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Synthesis of Bismuth Nanoparticles Using Microwave Irradiation Method

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ABSTRACT

The synthesis of bismuth nanoparticles has been successfully carried out using *Citrus limon* extract. This study uses the green synthesis method using microwave irradiation. Microwave irradiation can speed up reaction time and has the advantage of homogeneous heating which can directly affect the nucleation process of nanoparticle synthesis. The synthesis of bismuth nanoparticles was carried out by mixing nitrate pentahydrate powder (Bi $(NO_3)_3$). $5H_2O$ with *Citrus limon* extract and added with NaOH then heated with 450W microwave irradiation power at a heating time of 5 minutes. The formation of bismuth nanoparticles was indicated by a change in the color of the solution from orange to black and the UV-Vis spectrum of the nanoparticles showed a wavelength of 275 nm with an absorbance of 2.008 a.u. TEM characterization shows that the bismuth nanoparticle has a spherical shape with an average size of 35.56 nm.

Keywords: Bismuth nanoparticles, Microwave irradiation, *Citrus limon* extract, NaOH

I. INTRODUCTION

Nanoparticles are one of the nanotechnology products that are growing rapidly following the times. Nanoparticles are particles that have a size of 1-100 nm [1]. One type of nanoparticle that has received much attention as a breakthrough in research because of its unique properties of high stability, ease of functionalization, and relatively low toxicity, making it very suitable for biological applications is bismuth nanoparticles. In general, bismuth nanoparticles can be produced by physical methods (top-down) and chemical methods (bottom-up). However, physical methods have the disadvantage of requiring considerable costs and chemical methods have the disadvantage of potentially damaging the environment due to the use of chemicals [2].

Therefore, to minimize the cost and use of chemicals, the green synthesis method is proposed [3].

Green synthesis is a method that utilizes the content of organic chemical compounds in plants that can be used as reducing agents and stabilizing agents in the process of forming nanoparticles. The content of organic chemical compounds is terpenoids, flavonoids, enzymes, phenolic acids, and alkaloids [4]. One of the plants containing organic chemical compounds is Citrus limon fruit containing citric acid, ascorbic acid, flavonoids, and vitamins that can be used as reducing agents and stabilizing agents in synthesizing bismuth nanoparticles [5]. One of the factors that affect the synthesis of nanoparticles is temperature and reaction time. Conventional heating involves a vessel so that the material to be heated takes a long time to reach the optimal temperature level to produce the desired analyte [6]. In heating with microwave irradiations, only the material or solution is heated without involving a container, so the temperature will rise rapidly so that the desired analyte comes out of the cell faster.

Some ongoing research are developing microwave irradiation methods because microwave irradiation has the advantages of rapid nanoparticle growth, high concentration, and more uniform size distribution than to other heating methods. Heating with microwave irradiations can accelerate the reduction process which can have a direct effect on the nucleation process of bismuth nanoparticle synthesis, this is because the homogeneous heating process can increase the intensity of collisions to increase the possibility of interaction between reducing agents and precursors. Thus, this study aims to synthesize bismuth nanoparticles using *Citrus limon* extract with microwave irradiations.

II. METHODS AND MATERIAL

2.1 Material

Bismuth nitrate pentahydrate (Bi $(NO_3)_3$). $5H_2O$, Aquades, *Citrus limon*, and NaOH.

2.2 Methods

2.2.1 Preparation of *Citrus limon* extract

Citrus limon fruits are cleaned and then cut into pieces. Pieces of *Citrus limon* are further squeezed to obtain the extracts or fruit juice from *Citrus limon*. Then filtering 2-3 times to produce pure *Citrus limon* extract.

2.2.2 Synthesis of bismuth nanoparticles

A total of 0.121 grams of bismuth nitrate pentahydrate (Bi (NO3)3). 5H2O) was added to 60 ml of Citrus limon extract. Next, the pH was adjusted to 12 using 4 M NaOH. The solution is then heated by microwave irradiation for 5 minutes with a power of 450W. The formation of bismuth nanoparticles is characterized by a change in the color of the solution from orange to black. The scheme of the synthesis procedure is shown in Figure 1.

