FTIR, SEM and XRD analysis of activated carbon from sago wastes using acid modification

W R Kunusa¹*, H Iyabu¹ and R Abdullah²

¹ Department of Chemistry, Faculty of Mathematics and Natural Sciences, Gorontalo State University, Jl. Prof. Dr. Ing. B. J. Habibie, Indonesia
² SMAS Wira Bhakti Gorontalo, Jl. Pasar Minggu, Gorontalo, Indonesia

*rewinkunusa2014-gmail2@.com

Abstract. Activated charcoal bio adsorbent is a method that can be developed because the raw material is easy to obtain and does not require large costs such as processing sago. The stages of making activated carbon include the stages of dehydration, carbonization, and extraction of silica using 4% NaOH solution (1:10). The activation stage successively used 250 mL of 1M NaCl, 1M MgCl₂, 1M CaCO₃, 1M K₂Cr₂O₇ and the addition of 100 mL of 1M HNO₃, 3% H₂O₂, 1M H₃PO₄ and 4N H₂SO₄ respectively. The activation process uses a water bath at 700°C for 3 hours. In the conventional carbonization stage in a vacuum used drum for 1x24 hours. Physicochemical analysis of activated carbon products, namely moisture content, ash content, pH, Activated Carbon Adsorption Test against Iod Test and methylene blue. FT-IR analysis showed a wide band of 3442.94 cm⁻¹ to 3415.93 cm⁻¹ which showed the free O-H strain vibrations of the OH group in the cellulose molecule. 1178.51 cm⁻¹, –1039.53 cm⁻¹, –1024.20 cm⁻¹, C-C and C-O-C glycosidic ether band. SEM data describes the differences in the surface morphological structure of each sample and is supported by XRD data.

1. Introduction
The processing of sago, results in a large amount of solid waste that is produced, such as bark and in general, it is burned or comes from nearby rivers. Along with the long demand for sago starch, the sago starch industry is currently in management conflicts that have resulted in environmental pollution and health problems. Waste consists of starch, hemicellulose, cellulose, and lignin which can be validated into raw materials for economical products such as adsorbents, sugars, biofuels, nanomaterials, composites, and ceramics [1].

Agricultural waste-based activated carbon is one of the growing precursors in various researches. The development of natural synthesis methods to increase the selectivity of adsorption has been carried out through modification of various physical or chemical methods, either in salt, acid or alkaline solutions. Activated carbon is charcoal which is used as an adsorbent in the activated adsorption process. The character of activated carbon based on biomass is highly dependent on the carbonization process and the activation process, namely physical and chemical activation to activate the carbon material [2,3]. Carbon precursors need to be carbonized to produce charcoal and then activated. Activator using CO₂ or steam is called physical activation [4]. If the activator is a chemical substance it is called chemical activation [5].
Chemical activation basically uses acidic or basic compounds such as HCl, H2SO4, H3PO4, NaOH, ZnCl2 and other compounds that have often been used to produce activated carbon which has large pores and a wide surface to absorb. Activator ZnCl2 is a Dehydrating Agent that produces the highest yield compared to H3PO4 and KOH on activated charcoal / activated carbon. Activated carbon has high metal absorption and has a high yield of pure activated carbon, namely 24.45%. Synthesis and characterization of lignocellulosic activated carbon in sago waste was carried out in several steps, namely dehydration, carbonization, extraction of silica with NaOH, activation of 10% ZnCl2, in a modification of a solution of 1.5 HNO3 65% [6]. Activated carbon ZnCl2 has a surface area of 1737 m2/g [7]. Activated carbon from sago stem bark and dates through the carbonization and activation stages with ZnCl2 activator has a surface area of 1634 m2/g at an activation temperature of 700 °C for one hour [8]. The surface character of activated carbon significantly affects basicity, adsorption processes, electrical, electrochemical, catalytic, acid-base, redox reactions, hydrophilic and hydrophobic properties and other properties. The OH functional groups on the surface are classified according to their chemical properties, namely acidic, basic and neutral. Carboxylic acids or carboxylic anhydrides, lactones and phenolic hydroxyl are sources of surface acid properties of activated carbon [2].

