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Characterization of Activated Carbon from White Snapper Scales (*Lates calcarifer*) Waste

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ABSTRACT

Research on the characterization of activated carbon of white snapper (Lates calcarifer) scales waste through calcination and activation process. The purpose of the study was to conduct: a surface acidity test before activation, the calcination process and activation of activated carbon from fish scales, and an acidity test after activation. Fish scales were put in a furnace at 350 °C for 2 hours, cooled, and sieved with a 100 mesh sieve and characterized by XRD. The results obtained were three peaks with the strongest intensity at 2θ regions of 26.45°, 32.69°, and 33.60°, respectively. The surface acidity of carbon solids before and after activation were (4.48×10^{-3} and 7.77×10^{-3}) mol/g, respectively. The activation process of white snapper scales (Lates calcarifer), increases the surface area of carbon can react with other chemicals. Subsequently, it was calcined at 450 °C under vacuum conditions for 2 hours with N_2 gas, and characterized by XRD. The results showed a shift in 2θ peaks of 26.52°, 29.66°, and 32.77°, respectively. The carbon was activated by soaking with an activator 3 M KOH for 15 hours, washed with distilled water, and dried in an oven at 110°C for 1 hour, characterized by XRD, and the peaks with the strongest intensity were obtained at 26.22°C, 29.12°C, and 32.43°C, with increasing basal spacing.

1. Introduction

Carbon is the main element of activated carbon, which is about 85-95%. Activated carbon in the form of wood charcoal has been used for centuries. In Egypt, charcoal was used around 1500 BC as an adsorbent for medicinal purposes and also as a purification agent. Production of activated carbon on an industrial scale first began around 1900 and was applied to the sugar refining industry. Activated carbon is prepared through the carbonization of a mixture of materials of vegetable origin with metal chlorides or by activation of the materials through combustion which produces CO2 and water vapor. Carbon gas adsorbents became a concern during World War I because they were used as protective gas masks against the risk of harmful gases and vapors. Activated carbon is a microcrystalline, non-graphitic form of carbon that has been processed to develop its internal porosity. This porosity results in a surface area that provides the ability to adsorb gases and vapors from dissolved composite gases or substances dispersed from liquids. Almost any carbonaceous material can be used to produce activated carbon. Activated carbon has been made from animal bones, hard and softwoods, rice husks, refinery residues, peat, lignin, coal, tar, coal, field, and carbon black [1].

Unlike diamond and graphite whose composition consists of pure carbon, activated charcoal is a carbon material that is still mixed with other elements, usually hydrogen and oxygen or others depending on the elements that form it. Activated carbon is also called porous carbon solids, namely carbon in non-graphite form which has an internal surface area of 300-3500 m^2/g [2].

According to [3], the manufacture of activated carbon takes place in 3 stages, namely: dehydration, carbonization, and activation. This process is carried out by heating the raw material to evaporate all water content and lower the humidity in the raw material. The product produced in the dehydration process is dry raw material because the water content has evaporated.

The carbonization process is basically to reduce the hydrogen and oxygen elements bound in the raw material so that only carbon is the dominant element. During this process, non-carbon elements such as hydrogen and oxygen are released in the form of gases and liberated atoms. To determine the resulting crystal structure, the X-ray diffraction method is used. The basis of this method is the peculiarity of the distance between crystal planes in each different crystal. The carbonization process will produce 3 main components, namely carbon or charcoal, tar, and gas. The carbonization stage will produce carbon that has a weak pore structure because the crystal structure is irregular so there are voids that are still filled by the constituent elements of the raw material. These elements cover the pores so that the adsorption ability is low. Therefore, charcoal still requires improvement of its porous structure through the activation process [3].

In some regions in Indonesia, white snapper is known by several names such as pelak, petakan, cabek, cabik (Central Java and East Java), dubit tekong (Madura), talungtar, pica-pica, kaca-kaca (Sulawesi). The characteristics of white snapper are an elongated and flat body, blackish in color on the back, while the belly is white. The base of the caudal fin is widened. The caudal fin is rounded, while the dorsal fin has 3 hard fingers and 7-8 soft fingers. The mouth is wide with fine and sharp teeth. The eyes are bright red in color [4].