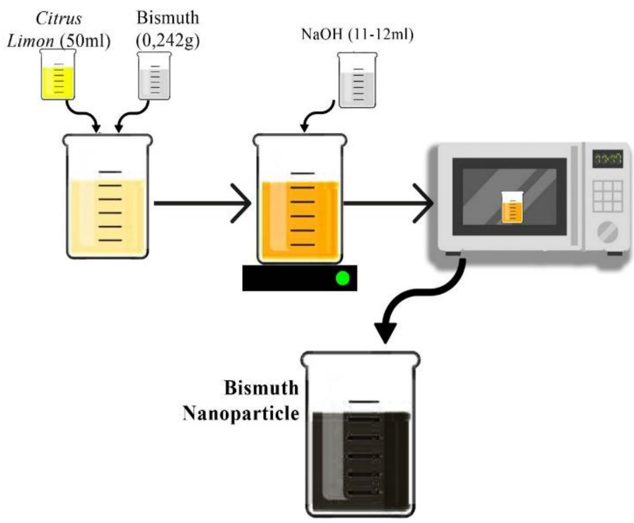


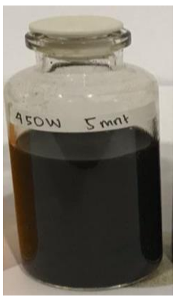
Figure 1: Schematic of bismuth nanoparticle synthesis procedure

III.RESULTS AND DISCUSSION

The synthesis of bismuth nanoparticles aims to make bismuth into nanometer-size particles by utilizing active compounds contained in plants which can be referred to as reducing agents. One of the requirements for natural ingredients that can be used as reducing agents is to contain active compounds such as flavonoids and citric acid. Where active compounds can play a role in the bismuth reduction process. One plant that contains active compounds is *Citrus limon* which can reduce bismuth (Bi^{3+}) to bismuth (Bi^0) with the help of microwave irradiation that can speed up the process reduction so that it can have a direct effect on the nucleation process of bismuth nanoparticle synthesis through a homogeneous heating process and can increase the intensity of collisions so that it can increase the likelihood of interaction between reducing agents and precursors.

The formation of bismuth nanoparticles can be known qualitatively by looking at the color change in the solution and quantitatively through characterization of the solution by Ultraviolet-Visible Spectroscopy (UV-Vis) testing as initial testing before proceeding to further testing. The results of the synthesis of bismuth nanoparticles are shown in Figure 2.

(a)



(b)

Figure 2: a) Solutions without microwave irradiation, b) Solutions with microwave irradiation

In Figure 2, it can be seen that the solution changes color from orange to black after heating with microwave irradiation. Color changes indicate the formation of bismuth nanoparticles according to Mahiuddin et al, (2021) namely the change of solution to black color [7].

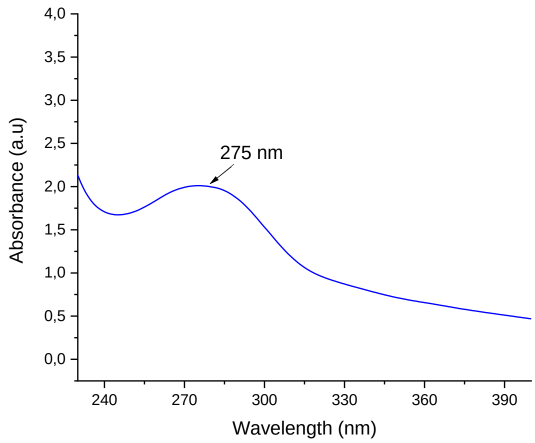


Figure 3: Spectrum UV-Vis of bismuth nanoparticles

Based on Figure 3, it can be known the wavelength value and absorbance resulting in the synthesis of bismuth nanoparticles. In this study using 450W power and a heating time of 5 minutes, the UV-Vis spectrum of the bismuth nanoparticles showed a wavelength of 275 nm with an absorbance of 2,008 a.u. The results of this study are in line with previous research conducted by Torrisi, et al (2018) who have succeeded in synthesizing bismuth nanoparticles using the pulse laser ablation method with a wavelength result of 269 nm [8]. According to J. A. Creighton et al., (1991) reported the absorption band for bismuth nanoparticles is around 270-280 nm [9]. Deepak et al, 2011 have suggested that at higher temperatures, more nucleation can occur to form smaller particles [10].