Various studies have been reported for the modification of activated carbon, natural zeolites, natural clays, biopolymers (chitosan, cellulose), metal oxide modified sands, silica and metal-organic for the purpose of expanding the surface, porosity, adsorption capacity and surface chemistry of natural materials. The role of metal ions, cations, metal oxides and polymers has an impact on changes in ion exchange properties. The nickel and cobalt examples show high electrochemical performance and redox reactions as binary metal oxides [9]. In addition, modification of functional groups such as amino, aldehyde, phenol, metal oxide, OH and CO groups aims to improve the surface properties of the adsorbent, namely adsorption selectivity, increase in surface area, pore volume, capacity and adsorption kinetics. Furthermore, surface modification through the esterification process will change the nature of the polar hydrophilic surface to organophilic. The polarity and aromaticity of the material affect the adsorption capacity. In addition, modifications using oxidizing agents such as (nitric acid, ammonium persulfate, hydrogen peroxide), sulfuration, nitrogenation and coordinate ligands greatly affect the surface properties of activated carbon. Activated carbon modified with SO2 gas as a sulfurizing agent is another technique to increase the efficiency of activated carbon. The chemical nature of the activity of the carbon-sulfur complex in the adsorption process is an important factor in determining the acid-base character of the carbon surface [4]. Likewise, the use of alkaline (KOH) has a higher pore surface area of 1124.4 m2/g compared to the acid treatment of H3PO4 and H2O2 with a pore surface area of 455.5 m2/g and 363.0 m2/g [7]. The resulting pore size distribution of activator H3PO4 45%. Modification of activated carbon from pomegranate pulp using H3PO4 was used as a chemical activation agent with a variety of impregnation ratios, namely 50%, 100% and 200%) impregnation time of 24 and 48 hours at 500°C and 700°C. The analysis results showed the maximum BET surface area at an impregnation ratio of 200%, namely 840 m2/g at a carbonization temperature of 700 °C and an impregnation time of 48 hours [9].

The electrochemical performance of activated carbon is significantly improved compared to that of inactive carbon. Although upon activation it produces high ash content, the production of volatile substances such as tar and highly corrosive chemicals to the equipment, but provides new insights for the development and comprehensive utilization of biomass waste to be of economic value. The use of analytical instruments in the carbonization process plays an important role and greatly affects the quality of activated charcoal. The modification of the activated carbon activation process which is microwave-induced with H3PO4 activator has a surface area of 1432 m2/g higher and the activation rate is greater than conventional heating. Another modification using the Microwave Ultrasonic System with KOH activator at a carbonation temperature of 800°C has the highest surface area of 1162.73 m2/g. [10]. Likewise, the use of CO2 gas under N2 gas flow of 600 mL/minute for 1 hour in the carbonization process is able to efficiently adsorb benzene and trichloroethylene [11]. FT-IR characterization on activated carbon activated KOH / NaOH illustrates the presence of functional groups (-OH, COOH, C = C, C-C). With the hydrothermal process treatment (390-500°C) produces activated carbon with a
surface area of 2425 m² / g [3]. KA activated 4M KOH for 24 hours has a surface area of 1259.048 m² / g capable of adsorbing Cr (VI).

2. Methods
The sample came from a sago processing factory from Pangi Village, Dulupi District, Boalemo Regency. The survey results show that 5 sago processing locations are ± 500 m² at each location point and are located in tributary areas. Locations I, II, III, IV ± 3 km from hilly areas. Pangi Village is one of the areas for developing National Food Security, apart from corn it is also a sago producing area with an area of ± 3.5 Ha. The processing capacity of sago reaches ± 15-30 tons / month, done manually and ± 45-60 tons / month using machines, even reaching ± 20 tons / week in each factory location. See figure 1 below.

