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Adsorption Power of Activated Charcoal from Coconut Shells on Lead Metal (Pb) in Well Water

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Abstract: This research aims to determine the results of the characterization and ability of activated charcoal (activated with KOH) to absorb lead metal (Pb). The procedure used is the process of carbonizing coconut shells in an airtight container at a temperature of 300ºC. Activation of coconut shell charcoal was carried out by immersing the charcoal powder in a KOH solution with variations of 1 M, 3 M, and 5 M. Characterization of activated charcoal powder was carried out on pore morphology using SEM-EDX, and XRD to identify the crystal phase, and AAS to determine the concentration lead metal (Pb). Based on the research results, the morphology of the charcoal before activation had a lot of impurities and the pores tended to be closed. After activation, the impurities disappeared and the pores were open. The crystal phase before and after activation was identified as 23.40% and 14.3% (calculated to be low), with the specific peak at 2θ, namely the corner area of 10º-80º. The adsorption capacity and efficiency obtained on activated charcoal with a KOH concentration of 1 M was 0.00194 mg/g and 23.66%, on activated charcoal the concentration of 3 M KOH was 0.00407 mg/g and 49.61%, the concentration of activated charcoal KOH 5 M is 0.00757 mg/g and 92.36%. Thus, the greater the KOH concentration, the greater the adsorption capacity and efficiency.

Keywords: AAS; Activated charcoal; Air well; Iodine; KOH; Pb metal; SEM-EDX; XRD

Introduction

Activated carbon is a type of carbon that can be produced from materials containing carbon or from charcoal that is specially processed to obtain a larger surface area. One adsorbent that is often used is activated charcoal from coconut shells. Coconut shells are mostly considered waste from the coconut processing industry, their abundant availability is considered an environmental problem, but they are renewable and cheap. Even though coconut shell charcoal can still be reprocessed into products that have high economic value, namely as activated carbon or activated charcoal (Agustikawati et al., 2022).

Activated charcoal is an amorphous carbon compound, which is produced from materials containing carbon or from charcoal that is specially treated to obtain a larger surface area. Activated carbon can adsorb certain gases and chemical compounds or its adsorption properties are selective, depending on the size or volume of the pores and surface. Activated carbon has many advantages, usually used in water purification, the sugar industry, wastewater treatment, and so on. In the industrial world, activated carbon is very important because it can absorb odors, colors, gases, and metals (Atmono et al., 2017).

Water is a source of life. Water pollution has become a major problem along with the increasing use of heavy metals in industry. One way to remove pollutants uses the adsorption method. Adsorption refers to the ability of the adsorbate to stick to the adsorbent. The strongest adsorbent is activated carbon because it has a large surface area so it has a high adsorption capacity (Sarah, 2018).

Dug well water is vulnerable to pollution because its construction is poor and its depth is poorly treated. from 15 meters, thereby allowing contaminants to enter the well (Dalmieda et al., 2019). Dug wells provide water that comes from shallow groundwater layers in the unsaturated zone so it is easily contaminated through

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seepage, potentially reducing water quality. It is feared that the quality of well water will decrease due to poor sanitation, such as seepage of household wastewater, chemical waste, laundry, etc. The most common pollution occurs due to water runoff from human or animal waste disposal facilities, which comes from less permanent septic tanks (Lensoni et al., 2023).

The decline in water quality is characterized by the discovery of many heavy metals such as Chromium (Cr), Lead (Pb), Copper (Cu), and other metals originating from industrial waste, final disposal sites (TPA), excessive use of fertilizers and household waste is a direct source of pollution, while an indirect source of pollution is surface water infiltration which penetrates groundwater or the atmosphere as rain (Handes et al., 2021). The presence of heavy metals in water at certain concentrations will accumulate in the water and the biota in these waters and can have toxic effects on the organisms they contain. Heavy metals can cause long-term damage to the environment because they can be toxic to living things even at low levels, this is due to the non-degradable nature of metals. Heavy metals in waters have a half-life of 32 × 103 years and in sediments 2.5 × 108 years longer than heavy metals on land and in the air (Meyla et al., 2016).

