

## Review article

# The Process of Activated Carbon from Coconut Shells Through Chemical Activation

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### Abstract

Carbon or charcoal can be made from biomass or cellulose-containing materials such as coconut shells or candlenut shells using a thermal process. One of the thermal process is pyrolysis, which in this process, the material is converted to carbon. The results of pyrolysis are in the form of three types of products namely solids (charcoal / carbon), gas (fuel gas) and liquid (bio-oil). Other products are gases such as carbon dioxide (CO<sub>2</sub>), methane (CH<sub>4</sub>) and some gases that have small contents. In general, the pyrolysis process takes place at temperatures above 300 ° C within 4-7 hours. Carbonized carbon or pyrolysis does not have a large adsorption capacity because the pore structure does not develop, so it is need activation process. One way to activate carbon is chemical activation. There is a need to know the best material for activating carbon through chemical process. This article aims to discuss the advantages and disadvantages of various types of chemical activation and to determine the promising chemical for activation. From various methods of chemical activation, the activator that promises to make activated carbon is Phosphoric Acid (H<sub>3</sub>PO<sub>4</sub>) because it can produce activated carbon which has a maximum micropore at operating conditions <450°C with a weight percent ratio between activator and sample around 29 - 52%.

## INTRODUCTION

Coconut is a multi-benefit plant that is quite familiar to most Indonesians. This plant is used almost all parts by human, so it is considered a multipurpose plant. Coconut (*Cocos nucifera*) is widely grown in most of the island and reaches a height of 30 m. Utilization of coconuts for various daily food needs as well as for agriculture-industrial commodities, leaving waste in the form of shell or coconut shell (Eskak, 2015).

The need for activated carbon is increasing due to its applications, such as for industries and various human aids. In industrial area, activated carbon can be used for drug industry, food, beverages, water

treatment (water purification) and others. Nearly 70% of activated carbon products are used for refining in the coconut oil, pharmaceutical and chemical sectors. Raw materials that can be made into activated carbon are all that contain carbon, both derived from plants, animals or mine carbon. These materials are various types of wood, rice husks, animal bones, coal, coconut shells, coffee bean skins. When compared these materials, coconut shell is the best material that can be made into activated carbon as they have a lot of micropores, low ash content, high water solubility and high reactivity (Pambayun *et al.*, 2013).

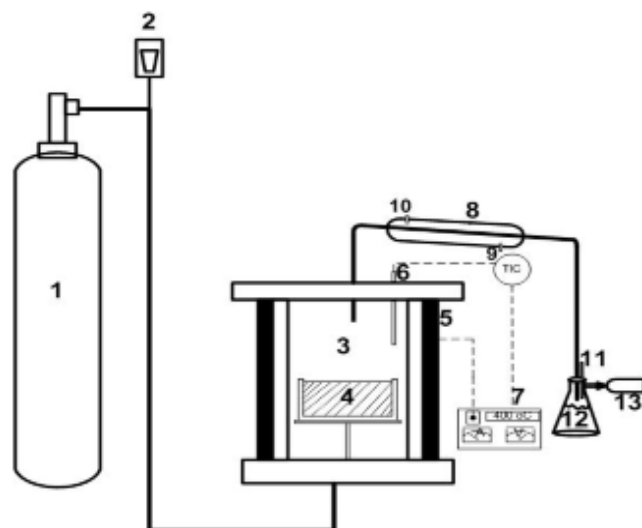
The effectiveness of activated carbon from various types of high carbon content mostly depends on the activation process. The activator can expand the surface of carbon by opening closed pores so that the adsorption power is increase. Activated carbon is composed of amorphous particles resulting in a complex arrangement of irregular shapes and interconnected with pores or surface area between the particles, both surface sizes of 300 - 2000 m<sup>2</sup>/g or those with sizes of more than 2000 m<sup>2</sup>/g. (Sani, 2011). Therefore, it is important to know the best activator when activating the carbon. This review paper will discuss and explain the benefit and drawback of the method for activating carbon through chemical process.

