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REVIEW

Adsorption characteristics of activated carbon prepared from spent ground coffee

Pasakorn Jutakridsada[1] · Chonticha Prajaksud[1] · Luksamone Kuboonya-Aruk[1] ·
Somnuk Theerakulpisut[2] · Khanita Kamwilaisak[1]

Abstract This paper reports results of a research project which attempts to produce low-cost activated carbon from agro-residue wastes. The ground coffee residue which is a by-product of coffee making was collected from coffee shops, prepared, and converted to activated carbon by a chemical activation method. The objective of this work is to investigate the effects of preparation conditions on properties of the activated carbon obtained. The preparation condition is defined by pyrolysis rate, concentration of $ZnCl_2$, impregnation time, and carbonization temperature. The pyrolysis rate was fixed at 10 °C min^{-1} for 4 h with three concentrations of $ZnCl_2$ (5, 10, and 15 wt%), three durations of impregnation time (8, 12, and 24 h), and three carbonization temperatures (400, 450, and 500 °C). The morphology and specific surface area were, respectively, determined using SEM and BET techniques. It was found in this study that the activated carbon with the best properties was obtained at the preparation condition given by 15 wt% of $ZnCl_2$, impregnation time of 24 h, and 500 °C carbonization temperature. On average, the activated carbon had a pore diameter of 0.61 nm, specific surface area of 831 m^2 g^{-1}, and a total pore volume of 0.44 cm^3 g^{-1}. It was also found that the adsorption isotherm of Cu (II) fitted well with Freundlich isotherm.

✉ Khanita Kamwilaisak
  khanita@kku.ac.th

[1] Department of Chemical Engineering, Faculty of
  Engineering, Khon Kaen University, Khon Kaen 40002,
  Thailand

[2] Department of Mechanical Engineering, Faculty of
  Engineering, Khon Kaen University, Khon Kaen 40002,
  Thailand

Introduction

Food waste has widely been used as a raw material for many applications such as bioethanol (Boochapun et al. 2014; Satarn and Kamwilaisak 2014) and adsorption agent (Kante et al. 2012; Gueye et al. 2014; Han et al. 2015; Jain et al. 2015). Recently, in Thailand coffee consumption increases annually. The increase of coffee consumption results in high quantity of spent ground coffee that needs to be adequately managed. This residue is usually collected with other organic wastes and disposed of by landfill where they decompose to produce methane and thus contribute to climatic change. The spent ground coffee is composed of high organic contents such as carbohydrate, protein, fiber, caffeine, polyphenols, tannins, and pectins (Pujol et al. 2013) that can be treated or valorized in a variety of ways, thus reducing its environmental impact (Caetano et al. 2014). The coffee residue was suitable as an animal feed, a soil conditioner, and an organic fertilizer (Braham 1979). Moreover, the coffee residue could be a renewable energy source such as biodiesel (Caetano et al. 2014) and bioethanol (Gurram 2015). The major compound of residual coffee is carbon element that can be used in adsorption process as an activated carbon. Activated carbon has successfully been used in many fields, including waste water, pharmaceutical waste, and odor treatment, gas separation (Prahas et al. 2008), electrode materials (Wang et al. 2013), and hydrogen storage (Zhu et al. 2014). Furthermore, the spent activated carbon can be regenerated by washing with organic solvents. A life-cycle of activated carbon in waste treatment depends on kinds and loading of pollutants. The

spent activated carbon could finally be used as a fertilizer. However, carbonization as a stand-alone process is not sufficient for efficient preparation of biomass as an adsorbent. Many researcher employed physical and chemical activation methods for improving activated carbon properties such as total pore volume, mesoporous volume, and specific surface area. The physical activation method uses steam to make carbonaceous materials porous. This method can create small pores but it requires high-pressure steam (Sun and Jiang 2010; Kopac and Toprak 2007). The chemical method uses chemical agents such as KOH, K_2CO_3, NaOH, $ZnCl_2$, H_3PO_4, and H_2SO_4 (Kiliç et al. 2012) to effect the activated surface, but it needs a washing process that generally results in low yield (Yakout and Sharaf El-Deen 2011). In particular, $ZnCl_2$ is a better activator giving higher yield of the activated carbon with greater capacity to encourage gas adsorption and liquid removal (Meng and Park 2014). The physical method was found to be more suitable for preparing activated carbon for odor reduction, while the chemical process could be for decolorization of wastewater treatment (Kamwilaisak 2013).

