The utilization of activated carbon from cassava stems on the glucose and cholesterol adsorption

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Abstract. The utilization of cassava stems from waste as an activated carbon raw material for glucose and cholesterol adsorption has been investigated. This study aims to know about the application of activated carbon from cassava stems waste on glucose and cholesterol adsorption with the variations of adsorbent weight and reaction contact time. The process of carbon acquiring was done by the pyrolysis method at a temperature of 400°C for 4 hours. After that, the process was followed by the chemical activation process using KOH as an activator. The carbon obtained was washed in KOH solution with a ratio of 1:2 w/w for ±24 hours, then filtered, washed again, and heated with an activation reactor at a temperature of 800°C. The application of activated carbon was carried out on glucose and cholesterol adsorption to produce glucose and cholesterol adsorption of 3.33 mg/g and 6.84 mg/g respectively. The optimum contact time for glucose and cholesterol adsorption was 45 minutes with the use of 0.1 g adsorbent.

1. Introduction
The cassava plants grow widely in Indonesia with a production range of about 1.9 million tons [1]. In general, the cassava plants only used in tuber part as an additional staple food, while the cassava stems become waste that not used optimally. This problem encourages the utilization of cassava stems to have added value as a raw material of activated carbon. It is expected that the waste can be an alternative precursor with low cost, high abundant, and renewable sources for activated carbon production.

Activated carbon is a product from pyrolysis and activation reaction with several methods, like the physical, chemical, or a combination of both. This process produces the activated carbon that has specific properties, such as high surface area, good surface reactivity, high physical-chemical stability, high pore size, and superior adsorption capacity [2]. The activated carbon with good properties is considered to be a promising material for industrial applications, such as for the adsorption process. Several findings of activated carbon application as adsorbent are for metal adsorption [3], methylene blue dan acid blue 29 [4], and synthetic organic contaminants [5,6].

On its development, the activated carbon is not only for metal or dyes adsorbent, but it can be used for health, including for poisoning removal pharmaceutical waste removal, such as antibiotics, beta-blocker, psychiatric drugs [7], and drugs delivery [8]. This research is aimed to study the potentials of activated carbon from cassava stems waste on glucose and cholesterol adsorption. This is due to the two compounds have an important role in the human body, while it is necessary to avoid body system
disorder. As we know that the excess glucose in our body causing diabetes mellitus disease with high rate of mortality [9]. While the cholesterol compound has a function in the digestion process, but if it is too high, it will cause a blockage in the blood vessels causing various disease, like hypertension, arteriosclerosis, and coronary heart disease, otherwise if it is too low, it causes anemia [10].

In the removal process of glucose and cholesterol, several materials have been used such as polysaccharide [11], chitosan [12,13,14], and alumina [15] but activated carbon utilization for this purpose is still limited. The production and application of activated carbon for glucose and cholesterol adsorption necessary to study the potential of cassava stems activated carbon as an adsorbent. This adsorption process was carried out by using various contact time and adsorbent weight to measure the optimum adsorption capacity.

2. Materials and methods

2.1 Materials and equipment

The materials used in this research were cassava stems waste, D-glucose (Merck, Darmstadt, Germany), KOH, FeCl₃·6H₂O, ethanol 95% (v/v), cholesterol powder, phenol 5% (b/v), CH₃COOH, H₂SO₄(p), Na₂S₂O₃ 0.1N, I₂ 0.1 N. The equipments used were Unitronic OR P shaker water bath (J.P. Selecta, Barcelona, Spain), rotor, furnace, analytical balance, porcelain curs, oven, 60 and 100 sieve mesh, glassware, and UV-Vis Spectrophotometer (Shimadzu-1700, Kyoto, Japan).

2.2 Method

2.2.1 Synthesis and characterization of activated carbon. The synthesis of activated carbon from cassava stems was carried out according to Prapagdee [3] method. Cassava stems were cleaned and chopped into the size of ±5cm, then dried with air dry process. The pyrolysis process was done at a temperature of 400°C for 4 hours, then the equipment was cooled. The carbon was collected and calculated. The carbon as a pyrolysis product was activated using a KOH activator with a ratio of 1:2 (w/w) (carbon soaked into KOH solution for 24 hours). The carbon was then dried for 24 hours using dry air process. The carbon produced was heated in an activation reactor at a temperature of 800°C and the activated carbon was obtained. The quality of activated carbon was analyzed based on Indonesian National Standard (SNI 06-3730 1995), including the moisture, ash content, volatile matter content, fixed carbon, and iod adsorption.

