Microwave assisted ZnCl$_2$ activation of salacca peel derived activated carbons as adsorbents for Cu(II) removal from aqueous solution

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Abstract. In this present study, salacca peel based activated carbons (SPAC) were prepared by microwave assisted ZnCl$_2$ activation method. The effects of microwave power, activation time and impregnation ratio on the characteristics of activated carbon were studied. The activated carbons were characterized by N$_2$ adsorption-desorption and scanning electron microscopy (SEM) instruments. The BET surface area of 1796 m$^2$/g were obtained at a microwave power of 540 W, activation time of 25 minutes with an impregnation ratio (ZnCl$_2$:salacca peel) of 4:1. The resulting activated carbon was used for removal of Cu(II) from aqueous solution. The prepared activated carbons were then used as adsorbents for removing Cu(II) metal ions from aqueous solutions. The adsorption equilibrium was investigated using using Langmuir, and Freundlich model equations. It was found that the adsorption equilibrium data followed the Langmuir isotherm equations with maximum capacity of 1262.62 mg Cu(II)/g SPAC at room temperature. The adsorption kinetics were also studied using the pseudo first order, pseudo second order and intraparticle diffusion models. The adsorption kinetics was shown to fit well with the pseudo second-order kinetic model.

1. Introduction

The mining, farming, metal plating and manufacturing industries usually produced waste water containing heavy metal ions such Cu (II) [1]. Copper is classified as one of toxic metal according to the World Health Organization (WHO) [2]. This metal is very hazardous, carcinogenic and mutagenic substance and furthermore it can accumulate in living organism including humans [3, 4]. The maximum amount of Cu(II) allowed in the surface water is about 1.3 mg/L according to the Environmental Protection Agency (EPA). If exceeding this limit, various diseases especially related to respiratory and digestion system can occur Cu(II) [5]. There are many types of treatment methods for the Cu(II) removal from industrial wastewaters such as electrocoagulation [6], ion exchange [7], chemical precipitation [8], oxidation – reduction [9], membranes [10] adsorption [11]. Adsorption can be one of the alternative method among others [11]. In the industry, adsorption has become a common unit operation used to remove heavy metal [12]. Therefore, it is very important to synthesize an effective and efficient adsorbent which can be prepared from abundant, low-cost and renewable resources, such as biomass [13].

It is well known that activated carbons are excellent materials for removal of toxic substances from wastewaters [14]. However, the drawbacks main for their application are on their high production cost, adsorbent regeneration after use and environmental problems. Hence related to those facts, the
utilization of biomass wastes as carbon precursors for the synthesis of activated carbons has become a hot topic in research in the area of adsorbent material technology. Recently, many types of biomass wastes have been used for the synthesis of activated carbons which have been found to be potential and low cost precursors as adsorbents and would solve the disposal problem of biomass waste ([15–18]).

Even though biomass has great potencies for activated carbons production, the processing steps are critical [19]. The activation process is the most important part in the production of activated carbon. This process opens or increases porosity on the surface of carbons. In general, activated carbon can be prepared by one of the following two methods of activation which are by physical or chemical activation ([20, 21]). Both of them however, consumed much energy and took more than 6 hours to complete [22].

Microwave (MW) heating process is one alternative method that can produce equally high quality activated carbon with less energy and shorter time [23]. In the MW device, the microwaves supply energy directly to the heated samples. Energy transfer is not by conduction or convection as in conventional heating, but microwave energy is readily transformed into heat inside the particles by dipole rotation and ionic conduction [24]. Microwave heating has been utilized to prepare activated carbon from many types of biomass wastes ([25–31]).

Salacca fruit or also known as snake fruit reddish-brown scaly skin is a typical tropical fruit cultivated in Indonesia especially in Bali, Lombok, Maluku and Sulawesi [32]. The fruit inside consists of three lobes with the two larger ones, or even all three, containing a large non-edible seed [33]. After consumption of the fruit, salacca peel has usually discarded as waste and until now, there has not been utilized as raw materials for useful products [34]. In order to make better use of this biomass waste, it is proposed to convert salacca peel to activated carbons. Our previous works focused on the application of SPAC as adsorbent material for removal of methylene blue [35].

