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Morphological study of fluorescent carbon Nanoparticles (F-CNPs) from ground coffee waste soot oxidation by diluted acid

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Abstract. Coffee ground waste utilization for fluorescent carbon nanoparticles (F-CNPs) through soot oxidation with diluted HNO$_3$ has been conducted. Soot was obtained through three different treatments to coffee ground waste; which was burned in furnace at 550°C and 650°C and directly burned in a heat-proof container. Then they were analyzed morphologically with Scanning Electron Microscope (SEM) instrument. Soot from direct burning indicated the optimum result where it has denser pores compared to other two soots. Soot obtained from direct burning was refluxed in diluted HNO$_3$ for 12 hours to perform the oxidation. Yellowish brown supernatant was later observed which lead to green fluorescent under the UV light. F-CNPs characterization was done in Transmission Electron Microscopy, which showed that 7.4-23.4 nm of particle size were distributed.

1. Introduction
Coffee for the last decade has become an important beverage around the globe. Drinking coffee has become a lifestyle for most societies either to kick start the morning or as afternoon snack companion. Since 2014, Indonesia has become one of the top countries for producing and exporting coffee beans. For the last decade, business in coffee world has also been growing in Indonesia which can be indicated from the increasing number of international coffee tenants and local coffee shops. This consequently increases the amount of coffee ground waste produced. Survey conducted to international and local coffee shops, in a day, at least 1 kg of coffee waste can be collected.

Ground coffee waste is the primary by-product derived from espresso extraction process at high temperature. It is considered as a potential waste with negative impacts. The big amount of coffee waste in landfill can produce methane and carbon dioxide, greenhouse gases that contribute to global warming. Furthermore, it also contributes to huge financial cost for its deposit. Although with the disadvantages it causes, ground coffee waste is a relatively unique organic waste. Elemental analysis to ground coffee waste shows coffee samples to possess high carbon (>58%), low nitrogen (<2%), and low ash (<1%) contents and low polarity coefficient. With that high organic and low inorganic material, ground coffee waste has plenty of uses.

Via pyrolysis, ground coffee waste can be made as soot for fluorescent carbon nanoparticles (F-CNPs) through soot oxidation methods. F-CNPs are biocompatible and chemically inert, which gives more advantages than the cadmium based fluorescent nanoparticles (F-NPs). As a result, the emergence of F-CNPs with better properties has increased the interest for F-CNPs synthesis.
F-CNPs derived from soot oxidation with nitric acid will give a green fluorescence and soluble in water. There are two steps of F-CNPs synthesis involving soot oxidation in nitric acid for 12 hours at 100°C by reflux and size separation of carbon particles. However, instead of using soot derived from candle burning, soot used in the synthesis process was obtained from the ground coffee waste collected. There are three different methods used to gain ground coffee waste soot which were then observed and compared by scanning electron microscopy (SEM) and F-CNPs/coffee soot was analyzed by transmission electron microscopy to observe the particle size distribution and using the UV light to check the fluorescence properties.

2. Materials and methods

2.1. Soot derived from ground coffee waste
There are two approaches to get soot from ground coffee waste; by furnace burning and direct burning in a heat proof container. Soot was black in colour and came in as powdery material. First, for furnace burning soot, ground coffee waste was dried in an oven at 120°C to lose the water content and putted into two porcelain dishes. Two furnaces were used, one was set to 550°C and the other was set to 650°C and ground coffee waste was putted for 4 hours to obtain a black powdery material. White powder might be formed due to the inorganic content in coffee waste collected. To separate the white powder, it was diluted into weak acid, HCl 2N and then was centrifuged at 4000 rpm for 10 minutes. The black soot was collected and dried in oven and characterized with SEM. Direct burning soot was obtained from ground coffee waste soaked in kerosene inside a heat-proof container. It was burned for approximately 20 minutes or until fire was out. Soot can be collected at the side of the container and finally characterized with SEM.

2.2. Soot oxidation
As many as 25 mg carbon soot derived from ground coffee waste was mixed with 15 ml of 5M nitric acid and refluxed at 100°C for 12 hours with magnetic stirrer. The solution was cooled and centrifuged at 3000 rpm for 10 minutes to separate the unreacted carbon soot. Then, yellowish brown supernatant was collected which gave a green fluorescence under UV exposure. It was mixed with acetone and centrifuged at 14000 rpm for 10 minutes to separate the excess nitric acid. Black precipitation was collected and the aqueous solution was discarded and dissolved in 5-10 ml water.

2.3. Size-based separation of carbon particles
Size-based separation of carbon particles was performed in a solvent mixture with the combination of high speed centrifugation. Carbon particles are soluble in water, ethanol and acetone but insoluble in chloroform. Therefore, solvent mixture of water-ethanol-chloroform was used where water-ethanol helped to solubilize particle whereas chloroform decreased the solubility. It was then followed by a step-by-step separation in different centrifugation speed from 4000-16000 rpm. The F-CNPs in water synthesized was then characterized by TEM to see the particle size distribution.

3. Result and Discussion

3.1. Soot morphology
With three different treatments to ground coffee waste, giving three different soot which for each were characterized by SEM to observe their morphology. There was a huge significant different among them. The coffee soot which was in furnace burning at 550°C has 8 pores per 10 cm length, while at 650°C, it has 18 pores per 10 cm. In contrast, the coffee soot obtained from direct burning has smaller pores per 10 cm length as shown in Figure 1. Based on the morphological study of the soot, it can be concluded that coffee soot from direct burning gave the most suitable carbon source to the synthesis of F-CNPs as the most pores it has, the more carbons can bind the nitrogen and oxygen from nitric acid.
Figure 1. The coffee ground waste soot morphology: (a) Soot derived from furnace burning at 550 °C, (b) Soot derived from furnace burning at 650 °C, (c) Soot derived from direct burning

3.2. F-CNPs from diluted HNO₃ oxidized ground coffee waste soot
After 12 hours refluxing the coffee soot with nitric acid, black solution was formed and was centrifuged at 3000 rpm for 10 minutes. Then, the yellowish brown supernatant was yielded. Under the UV light, the green fluorescent was observed as shown in Figure 2.

Figure 2. Fluorescent properties of F-CNPs obtained from ground coffee waste soot oxidation: (a) Yellowish brown supernatant, (b) Green fluorescent under the UV light

F-CNPs/coffee soot was also characterized by TEM to see the particle size distribution. The result showed that nanoparticles were formed with the size average of 7.4–23.4 nm. The result is shown in Figure 3.

Figure 3. TEM image of F-CNPs/coffee soot
4. Conclusion
Soot derived from ground coffee waste with direct burning method was the best method to the by-
product to get soot for F-CNPs. Although F-CNPs/coffee soot formed still had large distribution
particle size (7.4–23.4 nm), it still shows that coffee waste is promising as carbon source for F-CNPs.
However, further research to get better method and alternative for coffee waste utilization are still
needed to be conducted.

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