In an attempt to reduce this solid waste of spent ground coffee by producing useful material from it, this work aims to synthesize activated carbon from the residual ground coffee. The preparation parameters such as impregnation ratios, impregnation time, and carbonization temperature were investigated to determine the preparation condition which yields activated carbon with the best properties. Furthermore, adsorption capacity of Cu(II) ions, characterization of the obtained activated carbon, and adsorption isotherms were investigated.

Materials and methods

Materials and activated carbon preparation

Spent ground coffee was collected from coffee shops around Khon Kaen University, Khon Kaen, Thailand. It was washed with distilled water to remove water-soluble impurities, dried at 100 ± 3 °C for 2 h, and kept as a raw material in a desiccator. The raw material was then prepared by following the procedure recommended by Boonamnuayvitaya et al. (2004, 2005). Twenty-grams of the raw material was impregnated in 60 ml of $ZnCl_2$ with various concentrations (5, 10, 15 wt%) at ambient temperature for 8, 12, and 24 h. After that, the impregnated material was dried at 100 ± 3 °C overnight. Then, the impregnated samples were carbonized in a furnace with atmospheric air at 400, 450, and 500 °C. The heating rate was 10 °C/min from room temperature to the predetermined temperature. The carbonized samples were soaked

with 5 wt% of HCl for 2 h and then neutralized with distilled water. Afterwards, the samples were dried at 100 ± 3 °C until completely dried and then sieved with 60–80 mesh. All chemical reagents were supplied from Sigma (Singapore) in AR grade.

Physical characterization

The specific surface area and pore volume of the samples were determined by Nitrogen adsorption–desorption isotherms measured by gas sorption analyzer (micromeritics ASAP 2010) (Madani et al. 2016). Prior to the adsorption measurement, the samples were first outgassed at 110 °C under vacuum (<50 mmHg) for 12 h. The nitrogen adsorption–desorption isotherms were recorded at liquid nitrogen temperature (77 K). The specific surface area, S_{BET}, was calculated using the Brunauer–Emmet–Teller (BET) method. The micropore volume, V_{mic}, was calculated using Dubinin–Radushkevich (DR) equation. The total pore volume, V_T, was found from the amount of N_2 adsorbed at the relative pressure of 0.99. The mesopore and macropore volume, $V_{meso+mac}$, was obtained by subtraction of the micropore volume from the total pore volume. The average pore diameter, D_p, was calculated from $(4 \times V_T)/S_{BET}$. For comparison of untreated and treated residues, the surfaces of the materials were examined by scanning electron microscopy (SEM, HITACHI S-3000 N).

Thermogravimetric (TGA) analysis

In order to determine the carbonization temperature, the washed coffee grounds were characterized by TGA analysis (TGA 50, Shimadzu, Japan). A 200 mg sample of the material was then heated in a temperature range of 30–750 °C with a heating rate of 10 °C min^{-1} under a nitrogen flow.

Batch sorption testing experiment

Sorption tests were conducted in a batch reactor (500 mL) with a magnetic stirrer running at 200 rpm. The reaction mixture contained 2.5 g/L of activated carbon with various $CuSO_4$ concentrations (25–100 ppm) in a total volume of 200 mL. The sample was kept in the reactor for different time intervals, i.e., 0, 5, 10, 15, 30, and 60 min (Boonamnuayvitaya et al. 2004, 2005). The concentration of $CuSO_4$ solution was measured by an atomic absorption spectrophotometer (AAS) (PerkinElmer Analyst 100, USA) (air–acetylene flame, 15 mA/324.8 nm). The amount of heavy metal adsorption per unit weight of activated carbon at time t, q_t (mg/g), was calculated by

$$q_t = \frac{(C_o - C_t)V}{W},\tag{1}$$

where C_o and C_t are the metal concentrations (mg/L) initially and at time t, respectively, V is the volume of the metal solution, and W is the weight of AC.

