 cm^{-1} (-OH) and the presence of absorption peaks at 560 cm^{-1} indicating silver nanoparticles were successfully formed. XRD showed an average crystal size of silver nanoparticles of 15 nm with a face-centered cubic crystal shape. Results of PSA showed that the size distribution of silver nanoparticles is 43.5 nm. In contrast, TEM observation showed that shape of silver nanoparticles was a sphericale with an average particle size of 20 nm.

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