The aim of this work was to investigate the potential of SPAC prepared by microwave assisted ZnCl₂ activation method as adsorbent for the removal of Cu(II) from synthetic aqueous solutions. The effects of initial pH solution, initial Cu(II) concentration and SPAC dosage on the adsorption performance of SPAC were studied in further. The adsorption kinetics was analyzed using pseudo-first-order and pseudo-second-order models. Equilibrium data were modeled using two different equations such as Langmuir and Freundlich model.

2. Experimental

2.1. Preparation of SPAC

The salacca peel was collected from the local market in Bandung Indonesia and then washed with distilled water, and dried in an oven at 110°C for 24 h to remove the moisture content. The dried salacca peel were crushed and sieved to a uniform particle size of 100-200 meshes.

The microwave-assisted process was performed using a modified microwave oven (Electrolux). Mixtures of KOH and salacca peel powder with varying impregnation ratio of 2:1 and 4:1 were put in a quartz tube reactor placed in a microwave oven. The activation processes were then subjected to various microwave heating times (5, 15, 20,25 and 30 minutes) and microwave radiation powers (230,380, 540 and 700 W) under nitrogen flow. The resulting material was washed repeatedly with hot distilled water until solution pH reached 7.0. The obtained SPAC were dried at 80°C for 24 h and then stored for further analyses. Some text.

2.2. Structural and textural characterization of SPAC

Characterization in terms of specific surface area, pore volume, and pore diameter of the obtained activated carbons was determined by N₂ adsorption at with surface area and pore size analyzer using the Brunauer-Emmet-Teller (BET) method. Scanning electron microscopy (SEM) was used for observing the morphology of SPAC.
2.3. Adsorption of Cu(II) using SPAC

To study the effect of the initial metal ion concentration, 500 mL of Cu(II) metal ions at various concentrations (100-300 ppm) were mixed with 30 mg of SPAC in the Erlenmeyer flasks at room temperature. These mixtures were kept at room temperature with continuous shaking at 100 rpm. All samples were filtered and analyzed by UV-Vis spectrophotometer. To investigate the effects of initial pH solution (2-8), the pH was adjusted by 1 mol/L HCl and NH₄OH.

For all the adsorption experiments, they are conducted triplicate and the average values were used for the analysis. The adsorption performances were measured through the removal efficiency and adsorption capacity which can be calculated using the following equation (1) and equation (2):

\[ \%R = \frac{C_0 - C_e}{C_e} \times 100\% \]  
\[ q_t = \frac{C_0 - C_t}{m} \times V \]  

Where \( C_0 \) and \( C_t \) (mg/L) are the initial concentration and concentration at time \( t \) of Cu(II), respectively. \( V \) (L) is the total solution volume, and \( m \) (g) is the mass SPAC-adsorbent.

The study of adsorption kinetics was conducted at various initial Cu(II) concentrations of 10-50 ppm and 5 at room temperature. About 10 mg SPAC was put into Erlenmeyer flasks with 250 ml MB solution; the samples were separated by filtering at different contacting time. The residual concentrations of Cu(II) were measured, and adsorption capacity and removal efficiency of SPAC were calculated by equation (1) and (2).

The experimental data were then analyzed using the pseudo-first-order [36] and second-order kinetic models [37]. The equation of the pseudo first order model is given by equation (3):

\[ q_t = q_e (1 - e^{-k_1 t}) \]  

The form of the pseudo-second-order kinetic model is given by equation (4):

\[ q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \]  

Where \( k_2 \) is the pseudo-second-order rate constant (g/mg min).

The capacity of an adsorbent can be represented by its equilibrium adsorption isotherm. The Langmuir and Freundlich adsorption models are commonly used to study the adsorption behavior of materials and the correlation among adsorption parameters.

The Langmuir isotherm equation is represented by the following equation (5):

\[ q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \]  

Where \( C_e \) is the equilibrium concentration of (ppm), \( q_e \) is the amount of Cu(II) adsorbed (mg/g), \( q_m \) is the maximum adsorption capacity of Cu(II) (mg/g), and \( K_L \) is the Langmuir adsorption equilibrium constant (L/mg) related to the affinity of the binding sites.

