Fabrication and Characterization of Activated Carbon from Charcoal Coconut Shell Minahasa, Indonesia

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Abstract. Has performed the fabrication and characterization of activated carbon from charcoal coconut shell with the combination of activator HCl and NaOH. Fabrication is done in two stages, the characterization and activation. Conducted through pyrolysis carbonization batch at a temperature of 350 oC for 6 h to get charcoal. Activation is done chemically by immersing charcoal in the activator HCl for 24 h. Activated carbon with activator HCl back in activated with activator NaOH for 24 h. Characterization of activated carbon include the determination of functional groups with FTIR, morphology, and topography by SEM, the type of elements and elemental composition by EDS, specific surface area, total pore volume, and pore radius with methods BET and the identification of the structure crystallinity by XRD. Characteristics of activated carbon products with a combination of HCl and NaOH activator showed changes in the physicochemical properties of the base material charcoal coconut shell into activated carbon. The resulting activated carbon is polar with carbon element content of 94.93 % and has a smooth surface porous with a surface area of 5.041 m²/g, pore volume of 5.229 cc/g and pore radius of 20.74 Å. In addition, the activated carbon produced is also semi-crystal with a hexagonal crystal structure and pore structure of mesoporous size.

1. Introduction

Active carbon fabrication is done in two stages, carbonization and activation. Carbonization is done through pyrolysis process. Pyrolysis is a chemical decomposition of organic matter by heating at high temperatures with little or no oxygen or other reagents, in which the raw material will break down the chemical structure. Aims to release the combustible substances contained in biomass. In the pyrolysis process occurs the recycling of materials that can be described at high temperatures resulting in more valuable products.

Charcoal is the main product resulting from pyrolysis process with its by-products of liquid smoke and tar. Charcoal consists of several constituent components, namely bound carbon, ash, water, nitrogen, and sulfur. Most of the pores are still covered with hydrocarbons, tar, and other organic compounds. Charcoal of pyrolysis is potentially processed into activated carbon. Charcoal produced from the pyrolysis process has a bending nature such as activated carbon, but its surface area is still low and its adsorption power is very small. It is, therefore necessary to activate the process to enlarge the surface area and build porosity.
The activation process is a process of treating carbon products, causing the release of hydrocarbons, tar and other organic compounds that are still attached to carbonized charcoal. In this process occurs the formation of pores that are still closed and increasing the size and number of small pores that have been formed.

Chemical activation is activated by the use of chemicals called activators. Commonly used activators are alkali metal hydroxides, chlorides, sulfates, phosphates of alkaline earth metals and inorganic acids.

Chemical activation is carried out by gradually soaking the carbonized charcoal into the chemical solution HCl and NaOH; useful in inhibiting tar formation, eliminating other volatile products, and increasing the number of pores in activated carbon. The activation treatment of the activated carbon fabrication process is intended to investigate the relationship of microstructure, chemical composition, physical properties of the activated carbon chemistry.

The data obtained in the form of crystallographic system (crystalline structure) through X-ray Diffraction (XRD) technique, surface topography spectrum (microstructure) through Scanning Electron Microscopy (SEM) analysis technique, spectrum showing element composition based on energy level through Energy Dispersive Spectroscopy (EDS), a functional group with Fourier Transform Infra Red (FTIR) analysis.

2. Experimental Details

2.1. Material
The raw materials used in this study are coconut shell originating from Minahasa district as source of carbon raw material, HCl (p.amerck), PVA (p.amerck), NaOH (p.amerck), universal indicator, Whatmann no.42 paper, and aquades.

2.2. Tools/Instruments
The tool used includes a number of commonly used laboratory glassware, mortar agart, 100 mesh sieve (USA standard Testing Sieve), pyrolysis reactor, gravity vonvection model oven, electric furnace Carbolite model 2132 (Max Temperature 1200oC), Balance AND GR-200, thermometers, clamps, magnets, pellets, hot plate (stir & heat), Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) Bruker Tests Vega 3SB, Energy Dispersive Spectroscopy (EDS) JEOL JED-2300, X-Ray Diffraction (XRD) Rigaku Mini Flex II, Fourier Transform Infrared (FTIR) Shimadzu model IR-Prestige-21 and Quantachrome Instruments.

2.3. Sample preparation
The sample used is coconut shell of coconut-in type. The coconut shell was taken from the remaining copra production in the plantation in Minahasa district randomly. The coconut shell is cleaned from the coir using a sandpaper machine. Furthermore, coconut shell made into pieces with the smaller size. Then the coconut shell is dried in the sun for 3 days.

2.4. Active Carbon Fabrication
Samples of coconut shell pyrolysis batch for 6 hours at 350 oC using pyrolysis reactor. Pyrolysis charcoal is pounded into a powder using porcelain lumping. The charcoal powder is then sieved with a 100 mesh sieve.

The charcoal powder passage was activated with 0.1 M HCl at room temperature for 24 h with a volume ratio of 1:10 g / mL.

The charcoal powder of HCl activation is further washed with distilled water until the washing water shows a constant pH and filtered with filter paper. The activated charcoal HCl powder was dried in an oven at 110 °C for 2 hours and then cooled in the desiccator for 30 minutes.

