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Review on Methods For Activation and Characterization of Activated Carbon

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ABSTRACT
Activated carbon has become an ideal material for the separation of various chemical pollutants. Extensive use is limited due to production costs, which has triggered researchers on feasible options for unconventional and cost-effective production. The application of biomass waste has been widely explored as an alternative to expensive methods of producing activated carbon from coal. In this research, a detailed list of active carbon production methods from various biomass is presented systematically. Efforts have also been made to review physical properties, such as the final and closest analysis of various biomass materials. Finally, this review combines existing research papers on activated carbon derived from biomass waste to understand the effect of pyrolysis temperature, activation temperature, and the effects of various physical and chemical activation conditions on production, surface characteristics and behavior of activated carbon adsorption.

Keywords: Activated carbon, activation temperature, biomass waste, carbon adsorption pyrolysis temperature

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INTRODUCTION

Indonesia is an agrarian country where most people in Indonesia make a living in the agricultural sector. The potential of biomass waste in Indonesia has not been used optimally, one of which is activated carbon. Activated carbon is an amorphous compound produced from materials containing carbon or charcoal that is specially treated to get high adsorption power. Activated carbon can adsorb certain gases and chemical compounds or selective adsorption properties, depending on the size or volume of pores and surface area. Absorption of activated carbon is very large, which is 25-100% of the weight of activated carbon. Activated carbon is carbon that is free and has an internal surface, so it has good absorption. The active absorbability of this activated carbon depends on the amount of its kabon compound which ranges from 85% to 95% free carbon.

Activated charcoal is a carbon amorph compound, which can be produced from materials containing carbon or from charcoal that is treated in a special way to get a wider surface. The surface area of activated charcoal ranges between 300-3500 m^2/gram and this is related to the internal pore structure that causes activated charcoal to have an adsorbent characteristic. Activated charcoal can adsorb certain gases and chemical compounds or selective adsorption properties, depending on the size or volume of pores and surface area. Absorption of activated charcoal is very large, which is 25-1000% to the weight of activated charcoal.

Activated charcoal is divided into 2 types, namely activated charcoal as a pale and as an absorbent of steam. Activated charcoal as a pale, usually in the form of a very fine powder, pore diameter reaches 1000A0, used in the liquid phase, serves to remove the disturbing substances that cause unexpected colors and odors, freeing solvents from the disturbing substances and other uses, namely on chemical industry and new industries. Obtained from sawdust, pulp making or from raw materials which have a small density and have a weak structure [1].

Activated carbon is a good adsorbent. It has versatile applications in wastewater treatment and many other uses as an adsorbent. High-quality activated carbon can be obtained from wood materials such as bamboo with inherent mechanical strength, high carbon (40%) and low ash content. Bamboo-based carbon can function as a good adsorbent and can be regenerated and reused many times. In India, with its extensive bamboo reserves, there is an excellent scope for producing high-grade activated carbon bamboo.

Activated carbon has several important uses including refining solutions (such as in cleaning sugar cane, beets and corn sugar solutions), eliminating flavors and odors from domestic and industrial water supplies, vegetable and animal fats and oils, alcoholic beverages, chemicals and pharmaceuticals and in processing wastewater. It was found to be used in gas purification, liquid phase recovery, separation processes and as a catalyst and catalyst support. Many organic compounds such as chlorinated and non-chlorinated solvents, gasoline, pesticides, and trihalomethane can be absorbed by activated carbon. It's also effective at removing chlorine and effective enough to remove some heavy metals. This is used for liquid phase adsorption or decolourization; usually mild and fine powders are produced from low density materials such as sawdust or peat.

However, for gas phase adsorption, there is a need for hard and solid granular materials produced from high density raw materials such as bamboo, coconut shell, palm kernel shells, coal or coke [2].

In general, the texture and chemical characteristics of activated carbon are influenced by many factors such as the raw nature of the material, the activation method, the activating agent and the conditions of the activation process. Chemical activation methods are widely used to produce activated carbon from carbon-containing materials. In chemical activation, the starting material is impregnated with a chemical agent, and the impregnated material is heated for carbonization in an inert atmosphere. Chemical agents help develop activated carbon porosity by means of dehydration and degradation. After permeating the precursors by chemical agents and heat treatment of the mixture, the impregnating agent and its salts are removed by washing with acid / base and water, which makes the pore structure available in activated carbon. The advantages of the chemical activation process are lower activation temperatures (and energy costs), shorter activation times, one activation step (simultaneous carbonization-activation), higher activated carbon yields, large surface area, and developing microporosity. well. Among the disadvantages are the costs of the activating agent, the need for intensive washing to remove impurities generated by general corrosive processes and processes [3].