The Freundlich isotherm equation is described by the following equation (6):

\[ q_e = K_F C_e^{1/n} \]  

Where \( K_F \) and \( n \) are the Freundlich adsorption isotherm constants, which are indicators of adsorption capacity and adsorption intensity, respectively.
3. Results and Discussion

3.1. Characterization of SPAC
Figure 1 shows the N₂ adsorption desorption profiles of SPAC prepared by Microwave assisted ZnCl₂ activation method. It can be seen that the profiles follows the Type IV according to the International Union of Pure and Applied Chemistry (IUPAC) classification of adsorption isotherms [38]. This is a typical structure of mesoporous material and it is beneficial for adsorption purposes [39].

![Figure 1. Isothermal N2 Adsorption-Desorption of SPAC prepared by microwave assisted ZnCl2 Activation Method.](image)

Table 1 presented the BET surface area of SPAC prepared by microwave heating method at various impregnation ratio, activation time and microwave power. It can be seen that at the impregnation ratio of 1:2, the highest BET surface area was achieved at microwave power of 700 W and activation time of 20 minutes. While for the carbon samples obtained at impregnation ratio of 1:4 and microwave power of 700 W, their BET surface area increased as the activation time became longer from 5 to 25 minutes. Finally the highest BET surface area of about 1796 m²/g was obtained for SPAC prepared at impregnation ratio of 1:4, activation time of 25 minutes and microwave power at 540 W. For the adsorption of Cu(II) metal ions from aqueous phase, this carbon sample was then used as adsorbents.

| Sample | Impregnation Ratio | Activation Time (Minutes) | Microwave Power (W) | BET Surface Area (m²/g) |
|--------|--------------------|--------------------------|---------------------|------------------------|
| 1      | 1:4                | 5                        | 700                 | 1407                   |
| 2      | 1:4                | 15                       | 700                 | 1439                   |
| 3      | 1:4                | 20                       | 700                 | 1432                   |
| 4      | 1:4                | 25                       | 700                 | 1481                   |
| 5      | 1:4                | 25                       | 540                 | 1796                   |
| 6      | 1:4                | 25                       | 380                 | 1006                   |
| 7      | 1:4                | 25                       | 230                 | 260                    |
| 8      | 1:2                | 5                        | 700                 | 518                    |
| 9      | 1:2                | 15                       | 700                 | 717                    |
| 10     | 1:2                | 20                       | 700                 | 1385                   |
| 11     | 1:2                | 25                       | 700                 | 327                    |
3.2. Adsorption of Cu(II) from aqueous solution by SPAC

Figure 2 shows the effect of initial pH of Cu(II) solution on the adsorption performance of SPAC in terms of removal efficiency. The adsorption experiment was conducted at room temperature and initial Cu(II) concentration of 200 ppm. It can be seen that the removal efficiency was increased as pH increased from 2 to 5. The removal efficiency was decreasing as initial pH increased from 5 to 8. This phenomena is probably due to the adsorption competition between high concentration of H\textsuperscript{+} ions and Cu(II) metal ions. In the acid pH range, a high solubility and ionization of Cu(II) can occur and simultaneously, a formation of negatively charged surface area can also take place by the increasing pH solution. It can induce a higher Cu (II) ions removal. Actually, it was found that the highest removal efficiency of Cu(II) metal ions by SPAC obtained at In contrary to lower pH 5. At lower pH, the high H\textsuperscript{+} concentration can cause the surface of SPAC became more positively charge, so the attraction force between adsorbents and metal ions was getting weaker. Based on the highest percent, pH 5 was then selected for the next adsorption experiments by varying initial Cu(II) concentration, SPAC dosage and adsorption temperature.

