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Effect Of Microwave In The Green Synthesis Of Colloidal Bismuth Nanoparticles

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Abstract –In this study, bismuth nanoparticles were synthesized and prepared using citrus aurantifolia green synthesis method. The effect of microwave heating was examined. Citrus aurantifolia was used to reduce metal ions and nanoparticle formation due to the presence of various biomolecules needed. microwave was chosen because it can accelerate and form more stable nanoparticles. Bismuth nanoparticles were successfully synthesized with UV-Vis wavelength at 276 nm and nanoparticle average size is 4.66 nm. This result also shows a better TEM result image when with microwave because there is no impurity from bioreductor.

Keywords – Microwave; bismuth nanoparticles; citrus aurantifolia; green synthesis.

I. INTRODUCTION

Over the past few years, bismuth nanoparticle research has attracted significant attention due to the unique characteristics it possesses. One aspect that stands out is the extremely small particle size, which allows for a very large surface to volume ratio. Equally important is the very low toxicity of these bismuth nanoparticles [1,2]. Developments in the synthesis of bismuth nanoparticles have been a topic of interest in the research world, mainly due to their highly advantageous properties. Their small size allows for a large surface to volume ratio, which is very useful in various applications. Moreover, the fact that bismuth nanoparticles have a low level of toxicity makes them a promising option in various fields, such as medical science and environmental technology [3].

Various studies have revealed different synthesis methods for bismuth nanoparticles. Bismuth nanoparticles can be synthesized using green synthesis methods, namely the use of materials from nature, both plants and microorganisms. Saha et al. (2023) explored an easy and environmentally friendly synthesis to synthesize bismuth nanoparticles using green coffee bean extract [4]. Mahiuddin and Ochiai (2021) examined the use of lemon juice in the synthesis of crystalline bismuth nanoparticles (BiNPs), the study showed that BiNPs are limited by phytochemicals and can be dispersed stably in aqueous media. This study demonstrates that an environmentally friendly synthesis process based on plant sources can be an alternative choice to conventional chemical processes for synthesizing nanostructured bismuth and can even significantly affect the growth, morphology, and stability of BiNPs, as confirmed from XRD, DLS, SEM, and TEM analyses [5]. In the synthesis using plants, there is an additional method, namely microwave irradiation as a better method of synthesizing bismuth nanoparticles than conventional heating [1]. The results showed that microwave irradiation can accelerate reaction time and has the advantage of homogeneous heating which can directly affect the nucleation process in nanoparticle synthesis [6]. As the synthesis of bismuth ferrite nanoparticles made by microwave method in the research of Tadjarodi and Shahrab (2016) this method has the advantages of being simple, fast, energy efficient and environmentally friendly [7].

In this study, we have synthesized bismuth nanoparticles by green synthesis using Citrus aurantifolia assisted by a microwave. Citrus aurantifolia was chosen because it is easily available, does not cause excess waste, and is cheap. The nanoparticles were then analyzed using UV-Vis and TEM techniques without and with microwave process. This study aims to determine the effect of microwave irradiation on the characteristics of bismuth nanoparticles produced.

II. Experimental Procedure

2.1. Materials

Citrus aurantifolia from traditional market in Semarang, Indonesia, Bismuth nitrate pentahydrate ($Bi(NO_3)_3 \cdot 5H2O$), NaOH (Merck), aquades, microwave (Samsung 2450 MHz).

2.2. Methods of Synthesis of Bismuth Nanoparticles

Figure 1 shows procedure of bismuth nanoparticles synthesis. The synthesis of bismuth nanoparticles using three main components, precursors, reducing agents and stabilizers. The precursor used is Bi(NO3)3·5H2O and as a reducing and stabilizing agent it uses citrus aurantifolia. Total of 1 mM bismuth nitrate pentahydrate solution 30 mL was put into beaker glass. 10 mL of the bioreductor was mixed into a glass containing bismuth nitrate pentahydrate and then homogenized using a magnetic stirrer. 4 M NaOH was added to the mixed solution so that the pH was 12. The mixed solution was irradiated with microwaves in a microwave operating at a frequency of 2450 MHz and power 450 W and was subjected to microwave irradiation for 5 minutes.