Table 1. List of precursor materials from various agricultural sources for low carbon cost production of activated carbon

Raw material	References
Cassava peel	[4]
Jatropha curcas fruit shell	[5]
Spent coffee ground	[6]
Olive seeds	[7]
Rice husk	[8]
Corn cob	[9]
Coconut shell	[10]
Pecan shell	[11]
Rice straw	[12]
Macadamia nutshell	[13]
Fruit stones & nutshell (hazelnut shells, peanut hulls)	[14]
Apricot stones	[15]

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Almond shell	[16]
Grain of sorgum	[17]
Cedar wood	[18]
Date stone	[19]
Acacia mangium wood	[20]
Pomegranate seeds	[21]
Neem leaves	[22]
Palm oil shell waste	[23]
Wheat straw	[24]
Rice husk ash	[25]
Coconut coir dust	[26]
Natural condensed tannin	[27]
Vetiver roots	[28]

There are several sources of literature that have conducted experiments in the field of activated carbon is [29] investigate the physical properties of activated carbon derived from EFB. The initial ingredient is activated by potassium hydroxide (KOH) using a microwave at 720 W for 15 minutes. They report semi crystalline activated charcoal with a high surface area of 324.74 m^2/g. [30] also synthesized activated carbon using EFB at various temperature activations with KOH at 3 hours holding time. Research shows an inverse relationship between percentage yield and temperature. The carbonized sample at 800^{o}C produced the highest surface area of 1179 m^2 / g and an average pore diameter of 1.52-1.74 nm VFD also activated with ZnCl at 550^{o}C using different fertilization ratios (1: 1 and 2: 1). The same mass impregnated sample ratio produced the highest surface area of 1058 m/g and showed adsorption of 225 mg methylene blue per gram. In other studies by [31], activated carbon was prepared using microwave steam activation at 500^{o}C for 15 minutes, and a BET surface area of 570 m^2 / g was recorded.

1. Technology selection for the production of activated carbon

Activated carbon is an amorphous compound produced from materials containing carbon or charcoal that is specially treated to get high adsorption power. Activated carbon can adsorb certain gases and chemical compounds or selective adsorption properties, depending on the size or volume of pores and surface area.

Amorphous activated carbon which is ready to show a variety of porosity and extended surface area. This is the result of activating the carbon-rich material process. The wood is high in carbon content (50-90%) will be a suitable precursor to produce activated carbon. Activation begins with carbonization of the precursors to achieve high carbon content material [32]. Activated carbon with a

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large surface area and high mesoporous volume can function as an extraordinary adsorbent. Its large surface area and mesoporous activated carbon are useful for wide applications in separation, decoloration, deodorizing, purification and filtration operations [33] Its chemical composition, as well as its method and process conditions used during the activation of woody biomass, play an important role in the formation of pores, pore volume, and surface area. As a result, the activated carbon adsorption properties vary from one source of wood species to another [34].

In general the process of making activated carbon consists of three stages, namely

a. Dehydration is the process of removing the water content in raw materials by heating in an oven at 170°C. At temperatures around 275°C a carbon decomposition occurs and results such as tar, methanol, phenol and others are formed. Nearly 80% of the carbon element is obtained at temperatures of 400-600°C.