### PROCESSING OF CARBONIZATION

Carbon is a porous solid material that is the result of combustion of materials containing carbon elements, while activated carbon is carbon that is activated by immersion in chemicals or by flowing hot steam into the material, so that the pore material becomes more open with a surface area ranging between 300 to 2000 m<sup>2</sup>/g. The adsorption properties are selective, depending on the size or volume of the pores and surface area. Absorption of activated carbon is enormous, which is 25-100% of the weight of activated carbon. Carbon or charcoal can be made from biomass or cellulose-containing materials such as coconut shells or candlenut shells using a thermal process called pyrolysis, where crushed material becomes smaller and burned to produce carbon.

Some technological equipment regarding pyrolysis reactors with differences configuration has been proposed, designed, and tested. Erliyanti *et al.*, (2015) have designed and tested a pyrolysis reactor adapted from several report (Leung *et al.*, 2002; Pantea *et al.*, 2003). The reactor is a semi batch reactor where the temperature and heating rate can be controlled and carried out at 900°C. The pyrolysis reactor is shown in Figure 1.

Pyrolysis reactor consists of main components and supporting components. The pyrolysis reactor is equipped with a PID controller. The main component of a pyrolysis reactor is an unstirred semi batch reactor (1.150L) made of stainless steel (3). The supporting components are nitrogen tube (1), gas flow-meter (2), sample place (4), electrical heater (5), thermocouple (6), controlling instrument (7), condenser (8), cooler water inlet (9), warm water outlet (10), thermometer (11), liquid cross-section (12), and gass cross-section (13).



**Figure 1.** Semi batch pyrolysis reactor (Adapted from reference Erliyanti, *et al.*, 2015)

Pyrolysis is a process of decomposition of a material at high temperatures without the presence of air or with limited air. The thermal decomposition process in pyrolysis is also often referred to as devolatilization. Pyrolysis, also termed thermal distillation or can be called thermolysis is a chemical decomposition process by using heating without the presence of oxygen. Pyrolysis can be called a reverse polymerization process, thermal depolymerization or polymer cracking. One of the advantages of the pyrolysis process is that it can treat wastes, which they are difficult to recycle, so they can produce products that can be reused (Scheirs and Kaminsky, 2006). The results of pyrolysis are in the form of three types of products namely solids (charcoal /carbon), gas (fuel gas) and liquid (bio-oil). And generally, the pyrolysis process takes place at temperatures above 300 ° C within 4-7 hours. But this situation is very dependent on raw materials and how they are made (Demirbas, 2005).

The process of making activated carbon consists of three stages, which are:

1. Dehydration, is a process of removing water from the raw material when it is heated to a temperature of 170 °C
2. Carbonization, is the process of breaking down organic materials into carbon with temperatures used above 170 °C will produce CO and CO<sub>2</sub>, while at 275°C, decomposition produces ash, methanol and other by products, but the formation of carbon occurs at temperatures 400-600 °C.
3. Activation occurs through carbon decomposition and expansion of pores can be done with steam or CO<sub>2</sub> as an activator. (Arsyad & Hamdi., 2010).

**QUALITY STANDARD OF ACTIVATED CARBON**

Activated carbon that have functions as an adsorbent certainly has parameters for the activated carbon material produced, such as; has microporous structure, has large surface area, etc. Table 1 show the standard requirements of activated carbon.

**Table 1.** Properties of Activated Carbon

No	Description	Unit	Requirements	
			Granular	Powder
1	The lost part on heating 950°C	%	Max 15	Max 25
2	Water	%	Max 4,4	Max 15
3	Ash	%	Max 2,5	Max 10
4	Non-carbonated parts	-	There's no	There's no
5	Adsorption of I <sub>2</sub>	Mg/g	Min. 750	Min 750
6	Pure activated carbon,%	-	Min. 80	Min. 65
7	Adsorption od Benzene, %	-	Min. 25	-
8	Adsorption of metylen blue	MI/g	Min. 60	Min. 120
9	Bulk density	g/ml	0,45-0,55	0,30-0,35
10	Passed 325% mesh size	-	-	Min. 90
11	Mesh distance , %	-	90	-
12	Violence, %	-	80	-

**PROCESS OF ACTIVATION**

The carbon after pyrolysis does not have a large adsorption capacity because the pore structure does not develop, the pore structure will be improved during the activation process. The activation process aims to convert carbon to activated carbon which has a large number of pores that are randomly distributed in various shapes and sizes and result in a wide surface area. There are 3 types of activation processes, including physical process, chemical process, and physical - chemical process.

