วารสารวิทยาศาสตร์และเทคโนโลยี มรย.
YRU Journal of Science and Technology
ISSN : 2539-5688

Proximate Analysis and Structural Properties of Arabica Spent Coffee Grounds
การวิเคราะห์องค์ประกอบทางเคมีและสมบัติทางโครงสร้างของกากกาแฟพันธุ์อาราบิก้า

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(Recieived: Jan 14, 2020; Revised: Apr 23, 2020; Accepted: May 13, 2020)

Abstract

The chemical determination of spent coffee grounds in this research, focusing on proximate analysis and the structural properties. The proximate composition of spent coffee grounds were: 2.84±0.17% of moisture, 14.93±0.55% of ash, 10.93±0.03% of protein, 14.42±0.43% of oil, and 56.88% of carbohydrate. The morphology of spent coffee grounds before and after the oil extraction were observed through scanning electron microscopy (SEM). The image of before oil extraction sample exhibited the agglomeration of the spent coffee grounds particles. The sample of spent coffee grounds after the oil extraction appeared to have particle distribution because the oil drops drawn toward from the inside of the spent coffee grounds pores. The percentage of carbon and oxygen atom were found at the various sports surface of spent coffee grounds by using X-ray energy dispersive spectroscopy analysis (EDS). The main functional groups of spent coffee grounds were characterized by using the fourier transform infrared spectroscopy (FT-IR). The results of this experiment could be used as basic information that will be used in the further industrial level.

Keywords: Spent coffee grounds, Proximate chemical analysis, Structural properties

บทคัดย่อ

การวิเคราะห์ทางเคมีของกากกาแฟในงานวิจัยนี้ จะมุ่งเน้นไปที่การวิเคราะห์องค์ประกอบทางเคมีและสมบัติทางโครงสร้าง ซึ่งองค์ประกอบทางเคมีของกากกาแฟ พบว่ามีปริมาณความชื้นเท่ากับ 2.84±0.17% ปริมาณเถ้าเท่ากับ 14.93±0.55% ปริมาณโปรตีนเท่ากับ 10.93±0.03% ปริมาณไขมันเท่ากับ 14.42±0.43% และปริมาณคาร์โบไฮเดรตเท่ากับ 56.88% ลักษณะสัณฐานวิทยาของกากกาแฟก่อนและหลังสกัดไขมันเมื่อนำไปวิเคราะห์ด้วยกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด (SEM) พบว่ารูปที่ได้จากกากกาแฟก่อนสกัดน้ำมันอนุภาคจะรวมตัวกันเป็นกลุ่มก้อน เมื่อผ่านการสกัดน้ำมันจะเห็นว่าอนุภาคจะกระจายตัวแยกออกจากกัน สาเหตุเนื่องมาจากการที่หยดน้ำมันภายในอนุภาคได้ถูกสกัดออกมาจากรูพรุนของกากกาแฟ บนพื้นผิวของกากกาแฟพบปริมาณคาร์บอนและออกซิเจนโดยได้จากการวิเคราห์ผ่านอุปกรณ์ตรวจจับสัญญาณเอกซ์เรย์ (EDS) หมู่ฟังก์ชันหลักของกากกาแฟจะถูกนำไปวิเคราะห์ด้วยเครื่องฟลูเรียร์ทรานส์ฟอร์ม อินฟราเรดสเปคโทรโฟโตมิเตอร์ (FT-IR) ซึ่งผลจากการทดลองนี้สามารถนำไปเป็นข้อมูลเบื้องต้นในระดับอุตสาหกรรมต่อไป

คำสำคัญ : กากกาแฟ การวิเคราะห์องค์ประกอบทางเคมี สมบัติทางโครงสร้าง

Introduction

Spent coffee grounds, by-products of coffee fruit and bean processing, was generated from the coffee fruit residues amounting up to 50% of the fruit mass. Spent coffee grounds contain large amounts of organic compounds that can be exploited as a source of value-added products (Mussatto *et al*., 2011, pp. 368–374) Thus, the review of coffee residue has been reported about its components. For renewable source, Hamamre *et al*. (2012, pp. 70-76) studied to use the extracted coffee oil was converted into fatty acid methyl ester via single step esterification and two step Transesterification. They found that the two step transesterification process was met to be highly effective to convert spent coffee ground oils to fatty acid methyl ester. Lisowski *et al*. (2019, pp. 173-183) found that chemical composition of the spent coffee grounds confirmed the very good quality of the raw material as a biofuel, much higher than that for wood biomass. Moreover, the defatted spent coffee grounds had potential application to produce carbohydrates for ethanol production by fermentation (Rocha *et al*., 2014, pp. 343-348) In food industry, Hussein *et al*. (2019, p. 273-282) discovered the potential use of spent coffee grounds as a promising source of fiber and nutrition in the final product. They evaluate spent coffee grounds to use as functional food ingredient in sponge cake. In addition, Rahimnejad *et al*. (2015, pp. 257-264) reported on the use of coffee spent coffee ground as a feed ingredient for fish feed. They studied the terms of growth performance, feed utilization, body composition, and antioxidant enzyme activity.