Tabel 2. Drying methods are carried out in active carbon research

Raw Material	The method used	References
rubber wood sawdust	Dried at 105°C for 24 hours	[35]
Paulownia wood	Dry with a temperature of 110°C for 12 hours	[3]
tamarind wood	This wood is dried in the sun to remove moisture and soaked in ZnCl2 solution for 24 hours and dried again at a temperature of 150°C for 1 hour.	[2]
Jatropha curcas fruit shell	Dry with a temperature of 110°C for 48 hours	[5]
Olive-tree wood	Dry with a temperature of 120°C for 24 hours	[36]
Walnut wood	Dry with a temperature of 120oC for 24 hours	[37]
Oak Wood	Dry with a temperature of 110°C for 6 hours	[38]
Rice Husk	Dry with a temperature of 150°C for 15 hours	[39]
India shrub wood	Dry with a temperature of 800°C for 2 hours	[40]
coffee-shell	Dry with a temperature of 100°C for 12 hours	[41]

b. Carbonization is a process in which the elements of oxygen and hydrogen are removed from carbon and will produce carbon frames that have a certain structure. To produce charcoal suitable for activated carbon, carbonization is carried out at temperatures over 400°C but it also depends on the base material and the method used in activation. When carbonization occurs, several stages include removal of water or dehydration, the change of organic matter into carbon elements and the decomposition of tar so that the carbon pores become larger. Products from the carbonization process have little adsorption power. This is due to the low temperature carbonization process, a part of the tar produced is in the

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pore and surface so that the adsorption is blocked. Carbonized products can be activated by removing tar products by heating in an inert gas stream, or through extraction using an appropriate solvent such as selenium oxide, or through a chemical reaction. Activated carbon with a large adsorption power can be produced by the activation process of raw materials that have been carbonized with high temperatures.

The first set of experiments involved the preparation of activated carbon from African star apple seed husk, oil seeds and whole seeds using zinc chloride ($ZnCl_2$) and phosphoric acid H_3PO_4 Vertical stainless steel reactor placed on the table; 10 g of precursor is weighed and poured into it. The precursors weighed were carbonized at 500°C for 2 hours in the furnace [42].

c. Activation is a treatment of charcoal which aims to enlarge the pore by breaking the hydrocarbon bonds or oxidizing surface molecules so that the charcoal undergoes changes in both physical and chemical characteristics, ie the surface area increases and affects the adsorption power. The product of carbonization cannot be applied as an adsorbent (because the shaft structure does not develop) without additional activation. The basic activation method consists of treating the oxidizing gas at high temperatures. The activation process produces carbon oxides which are spread on the surface of carbon due to the reaction between carbon and oxidizing agent. The main purpose of the activation process is to increase or expand the pore volume and enlarge the pore diameter that has formed in the carbonization process and to create several new pores. The interaction between the activating agent and the structure of carbon atoms resulting from carbonization is the mechanism of the activation process. During activation, carbon is burned in an atmosphere of oxidation which will increase the amount or volume of pore and surface area of the product through the process of eliminating or removing volatile pyrolysis products [1].

Agricultural wastes such as rice husks, corncobs and wheat straw are used as precursors for the production of activated carbon by chemical activation with NaCl. This characterization gives an idea of the surface, texture and functional properties of the activated carbon produced. The adsorption experiment showed a good capacity of activated carbon prepared for adsorption of methylene blue. The equilibrium data for adsorption of methylene blue fit into the Langmuir isotherm. The results show that agricultural waste can be used to produce low-cost adsorbents using a simple method. This adsorbent has the potential to purify drinking water in remote areas [43].

2. Activation Method

In general, the perfect carbonization process is heating raw materials in the absence of air to temperatures high enough to dry and evaporate compounds in carbon. In this process the thermal decomposition of the carbon-containing

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material takes place, and removes the non-carbon species. The activation process aims to increase the volume and enlarge the pore diameter after undergoing the carbonization process, and increasing absorption.

The activated carbon activation method has two methods, namely activated carbon activation can be done in 2 ways, namely chemical activation and physical activation

a. Chemical Activation

Chemical activation is the process of breaking the carbon chains of organic compounds by the use of chemicals. Chemical activation usually uses activating ingredients such as calcium chloride (CaCl2), magnesium chloride (MgCl2), zinc chloride (ZnCl), sodium hydroxide (NaOH), sodium carbonate (Na2CO) and sodium chloride (NaCl). In addition to mineral salts, various organic acids and bases such as sulfuric acid (H2SO4), hydrochloric acid (HCl), hypochloric acid (H3PO4), potassium hydroxide (KOH), and sodium hydroxide (NaOH) are used. The disadvantage of using minerals as activators lies in the washing process of the mineral materials which is sometimes difficult to be eliminated again by washing. While the advantage of using mineral ingredients as activators is a relatively short activation time, more activated carbon is produced and the adsorption power of an adsorbate will be better [1].