Adsorption isotherms and modeling

The adsorption capacity of an adsorbent is an important information which can be provided by an equilibrium study. The adsorption isotherm is characterized by constant values which express the surface properties and affinity of the adsorbent. This type of adsorption isotherm is generally fitted to the Langmuir, Freundlich, and Elovich models. The Langmuir model is valid for monolayer adsorption onto a surface with a finite number of identical sites which are homogeneously distributed over the adsorbent surface. It is expressed as follows:

$$q_e = \frac{q_m b C_e}{1 + b C_e}, \tag{2}$$

where q_m and b are Langmuir parameters related to the maximum adsorption capacity and bonding energy of adsorption, respectively.

Equation (2) can be linearized as

$$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{b q_m}. \tag{3}$$

The Freundlich model is based on adsorption onto a heterogeneous surface suggesting that binding sites are not equivalent. It is characterized by the following equation:

$$q_e = K_F C_e^{\frac{1}{n}}, \tag{4}$$

where K_F and n are Freundlich parameters indicating the adsorption capacity and adsorption intensity, respectively. They were determined by plotting q_e versus C_e, using the following equation:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e. \tag{5}$$

The Elovich model is based on an increase of the adsorption sites exponentially with the adsorption process indicating multilayer adsorption. The model is mathematically represented by

$$\ln\left(\frac{q_e}{C_e}\right) = \ln(K_E q_m) - \left(\frac{q_e}{q_m}\right), \tag{6}$$

where K_E is the Elovich equilibrium constant (L mg^{-1}) and q_m is the Elovich maximum adsorption capacity (mg g^{-1}).

Results and discussion

Thermogravimetric analysis (TGA)

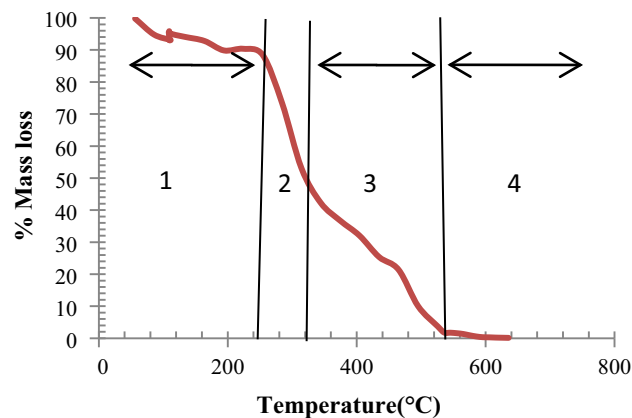Figure 1 shows the TGA curve which displays the weight loss of coffee grounds during the pyrolysis process at the

Fig. 1 Mass losses (%) against temperature (°C) under nitrogen condition for coffee grounds

heating rate of 10 °C min^{-1} under nitrogen condition. The curve explains the pyrolysis mechanism of these coffee grounds in 4 stages. The first stage took place in the temperature range of 30 to around 250 °C, during which the weight loss was caused by dehydration of the materials. The second stage was at about 250–320 °C. During this stage, initiation of carbonization was observed and the mass loss at the end of the stage was approximately 55 %. The mass loss was mainly volatile matter and tars. The third stage was observed to take place in the range of 320–530 °C, during which the mass loss was from carbonization of the coffee grounds. Finally, at the temperature above 530 °C, the coffee grounds underwent complete carbonization with the ash content of about 2.5 %. Similarly, Reffas et al. (2010) has reported the TGA of coffee grounds in the range of 25–550 °C with four stages of pyrolysis mechanism and similar temperature ranges in each stage.

SEM analysis

The SEM analysis (magnification 200x at 10 kv) of untreated and treated coffee grounds is shown in Fig. 2a, b, respectively. The treated coffee grounds were activated carbon which was prepared at the condition: 15 wt% of ZnCl$_2$, impregnation time of 24 h., and carbonization temperature of 500 °C (called AC15-24-500). From these figures, improvements in the number and sizes of porous sites can easily be observed.

