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## Synthesis of ZnO-gold nanoparticles composite using *Hibiscus* sabdariffa extract as reductor

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**Abstract**. One of the synthesis methods currently being carried out is green synthesis. One of the efforts to implement the green method is to use plant extracts that can be renewed. *Hibiscus sabdariffa* is one type of plant that is able to produce anthocyanin pigments. In this study, the synthesis of ZnO composites with gold nanoparticles will be carried out using *Hibiscus sabdariffa* flower extract. The composites of gold nanoparticles and ZnO were determined using XRD instrumentation where there were characteristic peaks of ZnO and gold nanoparticles.

#### 1. Introduction

Current technological developments are closely related to the development of inorganic materials. Some inorganic materials are reported to have various abilities such as adsorbents [1,2], catalysts [3], and photodegradators [4]. One of the frequently studied inorganic materials is ZnO because of the characteristics of the gap energy of the tape [5]. One type of research that is often found in the synthesis of ZnO and its composites with various methods because in metal oxide materials such as ZnO different synthesis methods can provide different characteristics [6].

One type of ZnO composite that is promising is the ZnO composite with gold nanoparticles [7]. Nanomaterials can be synthesized through two paths namely top-down and bottom-up. The top-down method is a method that cannot be used on a production scale because it requires high costs and takes a long time [8]. The bottom-up method is a method based on chemical and physical interactions on the formation of material from a smaller scale than a nanomaterial to the size of a nanomaterial through colloidal dispersion techniques. Nanomaterial synthesis with bottom-up method is modified in physics, chemistry, and green methods [9]. Green methods are a vigorous approach to make the process that is carried out more environmentally friendly [10]. The main principle of gold nanoparticles synthesis is the reduction of Au(III) to Au(0). The commonly used method is Turkevich where Au(III) from HAuCl4 is reduced by sodium citrate [11]. This process does not only reduce the oxidation number, but also resize gold particles in nanoscale.

The development of current research begins to lead to the synthesis of inorganic materials using natural materials to conform to the principles of Green Chemistry. Green synthesis of gold nanoparticles commonly used is using plant extracts such as the use of leaf extract [12] and flower extract [13]. Various plant extracts have been reported to be able to act as reducing agents of Au (III) in the synthesis of gold nanoparticles such as *Neem* [14], *Hibiscus rosa sinensis* [15], and *Coleus aromaticus* [16]. One of the contents of plant extracts that can act as Au (III) bioreductors is anthocyanin [17].

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Anthocyanin is a secondary metabolite that is widely found in vegetables and fruits [18]. *Hibiscus sabdariffa* is a plant that has bioactive compounds in the form of anthocyanin which is a compound with antioxidant properties or reductase so that it can act as a reducing agent for metal ions [17]. Anthocyanins are soluble in water and are polar in nature so they can be dissolved in polar solvents such as ethanol, acetone, chloroform, and chloride or formic acid [19]. The use of water solvents in anthocyanin extraction from *H. sabdariffa* in accordance with the principle of the fifth green chemistry [20].

Gold nanoparticles with ZnO composites were reported had various benefits such as solar cells [21], photocatalysts [22], and gas sensors [23] In general, the synthetic materials such as hydrazine hydrate [24] were used as reducing agents of gold nanoparticles syntheses. Anthocyanin has been known potential to replace the synthetic material as a reducing agent due to its high antioxidant activity [25]. The novelty of this research is the use of plant extracts immobilized in inorganic solid material for the synthesis of gold nanoparticles composites.

One of ways to produce gold nanoparticle composites on the surface of inorganic materials is through the principle of reductant immobilization [26]. Immobilization of reducing agents on the surface of inorganic materials can reduce the oxidation number of Au (III) to Au (0). This research was started by extracting *Hibiscus sabdariffa* using a water solvent to obtain anthocyanin and then proceed with immobilizing anthocyanin on the surface of ZnO. *H. sabdariffa* extract which was immobilized on ZnO will be reacted with Au (III) to produce Gold Nanoparticles-ZnO composites.