**Table 2.** Activator used by researchers.

Aktivation	Activator	Source
<b>Chemical</b>	ZnCl <sub>2</sub> dan CaCl <sub>2</sub>	Jamilatun & Setyawan, 2014
	NaCl, NaOH, HCl	Hartanto & Ratnawati, 2010
	HCl dan NaOH	Karyani & Malakauseya, 2014
	H <sub>3</sub> PO <sub>4</sub>	Kurniati,2008
	NaCl, NaOH, H <sub>2</sub> SO <sub>4</sub>	Setiawati & Suroto,2010
	KOH	Atmayudha,2007
	KOH	Zulkifli, <i>et al.</i> , 2015
<b>Physical</b>	Gas CO <sub>2</sub>	Ibrahim <i>et al.</i> , 2014
	Gas H <sub>3</sub> PO <sub>4</sub>	Hendra <i>et al.</i> , 2015
<b>Chemical - Physical</b>	ZnCl <sub>2</sub> , Na <sub>2</sub> CO <sub>3</sub> & Gas N <sub>2</sub>	Pambayun,2013
	NaOH dan O <sub>2</sub>	(Aryani <i>et al.</i> , 2019)

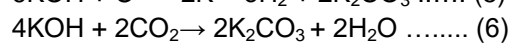
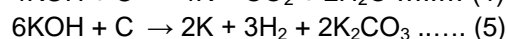
a. Physical Activation

The process of breaking the carbon chains of organic compounds with the help of heat, steam and CO<sub>2</sub>. In physical activation carbon is

usually heated in a furnace at a temperature of 800 - 900°C. Oxidation with air at low temperatures is an exothermic reaction so it is difficult to control it. While heating with steam or CO<sub>2</sub> at high temperatures is an endothermic reaction so it is easier to control and most commonly used (Arsyad & Hamdi., 2010).

b. Chemical Activation

Chemical activation is the process of breaking the carbon chains of organic compounds by the use of chemicals. Some of the advantages of chemical activation over physical activation are that the chemical activation process makes it possible to produce a very high surface area of activated carbon. Activators used are chemicals such as alkali metal hydroxides, carbonate salts, chlorides, sulfates, phosphates from alkaline earth metals especially ZnCl<sub>2</sub>, inorganic acids such as H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> (Udyani, 2019). The following is an example of a chemical reaction that occurs in the process of making activated carbon using KOH as an activator:



The following is an example of a chemical reaction that occurs in KOH added to carbon which will spread throughout the carbon surface, causing partial oxidation of carbon by oxygen. (Yuliusman, 2016)

c. Physicals – Chemical Activation

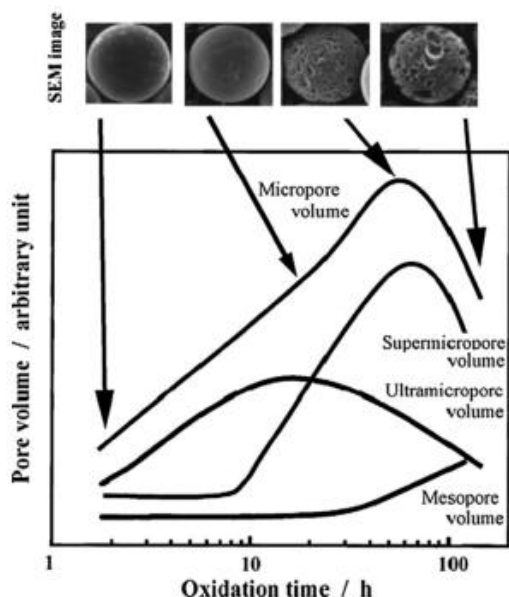
This activation is a combination of chemical activation and physical activation. Activation of activated carbon is carried out combustion with a certain temperature until it becomes carbon then soaked with chemical solution until it becomes activated carbon.

