The potential of arabica coffee grounds nanoparticles as an active compound of pharmaceutical preparations

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Abstract. Arabica coffee (Coffea arabica L.) grounds are kind of solid waste produced from filtering process containing many bioactive components and good for health. Nanoparticle technology can be used to produce nanoscale particles without separating the active compounds contained the rein. The aim of this study is to determine the potential of Arabica coffee grounds nanoparticles as active compounds to be used in pharmaceutical preparations. The research procedure was begun with collect the Arabica coffee grounds which was then processed into nanoparticles using the ball milling and ultrasonication technique. The nanoparticles were characterized using some techniques, namely particle size analysis using PSA (Particle Size Analyzer), phytochemical screening, total phenolics, pH, particle morphology using SEM (Scanning Electron Microscopy) and particle structure using FT-IR (Fourier Transform Infrared Spectroscopy). The results showed that Arabica coffee grounds nanoparticles has a particle size of 396.0 nm with a polydispersity index of 0.254, solubility of 70.680% and pH 5.33, the positive Arabica coffee grounds nanoparticles containing secondary metabolites of alkaloids, saponins and phenolics with total phenolics 1246.90 μgGAE/g. Morphology of Arabica coffee grounds nanoparticles was in irregular granules form with an asymmetrical size and has active functional groups such as OH, NH, CH alkane, C=N, C=O carboxylic acid, -C=C-, CO, and CC. It can be concluded that Arabica coffee grounds nanoparticles were potentially can be used as active compounds in various pharmaceutical preparations.

1. Introduction

Aceh is very famous for its distinctive Arabica coffee flavor. Arabica coffee is always available in coffee shops in all corners of Aceh and even has reached foreign countries. The existence of a coffee shop in Aceh has become a local wisdom that cannot be avoided for the local community. The culture of drinking coffee has become a hereditary tradition for Acehnese people. Acehnese people drink three to five glasses of coffee a day. It creates a lot of coffee grounds produced by each coffee shop from the
process of making Arabica coffee drinks. Arabica coffee grounds are still not utilized optimally thus its existence just piles up.

Arabica coffee grounds are kind of solid waste produced from filtering process containing many bioactive components and good for health. An Arabica coffee bean contains 3.43% oil[1] Arabica coffee powder contains 9.09% oil. 1,2-Benzenedicarboxylic acid, 20.48% bis(2- ethylhexyl) ester as main compound[2] and phenolic acid [3]. Coffee grounds contain oil with 15.3% residue of the sample weight [4]. Coffee oil also contains 81.3% triglyceride molecules containing 51.4% palmitic fatty acids and 40.3% linoleic acid. 15.9% free fatty acid [5] 48.93% palmitate, 10.41% stearic acid, 7.52% oleic acid, 10.46% linoleic acid, 4.48% arachdic acid [6]. In addition, coffee grounds contain active compounds such as phenolic groups, chlorogenic acids, flavonoids [7–10] as well as some compounds that act as antioxidants [11]. The bioactive content has biological activities that are very beneficial for health and can be used as active compounds in various pharmaceutical preparation.

The ability of the drugs to reach their site is often hampered, thereby reducing its efficiency. Particle size is one of the factors affects the efficiency of a compound or drugs [12], normally, only a small number of drugs can reach the target site, while most drugs are distributed throughout the body according to their physicochemical and biochemical content [13]. A tiny size (nano size) can help the distribution process of drugs reach their target [14].

Nanoparticle technology is a material technology related to the creation of tiny particles, namely nanometers (one billionth of a meter) that are more efficient in their use[15]. The use of nanotechnology has developed in various fields of research including drugs[16]. The utilization of nanoparticles as drugs carriers has been used in recent years [17]. The active ingredients of nano-sized drugs can pass through cell walls and membranes to reach the target cell. In addition, nano size also has solubility and high absorption efficiency [10,11].

Based on the above description, the aim of this study is to determine the potential of Arabica coffee grounds nanoparticles as active compounds to be used in pharmaceutical preparations. The method used for the manufacture of nanoparticles from coffee grounds is the ball mill and ultrasonication technique.

2. Material And Methods
2.1. Tools and materials
Tools used in this study were baker, graduated cylinders, and analytical balance. The instruments used were ball mill (High Energy Milling (HEM)), ultrasonicator (Branson 3510), UV-Vis spectrophotometer (UV-1700 pharma spec SHMADZU), particle size analyzer (Delsa™ Nano Beckman Coulter), scanning electron microscope (FEI Quanta 650) and Fourier transform infrared (IR-Prestige-21 SHIMADZU). Materials used in this study was sample of Arabica Coffee grounds from coffee shop in Banda Aceh, reagent test, and aquadest.

2.2. Research procedures
The research procedure was begun with collect the Arabica coffee grounds from a coffee shop in Banda Aceh. The coffee grounds was cleaned of dirt and dried in an oven at 80°C for 4 hours.