Nowadays, the world was aware to reduce waste from the social activities and increased the value added of the residue for reuse and recycling. Thus, the aim of the present study consisted in evaluating the proximate analysis and the structural properties of Arabica spent coffee grounds, as a low-cost and versatile resource materials at Khao Kho District, Phetchabun Province. Today, there was many coffee shops appearing as evidences. In addition, many tourists in Khao Kho like to drink coffee that caused a lot of coffee grounds after consumption. The spent coffee grounds could be used in multiple application about food fields and extracted coffee oil could be developed for the renewable energy industrial areas.

Objectives

1. To determine the proximate analysis of Arabica spent coffee grounds in Phetchabun Province
2. To study the structural properties of Arabica spent coffee grounds in Phetchabun Province

Material and Methods

1. Materials

Arabica spent coffee grounds after consumption, provided by a local rice mill from Narin Coffee Plantation, Amphoe Khao Kho, Phetchabun Province, Thailand was used as a raw material in this study. The spent coffee grounds were sun dried. Then, the spent coffee grounds were stored in a closed polyethylene bottle and kept at room temperature for later use. All chemicals in this study were purchased from Labscan (Thailand) and Merck (Thailand).

2. Methods

2.1 Proximate Analysis

Moisture, ash, oil and protein contents of spent coffee grounds were determined adapting to the method of AOAC (2000).

2.1.1 Moisture Content

Moisture content of spent coffee grounds were measures in terms of percentage quantified moisture containing in sample. This value was investigated by weighing about 5 g of sample into aluminum pan sealed with a cover which was partially closed. Then, aluminum pan was taken to the oven setted at the temperature of 105 °C. Finally, the pan was weighed every hour until the weight of sample was constant. The moisture content of the sample is calculated using Equation 1.

$$\%Moisture \quad \frac{A-B}{A}\times 100 \qquad \text{... (1)}$$

Where:

%Moisture = Percentage of moisture in the sample

A = Weight of wet sample (g)

B = Weight of dry sample (g)

2.1.2 Ash Content

Ash was determined by incinerating the 5 g of dry basis sample at 550 °C for 2 h in furnace (Scientific Promotion Co.Ltd., Thailand). The amount of ash was quantified by gravimetric method and calculated using Equation 2.

$$\%Ash = \frac{A-B}{A}\times 100 \qquad \text{... (2)}$$

Where:

%Ash = Percentage of ash in the sample

A = Weight of sample before incinerating (g)

B = Weight of sample after incinerating (g)

2.1.3 Protein Content

The content of total protein was determines by Kjeldahl methd by Foss/Kjeltec 8400, Cannada. The protein content of dry basis sample was calculated by using a conversion factor of 6.25 multiply by nitrogen content.

2.1.4 Oil Content

Spent coffee grounds were sun dried and stored in closed polyethylene bottle at room temperature for later use extraction. To determine the amount of extractable crude oil, a dried spent coffee grounds (5 g of dry basis sample) was packed into a cellulose thimble and plugged with cotton. The thimble was then loaded into a Soxhlet apparatus and extracted for 2 h using 150 mL of n-hexane as the solvent. At least three trials were done for seed sample. After extraction, the hexane solvent was evaporated under reduced pressure and the oil fraction was dried in an oven at 60 °C until constant weight. The total oil was calculated as a percentage of spent coffee grounds dry weight followed Equation 3.

$$\%Oil\ content = \frac{A}{B}\times 100 \qquad \text{...(3)}$$

Where:

% Oil content = Percentage of oil in the sample

A = Weight of extracted oil (g)

B = Weight of dry sample (g)

3. Scanning Electron Microscopy and Elemental Analysis

The physical appearances and percentage of each element of spent coffee grounds without the moisture content were observed by a scanning electron microscope (SEM), (JEOL Model JSM- 6010LV, USA). Prior observation, the samples were covered with gold and mounted over a carbon film.

4. Fourier Transform Infrared Spectroscopy (FT-IR) Analysis

The IR spectrum of spent coffee grounds without the moisture content were recorded with FT-IR instrument (PerkinElmer, USA). The sample was placed on diamond crystals for attenuated total reflectance spectroscopy. A background spectrum was taken before run sample spectrum. The spectrum was collected in the range of 4,000-400 cm^{-1}.

