Characteristics of activated carbon from coconut shell (*Cocos nucifera*) through chemical activation process

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Abstract. Activated carbon is a material that widely used in industries as an adsorbent and purifying material. Moreover, activated carbon has an essential role in improving product quality. Coconut shell has many advantages to be used as activated carbon sources as it has both high mechanical strength and high pore volume. This research aims to explore the coconut shell as an activated carbon material through immersion using a phosphoric acid solution. The activation process was conducted under different conditions, i.e. H₃PO₄ concentration of 1 M, 2 M, 3 M, 4 M and 5 M, and immersion time of 1 hour, 3 hours, 5 hours, 7 hours and 9 hours. The characteristics of the activated carbon were evaluated in terms of iodine number, density, water content and ash content. It could be concluded that the combination of 3M H₃PO₄ concentration and 9 hours immersion time was the best combination for producing desirable activated carbon from coconut shell. The characteristics of iodine number, density, water content and ash content are 248.5811 mg/g, 1.3613 g/ml, 5.3628% and 2.1239%, respectively. The phosphate acid concentration and the immersion time had a significant effect on the activated carbon characteristics such as iodine number, density, water content and ash content.

1. Introduction

Activated carbon has been widely used as an adsorbent and purifier in industrial processes to improve product quality, such as water treatment plant, sugar company, pharmaceutical industries and food companies. Many new processing industries in Indonesia have an increasingly higher demand for activated carbon. Thus, it makes an excellent opportunity to produce activated carbon from locally underutilized material such as coconut shell. The increasing demand for activated carbon encourages the government issue regulations on standards of activated carbon quality in the Indonesian Industrial Standard (SII). This standard contains the minimum quality requirement that has to be fulfilled by the producer of activated carbon. The drying process on average has been carried out following the usual standard reversal done in one day about 2-3 times process so that the heat received is more evenly distributed [1]. Generally, activated carbon used as absorbent, the surface area range of activated carbon was at 300-3500 m²/g, and this is related to the internal pore structure that makes the activated useful as an absorbent. Activated carbon could be used to absorb gas and some specific chemical substance.
Activated carbon can be prepared from many materials that have a high content of carbon. In recent years, various research on the production of activated carbon from renewable precursors have been reported, such as rice hull [2] com-cob [3], cattail [4], coconut husk [5], dates tree’s fronds [6] and pruning mulberry shoot [7]. There are a lot of cheaper and renewable materials that can be used to produce activated carbon; one of them is coconut shell. Indonesia is a country with the most significant area of coconut plant in the world. The planting area was approximately 3.8 million hectares with the total production of 3.2 million tons coconut, and most of the coconut production is in citizen farm.

Nowadays, the activated carbon consumption in Indonesia reaches 300,000 ton/year, 12% of the total consumption was contributed from coconut shell. In the technology aspect, producing activated carbon from the charcoal is very simple and can be done by a small and traditional company. Chemical activation has both cheaper cost and higher carbon product yield. Carbonization occurs in the presence of a dehydrating agent, e.g. ZnCl₂, H₃PO₄, and H₂SO₄, from both economic and environmental point of view, H₃PO₄ activation has been widely utilized [6].

This research is aimed to utilize the coconut shell as a primary material to produce activated carbon and to gain knowledge and method of producing activated carbon from coconut shell that could be developed for industrial scale, and finally to know the effect of H₃PO₄ concentration and immersion time on the characteristics of activated carbon from coconut shell.

2. Materials and Methods
The research used completely randomized block design of five levels of H₃PO₄ concentration (1 M, 2 M, 3 M, 4 M and 5 M) and five different immersion duration (1 hour, 3 hours, 5 hours, 7 hours and 9 hours) and was repeated three times. The materials used for this research were coconut shell, phosphate acid, triosulfate natrium, hydrochloric acid, starch, iodine and distilled water. The first step, coconut shells in the same maturity were collected and then burnt to produce charcoal, 10 grams of charcoal samples were immersed in H₃PO₄ solution following treatments. The samples were then dried in the oven to remove moisture, and then the product was characterized. The activated carbons resulted from the treatments were evaluated for iodine number, density, water content and ash content.

3. Results and Discussion

3.1 Iodine number
Activated carbon production can be divided into two processes, which are carbonization followed by activation. Carbonization process was performed in the absence of air to avoid oxidation, and this could be done by reducing the size of the coconut shell. Mature coconut fruits are essential to produce activated carbon as well as the cleanliness and dryness that allow the carbonating process to work correctly.

Figure 1 shows that longer immersion time resulted in higher iodine number. Iodine number is indicative of absorption capability of activated carbon. In chemical activation, carbonization occurs in the presence of a dehydrating chemical agent. Higher chemical solution affects to more intensive development of a pore structure. Generally, after 5 hours immersion time, iodine number tends to be constant. In comparison, the highest iodine removal was iodine number at 248.5811 mg/g.

This result is different from optimum activation time of Jatropha curcas fruit shell activated carbon, i.e. 120 minutes [8]. It is because different inorganic contents of raw materials and using high temperature in the activation process. Carbon from different agro-wastes was reported to remove iodine from aqueous solution in the range 50% to 95% [9]. The higher degree of iodine adsorption is indicative of a higher surface area and the presence of large microporous and mesoporous structures [10].

3.2 Density.
Density or specific gravity is a parameter showing the development of the porous activated carbon. Activated carbon activation using H₃PO₄ solution is to generate pores in the activated carbon produced. This is what causes the activation of activated carbon has large pores, as shown by the density data in Figure 2.
Figure 1. The effect of H₃PO₄ concentration and immersion time on the iodine number of the activated carbon.