Characterization is continued with TEM testing (*Transmission Electron Microscopy*) which aims to determine the shape, size, and size distribution. The results of the bismuth nanoparticle TEM test are shown in Figure 4 as follows. Based on the results of the analysis in Figure 4, it can be known the size of the nanoparticles and the shape of the bismuth nanoparticles formed. Based on the data obtained, it can be seen that the number of spherical nanoparticles is more than those that are trigonal or hexagonal. These results are supported by research conducted by Mahiuddin et al, 2021 which states that the shape of bismuth nanoparticles is spherical in size between 8 to 30 nm [7].

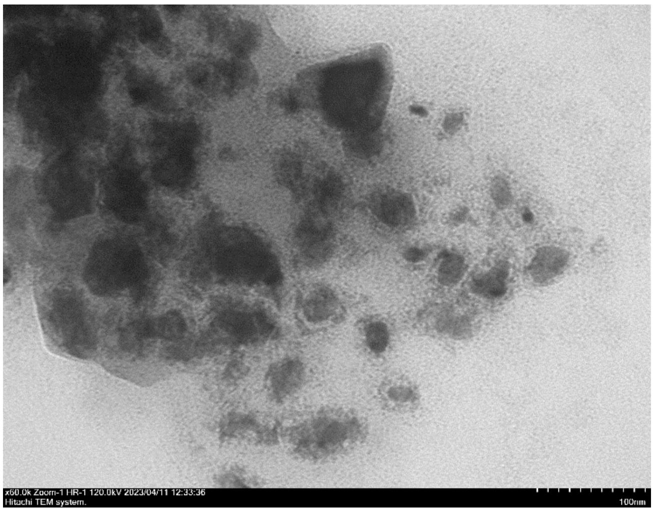


Figure 4: Morphology of bismuth nanoparticles

The size distribution of bismuth nanoparticles is shown in Figure 5. Based on Figure 5, it can be seen that the smallest bismuth nanoparticle size is 20 nm and the largest is 50 nm so the average bismuth nanoparticle size is 35.56 nm.

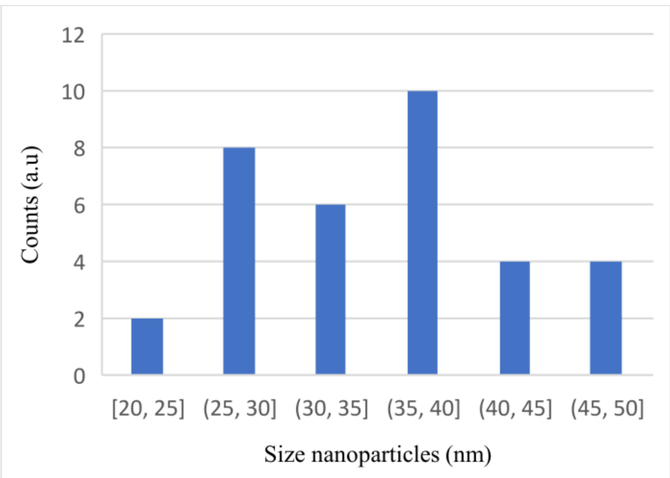


Figure 5: Histogram of bismuth nanoparticle size distribution

Based on the results of the particle size obtained, it can be seen that the resulting bismuth nanoparticle is stable with the average nanoparticle size being nano-sized, which is below 100 nm. This is thought to be because the provision of optimal microwave irradiation can accelerate the reduction process which can have a direct effect on the nucleation process of bismuth nanoparticle synthesis and prevent the occurrence of thermal degradation of the product as well as the effectiveness of *Citrus limon* extract which acts as a high stabilizing agent.

IV. CONCLUSION

Based on the research that has been done, it can be concluded that the synthesis of bismuth nanoparticles using *Citrus limon* extract with microwave irradiation has been successfully carried out by producing black bismuth nanoparticles in a round shape with an average size of 35.56 nm.

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