PERBANDINGAN KARAKTERISASI FTIR DAN SIFAT ADSORPSI LOGAM MERKURI TERHADAP KARBON AKTIF SEKAM PADI DAN KARBON AKTIF SEKAM PADI YANG TERIMPREGNASI KATALIS LOGAM FE

COMPARISON OF FTIR CHARACTERIZATION AND MERCURY METAL ADSORPTION PROPERTIES TOWARDS RICE HUSK ACTIVATED CARBON AND RICE HUSK ACTIVATED CARBON IMPREGNATED WITH FE CATALYST

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INFOARTIKEL

ABSTRAK

Penelitian tentang karbon aktif sekam padi dan karbon aktif sekam padi terimpregnasi dengan katalis logam Fe dilakukan. Sekam padi dilakukan proses karbonisasi pada suhu 400 ºC selama 2 - 2,5 jam di dalam furnace, dimana waktu dihitung pada saat suhu telah mencapai 400 ºC sehingga diperoleh karbon sekam padi. Setelah proses karbonisasi, karbon sekam padi yang terbentuk diaktivasi dengan menambahkan H_3PO_4 7% pada perbandingan 1 : 10. Karbon aktif sekam padi yang telah dikeringkan tersebut kemudian dipanaskan pada suhu 600 ºC selama 2 - 2,5 jam di dalam furnace, dimana waktu dihitung pada saat suhu mencapai 600 ºC. Karbon Aktif sekam padi yang terbentuk tersebut dihaluskan, kemudian disaring dengan menggunakan saringan 400 mesh. Karbon aktif sekam padi dilakukan impregnasi dalam tabung dan dikalsinansi dalam furnache selama 4 jam pada suhu 400 ºC, tanpa ada dialiri gas, dengan menggunakan katalis $Fe(NO_3)_3.9H_2O$. Hasil karbon aktif sekam padi dan karbon aktif sekam padi terimpregnasi dikarakterisasi dengan menggunakan FTIR. Pada karbon aktif sekampadi terimpregnasi menunjukkan adanya serapan pada daerah bilangan gelombang 1390 – 1300 cm^{-1} menandakan adanya gugus nitro yang ditunjukkan pada daerah serapan 1346.31 cm^{-1} dan 1381.03 cm^{-1} yang berarti adanya N=O simetri yang berasal dari $Fe(NO_3)_3$ yang digunakan pada impregnasi karbon aktif. Daya adsobsi terhadap logam Hg pada karbon aktif sekam padi sebesar 67,13% dan karbon aktif sekam padi terimpregnasi sebesar 71,60%.

Kata Kunci: Aktivasi, Impregnasi, Karbonisasi, Karbon aktif, Katalis, Sekam padi

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ABSTRACT

Research on rice husk activated carbon and rice husk activated carbon impregnated with Fe metal catalyst was conducted. Rice husk was carbonized at 400 ^{0}C for 2 - 2.5 hours in a furnace, where the time was calculated when the temperature reached 400 ^{0}C to obtain rice husk carbon. After the carbonization process, the former rice husk carbon was activated by adding 7% H3PO4 at a ratio of 1: 10. The dried rice husk activated carbon was then heated at 600 ^{0}C for 2 - 2.5 hours in a furnace, where the time was calculated when the temperature reached 600 ^{0}C. The formed rice husk activated carbon was pulverized, then filtered using a 400-mesh sieve. The rice husk activated carbon was impregnated in a tube and calcined in a furnace for 4 hours at 400 ^{0}C, without any gas flow, using Fe (NO3)3.9H2O catalyst. The rice husk activated carbon and impregnated rice husk activated carbon were characterized using FTIR. The impregnated rice husk activated carbon showed absorption in the wavenumber region of 1390–1300 cm-1, indicating the presence of nitro groups, as shown in the absorption regions of 1346.31 cm-1 and 1381.03 cm-1. This indicates the presence of N=O symmetry derived from the Fe (NO3)3 used in the activated carbon impregnation. The adsorption capacity of rice husk activated carbon for Hg metal was 67,13% and that of impregnated rice husk activated carbon was 71,60%