Effects of preparing conditions on properties of activated carbon

Figure 3 shows the N$_2$ adsorption–desorption isotherms of three samples of activated carbon at different ZnCl$_2$ concentrations (5, 10, and 15 wt%), impregnation time 24 h,
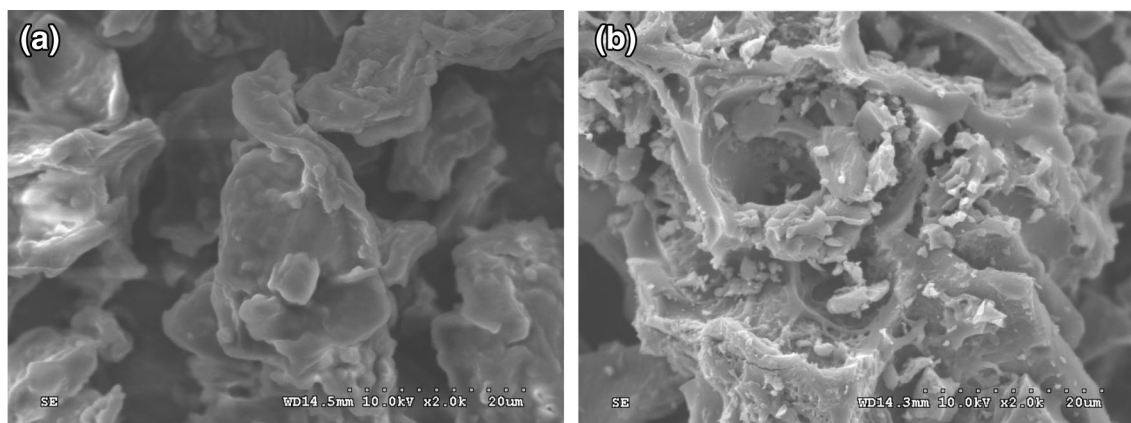
Fig. 2 SEM photograph of **a** untreated coffee ground and **b** treated coffee ground (activated carbon) AC15-24-500
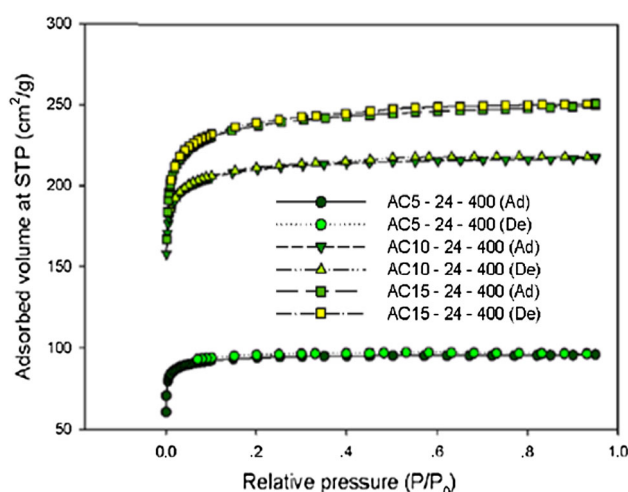

Fig. 3 Adsorption/desorption isotherms of N_2 at 77 K on $ZnCl_2$—activated carbons derived from coffee residue under different $ZnCl_2$ concentrations
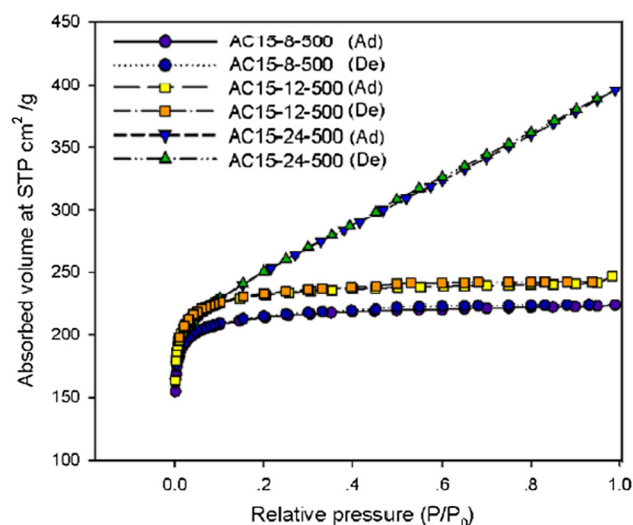