2.2.2 Glucose adsorption analysis. The glucose adsorption process using activated carbon was started with glucose content measurement by quantitative method (phenol sulphate method) based on [16]. The glucose standard solutions were made with a concentration of 5, 25, 50, 75, dan 100 µg/mL. The activity test of glucose adsorption using activated carbon was conducted with a contact time and adsorbent weight variations. The contact time variations were 30, 45 and 60 minutes while the adsorbent weights were 0.1, 0.3 and 0.5 g. The activated carbon was placed into a 10mL glucose solution with a concentration of 100 µg/mL, then shaken using shaker equipment follows the time contact and adsorbent weight variations. After the completed process, the solution filtered and then the glucose content was measured. The glucose measurement was done by 1 mL phenolic 5% and 5 mL H₂SO₄(p) added into blanc, standard, and sample solutions. The solutions were shaken and cooled for 10 minutes at room temperature. Then, the solutions were heated in the water bath for 15 minutes. The absorbance measurement analyzed by using UV-Vis spectrophotometer was done at 485nm wavelength.

The equation 1 of adsorption capacity [2]

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Q = \frac{V (Co-Ca)}{m}
\]
Q is adsorption capacity (mg/g), V is solution volume (L), Co is glucose/cholesterol initial concentration (µg/mL), Ca is glucose/cholesterol final concentration (µg/mL) and m is adsorbent weight (g).

2.2.3 Cholesterol adsorption analysis. Cholesterol adsorption process by activated carbon was done by using FeSac reagent. The process was initiated with FeSac reagent synthesis using 2.5 g of FeCl₃·6H₂O diluted in 25 mL glacial acetic acid. Then 1 mL of iron solution was placed into the H₂SO₄ solution. Meanwhile, a cholesterol solution was prepared by dissolving 25 mg of cholesterol in 250 mL of glacial acetic acid and dilute into concentrations (5, 25, 50, 75, and 100 µg/mL). The cholesterol adsorption process was conducted similarly with glucose adsorption, but 2 mL of FeSac added into blank, standard and filtered solution then those were shaken for 20 minutes and the absorbance was measured using spectrophotometer UV-Vis at 560 nm wavelength.

3. Results and discussion

3.1 Characterization of activated carbon-based on Indonesian National Standard
The synthesis of activated carbon in this research was started with the pyrolysis method at a temperature of 400°C. In this process, several stages of biomass raw material decomposition depended on pyrolysis temperature. In the initial stage at low temperature, the cellulose content tended to first decompose, then followed by hemicellulose and lignin at higher temperatures resulted in carbon as a product. The carbon product used as a precursor for the activating process with KOH activator and heated at a temperature of 800°C resulted in activated carbon with more excellent properties. The activated carbon from biomass generally prepared by chemical methods [17]. Table 1 shows the activated carbon quality based on proximate analysis using Indonesian Standard Method (SNI 06-3730 (1995)). From the table, we can see that the quality of activated carbon from cassava stems met SNI (moisture <15%; ash content <10%; Volatile matter content <25%, fixed carbon >65% and iod adsorption >750 mg/g).

| Carbon Activated carbon | Moisture (%) | Ash (%) | Volatile Matter (%) | Fixed Carbon (%) | Iod Adsorption (mg/g) |
|-------------------------|--------------|---------|---------------------|-----------------|-----------------------|
| Carbon                  | 2.49         | 4.38    | 30.17               | 65.45           | 173.136               |
| Activated carbon        | 14.2         | 7.75    | 24.85               | 67.4            | 979.119               |
| SNI                     | <15          | <10     | <25                 | >65             | >750                  |

