PAPER • OPEN ACCESS

Activation and characterization of waste coffee grounds as bio-sorbent

To cite this article: Mariana *et al* 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **334** 012029

View the article online for updates and enhancements.

Activation and characterization of waste coffee grounds as bio-sorbent

Mariana[1,2,4], Marwan[1], F Mulana[1], Yunardi[1], T A Ismail[3] and M F Hafdiansyah[3]

[1] Department of Chemical Engineering, Syiah Kuala University, Banda Aceh 23111, Indonesia
[2] Environmental and Natural Resource Research Center, Syiah Kuala University, Banda Aceh 23111, Indonesia
[3] Undergraduate Program of Chemical Engineering, Syiah Kuala University, Banda Aceh 23111, Indonesia

E-mail: mariana_hasyim@yahoo.com

Abstract. As the city well known for its culture of coffee drinkers, modern and traditional coffee shops are found everywhere in Banda Aceh, Indonesia. High number of coffee shops in the city generates large quantities of spent coffee grounds as waste without any effort to convert them as other valuable products. In an attempt to reduce environmental problems caused by used coffee grounds, this research was conducted to utilize waste coffee grounds as an activated carbon bio-sorbent. The specific purpose of this research is to improve the performance of coffee grounds bio-sorbent through chemical and physical activation, and to characterize the produced bio-sorbent. Following physical activation by carbonization, a chemical activation was achieved by soaking the carbonized waste coffee grounds in HCl solvent and carbonization process. The activated bio-sorbent was characterized for its morphological properties using Scanning Electron Microscopy (SEM), its functional groups by Fourier Transform Infra-Red Spectrophotometer (FTIR), and its material characteristics using X-Ray Diffraction (XRD). Characterization of the activated carbon prepared from waste coffee grounds shows that it meets standard quality requirement in accordance with Indonesian National Standard, SNI 06-3730-1995. Activation process has modified the functional groups of the waste coffee grounds. Comparing to natural waste coffee grounds, the resulted bio-sorbent demonstrated a more porous surface morphology following activation process. Consequently, such bio-sorbent is a potential source to be used as an adsorbent for various applications.

1. Introduction

Raw materials derived from organic materials can be converted into activated carbon since they contain carbon. Activated carbon is a porous solid produced from carbonaceous materials by heating at high temperatures. The more surface area of activated carbon the higher the adsorption capacity [1]. Previous research mentioned that the activated carbon from coffee grounds was able to adsorb iron ion up to 99,43% and was able to adsorb 99% mercury metal [2].

---

[4]   To whom any correspondence should be addressed.

The Province of Aceh, in the northern tip of Sumatera Island, Indonesia has been well known as the biggest Arabica coffee production in the country. In addition to coffee production, the region is also considered as the one with high consumption of coffee in the country. It is easily indicated by large number of traditional and modern coffee shops scattered in the capital city of Aceh Province, Banda Aceh. Every day, those shops produce a large quantity of spent coffee grounds as waste without any effort to convert them to various valuable products. In fact, since the used coffee grounds are wasted as trash, new environmental problems, such as odor and esthetics issues arise. Converting the waste coffee grounds into valuable products will not only provide economic potential, but also at the same time reduce the environmental problem.

In the Province of Aceh, particularly in Banda Aceh, a coffee drink is prepared in traditional way by pouring boil water through a small textile filter bag containing a bed of fresh roasted coffee grounds. A more concentrated coffee can be obtained by pouring the filtrate again and again through the filter bag. During the traditional dripping coffee making, water infiltrates into the coffee-ground and absorbs the liquid compounds and essence from the coffee, subsequently establishing the final, textural micro- or macro-pores within the coffee-grounds. Consequently, waste coffee grounds represent a porous material that has a potential for the use as a bio-sorbent to remove particular compounds from liquid phase.