Results and Discussion

1. Proximate Analysis

The Arabica spent coffee grounds showed the composition contents in Table 1. The ash content in this study exhibited the higher value compared in the previous work (1.49±0.06%wt). Brunerova *et al*. (2019, pp. 8-9) reported the low ash content and moisture content not exceed 15% in spent coffee grounds could be produced the positive behavior of fuel during burning. Moreover, Smaga (2016, p. 28) studied the coffee and tea grounds can be combined in suitable proportions with other components to produce the solid fuel in consideration of calorific value, moisture and ash content. The results of proteins and oil contents were similar according to Silva *et al*. (2018, pp. 221-222). They reported the proteins and fat contents were 10.03±0.28 and 14.70±0.56, respectively. In literature research, the proteins and fat contents in spent coffee grounds were a slightly different values that were reported by Ballesteros *et al*. (2014, pp. 3496-3497) & Somnuk *et al*. (2017, p. 182) These differences might be cause by the cultivated area conditions, the roasting conditions and extraction process (Kovalcik *et al*., 2018, pp. 104-119). The spent coffee grounds were rich in protein and oil content that could be considered as potential functional ingredients for the food industry such as feed utilization (Martinez-Saez *et al*., 2017, pp. 114-122) and resource of renewable energy such as raw material for liquid and solid fuel (Liu *et al*., 2017, pp. 157-161). Spent coffee grounds was composed of a variety of organic compounds that had carbohydrates being the most abundant. Rocha *et al*. (2014, pp. 343-348) presented the spent coffee grounds which can be used for fermentation by Saccharomyces cerevisiae to produce ethanol. Spent coffee grounds had the excellent potential for residue-based in various production.

Table 1 Proximate analysis of Arabica spent coffee grounds

Component	Composition of sample (%wt of dry basis)
Moisture	2.84±0.17
Ash	14.93±0.55
Proteins	10.93±0.03
Oil	14.42±0.43
Carbohydrates	56.88*

*Carbohydrate as 100-moisture-ash-protein-fat

2. Structural analysis

Figure 1 showed the shape and topology of spent coffee grounds before and after the oil extraction were observed through scanning electron microscopy (SEM). Spent coffee grounds before the oil extraction sample appeared to be formed out of the cluster than spent coffee grounds after oil extraction sample. The oil drops coagulated inside a particle of the sample. After the extraction, the high extraction temperature and non-polar of hexane solvent could be removed and drawn toward the oil drops from the inside of the spent coffee grounds pores. The sample of spent coffee grounds after the oil extraction appeared to have particle distribution, as shown in Figure 1. Somnuk *et al*. (2017, p. 188) reported the image of dried spent coffee grounds and defatted spent coffee grounds by SEM technique. They found that the after extraction sample had the many porous particles more than before extraction sample.

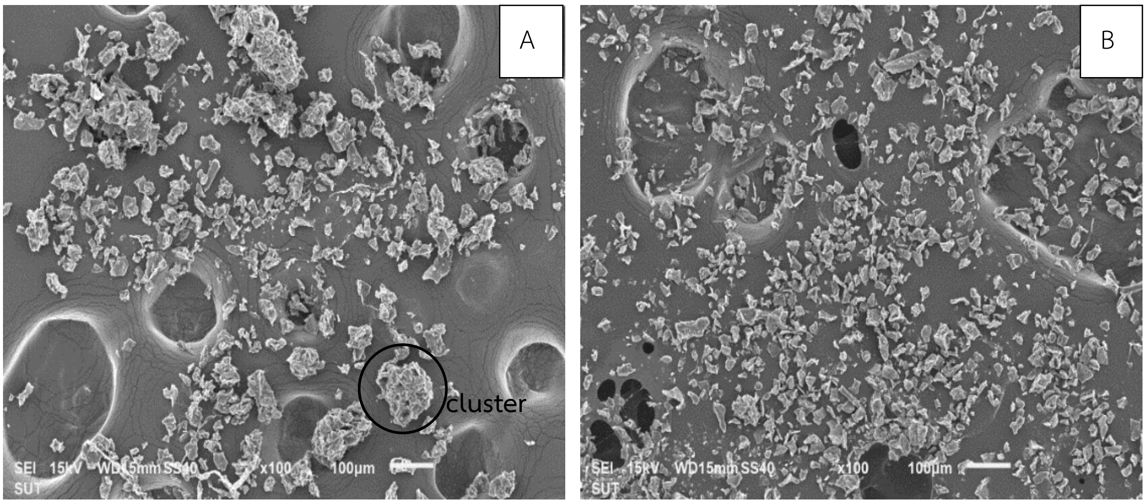

Figure 1 Scanning electron microscopy images

(A) spent coffee grounds before oil extraction (B) spent coffee grounds after oil extraction

3. Elemental Analysis

X-ray energy dispersive spectroscopy analysis (EDS) was performed in Table 2. The atomic % of carbon and oxygen on a particular area at the various sports surface of spent coffee grounds were 61.04±1.92 and 38.96±1.92, respectively. The carbon and oxygen elements were the main component of fat, protein and carbohydrate in the spent coffee samples. The analysis showed that the rich of carbon and oxygen indicated a great raw material to use in food industry and renewable energy resources. Zarrinbakhsh *et al*. (2016, pp. 7643-7644) found that the high concentrations of carbon and oxygen reflect the high content of carbohydrates found in the proximate analysis.

