Activated carbon preparation from bagasse and banana stem at various impregnation ratio

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Abstract. The effects of impregnation ratio on characteristics of activated carbon from bagasse and banana stem were analyzed. The precursors were impregnated using phosphoric acid (H\textsubscript{3}PO\textsubscript{4}) solution as the activator at a ratio of the activator to the precursor (w/w) of 1:1, 2:1 and 3:1. Pyrolysis was conducted at 400 °C for 15 minutes. The results showed that activated carbon from bagasse has better yield, surface area and total pore volume compared to activated carbon from banana stem. The increased of impregnation ratio decreased the yield and surface area, but increased the total pore volume and pore radius. The highest yield of activated carbon from bagasse and banana stem were 61.87% and 45.53%, respectively. Whereas the highest surface area of activated carbon from bagasse and banana stem were 1,070 m\textsuperscript{2}/g and 915 m\textsuperscript{2}/g, respectively. The activated carbon produced from both precursors contained both micropores and mesopores structures.

1. Introduction
Indonesia is an agrarian country with abandoned tropical plants including sugar cane and banana. In the utilization process, both plants leaves bagasse and banana stem as waste. These wastes are lignocellulosic materials which are very potential to be utilized as activated carbon precursors. The bagasse contains 42.16 \% cellulose, 36.0 \% hemicellulose, and 19.30 \% lignin [1], whereas banana stem contains 43.3\% cellulose, 20.6\% hemicellulose and 27.8\% lignin [2]. The lignocellulosic contents provide huge carbon source in producing activated carbon.

Activated carbon has beneficial properties such as the large surface area, well-developed porosity and rich surface group [3]. Hence, activated carbon has various applications in industrial scale primarily as adsorbent, catalyst, catalyst support, desiccants and electrode. It plays important role particularly in those concerning the efforts for environmental protection and technological development.

In this study, the effects of impregnation ratio on the characteristics of activated carbon from bagasse and banana stem using activator of H\textsubscript{3}PO\textsubscript{4} were analyzed. Impregnation ratio is defined as the ratio of the weight of the activating agent to the precursor [4]. It influences the yield, surface area, total pore volumes, and pore radius of the activated carbon. These properties are among important factors in supporting the wide range of activated carbon industrial applications.
2. Experimental Procedure

2.1. Activated Carbon Preparation
The same procedure was conducted in preparing the activated carbon from bagasse and banana stem as published earlier in [5-6]. The fresh bagasse and banana stem were initially washed using aquadest and then dried. The drying process performed under sunlight and continued by oven drying at 105°C. Both samples were then crushed, sieved to 32 mesh and then mixed with H₃PO₄ solution as the activator at various impregnation ratios of 1:1, 2:1 and 3:1 for 24 h. The samples were then filtered and carbonized at 400 °C for 15 minutes under nitrogen (N₂) flow. The activated carbon should be in neutral and dry condition before storing and characterizing. The yield of activated carbon could be calculated as the ratio of the product mass to the initial mass of the raw material used.

2.2. Activated Carbon Characterization
The Brunauer-Emmett-Teller (BET) surface area of sample was determined by applying the standard BET method to the nitrogen adsorption (Quantachrome Corp instrument by Nova-1200). The scanning electron microscopy (SEM) analysis (ZEISS) was used to get the surface morphological structure of the activated carbons.

3. Result and Discussion

3.1. Yield of Activated Carbon
The yield of activated carbon from bagasse was slightly higher than that of activated carbon from banana stem (Figure 1). The increasing of impregnation ratio gave fluctuation yield on activated carbon from bagasse. However, fewer yield of activated carbon from banana stem produced for all variation of impregnation ratio under study. The pyrolysis of the polymeric cellulose or lignin in carbonization process releases most of the non-carbon elements particularly hydrogen, oxygen and nitrogen, in the form of gases and tars [4]. In the dried raw material, found that bagasse had higher carbon content, thus in turn, lower non-carbon components compared to those of banana stem. Based on the EDX analysis (JEOL, JED 2300) to the dried samples, the carbon content in bagasse and banana stem were 95.85% and 83.06%, respectively. This might be contributed to higher yield of activated carbon from bagasse.