Natural bio-sorbent of waste coffee grounds requires improvement in terms of their adsorption capacity by increasing their accessible pores and adsorption surface area. Such improvement could be achieved through both physical activation and chemical activation. Physical activation can be done by heating the natural porous materials at high temperatures, while chemical activation can be completed by adding a chemical solution [1]. Previous research conducted by Adhitiyawarman [3] shows that activated carbon from shell activated by chemical solution has water content, ash content, surface area, surface acidity and better adsorption capacity than activated carbon. Other study conducted by Li [4] on the production of activated carbon from seaweed by carbonization and chemical activation processes. It was noted that the purpose of carbonization is to obtain an initial pore on the structure of the activated carbon layer for next activation process. While the purpose of activation is to create new pores and expand the specific surface area of the produced activated carbon. Therefore, in this study we used two processes to obtain activated carbon that is the carbonization pstage following by chemical activation using HCl. This study will compare the characteristics of bio-sorbent before and after the activation process. The chemical composition of the coffee grounds is shown in Table 1.

Table 1. Chemical composition of coffee grounds

Composition	Percentage
Ether extract	0.48
Crude fiber	21.40
Raw protein	10.10
Ash	1.50
Free nitrogen extract	31.30
Tannin	7.80
Peptic	6.50
Non producing sugar	2.00
Low sugar	12.40
Chlorogenic acid	2.60
Caffeine	2.30
Total caffeine acid	1.60

Source: Murthy K N, 200

2. Experimental procedure and methodology

2.1. Materials and equipment
The equipment and tools used in this research were a muffle furnace (Isuzu EPTR-26R), mechanical sieve of 100 mesh, multi-purpose balance (Acis-BC 500), hot plate & stirrer (Cimarec), desiccator, centrifuge, pH meter, electric oven (GE-171), shaker, *Scanning Electron Microscope* (SEM) JEOL-JSM 6510 LA, UV-Vis Spectrophotometer, FTIR Spectrophotometer Prestige-21 (Shimadzu) and X-Ray Diffraction (Shimadzu 6000). While the materials used in this study are the coffee grounds, obtained from the Solong coffee shop located in Lampineung Jl. Panglima Nyak Makam, Banda Aceh, Indonesia.

2.2. Activation of Waste Coffee Grounds
Activation of coffee grounds was performed physically and chemically. Physical activation was conducted through the following stages: the waste coffee ground powder was brewed with hot water for 10 minutes and then filtered. Subsequently, the material was dried under the sun then carbonized in a muffle furnace at 600 °C for 4 h. The carbonized waste coffee grounds then were cooled and weighted to compare their weight before and after carbonization. Furthermore, the carbonized coffee grounds were sieved with the size of 100 mesh [7,8]. The sieved activated coffee grounds were then subjected to chemical activation by soaking in a solution of HCL 0.1 M for 48 h. It then was drained and placed in an oven at 105 ºC for 24 h. The dried materials were stored in the desiccator.

2.3. Characterization of Bio-sorbent
The characterized parameters in this study were performed by the method of Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), X-Ray Fluorosence (XRF) and Fourier Transform Infrared (FTIR).

2.3.1. Water Content. The calculation of water content was performed according to Equation (1) where the weight of the activated carbon before and after heating at 105ºC for 3 h was noted.

$$Water\ Content\ (\%) = \frac{(a-b)}{a}x100\% \tag{1}$$

where:
a = initial carbon weights (g)
b = final carbon weight (g)

2.3.2. Ash Content. The calculation of ash content was performed according to Equation (2) where the weight of the dried activated carbon before and after heating at 600ºC for 4 h was noted.

$$Ash\ Content(\%) = \frac{a}{b}x100\% \tag{2}$$

where:
a = ash weight (g)
b = initial dry carbon weight (g)

2.3.3. Absorbability of Iodine. An amount of 2 g activated carbon together with 50 ml of 0.1 N iodine solvents was inserted into a dark and closed bottle, then shaken for 15 minutes. The solution was separated using a centrifuge and a 10 ml of filtrate that was then titrated with 0.1 N sodium thiosulfate solutions. If the yellow color of solvent is close to disappearing, an indicator of 1% amilum was added. The titration was continued until the right blue color disappeared. The capability of the bio-sorbent to adsorb Iodine is given by Equation 3.