Table 2 Elemental analysis of Arabica spent coffee grounds

Elements	Composition of sample (%wt of dry basis)
C	61.04±1.92
O	38.96±1.92

4. Fourier Transform Infrared Spectroscopy (FT-IR) Analysis

The FT-IR spectrum of sample was presented in Figure 2. It was important to establish that the FT-IR technique could be used to confirm the presence of the functional groups of the significant chemical composition in the sample. The spectrum of spent coffee grounds showed a broad peak from 3,000 to 3,600 cm^{-1}. Two strong bands between 3,000 to 3,600 cm^{-1} were observed, 3,342 and 3,014 cm^{-1}, that could be related to the O-H and N-H stretching vibration of the lignocellulosic components and the protein in sample (Ballesteros *et al*., 2014, pp. 3501). The peaks close 2,852 and 2,922 cm^{-1} observed in sample could be attributed to the symmetric and asymmetric stretching of C-H bonds. Moreover, the previous researches reported peaks at the same region which was related to the quantitative analysis of caffeine content (Craig *et al*., 2012, p. 507) The band between 1,600 to 1,700 cm^{-1} associated with the ester group of triglyceride in lipid that presented the stretching absorption of C=O group at approximately 1,708 cm^{-1}. The region of 1,600 cm^{-1} was C=C vibration of lipid and fatty acids. The band at around 1,400 cm^{-1} corresponds to C-H bending of CH_3 group. The broad band of C-O in C-O-H bond of sugar stretching vibration was shown at 1,029 cm^{-1} (Silva *et al*., 2018, p. 223). A comparison of the FT-IR spectrum in our work with the previous research showed that the nearly similar of the wave number.

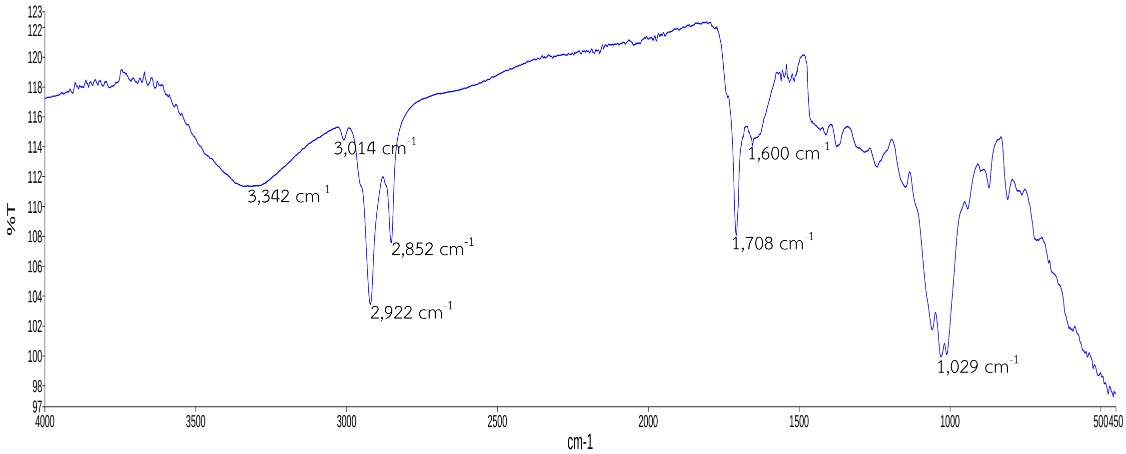

Figure 2 FT-IR spectrum of Arabica spent coffee grounds

Conclusion

The residue spent coffee grounds could be reused for possible applications because low cost and versatile resource materials. The proximate compositions in spent coffee grounds found that the high content of carbohydrates followed by ash, oil, proteins and moisture content, respectively. The spent coffee grounds were characterized to determine their surface properties, elemental and functional groups. The results found that obtained from this experiment in parts of proximate analysis and structural properties were interrelated. In the future work, we found that the spent coffee grounds represented to use in multiple application about the feedstock to produce the biodiesel. The by-product after oil extraction process, the defatted spent coffee grounds could be used as the biomass resource to produce the solid-fuel.

Acknowledgements

This study was financially supported by Phetchabun Rajabhat University (PCRU2562_KN037). The authors acknowledge Narin Coffee Plantation, Phetchabun, Thailand for spent coffee grounds and Miss Inthira Kalabut for assistance of this research. We also would like to thank the Faculty of Science and Technology, Phetchabun Rajabhat University for partial support of this research.

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