The dried coffee grounds were then processed into nanoparticles by put into a ball mill with a speed of 22000 rpm for 30 minutes with turning on two minutes and turning off ten minutes. Then they were sonicated using ultrasonicator. Sample was put into one container and added aquadest as a solvent, this process for 15 minutes.

The nanoparticles were then characterized using some techniques namely particle size analysis using PSA (Particle Size Analyzer), phytochemical screening, total phenolic using (UV-Vis spectrophotometer), pH, particle morphology using SEM (Scanning Electron Microscopy) and particle structure using FT-IR (Fourier Transform Infrared Spectroscopy).
3. Results and Discussion
The resulting nanoparticles were performed with two variations: nanoparticles without ultrasonication and nanoparticles with ultrasonication.

3.1. Particle Size
The size determination of Arabica coffee grounds nanoparticles was carried out using a PSA (Particle Size Analyzer) instrument. Based on the PSA test results showed that Arabica coffee grounds without an ultrasonication process had a particle size of 2923.4 nm with a polydispersity index of 1.002. While the size of the nanoparticles with the ultrasonication process had a particle size of 396.0 nm and a polydispersity index of 0.254 as shown in Fig 1.

Figure 1. PSA graphics of Arabica coffee grounds nanoparticles (a) without ultrasonication (b) with ultrasonication.
Ultrasonic wave is the propagation of mechanical momentum and energy, so a medium to propagate is needed[18], in this study the medium used is liquid (aquadest). The ultrasonication process is very effective in breaking down agglomerates, aggregates and primary particle materials, consequently it can produce particles of Arabica coffee grounds in nano size. The size of particles in nano form can make it easier for particles to enter the cell. The smaller the particle size, the easier it enter the cell hence its absorption increases in the body [10,11].

Arabica coffee grounds nanoparticles have homogeneous particle size distribution which is indicated by the polydispersity index value approaching to zero. The polydispersity index value approaching to zero indicates a homogeneous particle size dispersion while the polydispersity index of more than 0.5 indicates high heterogeneity [19].

### 3.2. Solubility

Table 1 shows the solubility results of Arabica coffee grounds nanoparticles with a ball mill process without ultrasonication and with the ultrasonication process. The process without ultrasonication produces coffee grounds nanoparticles with a solubility of 50.733%, while the nanoparticles with an ultrasonication process produce 70.680% solubility. High solubility can help the process of absorption of active compounds so that it can produce a better effect.

| Sample                      | pH   | Water Content (%) | Solubility (%) |
|-----------------------------|------|-------------------|----------------|
| Without ultrasonication     | 5.92 | 5.372             | 50.733         |
| With ultrasonication        | 5.33 | 4.329             | 70.680         |

### 3.3. pH Test

pH level of Arabica coffee grounds nanoparticles was measured using a pH meter. pH is a parameter used to express the acidity of a solution. The pH level is very important in the process of making pharmaceutical preparations, since it can affect certain organ functions. Based on the results of measurements, arabica coffee grounds nanoparticles with ultrasonication process have a pH of 5.33, while without the ultrasonication process it has a pH of 5.92 (Table 1). A low pH value is caused by many acid compounds contained in Arabica coffee grounds.

### 3.4. Phytochemical Screening

Phytochemical screening is a process to determine phytochemicals or active ingredients which are secondary metabolites in plants [20]. Phytochemical screening is carried out using a group detection reactor or reagent test on a drip plate or test tube. Phytochemical tests include: analysis of alkaloid, flavonoids, terpenoids, steroids, saponins, and phenolics compounds. Based on the results of a qualitative test of phytochemical screening with several reagents test, it was obtained that Arabica coffee grounds nanoparticles positively contains secondary metabolites of alkaloids, saponins and phenolics as shown in Table 2.

Alkaloids are compounds mostly containing basic nitrogen atoms, usually in the form of heterocyclic rings. Alkaloids have effects in body health such as nervous system triggers, increase blood pressure, reduce pain, antimicrobials, sedatives, drugs for heart disease[10,11].

Saponins are a complex glycoside compounds consist of compounds resulting from the condensation of a sugar with an organic hydroxyl compound and when hydrolyzed will produce sugar (glycone) and non-sugar (aglycone) [12,13]. The saponins structure causes they have similar characteristic like soaps or detergents, so saponins are called natural surfactants. The name saponins is taken from this main characteristic of “sapo” in Latin, which means “soap”. Saponins have various
biological properties such as hemolytic ability, antibacterial, antifungal, antimolluscular, antiviral, cytotoxic or anti-cancer, effects of hypocholesterolemia and antiprotozoal [21].