These activating substances function to degrade or hydrate organic molecules during the carbonization process, limit the formation of tar, assist the decomposition of organic compounds in subsequent activation, dehydrate water trapped in carbon cavities, help remove hydrocarbon deposits deposited during the carbonization process and protect the surface carbon so that the possibility of oxidation can be reduced.

b. Physical Activation

Physical activation is the process of breaking the carbon chains of organic compounds with the help of heat, steam and CO. Physical activation methods include using water vapor, carbon dioxide gas, oxygen, and nitrogen. These gases function to develop the existing cavity structure on the charcoal so that it expands its surface, eliminates volatile constituents and removes the production of tar or impurity hydrocarbons on the charcoal.

Physical activation can change carbonized material in a product that has an extraordinary surface area and pore structure. The purpose of this process is to increase the volume, expand the pore diameter that is formed during carbonization and can cause several new pores. Fluidized bed reactors can be used to process physics activation. This type of reactor has been used to manufacture activated carbon from rock.

The use of nitrogen gas during the activation process because nitrogen is an inert gas so that the combustion of carbon into ash and oxidation by heating can

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be further reduced, in addition to the activation of the gas will develop the existing cavity structure on the charcoal so that it expands its surface. An increase in the activation temperature in the range of 450°C-700°C can increase the specific surface area of activated carbon [1].

At this time many studies that have been used for activated carbon activation have been carried out such as

Table 3 activated carbon activation method from various sources

Source	Research That Has Been Done	Activated Carbon Activation Method	References
Tamarind wood	Characterisation of activated carbon prepared from tamarind wood for wastewater treatment	Chemical activation method using $ZnCl_2$ solution and carbonation with physical activation at temperatures between 350°C to 550°C.	[2]
Jatropha curcas fruit shell	Preparation of activated carbon derived from Jatropha curcas fruit shell by simple thermo-chemical activation and characterization of their physico-chemical properties	Chemical activation method using raw materials mixed with a solution of Potassium Bromide and carbonation with physical activation using nitrogen gas at a temperature of 300°C for 3 hours.	[5]
Olive-tree wood	Preparation of activated carbons from olive-tree wood revisited. I. Chemical activation with H_3PO_4	Chemical activation method using raw materials mixed with H_3PO_4 solution with a ratio of 3.5: 1 and carbonation with physical activation using nitrogen gas with a temperature of 350°C to 550°C with carbonation time for 1 hour.	[36]
Apamate wood	Synthesis and characterization of activated carbon from sawdust of Algarroba wood. 1. Physical activation and pyrolysis	The physics activation method is by carbonation using CO_2 and N_2 gas at temperatures of 200°C to 900°C. Carbonation time is from 1 hour to 2 hours.	[44]
Algarroba wood	Synthesis and characterization of activated carbon from	The physics activation method is by carbonation using CO_2 and N_2 gas at	[44]

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	sawdust of Algarroba wood. 1. Physical activation and pyrolysis	temperatures of 200°C to 900°C. Carbonation time is from 1 hour to 2 hours.	
Acacia mangium wood	Surface characterization and comparative adsorption properties of Cr(VI) on pyrolysed adsorbents of Acacia mangium wood and Phoenix dactylifera L. stone carbon	Physical activation method with carbonation process with nitrogen gas flow rate of 200 cm^3 / min in continuos with variable temperature and time of 300°C with a time of 30 minutes and 500°C with a time of 120 minutes.	[45]
Coconut Shell	Effect of Activation Temperature on the Quality of Coconut Shell-Based Activated Carbon	Physical activation methods are heating at temperatures of 500°C, 600°C, 700°C, 800°C, 900°C and 1000°C with a heating time of 3 hours using a furnace.	[46]
Walnut wood	Uptake of Reactive Black 5 by pumice and walnut activated carbon: Chemistry and adsorption mechanisms	Physical activation method of carbonation process using kilns at 900 ° C and a time of 2 hours.	[37]
Oak Wood	Local, cheep and nontoxic activated carbon as efficient adsorbent for the simultaneous removal of cadmium ions and malachite green: Optimization by surface response methodology	Physical activation method of the drying process at a temperature of 110°C and time for 6 hours.	[38]
rubber wood sawdust	Preparation of rubber wood sawdust-based activated carbon and its use as a filler of polyurethane matrix composites for microwave absorption	Chemical activation method using $ZnCl_2$ solution at a temperature of 500°C for 60 minutes. Comparison of $ZnCl_2$ / wood sawdust composition with a ratio of 1: 1, 1.5: 1 and 2: 1.	[35]
Paulownia wood	Preparation and characterization of activated carbons from Paulownia wood by	Chemical activation method using 85% H_3PO_4 solution.	[3]