Lead enters the body through the respiratory or digestive tract and then diffuses to various organs through the circulatory system. The accumulation of lead in the kidneys, liver, brain, nerves, and bones can last for a long time, and high concentrations of lead can damage nervous tissue and kidney function. Symptoms of chronic lead poisoning can cause a loss of appetite, constipation, headaches, anemia, paralysis, seizures, and vision problems. Lead causes hemoglobin synthesis and the performance of the central nervous system and peripheral nervous system (Baiq et al., 2016).

Therefore, researchers are interested in researching making activated charcoal from coconut shells to adsorb lead metal in well water based on variations in the concentration of the KOH activating solution. Activated charcoal characterization testing was also carried out using *Scanning Microscope Electrone* (SEM), *Energy Dipersive X-Ray* (EDX), *X-Ray Difracttion* (XRD), and the absorption capacity of activated charcoal on lead metal was tested using an *Atomic Absorption Spectrophotometer* (AAS).

Method

This research was conducted at the Chemistry Laboratory of Gorontalo State University, with a research time of ±5 months. The raw material used in this research is coconut shell.

Tools and Materials

The ingredients used are coconut shell, well water, NH3, Na2CO3, NaOH, KOH, Aquadest, Iodine, Sodium thiosulfate, starch, HNO3, and Pb(NO3)2. The tools used are pyrolysis equipment, analytical balance, oven, glassware, AAS, XRD, and SEM-EDX.

Work Procedures

Manufacture of Activated Charcoal

The shells are dried in the hot sun for ±3 days. Coconut shells are carbonized in an airtight burning vat, then carbonized for 4-6 hours at a temperature of 300-400°C. The resulting carbon is cooled, and after that, the charcoal is crushed and sieved using a 100-mesh sieve.

The charcoal samples were then weighed at 30 grams, then immersed in 100 mL of each KOH solution with varying concentrations of 1 M, 3 M, and 5 M. Then left for 24 hours. Then filtered until charcoal residue is obtained and the filtrate is discarded. The charcoal was then washed with distilled water and repeated until the pH was neutral. Then it is dried using an oven at 110°C for 4 hours, resulting in coconut shell-activated charcoal powder. Then the activated charcoal was tested using SEM-EDX and XRD to determine the activated charcoal pores and activated charcoal crystals (Verayana et al., 2018).

Activated Charcoal Absorption of Iodine

A total of 0.5 grams of activated charcoal was mixed with 40 mL of 0.1 N iodine solution and then shaken for 15 minutes. Then 10 mL of the filtrate is pipetted into an Erlenmeyer flask and titrated with 0.1 N sodium thiosulfate solution. If the solution has a faint yellow color, 1% starch solution is added as an indicator. Titrate the dark blue color again until it becomes clear. The absorption capacity of iodine can be determined using the equation:

$$Iodine\ Absorption\ Capacity = \frac{V\ (sample) - \frac{V\ (Na2SO3) \times N1}{N2}}{m\ (sample)} \times 126{,}93 \times fp \quad (1)$$

Information:

V (sample)	: Titrated filtrate volume (mL)
V Na_2SO_3	: Tritation volume Na_2SO_3 (mL)
N1	: Concentration Na_2SO_3 (N)
N2	: Iod concentration (N)
m (sample)	: Carbon weight
Iod Weight	: 126.93 (mg/mL)
fp	: Dilution factor

Activated Charcoal Absorption Test for Lead Metal (Pb) in Well Water

Weigh out 0.08 grams of activated charcoal, and add 50 mL of well water to the beaker. Shake for 40 minutes with a rotation speed of 180 rpm. Then filtered until the filtrate becomes clear, then add a drop of HNO3, then measured using AAS.