Keywords: Activation, Impregnation, Carbonization, Activated Carbon, Catalyst, Rice Husk

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I. INTRODUCTION

The composition and percentage of rice husk production are very high and abundant every year in Indonesia, reaching (millions of tons/year) along with existing rice production. Where rice is a staple food in Indonesia. Rice husk is a byproduct or abundant waste from rice milling. Currently, the utilization of rice husk waste is not optimal because of its kamba, hard, and fiber nature that cannot be processed into feed or paper products. If rice husk waste is not handled properly, it can cause major problems in the environment. Challenges faced in this case include farmers having difficulty marketing processed rice husk products such as briquettes, rural communities are less aware of how to process rice husks into high-value-added products, such as briquettes or bio silica and rice husk ash waste is still minimally utilized. This can cause environmental problems if not handled properly.

The main components of rice husk are cellulose, hemicellulose, and lignin. Rice husk contains several important chemical components such as Carbon 1.33%, Hydrogen 1.54%, Oxygen 33.64%, Silicon 16.98%, Lignin 25%, Cellulose 20% and organic components such as oil and protein as much as 3.51%. One of the unique chemical properties of rice husk is its silicon-cellulose content which is very different compared to other plant by-products. The high silicon content does not decompose even after complete combustion. The use of rice husk waste as carbon is starting to develop. Where rice husk carbon is easy to make and has economic value for daily needs. Rice husk charcoal is very useful for the community as a planting medium (loosening the soil, maintaining moisture, drainage), alternative fuel (charcoal briquettes), water purification materials (activated charcoal), plant strengtheners, even food packaging materials and lightweight bricks, all of which increase food security, the economy, and process agricultural waste into value-added products.

Rice husk carbon and rice husk activated carbon are very effective as adsorbents for treating liquid waste containing heavy metals such as mercury (Hg) due to their porous structure and richness in functional groups such as silanols, which are able to chemically bind metal ions, making them a promising natural adsorption solution. The activation process (physical and chemical) significantly increases its adsorption capacity compared to ordinary charcoal, with optimum conditions such as activation concentration, temperature, and stirring time can increase efficiency to more than 98%. Masdania (2016) has conducted research on the use of Fe catalysts on palm kernel shell activated carbon and rice husk activated carbon in the impregnation process.

Rice husk activated carbon can be impregnated to produce impregnated rice husk activated carbon, where the process is by placing the main active metal or catalyst in the catalyst support which is important in the manufacture of catalysts. The addition of metal to the catalyst support aims to expand (increase) the active site. The binding technique on the support affects the dispersion properties and interactions between the metal and the support, where the most commonly used are wet and dry impregnation and ion exchange. This catalyst is made by reducing the metal in the support material, then heated and calcined to modify the chemical composition, after which the metal as a support material is oxidized and reduced (Najam, 2012). Catalyst impregnation is one method used to incorporate a metal catalyst into a support material or porous media. It is expected that the active sites of the metal will be evenly distributed on the surface and pores of the support material in this way. The use of catalysts on activated carbon supports has many benefits, such as; to equalize the phase distribution throughout the carbon pores; Carbon provides a pathway for reactants to enter the active sites through diffusion within the pores; to reduce the possibility of heat loss in the reaction; to slow the sintering process, to increase resistance to catalyst poisoning and to increase catalyst activity. Activated carbon impregnated in the catalyst will help maintain the size of metal nanoparticles as the main active sites during calcination. Typically, calcination will cause the active site particles to sinter and this results in an increase in diameter. Calcination is a heating process to remove impurities to form metal oxides.