Figure 2. The effect of H₃PO₄ concentration and immersion time on the density of the activated carbon.

Figure 2 shows that the density of activated carbon decreased with increasing H₃PO₄ concentration from 1 M, 2 M to 5 M, although the decrease was not significant. At a concentration of 1 M H₃PO₄ with an immersion time of 1 hour, the density of the obtained activated carbon was of 2.3 g / ml. At a concentration of 3 M H₃PO₄ with an immersion time of 9 hours, the density of the obtained activated carbon decreased to 1.4 g / ml. The result shows that the density of activated carbon particles is inversely proportional to the volume of solids. The density of solid (particle density) is the quotient of the mass of particles with particle volume of a material. This density does not consider the pore volume of a material. So, the higher the volume of the particle, the lower the density.

3.3 Water content
The water content indicates the percentage of water content in the activated carbon. The presence of water is caused by nature hygroscopic activated carbon. So, if it reacts with air, the water vapour in the air will be absorbed in the pores of activated carbon. The presence of water in the activated carbon will occupy the pores of activated carbon and will lead deficiencies to the activated carbon adsorption. Treatment differences in the concentration of Activating Agent H₃PO₄ solution provide different water content on activated carbon. Following the test data moisture content of coconut shell activated carbon.
Figure 3. The effect of $\text{H}_3\text{PO}_4$ concentration and immersion time on the water content of the activated carbon.

Figure 3 shows that the concentration of $\text{H}_3\text{PO}_4$ gives different results on the water content of activated carbon. It can be seen that the relative decline in water levels at a concentration of $\text{H}_3\text{PO}_4$ 1 M, 2 M, 3 M and 4 M decline. However, at the $\text{H}_3\text{PO}_4$ concentration of 5 M, the value obtained was increasing from 6% to 9%. Thus, it can be concluded that increasing concentration of $\text{H}_3\text{PO}_4$ can give an increase in the water content of activated carbon. An increase in the water levels reached the highest value at a concentration of 5 M. For lower $\text{H}_3\text{PO}_4$ concentration under 4 M, the water content of activated carbon decreased. Water content value obtained is directly proportional to the iodine number of activated carbons. This is caused by the more significant number of activated carbon iodine, the more the pores found in activated carbon. The number of pores is what will affect the ability of activated carbon to absorb the particles are smaller in size, as well as water vapour. In other words, the greater the number of pores, the greater the activated carbon adsorption capacity and hygroscopic properties of activated carbon. The same finding also reported by [11], that the higher the iodine number, the more significant amount of the water that can be absorbed. Carbon with a high iodine number has a large surface area or pores that effective to absorb and store water from the air. Similarly, according to [5], activated carbon is allowed to stand 2-3 days in a desiccator would cause the interaction with the free air so that the activated carbon which has a high absorption will be able to absorb more water from its surroundings.

3.4 The ash content
The ash content has a significant impact on the quality of activated carbon. The existence of excessive ash can cause blockage to pores of activated carbon so that the surface area of activated carbon would be reduced [12]. Following is the test data on the ash content of coconut shell activated carbon.

Increased immersion time using phosphate acid ($\text{H}_3\text{PO}_4$) as activator material which is a water-absorbing compound or dehydrating agent affect the increasing water absorption and degradation of cellulose contained in a coconut shell. Consequently, the carbonization process by-product, i.e. tar that will cover pores of the carbon can be reduced or even eliminated. Activating agents can boost the intermolecular interactions between the activating agent and the biomass precursor. The impregnation time and activating agent are additional determinants of the characteristics of AC because of their direct influence on the pore sizes and thus release of volatiles [12]. Generally, this phenomenon resulting activated carbon has a large specific surface area which is shown generally by the increasing iodine number, decline the density (Figure 2), decline moisture content (Figure 3) and decline ash content (Figure 4). However, for 4 M and 5 M of $\text{H}_3\text{PO}_4$ concentrations, the phenomena were slightly different after reach 5 hours immersion time. $\text{H}_3\text{PO}_4$ promotes the pyrolytic decomposition of the starting material and the formation of the cross-linked structure. The cross-linked phenomenon is due to interactions...
between $\text{H}_3\text{PO}_4$ and biomass in the precursor. This leads to the formation of phosphate linkages between the fragments in biomass [13]. For specific use, characterization of activated carbon is essential. In marketing purpose, activated carbon is priced based on its moisture and absorption capacity. The results show that iodine number, density, water content and ash content affected by both, $\text{H}_3\text{PO}_4$ concentration and immersion time. It is predicted that the optimum immersion time reached faster in higher $\text{H}_3\text{PO}_4$ concentration.

Figure 4. The effect of $\text{H}_3\text{PO}_4$ concentration and immersion time on the ash content of the activated carbon.

The best results of activated carbon are shown in the treatment of 3 molar $\text{H}_3\text{PO}_4$ concentration and 9 hours immersion, i.e. the activated carbon which have 248.5811 mg/g iodine number, 1.3613 g/mL density valued, 5.3628 % water content and 2.1239 % ash content. These parameters are following Indonesian Industrial Standard of activated carbon.

4. Conclusions
The activated carbon can be produced using coconut shells as the raw material. The process involved cleaning the coconut shell, burning, milling, refining, activating with phosphate acid, immersion and drying. The phosphate acid concentration and the immersion time gave a significant impact on the activated carbon characteristics (iodine number, density, water content and ash content). The best result was gained using the 3 M phosphate acid concentration and 9 hours immersion time, resulting in the iodine number of 248.58 mg/g, 1.36 g/mL density, 5.36 % water content, and 2.12 % ash content. This result was satisfied the Indonesian Industrial Standard (SII) No.0258-79.

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