$$Adsorbability \quad Iod \left(\frac{mg}{g} \right) = \frac{\left\{ 10 - \left(\frac{N \times V}{0,1} \right) \right\}}{S} \times 12,69 \times 5 \tag{3}$$

where:

V	= required sodium thiosulfate solvent
N	= normality of sodium thiosulfate solvent
S	= weight of activated karbon (g)
12.69	= the amount of Iodine needed for a 1 mL sodium thiosulphate 0.1 N

3. Results and Discussion

### 3.1. Characterization of activated carbon

The analysis results of the activated carbon characteristics are shown in Table 2. Table 2 shows the activated carbon characteristics that including water content of 6.38%, ash content of 1.05% and iodine absorption of 856.58 mg/g. From those activated carbon characteristics, it can be concluded that the prepared activated carbon meets the permitted quality standard as in accordance with the quality requirements of active carbon as outlined in SNI No. 06-3730-1995.

Table 2. Characteristics of activated carbon from coffee grounds.

Parameter	Result analysis	Quality requirement of active carbon (SNI No. 06-3730-1995)
water content	6.38	Maximum 15 %
ash content	1.05	Maximum 2,5 %
absorbability of iodine	856.58	Minimum 750 mg/g

3.2. Morphology of activated carbon

Morphological structure of coffee grounds before activation and after activation was analysed using Scanning Electron Microscope (SEM). The characterization results from SEM were shown in Figure 1.
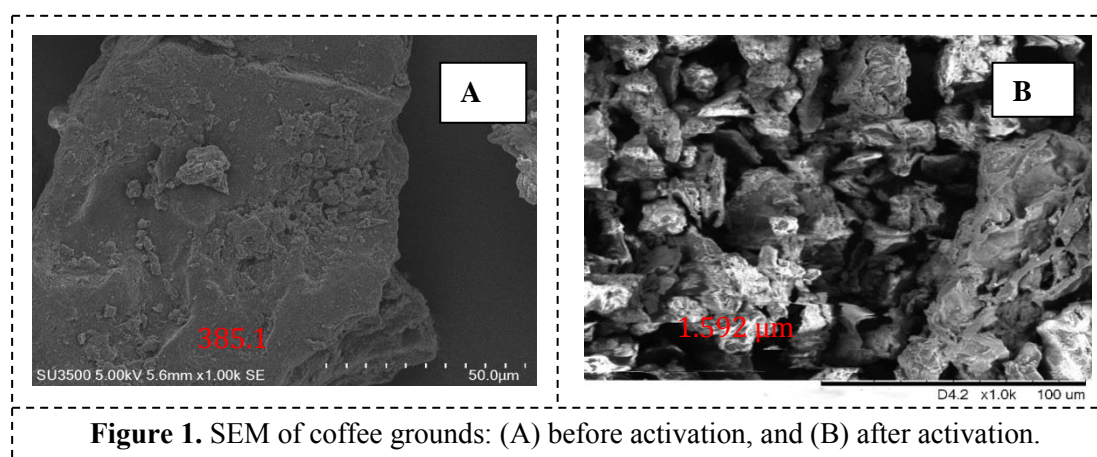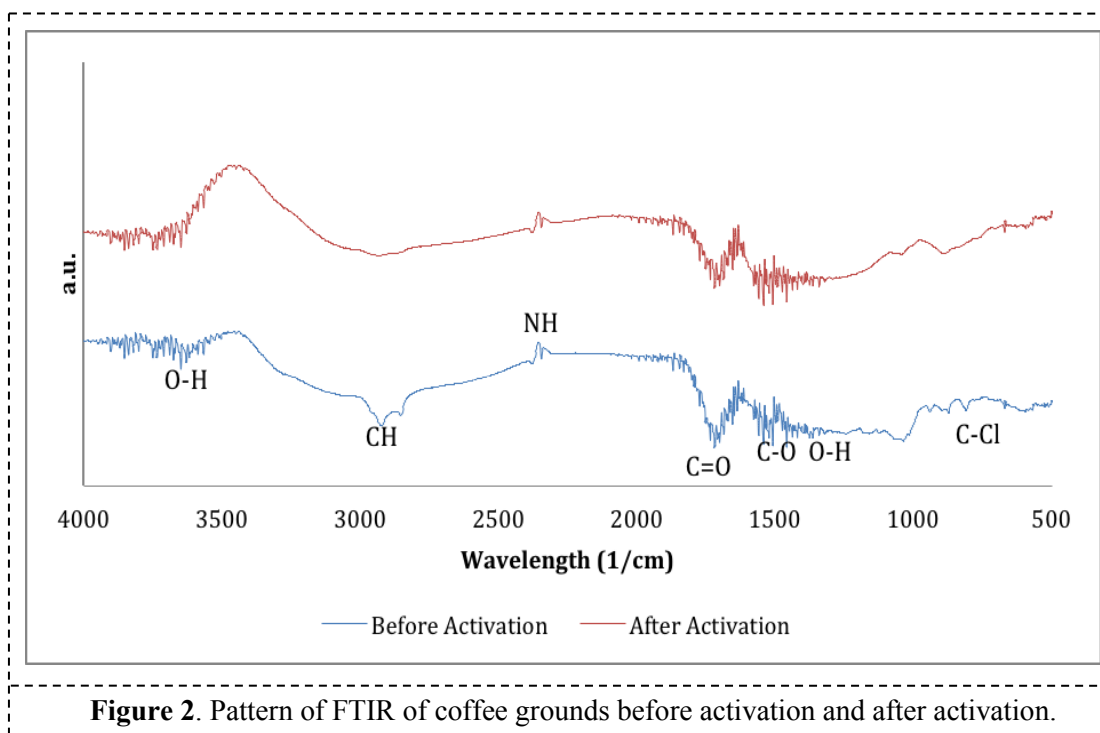