Adsorption is an effective and economical wastewater treatment method for addressing dye and heavy metal pollution. This process involves the attachment of atoms or molecules of one substance (the adsorbate) to the surface of another substance (the adsorbent) due to circulating forces at the surface. Adsorption can occur physically through van der Waals forces or chemically. Adsorption effectiveness is influenced by several factors, including the physical and chemical characteristics of the adsorbent (such as surface area and pore size), the chemical properties of the adsorbate (such as molecular size and polarity), the adsorbate concentration, environmental conditions (pH and temperature), and the duration of the adsorption process. Adsorption can occur between various phases, including gas-liquid, liquid-liquid, solid-liquid, and gas-solid (Anggriani et al., 2021). Adsorption methods excel in their effectiveness and efficiency in removing heavy metals from wastewater, even at low adsorbate concentrations (Safrianti et al., 2012). Other advantages include easy adsorbent regeneration, discontinuation, and simple system design. Adsorbents, as porous solid materials with active sites, play a crucial role in adsorbing adsorbate molecules. With these advantages, adsorption is a promising option for reducing the negative impacts of environmental pollution, particularly in the treatment of wastewater containing dyes and heavy metals. Activated carbon adsorbents and impregnated activated carbon adsorbents can be much more beneficial if used to reduce the levels of pollutants from liquid waste. Liquid waste as a byproduct of industrial activities often causes problems for the environment. Pollutants can be heavy metals. Basically, mercury (Hg) is a metal element that is very important in technology in this modern age. Mercury is an element that has an atomic number (NA = 80) and has a relative molecular mass (MR = 200.59). Mercury is given the chemical symbol Hg which is an abbreviation derived from the Greek Hydrargyricum, which means liquid silver. Its physical and chemical form is very advantageous because it is the only metal that is liquid at room temperature (25°C), has the lowest freezing point (-39°C), has a greater tendency to evaporate, easily mixes with other metals to form alloys (Amalgam/Alloy), and can also conduct electric current as a conductor of both high and low voltage electric currents.

However, in excessive concentrations, it can cause chronic or acute poisoning. Some toxic metals, such as mercury, have a long biological half-life and cause accumulation in the body (Alfian, 2006). Methyl mercury, an organic mercury, has always been a serious concern in toxicology. This is because methyl mercury can be absorbed directly through respiration with an absorption rate of 80%. Its vapors can penetrate the lung membrane and, when absorbed into the body, bind to sulfur-hydryl proteins such as cysteine and glutamine. In the blood, 90% of methyl mercury is absorbed into red blood cells, and methyl mercury is also found in hair. Most of the mercury found in nature is produced by industrial waste, amounting to approximately 10,000 tons annually. Mercury is widely used, with approximately 3,000 types of uses in the chemical processing industry, the manufacturing of medicines used by humans, and as a raw material for agricultural insecticides. All mercury components, both methyl and alkyl forms, that enter the human body continuously will cause permanent damage to the brain, liver, and kidneys. Mercury ions cause toxic effects by causing protein precipitation, inhibiting enzyme activity, and acting as a corrosive agent.

FT-IR spectroscopy is a commonly used analytical method for studying polymer materials and analyzing functional groups. This is done by determining and recording the results of residual spectra with energy absorption by organic molecules in infrared light. The infrared region is the part with a wavelength of 760–1 million nm. If the molecule is given energy in this region, the molecule can cause the bending and stretching of the bonds to increase, or this energy can cause vibrations in the molecules where the atoms in the molecule change their relative positions. Each functional group in a molecule generally has its own characteristics so that IR spectroscopy can be used to detect specific groups in polymers. The intensity of the absorption band is a measure of the concentration of the typical group owned by the polymer (Stevens, 2007).