As one of the export commodities, the demand for this type of fish is quite high in foreign markets. Increased production of sea bass will certainly lead to an increase in fish scale waste. As a result, this waste causes environmental problems such as unpleasant odors. Therefore, a study is needed on the utilization of fish scale waste as an adsorbent. The components contained in fish scales include 70% water, 27% protein in the form of collagen, 1% fat, and 2% ash content [5].

Based on the above background, this research aims to characterize the carbon scales of white snapper (*Lates calcarifer*), before and after activation, and test the acidity of its surface before and after activation.

2. Research Methodology

2.1. Tools and Materials

The tools used in the research include A set of glassware (Pyrex), an XRD diffractometer (Shimadzu XD 160), an Oven (Memmert), a Desiccator (Merck), a Scraper, a Sieve (100 mesh), Analytical balance, and Furnace (Barnstead Thermolyne 47900 Furnace). The materials used include white snapper scales (*Lates calcarifer*), distilled water, KOH (p.a Merck), nitrogen gas, and NH_3 (p.a Merck).

2.2. Sample preparation

White snapper scales (*Lates calcarifer*) were washed with distilled water and dried in an oven at 110 ^oC for 3 hours. After drying, it was put into a furnace at 350 ^oC for 2 hours. The resulting charcoal was then pulverized, sieved with a sieve (100 mesh), and characterized by XRD.

2.3. Surface acidity test before activation

Charcoal produced from the scales of white snapper (*Lates calcarifer*) was taken 0.2 g, put in a container, and placed in a desiccator containing ammonia adsorbate. The desiccator was closed for 24 hours, then removed and left for 2 hours in the open to release the physically adsorbed base. The weight of the carbon that had adsorbed the base was measured to obtain the moles of base adsorbed on the charcoal.

2.4. Calcination and activation of activated scales of white snapper carbon

The charcoal was placed in a calcination device heated at 450 ^{o}C and supplied with nitrogen gas, for 2 hours. The calcined charcoal was characterized by XRD. Activation was carried out by soaking 30 g of charcoal from the scales of white snapper (*Lates calcarifer*) in a 3M KOH activator reagent for 15 hours, filtered, and washed with distilled water. The resulting activated carbon was oven-dried at 110 ^{o}C for 1 hour, then cooled in a desiccator and characterized by XRD.

2.5. Acidity test after activation

Activated carbon was taken as much as 0.2 g and put into a container, then placed in a desiccator containing ammonia adsorbate. The desiccator was closed for 24 hours, then removed and left for 2 hours in the open to release the physically adsorbed base. The weight of the activated carbon that had adsorbed the base was measured to obtain the moles of base adsorbed on the carbon.

3. Result and Discussion

White snapper scales (*Lates calcarifer*) were washed with distilled water, dried in an oven at 110 ^{0}C for 3 hours. After the dehydration process, the *Lates calcarifer* scales were carbonised in a furnace at 350 ^{o}C for 2 hours. The carbonisation process is a process to convert organic material into carbon, so that the complex compounds that make up the organic material break down and obtain material that only contains carbon [2]. The resulting carbon is then pulverized, sieved with a sieve (100 mesh) to obtain a fine and homogeneous particle size. The more carbon pores will increase the surface area so that adsorption will be better. Xray diffraction analysis was performed using a Shimadzu XRD 160X-Ray Difractometer with voltage 40 kV, current 30 mA, slit 1, scan range 15-75 deg, scan speed 2 deg/min and Cu radiation source with wavelength 1.54060 Å. The results of carbon characterization by XRD are shown in Fig 1.