![Figure 1. Sampling and survey of sago processing locations.](image)

The stages of making activated carbon resulted from a modified procedure [6]. Includes steps of dehydration, carbonization, extraction of silica using 4% NaOH solution (1: 10). The activation stage successively used 250 mL of 1M NaCl, 1M MgCl₂, 1M CaCO₃, 1M K₂Cr₂O₇ solution and the addition of 100 mL acid solution respectively 1M HNO₃, 3% H₂O₂, 1M H₃PO₄ and 4N H₂SO₄. The activation process uses a water bath at 700°C for 3 hours (Table 1). In the carbonization stage, the production and burning of charcoal conventionally in a vacuum used drum for 1x24 hours. The next step is refining / sieving the bioarang which is produced for the preparation of activated charcoal. See table 1 and figure 2-4 below.

| No | Sample Code | Treatment | Treatment |
|----|-------------|-----------|-----------|
| 1  | Sample A   | + NaOH 4% | -         | -         |
| 2  | Sample B   | + NaOH 4% | + NaCl 1M | HNO₃ 1M   |
| 3  | Sample C   | + NaOH 4% | + MgCl₂ 1M| H₂O₂ 3%   |
| 4  | Sample D   | + NaOH 4% | + CaCO₃ 1M| H₃PO₄ 1M  |
| 5  | Sample E   | + NaOH 4% | + K₂Cr₂O₇ 1M | H₂SO₄ 4N |
**Figure 2.** Stages of making charcoal and activated charcoal.

**Figure 3.** Stages of making charcoal and activated charcoal.
2.1. Water content analysis
In the water content analysis, activated carbon as much as 5 grams put into a petri dish that has been known weight. It is then heated in an oven at a temperature of 100°C-110°C until the weight is constant and cooled within the eksikator for 20 minutes and then weighed. Do the same thing over and over again until the weight is constant. The analysis was done triplo (SNI-06-3730-1995). The water content is calculated by the following equation:

$$Water\ Content = \frac{a - b}{c} \times 100\%$$

where: sample weight and petri dish before drying (g), sample weight and petri dish after drying (g), sample weight before drying (g).

2.2. Ash content analysis
In the analysis of ash content, 5 gr of activated carbon is put into a petri dish that has been known weight. Then heated in a muffle furnace at 500°C for 3 hours until white ash is obtained. Petri dishes are cooled in the eksikator and weighed. Do the same thing over and over again until the weight is constant (SNI-06-3730-1995). Ash content is calculated by the following equation:

$$Ash\ Content = \frac{a - b}{c} \times 100\%$$

where: sample weight and petri dish before drying (g), sample weight and petri dish after drying (g), sample weight before drying (g).

2.3. Activated carbon adsorption test for Iod
The activated carbon is heated first at 115 °C for 1 hour, then weighed as much as 0.5 g and mixed with 50 mL of 0.1 N Iodine solution. Then shaken for 15 minutes, then centrifuged for 10 minutes. 10 mL of
filtrate is titrated with 0.1 N Na2S2O3 solution until it is light yellow then given a few drops of 1% starch solution and then re-titrated until the blue color disappears.

2.4. Activated carbon adsorption test for methylene blue
The activated carbon is heated first at 115 °C for 1 hour, then weighed as much as 0.5 g and mixed with 50 mL of 0.1 N Iodine solution. Then shaken for 15 minutes, then centrifuged for 10 minutes. 10 mL of filtrate is titrated with 0.1 N Na2S2O3 solution until it is light yellow then given a few drops of 1% starch solution and then re-titrated until the blue color disappears.