Fig. 4 Adsorption/desorption isotherms of N_2 at 77 K on $ZnCl_2$—activated carbons derived from coffee residue under different impregnation times

and carbonization at 400 °C (called AC5-24-400, AC10-24-400, and AC15-24-400). All the samples are the typical type I isotherm based on IUPAC (International Union of Pure and Applied Chemistry) classification, which corresponds to unimolecular adsorption (Rouquerol 1999). All the isotherms rise sharply at the beginning of adsorption due to the micropore filling effect. After that, adsorption of N_2 becomes constant. This result indicates the microporosity of all the three samples of activated carbon.

The adsorption/desorption isotherms of N_2 at 77 K on $ZnCl_2$-activated carbons prepared from coffee grounds at 15 wt% $ZnCl_2$ concentration and 500 °C carbonization temperature with various impregnation times (8, 12, and 24 h) (called AC15-8-500, AC15-12-500, and AC15-24-500) are shown in Fig. 4. The samples of AC15-8-500 and AC15-12-500 present type I isotherm, while AC15-24-500

is more complex with type II isotherm. All the isotherms increase sharply at the beginning of the adsorption. After that, the samples AC15-8-500 and AC15-12-500 reach a plateau which indicates the microporosity, whereas the isotherm of AC15-24-500 further rises sharply because of the multilayer adsorption of the BET type. Furthermore, Fig. 5 shows the adsorption/desorption isotherms of N_2 at 77 K on $ZnCl_2$-AC derived from coffee grounds at 24 h of impregnation time and 15 wt% $ZnCl_2$ concentration with various carbonization temperatures (400, 450, and 500 °C) (called AC15-24-400, AC15-24-450, and AC15-24-500). Type I isotherm is observed for the samples AC15-24-400 and AC15-24-450, but the AC15-24-500 displays type II isotherm.

It can be seen that the increase of $ZnCl_2$ concentrations (5–15 wt%) hardly affects the adsorption isotherm, while
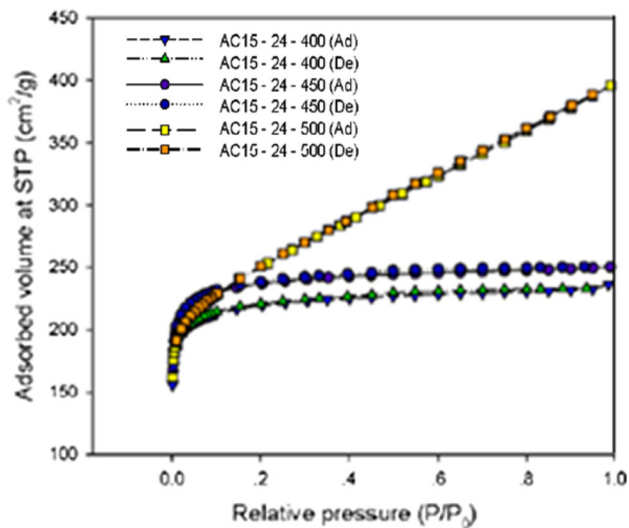
Fig. 5 Adsorption/desorption isotherms of N_2 at 77 K on $ZnCl_2$—activated carbons derived from coffee residue under various carbonization temperatures

the isotherm changes when impregnation time and carbonization temperature increases to 24 h and 500 °C, respectively. During activation or carbonization, polymeric structures in coffee grounds decompose and liberate most of the non-carbon elements, mainly hydrogen, oxygen, and nitrogen in the form of liquid and gases, leaving behind a rigid carbon skeleton in the form of aromatic sheets and strips (Yakout and Sharaf El-Deen 2011). This highly concentrated $ZnCl_2$ solution accesses the pores of the raw precursor, and two phenomena may occur. Firstly, $ZnCl_2$ is a salt and then water inside cells is released to the $ZnCl_2$ solution because of the osmotic pressure. Those cells were dehydrated which tend to increase the porosity of the raw

precursors. Secondly, water in the pores of the raw precursor (could be physisorbed water) is mixed with the $ZnCl_2$ solution inside the pores. Those phenomena may occur simultaneously and improve the contact between $ZnCl_2$ and a raw material (Cruz et al. 2015).