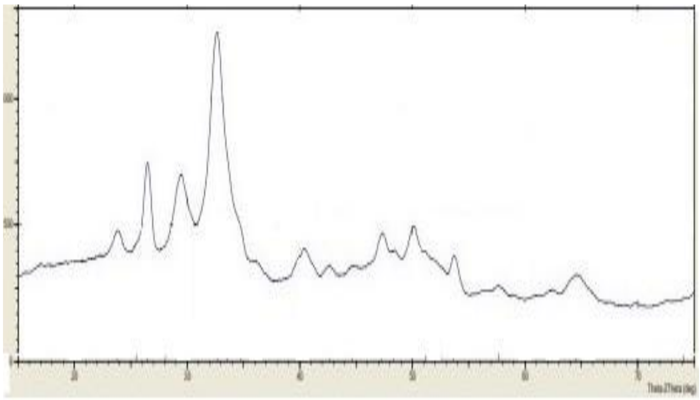

Fig 1. XRD diffractogram of fish scale charcoal

Based on the XRD diffractogram shown in Figure 1, the crystallinity data for the charcoal sample obtained three peaks with the strongest intensity at 2θ angles of 26.45^{o}, 32.69^{o}, and 33.60^{o}, respectively.

3.1. Determination of Surface Acidity of Solids

Determination of carbon surface acidity was carried out by gravimetric method, namely ammonia adsorption. The surface acidity of the activated carbon obtained is proportional to the amount of ammonia vapor adsorbed, where the adsorption process takes advantage of the vacuum state in the desiccator so that no other bases and water vapor are adsorbed besides ammonia. The results obtained are shown in Table 1.

Table 1. Comparison of the acidity of adsorbed ammonia-weight carbon before and after activation

Carbon Sample	Weight adsorbed (10^{-3}) (mol/g)
Before activation	4.86
After activation	7.77

Table 1. shows the acidity value of carbon before activation was 4.86×10^{-3} mol/g and after activation was 7.77×10^{-3} mol/g. This shows that the effect of activation increases the acidity of activated carbon. The acidic character of the activated carbon surface becomes more effective in absorbing ammonia bases. Activation causes the reduction of impurities that cover the acid centre (both Bronsted acid and Lewis acid sites), usually water which will increase the ability of active sites of adsorption and expand the pores of activated carbon. This is consistent with the carbon diffractogram data before and after activation, which shows a shift in the 2θ peak but with higher intensity.

3.2. Calcination and Activation of White Snapper Scales Carbon

The calcination process was carried out by heating the carbon at 450 °C for 2 hours. Carbon is placed in a reactor that has been connected to a nitrogen tube, placed in a furnace whose temperature has been set, also connected to a regulator to align the furnace temperature with a predetermined temperature logger, which is also connected to a stabilizer to stabilize the voltage entering the furnace. Upon reaching the target temperature, the system is supplied with nitrogen gas (N_2) to release the water content still present in the reactor tube. The resulting carbon was then characterized by XRD as shown in Figure 2.

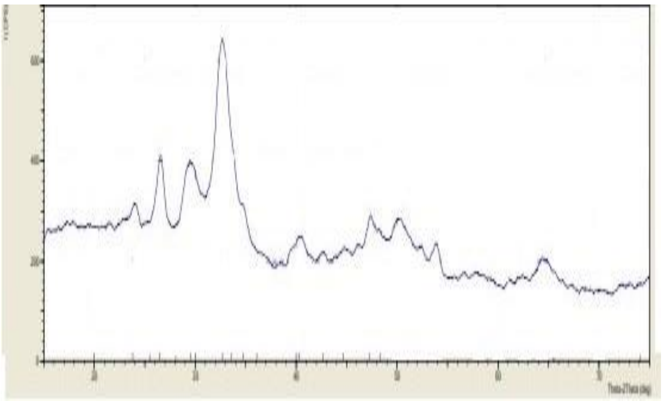

Fig 2. XRD diffractogram of carbon before activation

Based on the XRD diffractogram shown in Figure 2. the crystallinity data for the carbon sample after calcination obtained three peaks with the strongest intensity at 2θ angles 26.52°, 29.66°, and 32.77° respectively 2θ angle shifted. This indicates a difference in the distance between the faces of the carbon crystals as a result of the calcination process.