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	chemical activation with H_3PO_4		
India shrub wood	Preparation of the activated carbon from India shrub wood and their application for methylene blue removal: modeling and optimization	Physical activation method of carbonation process using Horizontal Furnace with a temperature of 550°C for 1 hour using nitrogen gas.	[40]
Rice Husk	Physical Activation of Rice Husk Pyrolysis Char for the Production of High Surface Area Activated Carbons	Physical activation method of carbonation process using Fix Bed Reactor with temperatures reaching 1000°C and nitrogen and carbon dioxide gas injection.	[39]
coffee-shell	Preparation of coffee-shell activated carbon and its application for water vapor adsorption	Physical activation method of carbonation process using Tube furnace 573 K for 10 hours and injected with nitrogen and carbon dioxide gas.	[41]

3. Future Work

Studies on the characteristics of activated charcoal produced from a variety of biomass wastes are still much needed. Utilization of biomass waste to date has not been maximized. Current plantation development will also indirectly increase the amount of biomass waste produced. Therefore, a new breakthrough is needed in order to process biomass waste so that it can be utilized and not wasted. This is because each type of biomass has different properties that require different treatment to get the quality of activated charcoal that is as expected.

The search for the most effective and efficient activation procedures at a low cost and using environmentally friendly consumers is the main focus for future research. A number of procedures that have been developed today still consume relatively large amounts of energy and chemicals.

It is necessary to study the durability and de-activation mechanism of activated charcoal. This type of study is still very limited. However, this study is needed to determine the lifecycle and lifetime of activated charcoal products.

At this point, we would like to underline a few important points that might help researchers in the future. The cost factor for activated carbon production must be considered. Research on the evaluation costs of activated carbon production from biomass waste needs to be done. As a low-cost activated carbon synthesis with a large surface area and high mesopore density the adsorbent is preferred. This is one of the important criteria for deciding whether the choice of biomass waste is suitable for producing activated carbon on a large scale. The regeneration of spent active carbon studies needs to be done in detail, because it will increase economic viability. Based on contemporary survey literature, not much recovery of pollutants and activated carbon research has been reported. Research is not limited to laboratory studies of scale or batch adsorption studies; there must be some column studies and industrial bed adsorption studies must be done. Implementation of activated carbon derived from biomass waste in a large scale industry is a necessity at this time.

In future investigations, researchers need to carry out pilot studies on real industrial wastewater treatment through activated carbon reports. Investigation of simultaneous removal of many existing pollutants is needed to be done. Despite some of the weaknesses and challenges that exist today, extensive and great progress in this field can be expected in the future.

CONCLUSION

Having agricultural biomass waste as a precursor for the preparation of activated carbon has been reviewed based on a large number of relevant articles published so far. Activated carbon derived from biomass waste is an efficient adsorbent that can separate various organic and inorganic pollutants. Many low-cost adsorbents from biomass waste have been synthesized and characterized in the past. However, activated carbon is known to have a far better performance in treating industrial contamination and small scale household waste. Because the precursor materials obtained from biomass waste can be freely and abundantly accessed, the cost of producing activated carbon must be lower than coal-based activated carbon. Therefore, it can provide potentially inexpensive substitutes for existing commercial coal-based activated carbon. The conversion of abundant agricultural biomass waste into activated carbon can have several applications such as the manufacture of gas filters, drinking water filter beds, municipal wastewater treatment plants, processing dyes and metal ions containing industrial waste. This will add value to biomass waste and help with additional markets for their agricultural by-products and solve waste management problems. It can also offer solutions to the problem of binding carbon and greenhouse gases, and help reduce waste disposal costs.

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