II. RESEARCH METHODS

2.1. Making Activated Carbon from Rice Husks

Rice husks are used to make activated carbon. The procedure is as follows: 300 g of the obtained rice husks are cleaned, placed in a container, and placed in an oven for 2 hours at 110°C to remove any moisture. The rice husks are then carbonized at 400°C for 2-2.5 hours in a furnace, with the time measured when the temperature reaches 400°C. After the carbonization process, the resulting charcoal is activated by adding 7% H3PO4 at a ratio of 1:10 (w/w). The mixture is then stirred for 30 minutes and soaked for 24 hours. The resulting mixture is filtered and dried in an oven at 120-150°C for 24 hours. The dried charcoal is then heated at 600 ^0C for 2 - 2.5 hours in a furnace, where the time is calculated when the temperature reaches 600 ^0C. After the heating process, the activated carbon obtained is then washed with 5N HCl several times to remove chloride elements, then washed using hot distilled water until the pH is neutral, and then washed using cold distilled water to remove phosphorus content. The activated carbon is dried in an oven at 120 - 150 ^0C, then crushed and blended. After the activated carbon is smooth, it is then filtered using a 400 mesh sieve (Najma, 2012).

2.2. Making Activated Carbon from Rice Husk Impregnation

50 g of activated carbon was placed in a tube, then the tube was placed in a heating furnace. The activated carbon in the tube was then calcined in a furnace for 4 hours at 400°C, without any gas flow. The Fe(NO3)3.9H2O catalyst was dissolved in 0.09 M acetone, then an impregnation mixture was prepared between the Fe catalyst and the calcined activated carbon by mixing 500 mL of the Fe solution with 50 g of activated carbon. The impregnation product was sonicated with an ultrasonic stirrer for 1 hour, accompanied by heating at 60-70°C until all the solvent had evaporated. After that, it was dried at 60-70°C in an oven for 12 hours. The result obtained is characterized with FTIR.

2.3. Preparation of Hg Standard Series Solutions

10 mL of the 1000 ppm Hg standard solution was pipetted into a 100 ml volumetric flask, diluted with distilled water to the mark, and homogenized to obtain a 100 ppm Hg standard solution. 10 mL of the 100 ppm Hg standard solution was pipetted into a 100 ml volumetric flask, diluted with distilled water to the mark, and homogenized to obtain a 10 ppm Hg standard solution. 10 mL of the 10 ppm Hg standard solution was pipetted into a 100 ml volumetric flask, diluted with distilled water to the mark, and homogenized to obtain a 1 ppm Hg standard solution. 50 ml of 1 ppm Hg standard solution was pipetted, then put into a 500 ml measuring flask then diluted with distilled water to the mark line, and homogenized to obtain a 100 ppb Hg standard solution. From the 100 ppb Hg standard solution, 25; 50; 75; 100; and 125 ml were pipetted successively, then each was put into a 250 ml measuring flask then diluted with distilled water to the mark line, and homogenized to obtain a 10; 20; 30; 40; and 50 ppb Hg standard series solution. Then the standard series solution was tested at a wavelength of 253.7 nm (Source: BSN, 2011).

2..4. Determination of Hg Ion

5 mL sample of gold mining liquid waste was placed in a dish, then placed in an oven at 70°C for 1 hour, then heated on a hotplate until it turned into charcoal. The sample was then oven-dried at 550°C until it turned to ash, placed in a desiccator for 1 hour, added with 2 mL of concentrated HNO3, dissolved in distilled water, and filtered through Whatman No. 42 filter paper. The filtered filtrate was transferred to a 50 mL volumetric flask, diluted with distilled water to the mark, and homogenized. A 10 mL sample of the diluted filtrate was taken and transferred to a test tube. Analysis was performed using an ICP-AES instrument at a wavelength of 253.7 nm for Hg ions. Measurements were performed using the ICP-AES three times (Source: BSN, 2011).

III. RESULTS AND DISCUSSION

3. FT-IR Characterisation Result

Characterisation using FT-IR is applied to rice husk carbon, rice husk active carbon and rice husk active carbon after impregnation with Fe metal.

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3.1. FT-IR Characterisation of Rice Husk Carbon

Data analysis from FT-IR Spectroscopy of rice husk carbon gives spectra with absorption peaks of different wavelengths, such as: 3406.92, 2931.8, 2866.22, 2314.58, 1612.49, 1099.43,802.39, 462.92 cm^{-1} which can be seen in figure 3.1.