![Figure 2 Effect of initial pH on the adsorption performance of Cu(II) ion onto SPAC (Initial Cu(II) concentration of 100 ppm, SPAC dosage of 0.3 mg, room temperature).](image)

Figure 3 shows the adsorption performance of SPAC as a function of the initial concentrations of Cu(II). The solution concentration of Cu(II) was varied in the range 100-500 ppm and the initial pH was fixed at 5.0. The removal efficiency of Cu(II) metal ions was found as 40% for initial concentration of 100 ppm. The removal efficiency was reduced to 20% at initial Cu(II) concentration increased from 100 to 300 ppm for adsorption of 300 minutes when the adsorption system reached the equilibrium state. It maybe due to the electrostatic forces between adsorbate and SPAC adsorbent.
Figure 3. Time profile of adsorption performance of SPAC for Cu(II) adsorption by varying initial metal ion concentration (Initial pH at 5.0, dosage of 30 mg, room temperature).

To investigate the effect of SPAC dosage on the adsorption performance of Cu(II) onto SPAC, varying SPAC dosage (10, 30, 60, 80 and 100 mg) was tested while keeping the volume of the metal solution constant at 500 ml, as shown in Figure 4. It can be seen that the removal efficiency increases within 10 – 60 mg before decreasing at SPAC dosage of 80 mg. It is maybe because of the increased adsorbent surface area and availability of more adsorption sites that gradual increase of adsorption performance was performed. However at SPAC dosage of 80 mg, the removal efficiency was reduced to 38% which is due to the adsorption sites become unsaturated.

Figure 4 Effect of SPAC dosage on the adsorption performance of Cu(II) ion in solution onto SPAC (Initial pH at 5.0, initial Cu(II) concentration of 100 ppm, room temperature)
In order to investigate the kinetic mechanism of Cu (II) ions sorption, two models were used in this study. The adsorption capacity data, as shown in Fig 5, was used to study the adsorption kinetic of Cu(II) onto the SPAC adsorbent.

The linear pseudo-first-order model of Lagergren [36] is given as equation (7)

\[ \ln(q_e - q_t) = \ln(q_e) - k_1 t \]  

(7)

Where, \( q_e \) and \( q_t \) are the amounts of copper ions absorbed onto the adsorbent (mg/g) at equilibrium and at \( t \), respectively. \( k_1 \) is the rate constant of first order adsorption (min \(^{-1}\)). The linear plot for calculating parameters in the pseudo first order model is given in Figure 5.

![Linear plot of pseudo first order model for Cu(II) adsorption on SPAC adsorbent by varying initial metal ion concentration.](image)

The pseudo-second-order kinetic model developed by Ho and Mckay [37] is based on the experimental information of solid-phase sorption. The linear pseudo-second-order model can be expressed as follows:

\[ \frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \]  

(8)

Where, \( k_2 \) is the rate constant of second-order adsorption (g/mg min\(^{-1}\)). The adsorption kinetic models were shown in Table 2. The rate constant (\( k_1 \) and \( k_2 \)), correlation coefficient \( R^2 \), and equilibrium adsorption density of \( q_e \) could be derived from the linear fitting of the data. Fig 6 shows the linear plot for determining the pseudo second order model.
The validity of the exploited models is verified by the correlation coefficient ($R^2$). For all variation of initial Cu(II) concentration, it can be seen from Table 2 that the $R^2$ values of the pseudo-second-order model are higher than those of pseudo-first-order model. This result confirmed that the adsorption of copper ion onto SPAC better fits to pseudo-second-order kinetic model. It implies that the predominant process is chemisorption, which involves in a sharing of electrons between the adsorbate and the surface of the adsorbent. Furthermore, most of the capacities calculated from the second order model are approaching the actual capacity of SPAC which is around 1000 mgCu(II)/g SPAC.