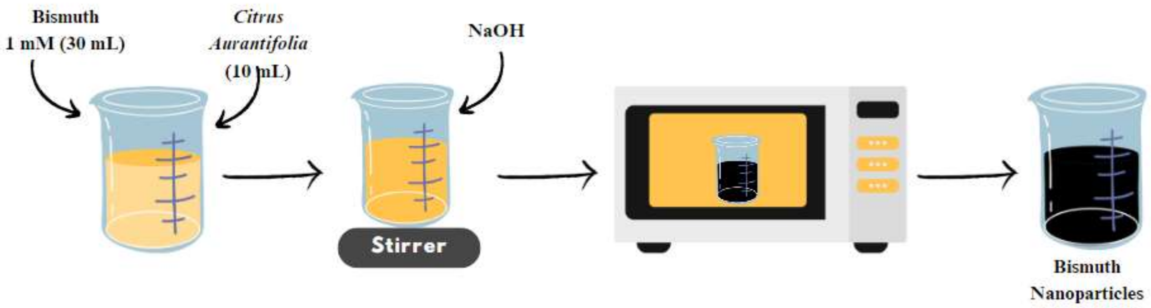

Fig. 1. Bismuth nanoparticle synthesis procedure

2.3. Nanoparticles characterization

UV-Vis spectrometer (Shimadzu). UV−Vis spectra were collected in an optical-quality quartz cuvette, requiring approximately 2 mL of solution. For the blank, the spectra used the aquades, always taken with the same cuvette as used for analysis. Solutions were diluted immediately before analysis, if required, in order to normalize absorbance to approximately 3 AU. Spectra were collected from 200 to 700 nm. Transmission Electron Microscopy (JEOL-JEM 1400). TEM analysis used to determine morphology of nanoparticles, the test was carried out with two samples, before being microwaved and after being microwaved. All sizing analysis performed using ImageJ software.

III. Result And Discussion

Figure 2 shows photographs of colloidal bismuth nanoparticles without and with microwave process. The picture shows that colloidal bismuth without microwave has a solid yellow color, while colloidal bismuth nanoparticles with microwave has a blackish color.

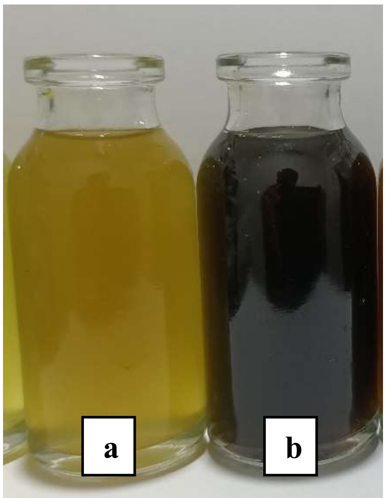

Fig. 2. Colloidal bismuth (a) without microwave (b) with microwave process

The observed phenomenon in Figure 2 can be attributed to the absorption of microwave radiation, leading to rotational motion of the atoms inside the colloidal system. This rotational motion induces collisions between the atoms, resulting in the generation of thermal energy. The process of nanoparticle synthesis is significantly influenced by homogeneous heating, which directly impacts the nucleation process and consequently accelerates the reduction process [8].

The resulting colloidal nanoparticles were then characterized with UV-Vis and TEM. First, we characterize using UV-Vis.