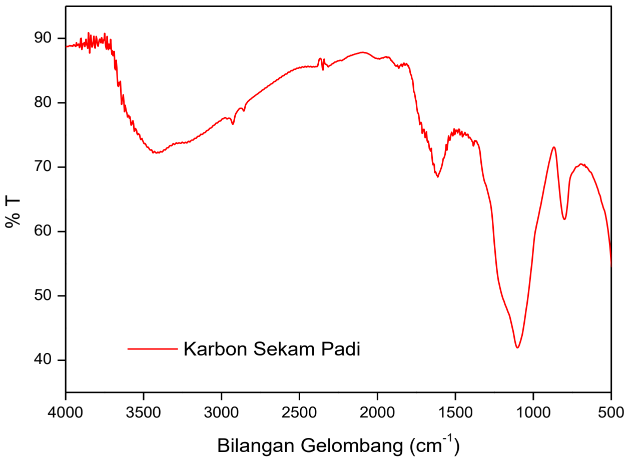

Figure 1. Rice Husk Carbon

Table 1. FT-IR Spectra for Rice Husk Carbon

Wave-length (cm-1)	Wave-length Range (cm-1)	Functional Groups
3406.29	3500-3200	OH stretching vibration
2931.80	2900-2800	Symmetric CH(- CH_2) vibration
2866.22	1820 – 1600	Carbonyl C=O stretching vibration
2314.58	2250-2100	Alkyne C = C stretching vibration
1612.49	1600-1400	Alkene C=C stretching vibration
1099.43	1075-1000	C-O stretching vibration
802.39	800-740	Bending = C-H

FT-IR spectroscopy analysis is used to determine if the substance obtained from the experiment is carbon. At first, carbon from rice husk is heated in a furnace at 400°C for 2.5 hours. From rice husk carbon FT-IR spectra (figure 3.1) shows sharp absorption and low intensity at 2314.58 cm^{-1} wavelength which shows unstable movement of $C\equiv C$ bonds, spectrum at 1612.49 cm^{-1} shows unstable C=C bonds, also supported by the existence of C-O bonds determined by the wide absorption with low intensity at 1099.43 cm^{-1}. Carbonisation process has also formed C=C bonds with the occurrence of spectrum at 1612.49 cm^{-1}. FT-IR result shows that sample tested is really pure carbon (Marham, 2009).

3.2. FT-IR Characterisation of Rice Husk Active Carbon

Data analysis from FT-IR spectroscopy of active carbon from rice husk carbon gives spectra with absorption peaks of : 3437.92, 2927.94, 2862.36, 2314.58, 1668.63, 1103.28, 798.53 cm^{-1} wavelengths (showed in figure 3.2.).

Rice *husk carbon is first activated with H_3PO_4, and then it is analysed with FT-IR spectroscopy which is then known that low intensity absorption occurred at wavelength of* 2314.58 cm^{-1} indicates movements of aromatic alkyne $C\equiv C$ compound. This shows that carbonisation and activation processes to form active carbon will increase aromatic compound which also indicated by the occurrence of peak with sharp but low intensity absorption at 2862.36 cm^{-1} and 2927.94 cm^{-1} wavelengths. The compound is CH(-CH_2) symmetry.

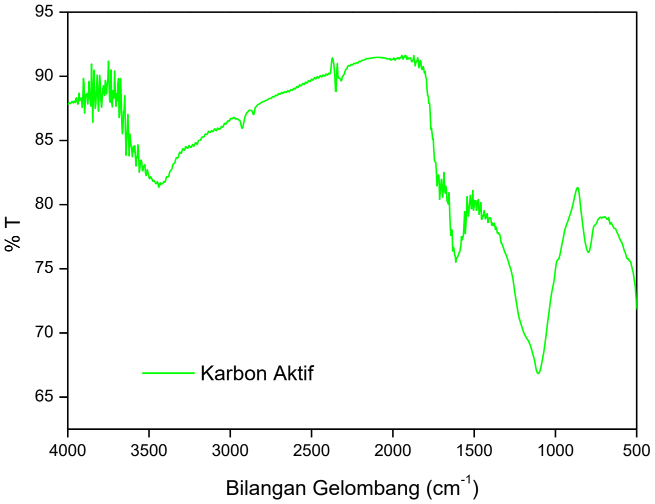

Figure 2. Rice Husk Activated Carbon

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Table 2. FT-IR Spectra for Rice Husk Activated Carbon