Then the amount of lead metal adsorbed per gram of adsorbent (Qe, adsorption capacity) is calculated using the formula:

$$Qe = \frac{(Co - Ci)}{W} \times V \quad (2)$$

Information:

Qe : Adsorption capacity (mg/g)
Co : Metal concentration before adsorption (mg/L)
Ci : Metal concentration after adsorption (mg/L)
V : Volume of metal solution (L)
W : Adsorbent mass (g)

Adsorption efficiency can be calculated using the following formula:

$$\%\,Efeciency = \frac{initial\ concentration - final\ concentration}{initial\ concentration} \times 100\% \quad (3)$$

Results and Discussion

Carbonization and activation

This process is the process of burning organic materials contained in the basic material for making carbon. This action can trigger a decomposition event of organic material in the raw material and the removal of impurities in the raw material. Some non-carbon elements will be lost in this process. The release of these volatile elements causes the formation of pore openings. Along with this process, there will be changes in the pore structure (Ramadhani et al., 2020).

According to Fessenden (Verayana et al., 2018) in the formation of carbon the carbonization process used is not a complete combustion process. Incomplete combustion, namely the combustion process with a limited oxygen supply, will produce CO or carbon in the form of charcoal. The combustion reaction that occurs is as follows:

$$C_nH_{2n+2} + O_2 \longrightarrow {}_nCO + ({}_n+1)\ H_2O$$
$$C_nH2_{n+2} + O_2 \longrightarrow {}_nC + ({}_n+1)\ H_2O$$

At this stage, the dried coconut shell is put into a burning barrel and then closed tightly to prevent oxygen from entering. At the top of the lid of the barrel, there is a chimney so that the smoke can come out through the chimney, then combustion occurs through the bottom of the barrel so that air enters the barrel during combustion. Then the resulting charcoal is crushed or crushed and sifted using a 100 mesh sieve.

The refinement process aims to reduce the size of the charcoal particles. The size of these particles will affect the surface area of the activated carbon produced. The smaller the size of the charcoal/carbon particles, the wider the carbon surface that can make contact during the activation process. Thus, more carbon is activated and more pores are formed on each carbon particle (Alfiany et al., 2013).

The activation process carried out in this research is a chemical activation process. Chemical activation is carried out by soaking the charcoal in a KOH solution for 24 hours. This activating agent acts as an activating reagent and this agent activates the carbon atoms. The activator has the property of binding water which causes the water that is tightly bound to the carbon pores which is not lost during carbonization to be released. In addition, the activating agent will enter the pores and open the closed charcoal surface. So when heated, the impurity compounds in the pores become more easily absorbed so that the surface area of the activated carbon becomes larger and can increase its adsorption capacity (Nurhayati et al., 2015).

KOH is a good chemical activator for carbon because it can increase its surface area up to 3000 m2/g. Apart from that, KOH as a strong base can also remove impurities in carbon resulting from imperfect combustion such as volatile substances and tar. The reaction of KOH with carbon also releases water (dehydration) because KOH is a hydrating agent. This dehydration reaction causes carbon to be eroded, causing an increase in the surface area of activated carbon due to the formation of more pores, so it is hoped that the adsorption efficiency of Pb2+ metal ions will increase. These pores play an important role in the process of absorbing Pb metal in well-water samples (Nurfitria et al., 2019).

In this research, immersion was carried out in KOH solutions with varying concentrations of 1.3.5 M for 24 hours, then filtered until the filtrate pH was neutral. The samples were then dried in an oven at 110ºC for 4 hours. The aim of this process is to remove the water content that is still in the activated charcoal so that the pores of the charcoal open and can increase its absorption capacity for metal.

Characterization of Activated Charcoal

Characterization Using SEM-EDX

Characterization using the Scanning Electron Microscopes (SEM) instrument is useful for determining the microstructure (including porosity and shape of cracks) of solid objects. With this test, pore formation can be determined before and after activation. Charcoal testing using SEM aims to determine the changes in the carbon surface before and after activation (Susmanto et al., 2020).

At a magnification of 20,000 times Figure 1, shows that the coconut shell charcoal before activation already had pores on the carbon surface, but the pores were not fully open. The charcoal pores still contain impurities. This is because the coconut shell carbon has not been activated first, so the pores are not completely open. The impurities in coconut shell carbon are produced from the carbonization process so that the impurities cover the carbon pores.

