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Synthesis and Characterization of ZnO Nanoparticles using the Duwet (*Syzygium cumini*) Leaves Extract as Bioreductor

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Abstract: The synthesis of ZnO nanoparticles was conducted using a green synthesis method that utilizes plants as bioreductors. The secondary metabolites contained in the plants can act as bioreductors in the reduction process of metal oxides and as capping agents in the production of ZnO nanoparticles. This research aims to determine the characterization of ZnO nanoparticles using *Syzygium cumini* leaves extract as bioreductor. The type of research is experimental research. In this study, the characteristics of ZnO nanoparticles were examined using extracts from the leaves of the duwet tree (*Syzygium cumini*). Synthesis was performed with varying compositions of zinc acetate dihydrate solution and duwet leaf extract at ratios of 1:1, 1:2, 1:3, and 1:4, as well as pH levels of 7, 8, 9, and 10. The nanoparticles were then characterized using UV-Vis spectrophotometry and a Particle Size Analyzer (PSA) to determine the optimal conditions. The optimal conditions for synthesizing ZnO nanoparticles were obtained with a maximum wavelength (λmax) of 368 nm at a ratio of 1:2 and pH 8. The particle size distribution analysis from the PSA indicated an average size of 19.52 nm, with a Poly Dispersity Index (PDI) value of 0.2491. The analysis results using XRD showed the synthesized nanoparticles showed the typical peak for ZnO with 2θ values of 31,7680°, 34,3699 °, and 36,2281°, confirming that the synthesis of nanoparticles had successfully produced ZnO nanoparticles. The UV-Vis, FTIR, and XRD characterization results showed that the nanoparticles produced were pure ZnO nanoparticles.

Keywords: Bioreductor; Duwet Leaf; Zink Acetate Dihydrate; ZnO Nanoparticles.

Introduction

Technological advancement is closely linked to the global expansion of science and industry, which has increasingly directed attention to nanotechnology [1][2]. This field has emerged as a significant branch of science that considerably impacts various life aspects [3][4]. Nanoparticles have unique physical and chemical properties due to their high surface area and nanoscale size [5][6]. Nanoparticles have a high surface area and are sized in the nanometer range. Nanoparticles are materials with sizes ranging from 1 to 100 nanometers [7][8]. Products derived from the application of nanotechnology can be categorized as nanomaterials, such as nanocomposites, nanotubes, nanoparticles, etc [1]. One of the widely used nanoparticles is ZnO nanoparticles. ZnO nanoparticles have non-toxic properties, the synthesis process is easy, and the costs used are relatively cheap, so they are widely preferred [9][10][11][12].

Methods that can be used to synthesize ZnO nanoparticles are physical and chemical methods [13][14]. However, these methods consume a lot of energy, time, and cost and are not environmentally friendly [15][16]. Therefore, a more environmentally friendly green synthesis method is a better alternative for synthesizing ZnO nanoparticles [17][18]. The green synthesis method requires a bioreductor for the synthesis of ZnO nanoparticles. Bioreductors used can be bacteria, algae and plants. In this study, the bioreductor used is a plant [19].

Plant extracts can reduce metal ions as stabilizers [20][21]. This is due to the content of secondary metabolite compounds such as alkaloids, flavonoids, proteins, terpenoids, tannins, and phenolics [20][21][22]. Duwet leaves are one of the plants that contain this content. Duwet leaves contain alkaloid, flavonoid, tannin, and saponin compounds that can help reduce Zn2+ ions in forming ZnO nanoparticles [23].

There is no research report on the synthesis of nanoparticles using duwet leaf extract, especially ZnO nanoparticles. Therefore, this study will synthesize ZnO nanoparticles using duwet leaf extract (Syzygium cumini) as a bioreductor and stabilizing agent. In addition, ZnO nanoparticles will also be characterized using a UV-Vis Spectrophotometer instrument to determine the maximum absorption wavelength of nanoparticles and a Particle Size Analyzer (PSA) to determine the size distribution of particles.

Research Methods

This experimental research was conducted at the Organic Chemistry Laboratory of the State University of Surabaya. The tools used in this research include UV-Vis (Shimadzu UV-1800), FTIR spectrophotometer (PerkinElmer), PSA (Biobase), XRD (PANalytical), analytical balance (Adventurer Ohaus), magnetic stirrer

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(Thermo scientietic), Erlenmeyer, beaker, measuring cup, volume pipette, dropper pipette, test tube, stirring rod, and spatula. At the same time, the materials used in this study include duwet leaf powder obtained from Herbal Materia Medica Laboratory, Batu, East Java, zinc acetate dihydrate ($Zn(CH_3COOH)_2.2H_2O$), aquabides, sodium hydroxide (NaOH), Whatman filter paper No. 1. The research data were analyzed using quantitative and descriptive analysis.