#### 2. Methods

#### 2.1. Extraction of H. sabdariffa Flowers

The flowers of *H. Sabdariffa* were obtained from Bojonegoro local market. Amount of 0.5 g of *H. Sabdariffa* flowers were added by 50 mL of aqua destilata by maceration process. The extraction was carried out with sonication at a frequency of 24 kHz in 5 minutes and a temperature of 30° C. The extraction was optimized with frequency variations from 24 to 40 kHz and then continued with variations in extraction time from 1 to 5 minutes. The optimal results from frequency and time variations continued by temperature variations from 30 to 70° C. At the end, continued by variations of solvent and flower extracts ratio from 1:25 to 1:125. The concentration of extract was compared using a UV-Vis spectrophotometer. The filtrate was separated using Whatman filter number 42. The optimum extraction results were selected based on the highest absorbance according to the Lambert-Beer law which the absorbance is proportional to the concentration of the solutions.

#### 2.2. Synthesis of HAuCl4

One gram of Gold (UBS) is dissolved in aqua regia, 60 mL of aqua regia is then left to dissolve. The gold solution is diluted to produce 1000 mL of 1000 ppm HAuCl<sub>4</sub>.

#### 2.3. Synthesis of AuNPs-ZnO

ZnO is synthesized by mixing 2 g of zinc acetate in 15 mL and 8 g of sodium hydroxide in 10 mL of distilled water. The sodium hydroxide solution is poured into the zinc acetate solution slowly then 100 mL ethanol is added slowly to the solution. The precipitate formed was separated using Whatman no 42 filter paper. ZnO solids were dried at 110° C for 24 hours.

Composite synthesis was carried out by means of a reductive adsorption of Au (III) using *H. sabdariffa* extract immobilize on ZnO. The mixture is set to 80° C and stirred using a magnetic stirrer for 2 hours. The deposits and filtrate are separated by using centrifuge. The precipitate is separated by Whatman filter paper, then the precipitate was washed and filtered. The filtrate was observed using AgNO<sub>3</sub> as a chloride ion detector, washing ended when no AgCl precipitate was formed. The precipitate was separated again using a centrifuge then dried at 110° C for 24 hours.

#### 3. Results and discussions

#### 3.1. Extraction of H. sabdariffa flowers

The flowers of H. sabdariffa were crushed by using blender before mix with solvent. The extraction of anthocyanin pigments from H. sabdariffa flowers was then carried out by the sonication method on various variables, namely wave frequency, process temperature, extraction time, and the ratio between Hibiscus sabdariffa flower mass and aqua destilata solvent. Aqua destilata was used as a polar solvent to dissolve anthocyanin from H. sabdariffa flower [27]. Sonication is an extraction method that can increase the amount of anthocyanin in solution [28]. The result of H. sabdariffa extraction are presented in Figure 1.

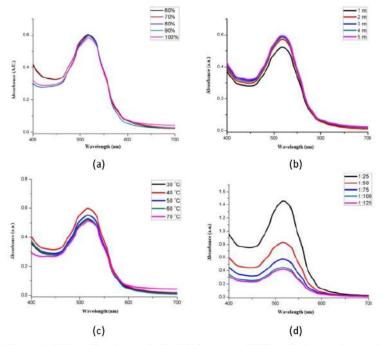


Figure 1. Effect of various variable (a) Frequency (b) time (c) temperature and (d) ratio on anthocyanin of H. Sabdariffa extraction.

Figure 1 shows that (a-d) will use a 60% or 24 kHz frequency to prevent damage the pigment of the pigment in high frequency (100% or 40 kHz). The same reason for utilizing 40° C temperature because the pigment will damage in high temperature. At high temperatures it will damage delfinidin 3-Osambubiose and cyanidin 3-O-sambubiose which is an anthocyanin group into protecuat acid and gallic acid, while those that are more easily degraded are delfinidin 3-O-sambubiose compared to cyanidin 3-O-sambubiose[23]. For optimal time it is done in 5 minutes, so that the longer the time the concentration of anthocyanin pigment will also increase. Then for the ratio used to obtain optimal results using a 1:25 ratio it will get optimal extraction and have a high anthocyanin concentration.