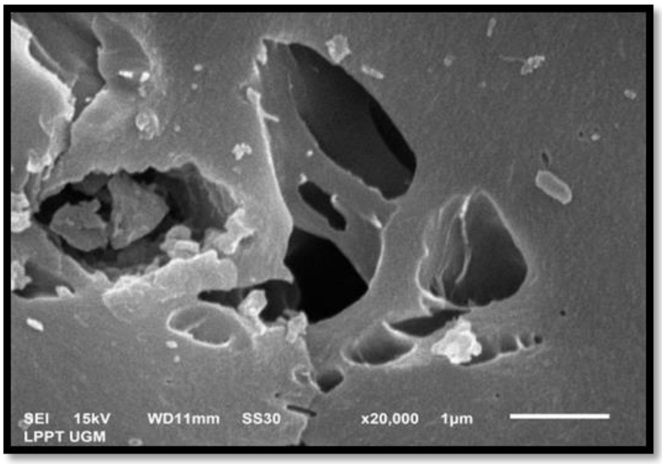

Figure 1. Charcoal pore morphology before activation

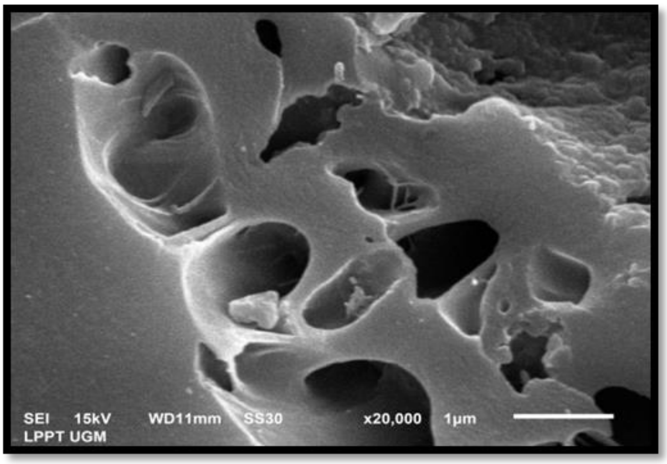

Figure 2. Charcoal pore morphology after activation

Meanwhile, Figure 2 shows that the surface of the pores is increasingly open, which is spread over the surface and cavity walls of the coconut shell-activated charcoal, and the impurities are gone. Increasing the number of pores on a surface increases its adsorption capacity because the more pores formed, the more active sites are available (Cintia et al., 2022).

The EDX spectrum results show that the percentage of carbon (C) in coconut shell activated carbon is most abundant in the carbon before activation, 73.00%, and the carbon after activation has 69.32% carbon (C). Other constituent compounds in coconut shells are O, K, Ca, Cu, and Zn. The content of elements such as element C is obtained from the cellulose, hemicellulose, and lignin content in coconut shells. The O element content is obtained from metal oxides and water molecules that bind to the support pores (Bakti et al., 2022).