Results and Discussion

Extraction of Duwet Leaves Extract

Extraction was carried out using duwet leaves powder and deionized water solvent. Fine powder is used to increase the sample's surface area so that the solvent is more straightforward and can attract the chemical compounds contained in the sample more optimally [24]. The solvent used in this extraction is aquabides. Aquabides are used because aquabides are polar, so they can attract secondary metabolite compounds in duvet leaves, which are also polar [25]. The extraction results were then filtered using Whatman filter paper no. 1 to obtain the filtrate. The extract can be preserved at 4ºC [26].

Phytochemical Screening

Phytochemical screening of the duwet leaves extract is a fundamental step in identifying the bioactive compounds contained within it. The results of this study's phytochemical screening of the duwet leaves extract indicate a positive presence of alkaloids, flavonoids, saponins, phenolics, and tannins [27]. The results of the phytochemical screening of the duwet leaves extract can be seen in Table 1.

Table 1. Phytochemical Screening of Duwet Leaves Extract

Test	Observation	Results
Alkaloids	Orange precipitate with Dragendorff reagent, white precipited with Mayer reagent, brown precipited with Wagner reagent	Positive
Flavonoids	Formed a orange color	Positive
Phenolics	Formed a dark blue color	Positive
Saponins	Formed a stable foam	Positive
Steroids	Formed a pale yellow colour	Negative
Tannins	Formed a blackish blue colour	Positive

Based on research conducted by Hasanuzzaman, Duwet leaf extract contained alkaloids, phenolics, saponins, and flavonoids [28]. Meanwhile, Pujiastuti also reported that Duwet leaves contained phenolics, alkaloids, tannins, saponins, and flavonoids [29]. So, previous studies supported the results of the phytochemical screening of duwet leaves extract.

Synthesis of ZnO Nanoparticle

The synthesis of ZnO nanoparticles was conducted using hydrated acetate precursor and duwet leaf extract at volume ratios of 1:1, 1:2, 1:3, and 1:4. The most optimum result can be determined using a UV-Vis spectrophotometer. Subsequently, the pH was optimized at 7, 8, 9, and 10 to identify the most optimal pH using the UV-Vis spectrophotometer.

The synthesis results with an optimum pH can be tested further using PSA. Other results were centrifuged at room temperature (around 25 ℃) at a speed of 1000 rpm. After forming a precipitate, the precipitate was washed with distilled water and then dried at 80 ℃ for 3 hours in an oven to remove the water content. The solution was crystallized using a furnace at 400 ℃ for 4 hours to dry [22][30]. The result can be further tested with XRD.

Characterization of ZnO Nanoparticle

Spectrophotometer UV-Vis

The results of the volume comparison optimization using the UV-Vis spectrophotometer are shown in Figure.

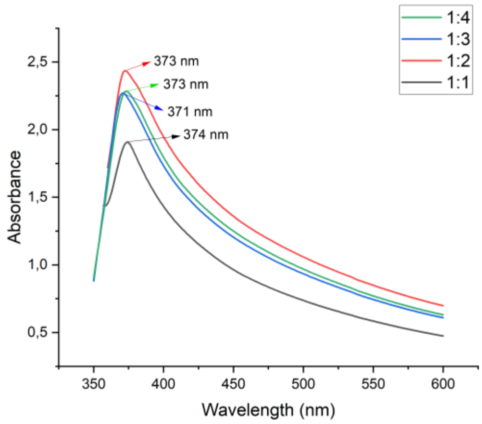

Figure 1. UV-Vis spectrum of synthesized ZnO nanoparticles

It can be seen in the figure that the most optimal ratio is 1:2, with an absorbance of 2.43 (Table 2).

Based on previous research, the most optimal comparison was 1:2. Because the more optimal the volume of bioreductor, the greater the ability of the extract to reduce ZnO. This is because the bioreductor of duwet leaf extract acts as a capping agent, which functions to maintain the stability of nanoparticles. However, there were certain limitations regarding the ability of bioreductor to reduce ZnO depending on the bioreductor used [31][32].

Table 2. Results of UV-Vis spectrum with volume variation

Ratio	Wavelength (nm)	Absorbance
1:1	374	1.90
1:2	373	2.43
1:3	371	2.25
1:4	373	2.28

Next, this optimal ratio (1:2) will be varied at pH levels 7, 8, 9, and 10, resulting in data as shown in Figure 2.