Figure 1. SEM of coffee grounds: (A) before activation, and (B) after activation.

Figure 1 (A) shows that the bio-sorbent coffee grounds that have not been activated yet have irregular pores with an average pore size of only 385.1 μm. The results of this measurement are also in accordance with the results of research that has been done by other researchers [9]. They stated that the adsorbents that have not yet been activated have irregular pores so that the cavity on the pore is still rough and loose. Figure 1 (B) shows that the sorbent that has been activated both physically and chemically has a pore size that is regular and porosity is arranged with the average pore size of 1.592

μm. The above results indicate that bio-sorbent activation can form the distribution of the pores over the entire surface of the adsorbent which can improve the performance of the sorbent.

3.3. Functional groups analysis of bio-sorbent
The functional groups of bio-sorbent of coffee grounds before and after activation were identified using fourier transformation infrared (FTIR) in the range of 400 to 4000 cm^{-1} as shown in Figure 2 and Table 2.

Figure 2. Pattern of FTIR of coffee grounds before activation and after activation.

Table 3. FTIR spectrum results of coffee grounds before activation and after activation.

Functional Cluster	Bonded	wavelenght / cm^{-1}	
		before activation	after activation
Alkanes (-CH-)	CH	2905-2875	2905
Carboxylic Acids, Free OH	O-H	3550-3500	3550
Charged Amines (C=NH$^+$)	NH$^+$	2500-2325	2400
α-amino acid hydrochloride	C=O	1755-1730	1755-1730
-COOH	C-O str plus O-H def	1440-1395 1320-1310	1440-1395
Polyclorinated	C-Cl	800-700	790

Table 3 shows the FTIR analysis of coffee grounds before activation and after activation. Table 3 shows that the spectra before activation and after activation of samples have different patterns. The occurrence of this pattern change is caused by the effect of activation which results in the presence of

O-H group which is hydroxyl group at peak of 1320 - 1310 cm^{-1}. The infrared spectra of the activation sorbent also show the presence of a specific absorbance band of C - O carbons at peak region around 1440 - 1395 cm^{-1}. This phenomenon proves that the bio-sorbent after activation has an R-COOH functional group of carboxylic acids group which can improve the performance of the bio-sorbent when used in the adsorption process.