Thus, at a higher concentration of $ZnCl_2$ and carbonization temperature, the chemical reaction is stronger on the surface of coffee grounds resulting in greater microporosity on the surface of coffee grounds. Furthermore, the microporosity of an activated carbon can be collapsed to become mesoporous and macroporous because of the high carbonization temperature. However, both types I and II are desirable because they exhibit strong adsorption. The values of specific surface area, mesopore volume, total pore volume, and average pore diameter of all activated carbons are shown in Table 1. The largest specific surface area was obtained at 831 m^2 g^{-1} for AC15-24-500. The value of the specific surface area in this study was in the range of commercial activated carbon (400–1200 m^2 g^{-1}) (Enley et al. 2011) which can be used to treat many compounds in waste water and water treatment such as reactive dye, heavy metal, phenol, formaldehyde, and toluene (Yuvarat Ngernyen and Kamwilaisak 2013).

Furthermore, the activated carbon prepared from our work may be used for CO_2 removal due to its capability to retain gases with small molecules such as CO_2. Similar research work has been reported by Rashidi et al. (2014). However, the yield of AC in our research was low. This could be due to the effect of thermal decomposition at high carbonization temperature (Srenscek-Nazzal et al. 2013). In the future, the preparation process should be improved for higher productivity with the consideration of climate change, loss of biodiversity, and stratospheric ozone depletion (Sikdar 2007).

Table 1 Porous properties of activated carbon under impregnation ratios, carbonization temperatures, and different impregnation times

Activated carbon	Yield (%)	Specific surface area (m^2 g^{-1})	Mesoporous volume (cm^3 g^{-1})	Total pore volume (cm^3 g^{-1})	Pore diameter (nm)
$ZnCl_2$ concentration					
AC5-24-400	15.99	308	0.13	0.02	0.15
AC10-24-400	21.24	680	0.28	0.06	0.34
AC15-24-400	22.95	711	0.30	0.08	0.38
Carbonization temperature					
AC15-24-400	22.95	711	0.30	0.08	0.38
AC15-24-450	15.53	768	0.30	0.12	0.42
AC15-24-500	6.22	831	0.17	0.44	0.61
Impregnation time					
AC15-8-500	7.21	694	0.27	0.07	0.34
AC15-12-500	9.50	751	0.29	0.09	0.38
AC15-24-500	6.22	831	0.17	0.44	0.61

Absorption test

In this study, the adsorption capacity of an activated carbon was investigated by copper aqueous solution. The copper absorption of AC15-24-500 is shown in Fig. 6. The percentage of Cu(II) adsorption sharply increased at the beginning of adsorption and then became constant after 30 min. The maximum adsorption obtained was approximately 18 % with 100 ppm copper aqueous solution which is considered low as compared with the results of others (Boudrahem et al. 2011). This could be the effect of the increase in mass transfer driving force due to the increase of the initial concentration. However, this work used $ZnCl_2$ as the chemical activator yielding Zn^{2+} which has the same charge as copper. Consequently, a low efficiency of copper removal was obtained.