The carbon produced through the carbonization process of the raw materials, most of its pores are still covered by hydrocarbons, tar, and other components, such as ash, water, and sulfur, resulting in low absorption capacity. Therefore, the carbon was activated using a 3 M KOH solution and soaked for 15 hours, then filtered and washed with distilled water. KOH solution is a strong and corrosive base that can remove impurities in carbon, resulting in ash that is released from carbon. In the activated carbon product resulting from chemical activation, there is a little white powder like ash. This indicates that the reaction of carbon with KOH produces carbonate compounds (K_2CO_3) [6]. As a result, the physical specific surface area of the pores is increased. The resulting activated carbon was further characterized by XRD which is shown in Figure 3.

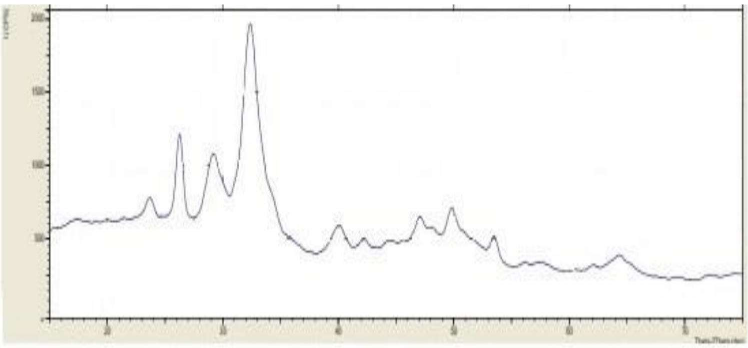

Fig 3. XRD diffractogram of carbon after activation

Based on the XRD diffractogram shown in Figure 3, the crystallinity data for the charcoal sample after activation obtained three peaks with the strongest intensity at 2θ angles of 26.22°, 29.12°, and 32.43°, respectively. The X-ray diffraction pattern of fish scale carbon, under different conditions, compared with the X-ray diffraction pattern of graphite is shown in Figure 4.