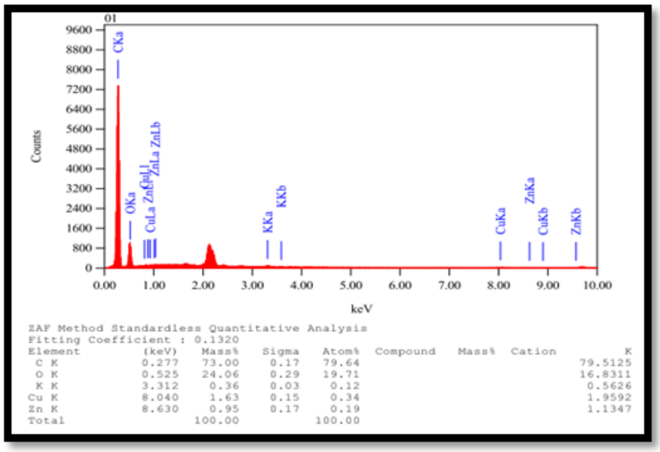

Figure 3. Charcoal EDX graph before activation

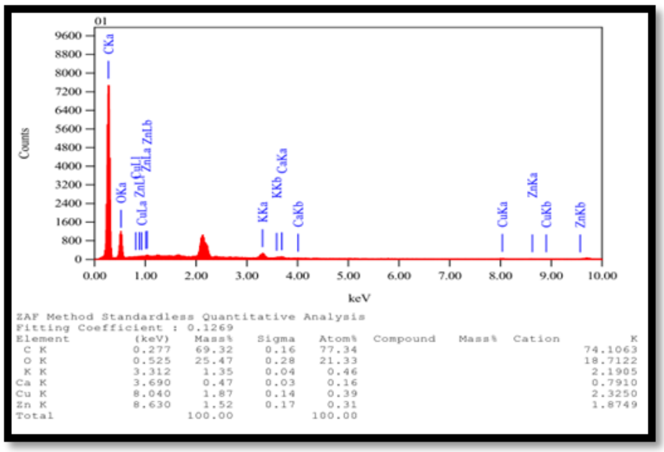

Figure 4. Charcoal EDX graph after activation

Characterization Using XRD

XRD analysis was carried out to identify the crystallinity of the sample. The samples used in XRD analysis are charcoal samples before activation and charcoal samples after activation. In the characterization using XRD, the diffractogram of the activated carbon sample before and after activation was observed which is shown in figures 5 and 6.

Figure 5 shows the x-ray diffraction pattern of carbon before activation with a crystalline degree of 23.4%, while Figure 6 shows the diffraction pattern of charcoal after activation with a crystalline degree of 14.3%. The results of this analysis show that the sample also shows an amorphous phase and a crystalline phase. Where the results of calculating the area show that the charcoal before activation has an amorphous area value of 76.6% and the charcoal after activation is 85.7%. The degree of crystallinity of the sample was calculated using the regional area comparison method. The diffractogram results show a specific peak that appears at 2θ, namely the angle area of 10°-80°m (Ramadani, 2021).