**Table 2** Adsorption kinetic parameters

| $C_0$ (ppm) | $k_1$ (minute$^{-1}$) | $q_e$ (mg Cu$^{2+}$/g SPAC) | $R^2$ | $k_2$ (g/mg min$^{-1}$) | $q_e$ (mg Cu$^{2+}$/g SPAC) | $R^2$ |
|-------------|-----------------|-----------------|------|-----------------|-----------------|------|
| 100         | 0.011           | 433             | 0.6519 | 0.0000624       | 769.23          | 0.9735 |
| 150         | 0.014           | 495             | 0.6565 | 0.0000646       | 869.56          | 0.9469 |
| 200         | 0.011           | 596             | 0.7403 | 0.000043        | 988.14          | 0.9687 |
| 250         | 0.011           | 354             | 0.5239 | 0.0001118       | 1009.08         | 0.9499 |
| 300         | 0.017           | 526             | 0.5879 | 0.0001136       | 1021.45         | 0.9662 |

The capacity of an adsorbent can be represented by its equilibrium adsorption isotherm. The Langmuir and Freundlich adsorption models are commonly used to study the adsorption behavior of materials and the correlation among adsorption parameters. Accordingly, equilibrium data, as presented in Figure 7, were fitted by the Langmuir and Freundlich models.
The linear plot for Langmuir model is shown in Figure 8.

The linear plot for Freundlich model is given in Figure 9.
The corresponding values of Langmuir and Freundlich isotherms for Cu(II) ions adsorption on SPAC were shown in Table 3. The results indicate that the Langmuir isotherm fits better than the Freundlich isotherm, which may be due to the homogeneous distribution of active sites onto SPAC. The maximum adsorption capacity obtained from the Langmuir isotherm is 1262.62 mg/g. The results show that activated carbon salacca peel should be a promising adsorbent for removal of Cu(II) ion in aqueous solution. Table 4 presents the result comparison, related to the adsorption of Cu(II) from the aqueous solution, between this work and those found in the literature. It can be seen that the adsorption capacity obtained in this work is relatively higher than those achieved in the previous references.

Table 3 Isothermal Adsorption Parameter Models

| Langmuir model | K_L (L/mg) | R² | Freundlich model | K_F (mg/g) | n | R² |
|----------------|------------|----|-----------------|------------|----|----|
| q_m (mg/g)     | 1262.62    | 0.0216 | 0.9912          | 239.44     | 3.645 | 0.8879 |

Table 4 Comparison of adsorption capacity of SPAC with other biomass based activated carbons for Cu(II) removal

| Raw materials for Activated Carbons | BET Surface Area (m²/g) | Maximum Adsorption Capacity (mg/g) | Optimum pH | Fitted Adsorption Isotherm Model | Reference |
|-------------------------------------|-------------------------|------------------------------------|-------------|---------------------------------|-----------|
| Green Vegetable                     | -                       | 75                                 | 2.0 - 4.5   | Langmuir                        | [40]      |
| Banana Peel                         | 63.5                    | 14.3                               | 6.5         | Langmuir                        | [16]      |
| Grape Bagasse                       | 1455                    | 43.47                              | 5           | Langmuir                        | [41]      |
| Pomegranate Wood                    | -                       | 1.683                              | 6           | Langmuir                        | [17]      |
| Typha Latifolia                     | 130.41                  | 34.48                              | 6           | Langmuir                        | [42]      |
| Corn Cob                            | 88                      | 38.61                              | 7           | Langmuir                        | [43]      |
4. Results and Discussion
In this study, activated carbon was successfully synthesized from salacca peel wastes by the Microwave assisted ZnCl$_2$ activation process. The synthesized carbons were characterized by BET and SEM. An application study was then conducted on the adsorption performance of salacca peel based activated carbons for the removal of Cu(II) metal ions from an aqueous solution.

BET surface analysis showed that the synthesized carbons is a mesoporous solid with a high surface area of 1796 m$^2$/g and from the SEM images, it can be seen that well-developed pores were found on the surface of the activated carbons.

The adsorption equilibrium data was represented well by the Langmuir model results fit with a maximum adsorption capacity of 1262.62 mg Cu(II)/g activated carbon. The large surface area and mesoporous nature of activated carbons derived from salacca peel contribute to the high adsorption. A kinetic study suggested that the adsorption properties of the activated carbon followed the pseudo-second-order kinetic model. Based on the experimental results, it can be said that the salacca peel based activated carbons prepared by microwave heating method have promising potential to be used as an efficient, effective, low cost and green adsorbent for removal of Cu(II) metal ions from aqueous solutions.

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