Table 2. Phytochemical Screening results of Arabica coffee grounds nanoparticles.

| Chemical Composition | Reagen Test | Result |
|----------------------|-------------|--------|
| Alkaloids            | Mayer       | -      |
|                      | Wagner      | +      |
|                      | Dragendorff | +      |
| Flavonoids           | Mg+HCl      | -      |
| Steroids             | Lieberman Burchard | - |
| Saponins             | Aquadest    | +      |
| Phenolics            | FeCl₃       | +      |

Phenolic is a compound have one or more hydroxyl groups attached to aromatic rings[3]. There are three main groups of phenolic, namely flavonoids, phenolic acids and polyphenols[15,16]. Phenolic acid has a variety of biological activities in human body, such as increasing bile secretion, lowering blood cholesterol levels, having antimicrobial activity, for example in the Staphylococcus aureus bacteria. Phenolic acid also has several biological activities, such as antiulser, anti-inflammatory, antioxidant, cytotoxic and antitumor, antispasmodic, and antidepressant[22].

Flavonoids, terpenoids and steroids have negative results on Arabica coffee grounds nanoparticles phytochemical screening. This is probably caused by a long coffee production process, including drying, roasting, grinding and extracting coffee. Many compounds are missing.

3.5. Total Phenolics
Analysis of total phenolics of arabica coffee grounds nanoparticles using VU-Vis spectrophotometry at a wavelength 725 nm, the total phenolics content expressed as gallic acid equivalent (GAE). GAE is a reference in the analysis of total phenolics contained in a substance [23]. Arabica coffee grounds nanoparticles have a high phenolic total of 1246.90 μgGAE/g, the high total phenolics in arabica coffee grounds nanoparticles is caused by the process of making nanoparticles which destroy particles into very small sizes without removing the components contained therein. High total phenolics in Arabica coffee grounds nanoparticles is one of the potential of Arabica coffee grounds nanoparticles as a source of active compounds in pharmaceutical preparations.

3.6. Particle Morphology
Surface morphology of Arabica coffee grounds nanoparticles was analyzed using SEM (Scanning Electron Microscopy). The measurement of SEM image from Arabica coffee grounds was carried out using the LFD detector and 12.50 KV HV on the 50 μm scale bar. Based on the SEM results shown in Fig 2, it can be seen that the morphological state of Arabica coffee grounds nanoparticles at 500x magnification in the form of irregular granules form with an asymmetrical size. The asymmetry of particle size is due to clumping in the sample.
3.7. Particle Structure

Particle structure of Arabica coffee grounds were analyzed using FT-IR (Fourier Transform Infrared Spectroscopy) instrument. FT-IR is one instrument widely used to determine the molecular vibrational spectrum and also can be used to detect the molecular structure of compounds through the identification of functional groups of compounds[24].

Based on analysis using FT-IR, Arabica coffee grounds nanoparticles were performed absorption at wave number of 3381.21 cm\(^{-1}\); 3367.71 cm\(^{-1}\); 2926.01 cm\(^{-1}\); 2295.26 cm\(^{-1}\); 1707.00 cm\(^{-1}\); 1625.99 cm\(^{-1}\); 1600.92 cm\(^{-1}\); 1255.66 cm\(^{-1}\); 1064.71 cm\(^{-1}\); 893.04 cm\(^{-1}\). The infrared spectrum result of Arabica coffee grounds nanoparticles is shown in Fig 3. The infrared spectrum obtained then was compared to the correlation table to obtain information about functional group structure on Arabica coffee grounds nanoparticles. Based on these result, it can be identified that Arabica coffee grounds nanoparticles have functional groups OH, CH alkane, C=\(\equiv\)N, C=O carboxylic acid, -C=\(-\), CO, and CC as shown in Table 3. These functional groups are an active functional group of compounds, so that the compounds in the Arabica coffee grounds nanoparticles, can be used as active compounds in various pharmaceutical preparation.

Table 3. Characterization of absorbance from FTIR spectrum peak of Arabica coffee grounds nanoparticles.

| Wave number (cm\(^{-1}\)) | Functional Groups          |
|---------------------------|-----------------------------|
| 3381.21                   | O-H                         |
| 3367.71                   | N-H                         |
| 2926.01                   | C-H alkene                  |
| 2295.26                   | C=\(\equiv\)N               |
| 1707.00                   | C=O carboxylic acid         |
| 1625.99                   | -C=\(-\)                    |
| 1600.92                   | -C=\(-\) aromatic           |
| 1255.66                   | C-O strech                  |
| 1064.71                   | C-O-H deformation           |
| 893.04                    | C-C                         |
4. Conclusions

Arabica coffee grounds nanoparticles have a particle size of 396.0 nm and positively contain secondary metabolites in the form of alkaloids, saponins, and phenolics which have many biological activities for health. Arabica coffee grounds nanoparticles were potentially can be used as active compounds in various pharmaceutical preparation like the preparation of antioxidant masks and anti-inflammatory gels.

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