**ACTIVATED CARBONS**

Activated carbons is indicated by a large number of open pores on the carbon form. This is usually indicated by surface area and pore size distribution (Marsh and Rodriguez-Reinoso, 2006). Activated carbon is mostly used as an adsorbent, so pore texture is the most important thing to be controlled. To develop a micropores with a width of less than 2 nm, a process called an activation process is needed.

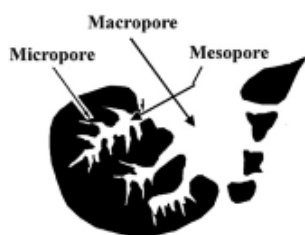
Activation process can be carried out physically (gas activation, e.g., diluted oxygen gas, air, steam, CO<sub>2</sub>, etc.) and chemical activation using H<sub>3</sub>PO<sub>4</sub>, ZNCl<sub>2</sub>, KOH, etc. (Inagaki, 2013). During the gas

activation process, the size of the pores changes from micropores to mesopores and finally become macropores. Figure 2 shows the changes in pore size during air activation of glass-like carbon spheres (Adapted from references Inagaki, 2013 and Inagaki *et al.*, 2006).



**Figure 2.** Pore development during gas activation on glass-like carbon sphere (Adapted from references Inagaki, 2013 and Inagaki *et al.*, 2006)

The structure of activated carbon pores is shown in Figure 3 (adapted from reference Inagaki, 2013), where macropores (width > 50 nm) and mesopores usually are coexisted with micropores.



**Figure 3.** Model of pore structure (Adapted from reference Inagaki, 2013)

Activation of carbon using the impregnation method with the addition of activators  $ZnCl_2$ ,  $KOH$ , and  $H_3PO_4$  has been reported by Esterlita and Herlina (2015). A comparison between carbon activations using activators and without activator is shown in Figure 4 (adapted from reference Esterlita and Herlina, 2015).

In Figure 4 shows that the activation of carbon using several activators produces black activated carbon. Carbon colors without activators tend to be gray. This is caused by the amount of ash contained on the carbon produced.

## THE USE OF ACTIVATED CARBON

Activated carbon is used in various fields, including:

### 1. Industry

More than 70% activated carbon products are used in the industrial sector. The main uses of activated carbon are for purification of solutions, such as the sugar industry, syrup, drinking water, vegetables, fats, oils, alcoholic beverages, chemicals and pharmaceuticals; absorbent poison gas in the mask; deodorizing in refrigeration systems; absorbers of fuel vapor emissions in the automotive as well as cigarette filters.

### 2. Health

In the health sector, activated carbon is used in the treatment of external poisoning and secretion diarrhea therapy. In oral poisoning, to avoid the absorption of a number of poisons that still exist in the digestive tract can be done by giving adsorbents. The most effective and less dangerous adsorbent so that the most widely used is activated carbon.

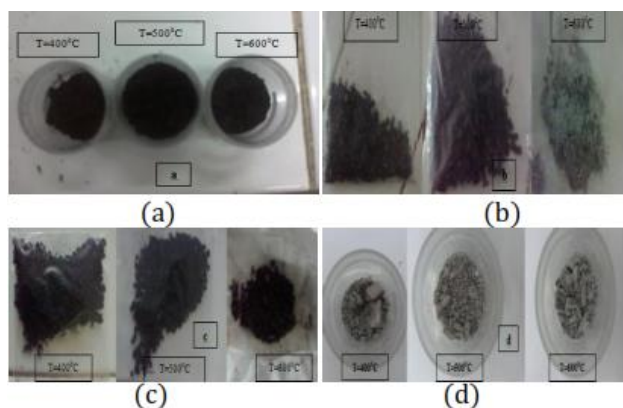
### 3. Environment

Kadirvelu *et al.*, (2001) have proven the ability of activated carbon as an adsorbent for Hg, Pb, Cd, Ni, Cu metals in industrial wastewater in radiators, nickel plating and copper coatings. The ability of activated carbon as a metal remover is influenced by the pH and carbon concentration. An increase in carbon content increases the percent adsorption of activated carbon to metal ions. The use of activated carbon is very important in the process of purifying water and air. In the water purification process, activated carbon in addition to adsorbing metals such as iron, copper, nickel also can eliminate odors, colors and flavors found in a solution or wastewater. In some countries activated carbon is reported to have been used as an absorber of pesticide residues in the water purification process to get pesticide-free drinking water.