The activated charcoal HCl powder was reactivated with 1M NaOH at room temperature for 24 hours with a volume ratio of 1:20 g / mL. The charcoal powder resulted from activation of HCl and
NaOH is then washed with distilled water until the washing water showed a constant pH and filtered with filter paper. The charcoal powder resulted from activation of HCl and NaOH was dried in an oven at 110 °C for 24 hours and then cooled in the oven for 1 hour. The activated charcoal powder is called activated carbon.

3. Results and Discussion

3.1. Fourier Transform Infra Red (FTIR) Analysis of Coconut Shell Charcoal Before and After Activated by Combination of HCl and NaOH Activators.

Figure 1. Infrared spectral band absorption band numbers before and after activation with a combination of HCl and NaOH activators showed a shift in absorption band, the appearance of new absorption bands and the loss of absorbing bands indicated that the activation process could remove the impurity group present on the charcoal surface and form new pores on the charcoal surface. The serpent band showing the vibration of the -OH, C = C, C = O and C-O groups indicates that charcoal before and after activation tends to be polar. In addition, the presence of aromatic compounds shows the preparation of hexagonal structures in charcoal before and after activation.

![Figure 1. Rampe et al.](image)

3.2. Scanning Electron Microscopy (SEM) Analysis of Coconut Shell Charcoal Before and After Activated by Combination of HCl and NaOH Activators

Figure 2 shows the distribution of unfilled particles, coarse charcoal surfaces, and pore visible on the charcoal surface. This is because the charcoal surface that has not been activated there are impurities in the form of hydrocarbons, tar and other compounds formed at the time of carbonization so that the charcoal surface becomes rough and cause the closed pore of the charcoal.

![Figure 2. Rampe et al.](image)

Figure 3 shows a nearly uniform particle distribution, a smooth charcoal surface and a pore visible on the charcoal surface. This is because the impurities found on the charcoal surface have been lost
during the activation process. During the activation process, the activator reacts with the charcoal oxidizing and eroding the hydrocarbons, tar and other compounds attached to the charcoal surface so that the charcoal surface becomes smooth, the pores on the charcoal surface become exposed, the formation of new pores and the increase in pore diameter and volume on the charcoal surface.

**Figure 3.** Rampe et al

3.3. *Analysis of Energy Dispersive Spectroscopy (EDS) Coconut Shell Before and After Activated by Combination of HCl and NaOH Activators*

Figure 4. Energy Dispersive Spectroscopy (EDS) analysis results indicated that charcoal activated with combination of HCl and NaOH activators increased carbon purity by 6.67%. Increased purity of carbon affects the adsorption capacity of an activated carbon, the higher the carbon purity the greater the adsorption power.

**Figure 4.** Rampe et al

3.4. *X-ray Diffraction (XRD) Analysis Coconut Shell Before and After Activated by Combination of HCl and NaOH Activators*

Figure 5 shows 2θ per corner shift and a decrease in the intensity of the main peak indicates that during the activation process the chemical compounds present in the charcoal begin to degrade but have not changed the crystallinity structure. The occurrence of 2θsudut angle shift and decreased intensity is thought to be a reflection of the plane of the hexagonal crystallinity structure. The blunt main peak with low intensity indicates the presence of low crystalline (semi-crystalline) structures, where the number of semi-crystalline phases formed is directly related to the many mesoporous structures present in the charcoal.

**Figure 5.** Rampe et al.
The result of X-ray Diffraction (XRD) analysis showed that charcoal before and after activation was the semi crystalline with hexagonal-shaped crystal structure and mesoporous pore structure.

3.5. *Analysis of Brunauer-Emmet-Teller (BET) Coconut Shell Before and After Activated by Combination of HCl and NaOH Activators*

| Sample                        | Surface Area (m²/g) | Pore Volume (cc/g) | Pore Radius (Å) |
|-------------------------------|---------------------|--------------------|-----------------|
| Charcoal before activation    | 2.776               | 3.240              | 23.34           |
| Charcoal after activation     | 5.041               | 5.229              | 20.74           |

Table 1 shows activated charcoal with HCl and NaOH combination activators having a larger surface area and pore volume, but the pore radius is smaller than that of unactivated char. The greater surface area and the volume of activated charcoal pores are caused due to impurities present on the surface and the pore of charcoal has disappeared. The smaller activated charcoal pore radius is due to the erosion and oxidation of the charcoal surface by activators during the activation process. Charcoal pore size distribution before and after activation with activator combination of HCl and NaOH was categorized as having mesoporous pore size because it has pore radius> 20 Å.

4. Conclusion

In conclusion, this study shows characteristics of activated carbon products with a combination of HCl and NaOH activator showed changes in the physicochemical properties of the base material charcoal coconut shell into activated carbon. The resulting activated carbon is polar with carbon element content of 94.93 % and has a smooth surface porous with a surface area of 5.041 m²/g, pore volume of 5.229 cc/g and pore radius of 20.74 Å. In Addition, the activated carbon produced is also semi crystal with a hexagonal crystal structure and pore structure of mesoporous size.

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