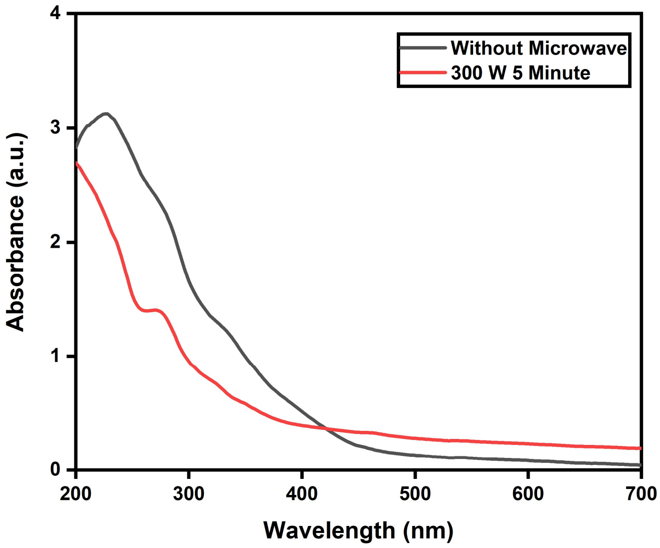

Fig. 3. UV-Vis Spectrum of bismuth nanoparticles

Figure 3 presents the UV-Vis spectroscopy outcomes for samples subjected to two different conditions: one without the application of microwave energy, and the other with microwave energy at a power level of 300 W for a duration of 5 minutes. The sample subjected to microwave at a power of 300 W for a duration of 5 minutes exhibits a prominent absorption peak at a maximum wavelength of 276 nm, with absorbance of 1,400. This observation suggests the successful formation of bismuth nanoparticles in the treated sample. Previous studies have demonstrated a positive correlation between microwave time and both wavelength and absorption value. Specifically, an increase in microwave time leads to an increase in wavelength and absorbance value. A higher absorbance value is indicative of a greater concentration of nanoparticles being created [9].