## ACTIVATOR COMPARISON

In the research of Jamilatun & Setyawan (2014) activating activated carbon using  $ZnCl_2$  and  $CaCl_2$  compounds obtained the best quality of activated carbon was obtained at  $800^\circ C$  with a moisture content of 1.3%, ash content of 0.60% met SNI 0258-79 standards and had absorption of iodine levels of 580.0 mg/g that meets SNI 06-3730 standards. In Hartanto and Ratnawati (2010) research activating carbon using  $NaCl$ ,  $NaOH$ ,  $HCl$  compounds, the results of which are activated charcoal with the best characteristics are  $NaOH$

activator for 4 hours, with the result that the water content of activated carbon is 3.6%, absorption of I<sub>2</sub> is 851 , 8797 mg/g and these results are in accordance with SNI. In Karyani & Malakauseya research (2014) activating carbon using HCl and NaOH compounds, the results of which were missing parts of 10.02%, water content of 2.91%, ash content of 1.10% and absorption of I<sub>2</sub> solution is 800 mg / g.



**Figure 4.** Activated carbon using an activator: (a) KOH, (b) ZnCl<sub>2</sub>, and (c) H<sub>3</sub>PO<sub>4</sub>, (d) carbon without activator (adapted from reference Esterlita and Herlina, 2015).

Kurniati (2008) reported that to activate carbon using H<sub>3</sub>PO<sub>4</sub> obtained the best condition at 22 hours immersion with 9% activator concentration, with the results: Moisture content; 7.36%, ash content; 2.77%, Volatile Matter; 8.21%, Iodine absorption 19.80%. In the research of Setiawati & Suroto (2010) activating carbon using NaCl, NaOH, H<sub>2</sub>SO<sub>4</sub> compounds, the best quality of activated carbon was obtained from NaCl concentration of 20% with characters in the form of water content of 4.88%, ash content of 3.54%, the part lost on heating 950<sup>0</sup>C 14.63%, and the absorption of iodine 761.07 mg / g. In Atmayudha's research (2007) activating carbon using KOH compound, the results were surface area of 111.9 m<sup>2</sup> /g, total pore volume of 0.03013 cm<sup>3</sup>/g, average pore diameter of 80.8. In the Zulkifli *et al.*, (2015) study activating carbon using KOH compounds, the results obtained are coconut shell activated carbon which has an amorphous structure marked by the formation of two sloping peaks at an angle of 2θ = 23.586° and 44.203° at the concentration of 1 M KOH activating agent, while at concentrations with activating agents 3 M KOH angles of 2θ = 24,659° and 45,438°.

As mentioned above, there are 2 kinds of carbonization process, as called; the carbonization process and pyrolysis or thermal processes. The advantage of this thermal process is that it uses a

pyrolysis process which is much simpler in operation and cheaper than other processes and can use raw materials that are less expensive and environmentally friendly. While the disadvantage of this process is controlling the oxygen supplied to the furnace / kiln. As for the activated carbon activation process, there are 3 kinds of processes including physical, chemical, and physical-chemical activation. The advantage of the chemical activation process is that it is easier than others, first by soaking / stirring for several hours, then the second chemical compound used as an activator is easy to obtain. The last benefit is the one of the chemical compounds such as ZnCl<sub>2</sub> acts as an inhibitor of carbon formation, and the equipment used is easily assembled.

## CONCLUSION

Based on discussion above about the various methods of chemical activation, it seems that the promising activator for activated carbon is Phosphoric Acid (H<sub>3</sub>PO<sub>4</sub>). There are some benefits of using Phosphoric Acid. Firstly, H<sub>3</sub>PO<sub>4</sub> activator can produce activated carbon which has a maximum micropore at operating conditions <450<sup>0</sup>C with a weight percent ratio between activator and sample around 29 - 52%. Secondly, because H<sub>3</sub>PO<sub>4</sub> is liquid with a solubility of 5.48 g / ml than ZnCl<sub>2</sub> which has a solubility of 4.32 g / ml. Thirdly, the price of H<sub>3</sub>PO<sub>4</sub> is cheaper, so it is more economical.

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