Wave-length (cm-1)	Wave-length Range (cm-1)	Functional Groups
3437.15	3500-3200	OH stretching vibration
2927.94	2950-2875	CH(- CH_2) stretching vibration
2862.36	2950-2875	CH(-CH_2) stretching vibration
2314.58	2250 – 2100	Alkyne $C\equiv C$ stretching vibration movement
1668.63	1500-1600	Alkene C=C stretching vibration
798.53	800-740	Bending =C-H

3.3. FT-IR Characterisation of Active Carbon after Fe Metal Impregnation

Data analysis from FT-IR spectroscopy of active carbon after Fe metal impregnation gives spectra with absorption peaks of: 3491.16, 3363.86, 2353.16, 1728.22, 1608.63, 1381.08, 1346.31,837,11, and 798.53 cm^{-1}wavelength (showed in figure 3.3.).

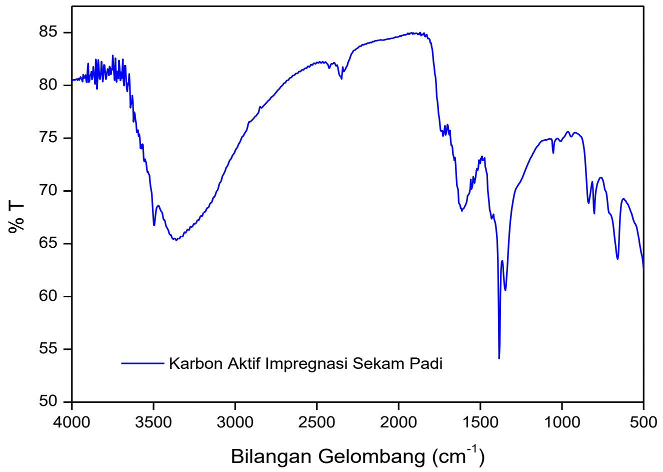


Figure 3. Impregnated Rice Husk Active Carbon

Impregnated active carbon analysis using FT-IR spectroscopy is done by calcining active carbon and $Fe(NO_3)_3.9H_2O$ catalyst is added. Next, it is sonicated to get homogenised solution and it is dried at 60 - 70ºC.

Table 3. FT-IR Spectra for Impregnated Active Carbon with Fe Metal

Wave-length (cm-1)	Wave-length Range (cm-1)	Functional Groups
3363.86	3500-3100	OH stretching vibration
2353.16	2250-2100	Alkyne C= C stretching vibration movement
1728.22	1820-1600	Carbonyl C=O stretching vibration
1608.63	1650-1450	Alkene C=C stretching vibration
1381.03	1390 – 1300	N=O symmetry stretching vibration
1346.31	1390 – 1300	N=O symmetry stretching vibration
837.11		
798.53	800 – 740	Bending =C–H

Impregnated active carbon analysed with FT-IR shows absorption at 1390 - 1300 cm^{-1} of wavelength indicating that there is nitro functional groups, showed in 1346.31 cm^{-1} and 1381.03 cm^{-1} absorption area which means that N=O symmetry from $Fe(NO_3)_3$ used in impregnating active carbon exists. There is also a bending of =C - H out of the plane im 800 - 740 cm^{-1}, which evidently showed with the spectrum of 798.53 cm^{-1} wavelength.

Data analysis from FT-IR spectroscopy gives an illustration that in this research, changing of functional groups takes place starting from carbon, active carbon and Fe impregnated active carbon (showed in Figure 3.4.).