Adsorption isotherms

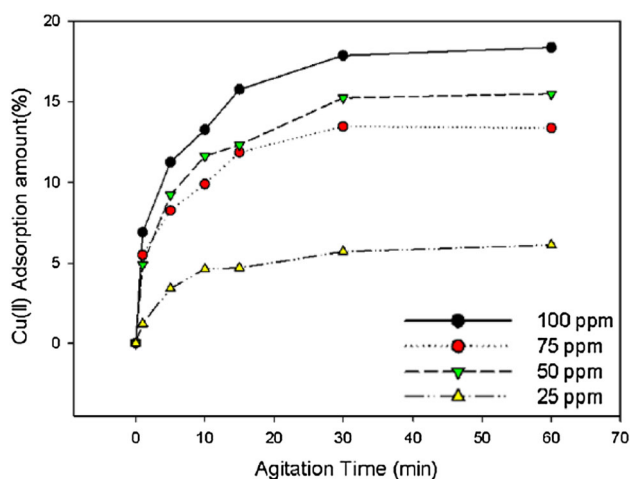The adsorption isotherm of Cu onto AC15-24-500 was characterized by using well-known models. The Langmuir, Freundlich, and Elovich models were used to explain the surface properties and affinity of the adsorbent with regression analysis. The sorption data of Cu(II) onto AC15-24-500 were plotted with Langmuir, Freundlich, and Elovich isotherms in Fig. 7 a, b, c, respectively. The data were fitted with linearized expressions for all the isotherms. Table 2 presents all values of Langmuir, Freundlich, and Elovich models. The Freundlich adsorption model was found to have the highest R^2 value of 0.9895 and hence the best fit. The adsorption capacity (KF) and the adsorption intensity (n) of this system were 38.25 and 1.81, respectively. Furthermore, the $1/n$ value of Freundlich model is a function of the strength of adsorption in the adsorption process. This value is below one which indicates a normal adsorption.

Conclusion

Activated carbon from spent ground coffee has successfully been prepared. It was found that the activated carbon was best produced when 15 wt% of $ZnCl_2$ was used with an impregnation time of 24 h and a carbonization temperature of 500 °C. This preparation condition yielded the average specific surface area of 831 $m^2 g^{-1}$ and average

Fig. 6 Percentage of Cu(II) removal against agitation time (min)

Table 2 Parameters of Langmuir, Freundlich, and Elovich isotherms for the adsorption of Cu(II) onto prepared AC

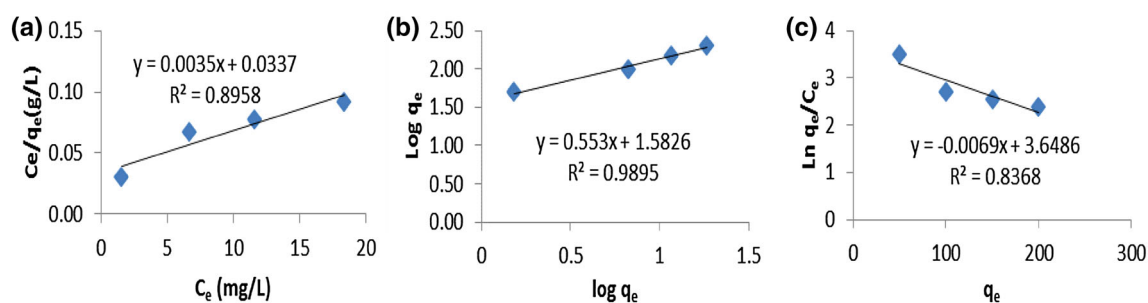| Isotherm	Parameters	Value
Langmuir	b (L mg^{-1})	0.104
	q_m (mg g^{-1})	285.71
	R^2	0.896
Freundlich	K_F	38.25
	n	1.81
	R^2	0.9895
Elovich	K_F (L mg^{-1})	0.27
	q_m (mg g^{-1})	144.93
	R^2	0.8368

Fig. 7 Adsorption isotherms of Cu(II) onto prepared AC: a Langmuir, b Freundlich, and c Elovich isotherms

pore volume of 0.44 cm^3 g^{-1}. The adsorption capacity of this carbon with Cu(II) was ca 18 % removal with 100 ppm of CuSO$_4$. It was also discovered that the adsorption isotherm of Cu onto AC15-24-500 fitted linearly very well with the Freundlich model. The value of specific surface area of the obtained activated carbon was in the commercial range (400–1200 m^2 g^{-1}) which could be used to treat many compounds in waste water and water treatment.

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