*3.4. X-Rays diffraction (XRD) and X-Ray Fluorosence (XRF) analysis*

Figure 3 shows the XRD patterns of both prepared bio-sorbent samples before and after activation. A very significant difference can be seen in Table 3 (A) and (B) where the Cl_2 compound increases when the sample of coffee ground has been carbonized and activated. Figure 3 shows a graph of the analysis results where a very significant difference can be seen in Table 2 (A) and (B). SO_3 compound increases when the sample of coffee ground has been carbonized. It could be happen due to the evaporation of the water content at the time of physical activation, where the space that was occupied by the free and bonded water molecules becomes empty so that the sorbent adsorption capacity increases [11].

The activation process with HCl will decrease the levels of Ca, K and Na that are impurities which bind to the surface of the adsorbent that act as the cation exchange material. With the decreasing levels of Ca, K and Na inside the bio-sorbent there will be a lot of free space on the surface of the bio-sorbent so that the ability of the bio-sorbent to adsorb would also increases.

Performance of the bio-sorbent after activation would increase compared to without the activation process. This is because the bio-sorbent that has been treated by the activation process is generally dominated by Cl_2 about 33.2% of which is amorphous and porous solid and has an inert, neutral, and large surface area so it has large adsorption capacity properties. In addition, the activated bio-sorbents are also dominated by SO_3 of about 32% of which alumina can adsorb cations and anions with a cation adsorption sequence by alumina [10].

Table 4. Data of XRF.

Compounds	Content (%)	
	Before Activation (A)	After Activation (B)
SO_3	27.7	32
$PbCl_2$	7.5	7.9
Cl_2	20.6	33.2
KNa_2	18.9	13.9
N_2	9.4	9.5
Ca	16	3.4
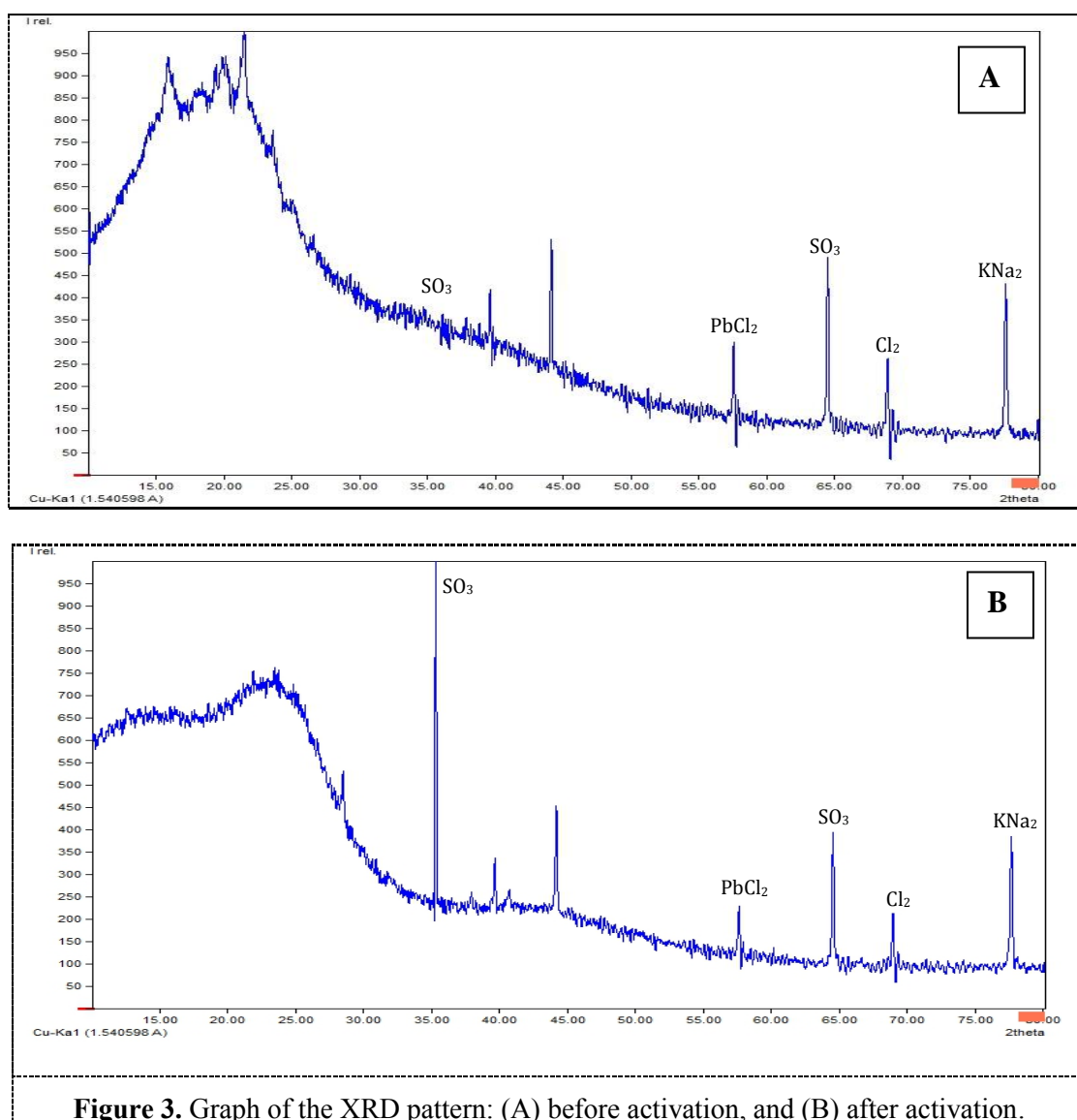
Figure 3. Graph of the XRD pattern: (A) before activation, and (B) after activation.