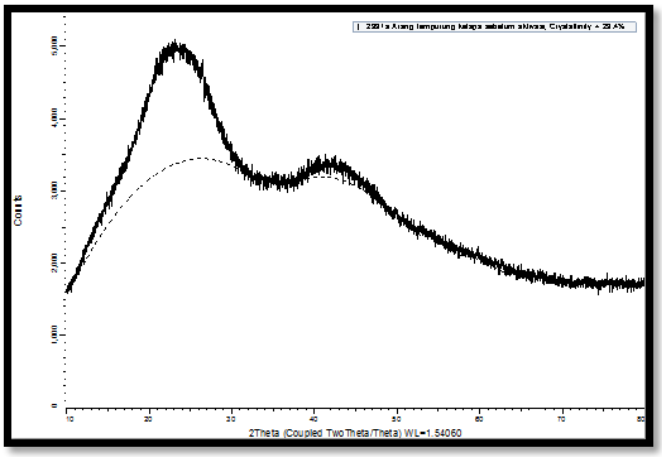

Figure 5. Charcoal XRD graph before activation

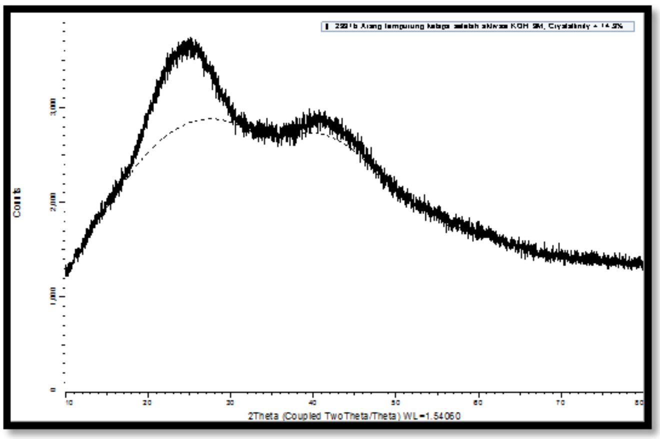

Figure 6. Charcoal XRD graph after activation

Activated Charcoal Application Stage

Absorption Power of Activated Charcoal on Iodine

Activated carbon has a high iodine absorption capacity, indicating that it has a larger surface area and wider microstructure and pores. This is related to the absorption of the adsorbent with the substance being absorbed. Iodine number (I_2) is defined as the number of milligrams of iodine (I_2) that can be absorbed by one gram of activated carbon. Adsorb ate in this case is an iodine solution that will be absorbed by activated carbon as an adsorbent. Factors that influence the ability of activated carbon to absorb iodine may be caused by residual hydrocarbon compounds on the surface of the charcoal which are removed during activation so that the surface becomes active (Chrisnandari et al., 2022).

Iodine absorption capacity indicates the ability of activated charcoal to absorb substances with a small molecular size, namely less than 10 Å or showing a number of pores with a diameter of 10-15 Å. The higher the iodine absorption capacity, the better the quality of activated charcoal (Kadang et al., 2020).

One method used to analyze the absorption capacity of activated charcoal for iodine is the iodometric titration method. The reactivity of activated carbon can be observed from its ability to absorb the substrate. The adsorption power can be observed using the iodine number, which shows how much the adsorbent can absorb iodine. The greater the value of the iodine number, the better the quality of the activated charcoal used (Dewi et al., 2020).

Table 1. Results of Iodine Absorption

Concentration KOH (M)	Iodine Absorption (mg/g)
Activated charcoal 1M	1277.76
Activated charcoal 3M	1565.47
Activated charcoal 5M	1523.16

At KOH concentrations of 1 M and 3 M, it shows that the higher the concentration, the more activated charcoal pores are formed so that the amount of iodine absorption is also greater. However, a KOH concentration that is too high can damage the pores formed because the activator solution has reached the saturation point so that it is not optimal in activating the activated carbon, which ultimately results in the value of the adsorption capacity for iodine decreasing (Erlina et al., 2015). The small adsorption capacity of activated charcoal on iodine illustrates that the micropore structure formed is small and provides the size of the pores that the molecules enter which are no smaller, resulting in a small iodine absorption capacity (Rachmawati et al., 2018).

The absorption characteristics of activated charcoal are not only influenced by pore size but are also influenced by the chemical composition of activated charcoal such as functional groups that act as active groups and the surface shape of activated charcoal. The activation process affects pore formation, where the stronger the activator used, the more damaged the surface and pores of the activated charcoal will be (Rachmawati et al., 2018). The activated charcoal used in this research complies with SNI-06-3730-1995 with a minimum value of 750 mg/g.

Adsorption Power of Activated Charcoal on Pb Metal in Well Water

The aim of applying activated charcoal to well water is to see how much active charcoal works in absorbing lead metal in well water. This study was grouped into two treatments, namely well water before adding activated charcoal and well water after adding activated charcoal. From this test, the greater the concentration of the activator, the lower the lead content (Apriani et al., 2013).

Adsorption capacity is the ability of an adsorbent to absorb or adsorb adsorbate. Determination of adsorption capacity was carried out to determine the ability of coconut shell-activated charcoal to absorb or adsorb Pb^{2+} metal ions (Deviyanti et al., 2014). Adsorption capacity is expressed in mg/g, which means the amount of metal (mg) that can be adsorbed by one gram of adsorbent (gram of activated charcoal) (Jasmal et al., 2015).

The highest value of adsorption capacity and % adsorption efficiency for activated charcoal is the value for activated carbon which has the best ability to adsorb Pb2+ metal ions. The influence of the use and concentration of KOH as a carbon activator also affects the absorption capacity of metals. The higher the KOH concentration, the higher the absorption capacity for metals. This is because increasing the concentration of the activating compound will make it easier for the tar compounds to be bound out through the pores of the activated carbon so that the surface of the activated carbon becomes wider or larger so that the absorption capacity of the activated carbon becomes greater (Alfiany et al., 2013).