2.5. Fourier Transform Infrared (FTIR) analysis
FT-IR spectroscopy analysis was used to determine the activated carbon functional group were carried out using (Shimadzu IR-Prestige21, Instrument Laboratories Brawijaya University, Malang). Bands were recorded in the region from 4000 to 500 cm⁻¹. The process of chemical compound analysis is done by using Fourier Transform Infra-Red (FTIR). The activated carbon analysis using FT-IR was performed by mixing 0.2 mg activated carbon with 2 mg KBr and formed into pellets. The pellet of the sample is then fed to the FT-IR instrument with λ 4000-400 cm⁻¹.

2.6. SEM analysis
SEM analysis on activated carbon is done by freezing the cellulose activated carbon powder over the aluminium to dry. Then sprinkle the sample with gold for 30 seconds using polaron. The analysis results are displayed in stereoscopic. SEM was used to examine the microscopic structure and the surface morphology of activated carbon fiber. The instrument used was SEM-EDS JEOL JSM-6360LA, Japan, at Geologi Laboratories, Bandung.

2.7. XRD analysis
The activated carbon XRD analysis was performed by heating activated carbon and firing X-rays with wavelengths of 10-10 s/d 5-10 nm, having a frequency of 1017-1020 Hz and having 103-106 eV energy. Morphological changes in the crystalline structure of MCC fiber were analyzed using a high-resolution X-ray diffractometer (PANalytical, X’Pert HighScore, at Geologi Laboratories, Bandung).

3. Results and discussion
In the pre-activation process for samples A, B, C, D and E, 4% NaOH was used as a strong base that can remove impurities in the carbon from the pre-carbonation process such as volatile and tar. Alkali bases are good activators for the formation of pores of activated carbon [7]. The resulting product has a water content of 6% and an ash content of 17%. See figure 5 below.

Figure 5. Charcoal product based on sago pulp.

Good quality activated carbon products have low ash content and high carbon and oxygen content. Increasing the activation temperature reduces the content of activated carbon groups. Carrying out the activation process at 850 °C will prevent the formation of surface acid groups [2]. The following is the test data for the adsorption power of activated carbon on iodine solution shown in Table 2.
Table 2. Testing of the activated carbon adsorption power of Iod solution.

| No | Sample Code | Treatment | Power Adsorption Iod |
|----|-------------|-----------|----------------------|
| 1  | Sample A   | + NaOH 4% | -                    | 1903.35 mg/g         |
| 2  | Sample B   | + NaOH 4% | + NaCl 1M            | 393.483 mg/g         |
| 3  | Sample C   | + NaOH 4% | + MgCl₂ 1M + H₂O₂ 3% | 1675.476 mg/g        |
| 4  | Sample D   | + NaOH 4% | + CaCO₃ 1M + H₃PO₄ 1M | 1700.862 mg/g        |
| 5  | Sample E   | + NaOH 4% | + K₂Cr₂O₇ 1M + H₃SO₄ 4N | 1726.248 mg/g        |

Based on the data in Table 2, activated carbon without salt activation and the addition of an acid solution gave the highest iodine adsorption power, namely 1903.35 mg/g at the time of extraction of silica in 4% NaOH solution. Activation using 1M K₂Cr₂O₇ addition of sulfuric acid, H₂SO₄ 4N resulted in an adsorption power of 1726,248 mg/g and activation using 1M CaCO₃ with the addition of 1M H₃PO₄ acid and the addition of 1700,862 mg/g of acid, then 1M NaCl activation in the modification of 1M HNO₃ acid, the iodine adsorption power is 393,483 mg/g. The following is the test data for the adsorption power of activated carbon against the methylene blue solution shown in Table 3.