Based on the FT-IR spectra of Hibiscus sabdariffa extract in Figure 2, there is a C = O group at 1631.83 cm-1, C-O-C at 1093.67 cm-1, and O-H at 3446.91 cm-1. The groups that appear in the FTIR spectra reinforce the assumption that Hibiscus sabdariffa extract contains anthocyanins. Anthocyanin is very easy to oxidize so that it can act as a reducing agent for Au (III) to Au (0).

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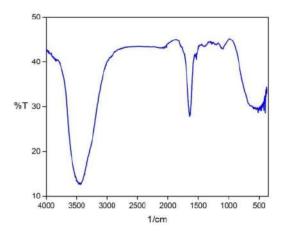


Figure 2. Spectra of FT-IR H. sabdariffa extract.

#### 3.2. Synthesis of HauCl4

The gold source used in the synthesis of gold nanoparticles is HAuCl4. HAuCl4 is formed by dissolving gold in aqua regia. The UV-Vis spectra resulting from the dissolution of gold showed that there were peaks at a wavelength of 309 nm which indicated the formation of a complex [AuCl4]. The complex [AuCl4] is a complex consisting of Cl ligands and central Au (III) ions. The central ion Au(III) of the complex [AuCl4] which will be reduced using *H. sabdariffa* extract.

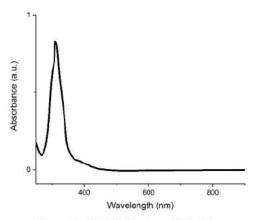
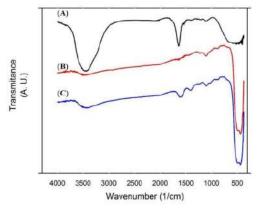


Figure 3. UV-Vis Spectra of HAuCl<sub>4</sub>.

Table 1. Characteristics of ZnO.

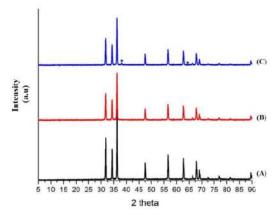
Data	ZnO
a (Å)	3.2494
b (Å)	3.2494
c (Å)	5.2038
Crystal system	Hexagonal

ZnO was characterized using XRD to find out the characteristic peaks and it was found that there was a peak at 36.3° which indicated hkl 111, and 47.5° which showed hkl 200. XRD results showed that ZnO was successfully synthesized, then the synthesis results were reused using Match software to get the values of a, b, c and other crystal characteristics. The characteristics of ZnO crystals are presented in table 1.



**Figure 4.** FTIR spectra of (a)*Hibiscus sabdariffa extract* (b) ZnO and (c) Immobilized *H. sabdariffa* extract on ZnO.

The immobilization process of the extract was carried out by macerating the extract on ZnO. *H. sabdariffa* extract which was immobilized on ZnO was then put into a solution of HAuCl<sub>4</sub> to reduce Au (III) to Au (0). Based on the results of FT-IR seen immobilization of *H. sabdariffa* extract was successfully carried out, in the FT-IR ZnO-HS spectra (Figure 5. B) it was seen the emergence of absorption of C = O at 1618.33 cm<sup>-1</sup> and C-O-C at 1101.39 cm<sup>-1</sup>. The diffractogram of the synthesis of AuNPs-ZnO showed that ZnO which had immobilized *H. sabdariffa* extract was able to reduce Au (III) on the surface of ZnO. The diffractogram results in the appearance of Au(0) peaks in the Au-ZnO diffractogram.



**Figure 5.** XRD diffractogram of (a) ZnO (b) Immobilized *H. sahdariffa* extract on ZnO and (c) AuNPs-ZnO.

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#### 4. Conclusion

Hibiscus sabdariffa was successfully extracted optimally at 40° C and 24 kHz for 5 minutes. Immobilization of *H. sabdariffa* extract in ZnO material has been confirmed using FTIR which is characterized by the emergence of absorption of characteristic functional groups of anthocyanins. The formation of gold nanoparticles and ZnO composite was shown by showing the peak gold characteristics in the XRD results.

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