In general, the pyrolysis from biomass that involved decomposition reaction would produce carbon and liquid smoke as by-product. Carbon as a pyrolysis product could be activated with a chemical or physical activator. The activation process would produce activated carbon with better characteristics. From figure 1, it can be seen that the proximate analysis of activated carbon was different than those of without the activation process. The activation process with KOH as an activator compound could reduce the impurity and ash content, also open carbon pores [18]. This resulted in activated carbon with higher iod adsorption. With the utilization of a temperature 800°C, the potassium boiling point could be reached and the diffusion of potassium compound into the surface internal structure would break the chemical bond on biomass and cause the volatile matter apart from the surface. KOH activator utilization play an important role in the forming and developing of pore structure on the surface of activated carbon, it will be accessible and adsorbate molecules easier to diffuse [19]. The carbon activation process using KOH forms micropores more than other activators, so this is an advantage for adsorption [20], like in methylene blue adsorption [17]. KOH and heating process used at a temperature of 800°C, and it’s increasing the surface area and pore of activated carbon so it is
potential to become CO₂ adsorbent [21]. In other research, the activated carbon used for rhodamine B adsorption with an adsorption capacity of 123.46 mg/g was reached in 12 minutes contact time and can be regenerated until 7 times [22].

3.2 Glucose and cholesterol adsorption
Glucose and cholesterol have hydrocarbon groups so that most of the activated carbon surface can function as effective adsorption sites for glucose (table 2). The difference in the presence/absence of OH groups will affect the saturation capacity, the more OH groups presence, the higher the saturation. Molecule charges contribute to the electrochemical resistance on pore channels. The adsorbate polarizability can affect toward adsorption process. The aromatic compounds, in general, has a higher of PKa and usually do not have charges so the polarity is more dominant than charges. The polarity influenced by molecule size, the higher molecule size, the higher the polarity of the compound. The adsorption capacity of activated carbon to glucose is lower than cholesterol, it is caused by OH groups, size, and mass molecule difference from both of the compounds. The compound with higher molecule size tends to has a higher affinity and more adsorbed in adsorbent surface than the pore, while the compound with lower molecule more adsorbed into pores [4].

Table 2. Characteristics of compounds studied.

| Compounds   | Chemical formula | Structure | Mass (g/mol) | \( \Lambda_{max} \) (nm) |
|-------------|------------------|-----------|--------------|--------------------------|
| Glucose     | C₆H₁₂O₆          | ![Glucose](image) | 180,156 g/mol | 485                      |
| Cholesterol | C₂₇H₄₆O          | ![Cholesterol](image) | 386.65 g/mol | 560                      |

The increasing of oxygen in the external surface of activated carbon causes a higher negative charge and it will increase water molecule bond and form a barrier on the hydrophobic molecule adsorption process [5]. The negative surface and acidity tend to associate with low adsorption capacity. The carbon surface with negative charge tends to more beneficial for adsorption of adsorbate with positive charges. In this study, the maximum glucose and cholesterol adsorption capacities were 3.3 mg/g and 6.84 mg/g respectively. In other research, the activated carbon utilization with KOH activator showed the adsorption capacity of methylene blue of 642-683 mg/g [23] and H₂S of 98 mg/g [19]. The cholesterol removal from dairy products reached 97% (2.2 mg/g to 0.1 mg/g) using combination methods with supercritical and alumina as an adsorbent but the weight of alumina used is 50 g [15]. The activated carbon with KOH and EDTA combination also can adsorb the phenolic compound with an adsorption capacity of 194 mg/g [24] and used for butyric acid adsorption with 98% adsorption [25].

The contact time variations showed different adsorption results. From figures 2 and 3, we could see that the increasing contact time used can increase the adsorption capacity but in a certain time the adsorption will decreased, this is due to the fast adsorption on adsorbent in initial stages until equilibrium reached. After the optimum time passed through, the adsorption would be decreased due to the active sites was occupied with adsorbate molecules and the desorption process reached. The presence of empty sites on adsorbent in initial reaction causing the adsorption process to increase, but later then there was a rejection force between adsorbate and adsorbent molecules. In general, when the
adsorption doses increased, the adsorption process will be increased. This can be explained by an increasing number of activated carbon sites, so the adsorbate will be easier to adsorbed, but if it is too much, the saturation will be reached so the adsorption will be not optimal [22].

Figure 1. The glucose adsorption capacity of activated carbon.

Figure 2. The cholesterol adsorption capacity of activated carbon.

4. Conclusion
The activated carbon from cassava stems waste is potential to be utilized on glucose and cholesterol adsorption process. The adsorption capacity of activated carbon on glucose and cholesterol adsorption were 3.33 mg/g and 6.84 mg/g respectively. The optimum contact time for glucose and cholesterol adsorption was 45 minutes with the addition of 0.1 g adsorbent.

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