Table 3. Testing of activated carbon adsorption power on methylene blue solution.

| No | Sample Code | Treatment | % Power Adsorption MB |
|----|-------------|-----------|-----------------------|
| 1  | Sample A   | + NaOH 4% | -                     | 41.35%                |
| 2  | Sample B   | + NaOH 4% | + NaCl 1M + HNO₃ 1M   | 91.17%                |
| 3  | Sample C   | + NaOH 4% | + MgCl₂ 1M + H₂O₂ 3%  | 79.60%                |
| 4  | Sample D   | + NaOH 4% | + CaCO₃ 1M + H₃PO₄ 1M | 98.45%                |
| 5  | Sample E   | + NaOH 4% | + K₂Cr₂O₇ 1M + H₃SO₄ 4N | 98.45%               |

Based on the data in Table 3, activated carbon without salt activation and the addition of an acid solution actually gives the lowest adsorption power of Methylene Blue (MB), which is different from its adsorption power in iodine solution which is 41.35%. Activation using 1M K₂Cr₂O₇ addition of H₂SO₄ 4N, 1M CaCO₃ activation with the addition of 1M H₃PO₄ acid and 1M NaCl activation with a modification of the addition of 1M HNO₃ acid gave adsorption power to methylene blue of 98.45%, 98.45%, and 91.17%, respectively. For activation of 1M MgCl₂ in 3% H₂O₂ modification, it had a percentage of adsorption capacity of 79.60%.

3.1. FT-IR spectra data

FTIR analysis of samples A, B, C, D and E showed similar peaks with the only difference being the slight change in peak intensity. All spectra show a wide band in the region 3442.94 cm⁻¹ to 3415.93 cm⁻¹ which shows the O-H strain vibrations free of OH groups in the cellulose molecule. FTIR results reveal that the absorption band at 1178.51 cm⁻¹, -1039.53 cm⁻¹, -1024.20 cm⁻¹ corresponds to CC and the ether glycosidic COC band is at wave number 1105 cm⁻¹ this peak gradually disappears in activated carbon samples activated by 1M CaCO₃ in H₃PO₄ 1M acid modification. See figure 6 below.
3.2. SEM data interpretation

Based on SEM data, sample (c) MgCl₂ + H₂O₂ sample (d) CaCO₃ + H₃PO and sample (e) K₂Cr₂O₇ + H₂SO₄ did not show any cavities on the morphological surface. For samples A and B, namely without acid treatment and modification of NaCl + HNO₃, SEM results showed several small white particles and the sample surface was porous and had a hollow shape even though the number of pores produced was small because there were still many impurities that closed the pores of the activated carbon, so that the pores that are formed are still small. The differences in pore size and shape observed on the surface of activated carbon are due to depolymerization and release of volatile substances from organic substances during the carbonization process [9]. XRD analysis of char-pyrolysis and low temperature hydro-charcoal can predict the porosity of the activated carbon produced. The SEM reading results are shown in Figure 7.
3.3. XRD data interpretation
Microstructure characterization of activated carbon was carried out using X-Ray Diffraction (XRD). This characterization was carried out to determine the distance between planes (d002 and d001) and the micro-crystalline dimensions which included the height value of the Lc layer, the width of the La layer and the number of Np layers. The X-ray diffraction pattern showed that the sample had an amorphous structure before modification and began to form exfoliated graphite crystals, as indicated by the peaks at 2θ 31.64460 and 36.39390. Significant results were obtained from a series of carbonization, extraction, activation, and modification processes using 1.5 mL HNO₃ (CEA 1.5) which produced nanopore particles with uniformly homogeneous shapes in the 200 nm size range as indicated by scanning electron. Images. Finally, the infrared spectrum of the activated and modified samples ensures that the oxygen-containing group has increased [6].

Figure 7. FT-IR readings.
Based on the data in Figure 8 that activator and acid modification can affect the resulting microstructure. The increase in crystallinity occurs due to the shrinkage of the crystallite structure of activated carbon which is increasingly regular, which will result in a wider gap between the crystallites and the pores that are formed will increase so that it will increase the surface area of the carbon. The sharp peak formed at an angle of $2\theta$ is the impurity compound in the activated carbon produced [12].