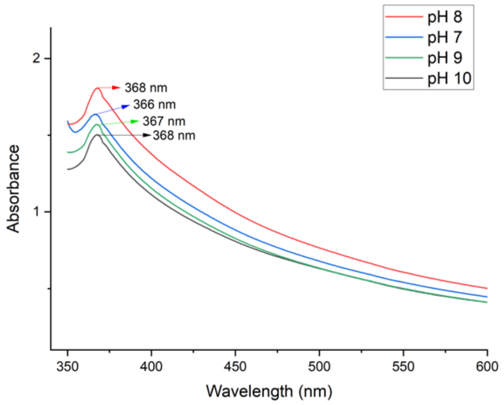

Figure 2. UV-Vis spectrum of synthesized ZnO nanoparticles

The most optimum pH optimization is 8, with an absorbance of 1.90, as shown in Table 3.

Based on previous research, pH 8 was the optimum pH in the synthesis of ZnO nanoparticles. At pH 8, it had a better and more homogeneous particle size distribution than other pH [33][34].

Table 3. Results of UV-Vis Spectrum with pH Variation

pH	Wavelength (nm)	Absorbance
7	366	1.64
8	368	1.90
9	367	1.57
10	368	1.50

Particle Size Analyzer (PSA)

Based on measurements using a Particle Size Analyzer (PSA) for ZnO nanoparticles from duwet leaves extract (*Syzygium cumini*), the average particle diameter was found to be 19.52 nm, which falls within the nanoparticle size range of 1-100 nm, thus confirming that the synthesized ZnO nanoparticles meet the nanoparticle size criteria. The PSA measurements yielded a polydispersity index (PDI) of 0.2491. A PDI value approaching zero indicates that the particle distribution is increasingly uniform [35][36].

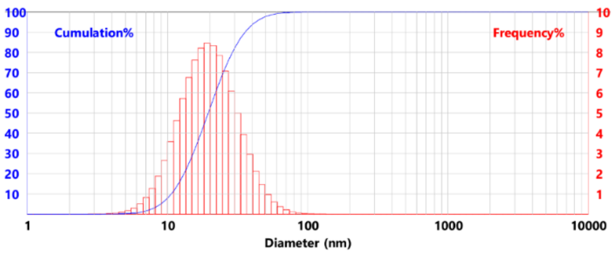

Figure 3. PSA of synthesized ZnO nanoparticles

X-ray diffraction (XRD)

Based on the results of XRD measurements (Figure 4), it was found that the X-ray diffraction results had diffraction peaks at 2θ angles of 31.7680º, 34.3699º, 36.2281º, 47.5745º, 56.5552º; 62.8643º; 68.0114º; 69.1933º.

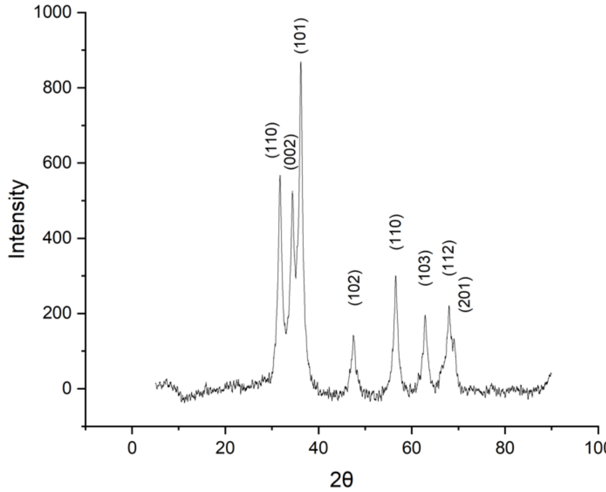

Figure 4. XRD of synthesized ZnO nanoparticles

The resulting diffraction pattern is by ICDD No. 00-005-0664. The synthesized nanoparticles showed the typical peak for ZnO at 2θ value of 31,7680º, 34,3699 º, and 36,2281 º. Thus, it can be concluded that the synthesis of ZnO nanoparticles was successful.

Conclusion

This study synthesized ZnO nanoparticles using duwet leaves extract (*Syzygium cumini*). The compounds in the duwet leaves extract include alkaloids, flavonoids, tannins, saponins, and phenolics, which serve as reducing and capping agents in synthesizing ZnO nanoparticles. The synthesis of ZnO nanoparticles under optimal conditions was achieved with a volume ratio of 1:2 at a wavelength of 373 nm and a pH of 8 (368 nm). Particle size analysis using PSA revealed an average size of the copper nanoparticles of 19.52 nm, with a PDI value of 0.2491 nm. The analysis results using XRD showed the typical peak for ZnO with 2θ value of 31,7680º, 34,3699 º, and 36,2281º.

Author's Contribution

Kholif Evawati: research, data collection, wrote the draft manuscript; Suyatno Suyoto: helped guide, direct and evaluate author one in the process of creating the article.

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