In this adsorption process, on the surface of the activated carbon or adsorbent, a monolayer of Pb^{2+} metal ions will form which undergoes a diffusion process towards the active carbon pores. This process is caused by differences in the concentration of Pb^{2+} metal ions between the activated carbon surface and those dissolved in the water sample. The Pb^{2+} metal ion in water samples is found in solvated form with water molecules (H_2O) so it will be polar. Because the surface of activated carbon is non-polar and water molecules (H_2O) are polar molecules, the Pb^{2+} metal ion in the water sample which is in its solvated form will be able to be adsorbed by active carbon (Nurfitria et al., 2019). The following is a table of well water test results before and after treatment:

Table 2. Results of Adsorption Power of Activated Charcoal in Well Water

Sample Name	Concentration (mg/g)	Adsorption Capacity (mg/g)	Adsorption Efficiency (%)
Well Water	0.0131	-	-
Well Water + Activated Charcoal 1 M	0.0100	0.00194	23.66
Well Water + Activated Charcoal 3 M	0.0066	0.00407	49.61
Well Water + Activated Charcoal 5 M	0.001	0.00757	92.36

From Table 2, the results show that the metal content in well water before adding activated charcoal was 0.0131 mg/g. After adding activated charcoal with a KOH concentration of 1 M, the metal content in well water decreased to 0.0100 mg/g. Inactivated charcoal with a KOH concentration of 3 M, the metal content in well water decreased to 0.0066 mg/g, and in activated charcoal with a KOH concentration of 5 M, the metal content in well water decreased to 0.001 mg/g.

This is inversely proportional to the action of activated carbon on iodine, where the results obtained are that activated charcoal with a KOH concentration of 3 M has the highest iodine number compared to activated charcoal at a concentration of 5 M. This happens because, during the absorption process, the Pb^{2+} ion has a narrow radius. larger and capable of forming smaller hydration molecules, so that it can fill various types of pores in activated carbon, including micropores, mesopores, and macropores (Tejawati et al., 2017).

Adsorption capacity and efficiency increased along with increasing concentrations of activated charcoal used. When adding activated charcoal with a KOH concentration of 1 M to well water, the adsorption capacity value was 0.00194 mg/g and the adsorption efficiency was 23.66%. Inactivated charcoal with a KOH concentration of 3 M, the adsorption capacity and efficiency values were 0.00407 mg/g and 499.61%. Meanwhile, for activated charcoal with a concentration of 5 M, the adsorption capacity and efficiency values were 0.00757 mg/g and 92.36%. In this study, charcoal with a KOH concentration of 5 M was more selective in absorbing Pb^{2+} metal ions (Nurfitria et al., 2019).

Conclusion

In testing using the SEM-EDX instrument, activated charcoal before activation already had pores but there were still impurities on the surface of the charcoal, and had a mass of Carbon (C) elements of 73.00%. Meanwhile, after the activation of charcoal, it can be seen that the impurities attached to the activated charcoal have been reduced with a mass of Carbon (C) elements of 69.32%. In the XRD instrument test, the charcoal before activation had a crystalline degree of 23.4%, while the charcoal after activation had a crystalline degree of 14.3%. The ability of coconut shell activated charcoal to absorb iodine in activated charcoal with a 1 M KOH concentration is 1,277,762 mg/g, 3 M KOH 1565.47 mg/g, 5 M KOH experienced a decrease in iodine number with a value of 1523.16 mg/g. Meanwhile, the absorption capacity of activated charcoal towards lead metal in well water increases along with the increase in KOH concentration used, so that Pb metal in well water decreases. With an adsorption capacity value of 0.00757 mg/g and an adsorption efficiency of 92.36% at 5 M

KOH, at 3 M KOH, it has an adsorption capacity value of 0.00407 mg/g and an adsorption efficiency of 49.61%, and at 1 M KOH, it has a value the adsorption capacity is 0.00194 mg/g and the adsorption efficiency is 23.66%.

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