Table 4. Sample A Activated carbon product without acid modification treatment.

| Pos. $[^\circ\text{2Th.}]$ | Height [cts] | FWHM $[^\circ\text{2Th.}]$ | d-spacing [Å] | Rel. Int. [%] | Tip width $[^\circ\text{2Th.}]$ |
|-----------------------------|-------------|----------------|-------------|---------------|----------------|
| 20.7440                     | 35.38       | 0.4896         | 4.27852     | 15.91         | 0.5875         |
| 26.5182                     | 222.30      | 0.4080         | 3.35855     | 100.00        | 0.4896         |
| 36.4562                     | 9.74        | 0.3264         | 2.46258     | 4.38          | 0.3917         |
| 49.1766                     | 5.68        | 0.2856         | 1.85126     | 2.56          | 0.3427         |
| 59.9507                     | 10.37       | 0.4896         | 1.54175     | 4.66          | 0.5875         |
| 62.5736                     | 13.32       | 0.2448         | 1.48328     | 5.99          | 0.2938         |
| 63.9859                     | 16.87       | 0.2448         | 1.45390     | 7.59          | 0.2938         |
| 66.2614                     | 19.28       | 0.2448         | 1.40938     | 8.67          | 0.2938         |
| 68.0087                     | 9.30        | 0.6528         | 1.37737     | 4.18          | 0.7834         |

Table 5. Sample B Activated carbon product of NaCl with modification of HNO$_3$ acid.

| Pos. $[^\circ\text{2Th.}]$ | Height [cts] | FWHM $[^\circ\text{2Th.}]$ | d-spacing [Å] | Rel. Int. [%] | Tip width $[^\circ\text{2Th.}]$ |
|-----------------------------|-------------|----------------|-------------|---------------|----------------|
| 12.2614                     | 11.29       | 0.2856         | 7.21273     | 7.32          | 0.3427         |
| 20.6827                     | 24.60       | 0.4896         | 4.29107     | 15.94         | 0.5875         |
| 23.0161                     | 24.54       | 0.2448         | 3.86105     | 15.90         | 0.2938         |
| 26.5498                     | 154.35      | 0.2856         | 3.35462     | 100.00        | 0.3427         |
| 27.3925                     | 16.68       | 0.2448         | 3.25330     | 10.80         | 0.2938         |
| 31.6446                     | 113.08      | 0.2448         | 2.82518     | 73.27         | 0.2938         |
Based on XRD data in Tables 4-8, Sample [A]. Without acid treatment it has a 100% intensity at angle $^{\circ}$ 2Th. 26.5182. For sample [B], namely the activation of NaCl with HNO$_3$ acid modification has a% intensity of 100% at an angle of $^{\circ}$ 2Th. 26.5498. Furthermore, for sample [C] which was activated with MgCl$_2$ in modified H$_2$O$_2$ acid solution had a% intensity of 100% at an angle of $^{\circ}$ 2Th. 26.5509. Sample [D] of activated charcoal CaCO$_3$ in modified H$_3$PO$_4$ acid solution has% Intensity 100% at angle $^{\circ}$ 2Th. 26.6458. The activated carbon product was further activated in K$_2$Cr$_2$O$_7$ solution for sample [E] in a modified H$_2$SO$_4$ acid solution having a% intensity of 100% at an angle of $^{\circ}$ 2Th. 26.6720. XRD data illustrates that Samples A, B, C, D and E have a% intensity of 100% at an angle of $^{\circ}$ 2Th. 26.5182 to $^{\circ}$ 2Th. 26.6720.

4. Conclusion
Analysis of Activated Carbon from Sago Waste with Acid Modification in FT-IR Analysis showed a band width of 3442.94 cm$^{-1}$ to 3415.93 cm$^{-1}$ which indicated the presence of free O-H strain vibrations from the OH group in the cellulose molecule. 1178.51 cm$^{-1}$, –1039.53 cm$^{-1}$, -1024.20 cm$^{-1}$, C-C and C-O-C glycosidic ether bands. SEM data illustrates the differences in the surface morphological structure of each sample and is supported by XRD data.
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