![Figure 1. Activated carbon yield at various impregnation ratios.](image-url)

The decreased yield as the increasing impregnation ratio was exhibited by previous studies [7-9]. The phenomena was attributed to the continuous removal of tar material from the pores [10]. It also due to the enhancement of carbon burning-off by extra activator [8].
3.2. \( \text{N}_2 \) Adsorption-Desorption Isotherms

Nitrogen adsorption isotherms of these activated carbons can be properly classified as a mixture of type I and type IV isotherms. It was exhibited clearly in Figure 2 that the amount of adsorbed nitrogen increased rapidly at relative pressure lower than 0.1. In the initial part of each isotherm, all isotherms are type I, with an important uptake at low relative pressure, the characteristics of microporous materials. Moreover, as the relative pressure increases to about 0.4, the isotherms showed apparent hysteresis loops. This adsorption behavior exhibits a type IV isotherm as characteristic of mesoporous structure. The slope of the plateaus in these isotherms gradually increases with increasing relative pressure. According to IUPAC classification, type I isotherm can be associated with microporous structure while type IV isotherm with mixture of microporous and mesoporous structures [11]. Therefore, the activated carbons obtained here show both microporous and mesoporous structures.

The increasing impregnation ratio for bagasse showed slightly lower curve at relative pressure up to 0.5. For banana stem, in Figure 2 showed that with increasing impregnation ratio up to 2:1 there was an upward trend in adsorption. This showed that the surface area increased. As the impregnation ratio increased from 2:1 to 3:1, the amount of nitrogen adsorbed by the activated carbons decreased.

![Figure 2. \( \text{N}_2 \) adsorption-desorption isotherms of the activated carbons from bagasse (left) and banana stem (right) at various impregnation ratios.](image)

3.3. Surface Area and Pore Volume

It demonstrated in Figure 3 that activated carbon from bagasse have much larger surface area than that of banana stem. Both activated carbons exhibited the same trends in total pore volume and the pore radius as the increasing impregnation ratio. From the pore radius data, all of the activated carbons were dominated with mesopore. According to IUPAC, activated carbons are classified into three groups based on its pore diameter: micropore (diameter < 2 nm), mesopore (2-50 nm) and macropore (> 50 nm) [12].
3.4. Surface Morphology

SEM provides wide information, not only the surface morphology but also the pore structure, surface structure and pore arrangement of a material surface. The use of H3PO4 is very effective in creating high surface area with well-developed pores on the surface of the activated carbon. Upon the activation using H3PO4, more porous structures begin to appear which can be accredited to the dehydration effect of H3PO4 and the oxidation of organic compounds in the carbonization step. New pores were also created due to the reaction between carbon and the activator [7].

As the impregnation ratio was increased from 1:1 to 3:1, the pore volume and pore radius generally increased (Figure 4). The surface of the activated carbon at the impregnation ratio of 1:1 was fairly smooth except for some occasional cracks. As the impregnation ratio was increased to 2:1 and 3:1, activation process was able to create pore structure within the carbon, indicating higher surface area. It seems that the pores and cavities on the surfaces of carbons resulted from the evaporation of the activating agent in this case is phosphoric acid during carbonization, leaving the space previously occupied by the activating agent [13].

Figure 3. Effect of the impregnation ratio on the surface area, total pore volume and average pore radius.
4. Conclusion

The impregnation ratio showed an important effect on the development of porosity. Higher yield and surface area were obtained with bagasse as the precursor of activated carbon compared to banana stem. The maximum yield and BET surface area of activated carbon from bagasse were 61.87% and 1070 m²/g, respectively. The N₂ adsorption exhibited that the activated carbons had both micropores and mesopores structures with dominated by mesopores. It was revealed that both raw materials of bagasse and banana stem are potential precursor for activated carbon preparation.

5. References

[1] Chen C X, Huang B, Li T and Wu G F 2012 Preparation of phosphoric acid activated carbon from sugar cane bagasse by mechanochemical processing BioResources 7 5109–5116
[2] Noeline B F, Manohar D M and Anirudhan T S 2005 Kinetic and equilibrium modelling of lead(ii) sorption from water and wastewater by polymerized banana stem in a batch reactor Separation and Purification Technology 45 131–140
[3] Xu J Z, Chen L Z, Qu H Q, Jiao Y H, Xie J X and Xing G 2014 Preparation and characterization of activated carbon from reedy grass leaves by chemical activation with H₃PO₄ Applied Surface Science 320 674-680
[4] Yahya M A, Al-Qodah Z and Zanariah Ngah C W 2015 Agricultural bio-waste materials as potential sustainable precursors used for activated carbon production: A review Renewable and Sustainable Energy Reviews 46 218
[5] Misran E, Maulina S, Dina S F, Harahap S A A and Nazar A 2017 Characterizations of activated carbon produced from bagasse and banana stem using H₃PO₄ as activating agent Journal of Engineering and Applied Sciences 12(15) 3839-3842
[6] Misran E, Maulina S, Dina S F, Harahap S A A and Nazar A 2018 Activated carbon production from bagasse and banana stem at various times of carbonization IOP Conf. Series: Materials Science and Engineering 309 012064
[7] Demiral İ and Şamdan C A 2016 Preparation and characterisation of activated carbon from pumpkin seed shell using H₃PO₄ Anadolu University Journal of Science and Technology A-
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