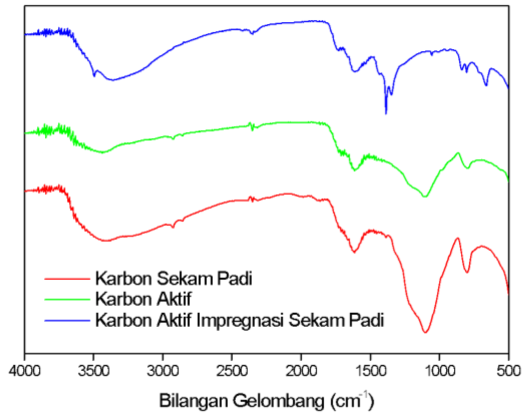


Figure 4. FT-IR of rice husk carbon, rice husk active carbon and impregnated active carbon from rice husk.

3.4. Calculation of Hg Ion Absorption in Gold Mining Liquid Waste Samples with Several Adsorbents

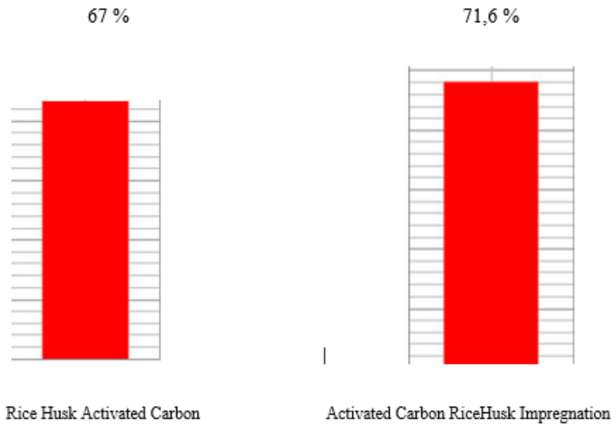
The results of the calculation of the Hg ion concentration can be determined using the calibration curve method for the Hg 0.0065 standard by substituting the Y value obtained from the measurement results into the regression equation of the calibration curve, so that the Hg ion concentration is obtained and for the absorption of Hg ions in gold mine liquid waste by the adsorbent using the following equation: hrough the regression equation and calculations, the percentage value of Hg ion absorption for each adsorbent.

The following equation:

$$\% \, Penyerapan \quad ion \, Hg = \left[\frac{Konsentrasi \, ion \, Hg \, awal - Konsentrasi \, ion \, Hg \, akhir}{Konsentrasi \, ion \, Hg \, awal} \right] x \, 100\%$$

Tabel 4. Data on the Percentage of Hg Ion Absorption by Several Adsorbents with a Contact Time of 15 Minutes

No	**Adsorben (3 g)**	**Initial Concentration of Hg Ions (mg/L)**	**Final Concentration of Hg Ions (mg/L)**	**Hg ion absorptin (%)**
1	Karbon Aktif	9,0352	2,9698	67,13
2	Karbon Aktif Impregnasi	9,0352	2,5652	71,60



The comparison of adsorption capacities for Hg metal between rice husk activated carbon and rice husk activated carbon impregnated with Fe catalyst shows that the adsorption capacity of impregnated activated carbon is higher, reaching 71.60%, compared to the adsorption capacity of rice husk activated carbon. This increase is due to the impregnation process (the addition of active chemical agents such as Fe) which modifies the surface of the activated carbon, enhancing its affinity for heavy metal Hg ions, leading to a greater absorption of Hg metal.

IV. CONCLUSION

Impregnated activated carbon analysed with FT-IR shows absorption in 1390 - 1300 cm-1 wavelength which indicated the existence of nitro functional group which is given by the absorption wavelength at 1346.31 cm-1 and 1381.03 cm-1. With the absorption at that area which means that N=O symmetry from $Fe(NO_3)_3.9H_2O$ used in impregnating active carbon exists. Therefore in this case, characterisation with FT-IR has shownthat Fe has been impregnated to rice husk active carbon. The adsorption properties of impregnated activated carbon are higher than rice husk activated carbon, namely in impregnated activated carbon 71.6088% while rice husk activated carbon has an absorption capacity of 67.1307%.

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