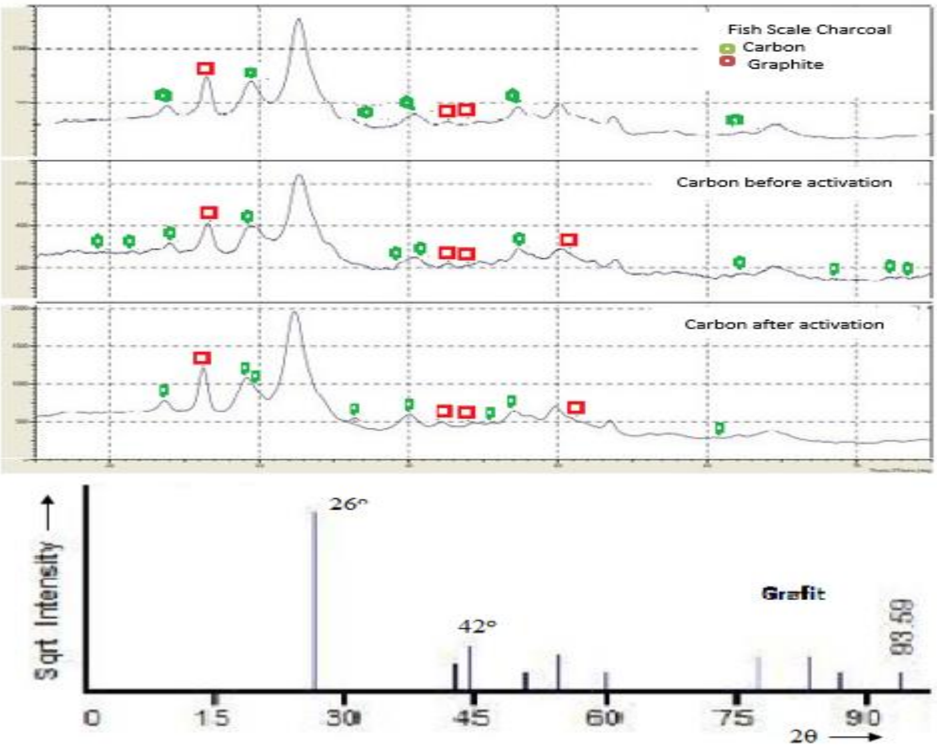

Fig 4. X-ray diffraction pattern of fish scale carbon, at different conditions, compared with the X-ray diffraction pattern of graphite [7].

The diffraction patterns shown in Fig. 4 show that there are changes in the microstructure of fish scale charcoal and carbon before activation, and after activation. The diffractograms of each carbon compared with the JPDS (*Joint Committee on Powder Diffraction*) standard data show that the patterns of all three are close to the graphite structure. This can be proven by the appearance of peaks at an angle of $2\theta=26^{o}$ and an angle around $2\theta=42^{o}$, both of which are characteristic peaks of graphite in the plane (*planar,* L_a), and at an angle of 25-26° are graphite crystals in the vertical plane (C-*axis,* L_c) (Sonibare et al., 2010). In addition to these two angles, graphite peaks also appear at other 2θ angles with low intensity.

The characterization results also indicate the formation of carbon in the charcoal. In the diffraction pattern of fish scale charcoal, the *lonsdaleite* phase (carbon phase) is characterized by the appearance of a peak at an angle of $2\theta = 29.52^{o}$ with a fairly strong intensity and has formed crystals with a hexagonal structure according to JCPDS 20-0258 data. In carbon after calcination, there is an increase in the number of crystalline peaks. In the initial state, 25 crystalline peaks with two phases were found. After calcination, the number of crystalline peaks increased to 39 peaks with the same number of phases, and more carbon and graphite phase components were identified. The peak with the second strongest intensity in fish scale charcoal ($2\theta=33.60^{o}$) also did not appear after the carbon was calcined. This is possible as a result of increasing the carbonization temperature resulting in the decomposition of components followed by the formation of new crystallite structures. The peak at 2θ=29.66o according to JCPDS 72-2091 is characteristic of C8 [8].

In the activated carbon, there is a reduction in the number of crystalline peaks, which becomes 24 crystalline peaks with two phases. In addition, the strongest peak is still at the same 2θ angle as the carbon before activation, but there is a slight shift with the resulting intensity getting higher. This shows that during activation, the rearrangement process of carbon atoms occurs, and the crystallite carbon plates become open and push the hydrocarbon residues. The shifting of the carbon plates creates new pores. The greater distance between crystallites also indicates an increase in carbon pore surface area [9].

The presence of two phases identified in the diffractogram of fish scale carbon in the form of graphite and carbon phases indicates an intermediate structure called turbostatic between crystalline and amorphous. Turbostatic is a hexagonal network that is still parallel, not having the right arrangement with each other [10]. So, the phase formed in this study is still turbostatic.

4. Conclusion

Characterization of scale carbon of *white snapper (Lates calcarifer)* using by XRD displayed three peaks with the strongest intensity at 2θ angles of 26.45^{o}, 32.69^{o}, and 33.60^{o} respectively. Moreover, after calcination, there was a shift in the 2θ peak with the strongest intensity at 26.52^{o}, 29.66^{o}, and 32.77^{o}. The surface acidity before and after activation were $4.86\text{x}10^{-3}$ mol/g and $7.77\text{x}10^{-3}$ mol/g, respectively. In addition, after carbon activation, the strongest intesity of peak was still at the same 2θ angle as carbon before activation, but there was a slight shift with higher intensity. This shows that during activation, the rearrangement process of carbon atoms occurs, the crystallite carbon plates become open and push the hydrocarbon residues. The shifting of the carbon plates results in a new pore. The greater distance between crystallites also increases the surface area of the carbon pore and its adsorption ability.

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