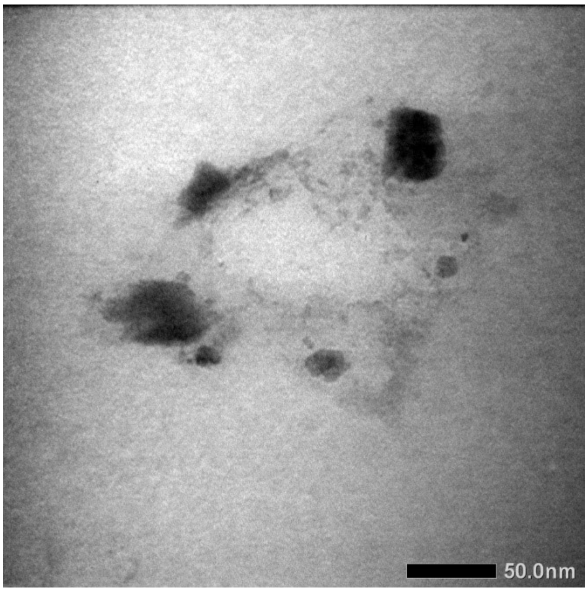

Fig. 4. TEM without microwave

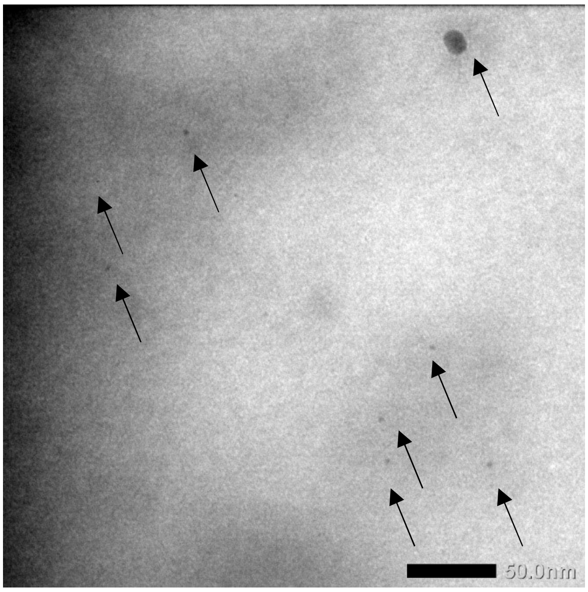

Fig. 5. TEM with microwave

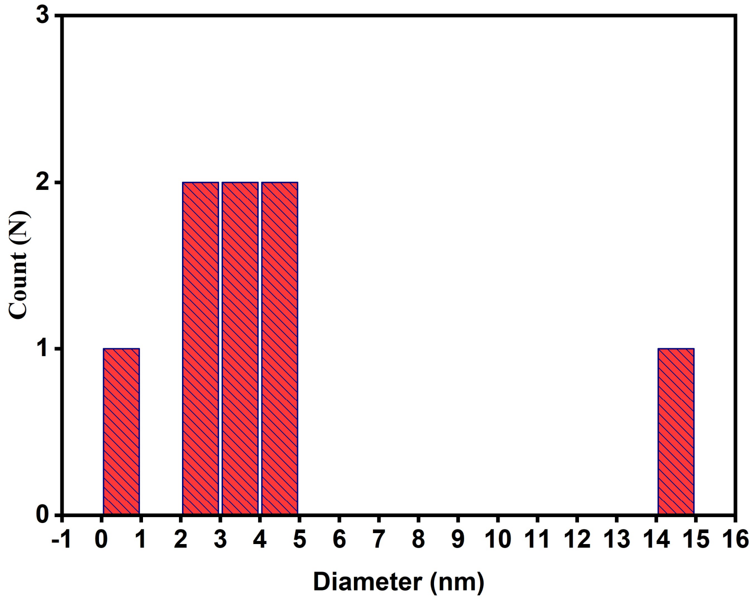

Fig. 6. Size distribution of bismuth nanoparticles

Furthermore, a characterization analysis was performed using transmission electron microscopy (TEM). The TEM image in Figure 4 and Figure 5 clearly depicts morphology of the sample, with a scale of 50 nm. Figure 4 shows an image that still has impurities from the bioreductor used which causes the image to be like this and has not formed bismuth nanoparticles. Figure 5 shows a clean image and only shows the image of bismuth nanoparticles with a fairly small concentration, this is due to using a fairly low power of 300 W. In some studies, it was found that the higher the power will make the concentration of nanoparticles increase. For bismuth nanoparticle distribution was quantified using ImageJ software, allowing the creation of a histogram representing their size distribution. This was achieved by measuring the diameter of each circular pattern observed in the TEM image. Figure 6 shows the results of the histogram analysis, indicating a monodispersed distribution of the nanoparticles, aligning closely with their respective diameters. The nanoparticle diameter ranges from 0.67 to 14.38 nm, with an average diameter of 4,66 nm. One significant advantage of these monodispersed particles with a narrow size distribution is their improved colloidal stability.

It's worth noting that the diameter of these nanoparticles is notably smaller compared to prior studies on bismuth nanoparticles. For example, in a study by Kazemi and Yaqoubi (2019), particle measurements reached 120 nm using a heating method for 24 hours at 90°C. Similarly, Das et al. (2020) employed a hotplate stirrer heating method at 60°C for 3 hours, resulting in measurements ranging from 40.4 to 57.8 nm. Additionally, Mahiuddin and Ochiai (2021) reported measurements of 8-30 nm using a hot water bath at 80°C for 2 hours [10,11,5]. This finding highlights that the use of microwave irradiation for nanoparticle synthesis can yield smaller nanoparticle sizes compared to other methods involving microwave use in the synthesis of bismuth nanoparticles [10].

IV. Conclusions

We have successfully synthesized bismuth nanoparticles using green synthesis from Citrus aurantifolia assisted by a microwave. The research results show that the nanoparticles formed have a spheric size distribution with a diameter between 0.67-14.38 nm with an average diameter of 4.66 nm. The TEM image wihout microwave has impurities from the bioreductor but the TEM image with microwave produces an image that only shows bismuth nanoparticles

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