4. Conclusions

The characteristics results of activated carbon prepared from coffee grounds shows that the activated carbon meets the permitted quality standard according to SNI 06-3730-1995. SEM analysis shows that the pore size of bio-adsorbent of coffee grounds before activation is only about 385.1 μm, while after activation it increases to a pore size of 1.592 μm. FTIR analysis shows the appearance and disappearance of functional groups on bio-sorbent from coffee grounds after activation. The appearance of O-H bond at peaks of 3550 – 3500 cm^{-1} indicated that the surface of the bio-sorbent of activated coffee grounds allows to bind ions. X-Ray Diffraction (XRD) analysis show that Cl_2 and SO_3 intensity increased after activation process that would also increase adsorption capacity of activated bio-sorbent. The impurities contained in the coffee grounds can be minimized through the activation process and carbonization process.

References

[1]   Sembiring M T and Sinaga T S 2003 *Arang Aktif*
[2]   Lubis S and Nasution R 2002 *Jurnal Natural* **2** 2 12
[3]   Adhitiyawarman A I 2015 *Jurnal Kimia Khatulistiwa* **4** 2

[4]   Li S, Han K, Li J, Li M and Lu C 2017 *Microporous and Mesoporous Materials* **243** 291-300.
[5]   Murthy K N, D'Sa A and Kapur G 2004 *Arabica*, **167** 121-050.
[6]   Rizki A P 2015 *Paper presented at the Seminar Nasional Teknik Kimia Kejuangan*
[7]   Irmanto I and Suyata S 2010 *Molekul* **5** 1 22
[8]   Alfiany H Bahri S and Nurakhirawati N 2013 *Journal of Natural Science FMIPA* **2** 3
[9]   Wang Man 2017 *Ecotoxicology and Environmental Safety* **139** 36
[10]   Utari Tresye 1994 *Pembuatan Adsorben Alumina Dari Kaolin*. Diss. **27164**
[11]   Sari, Wulan T I, Muhsin M and Wijayanti H 2016 *Konversi* **5.2** 20-25