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SYNTHESIS OF $ZnCl_2$-ACTIVATED COCONUT SHELL ADSORBENT FOR MERCURY REMOVAL

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ABSTRAK

Kontaminasi merkuri dalam air menimbulkan risiko besar bagi organisme dan ekosistem. Pengurangan merkuri dari air yang terkontaminasi sangat penting untuk meningkatkan kualitas air. Metode adsorpsi, khususnya menggunakan adsorben yang berasal dari limbah biomassa, telah menunjukkan hasil yang menjanjikan dalam proses penjernihan air. Adsorpsi logam merkuri menggunakan arang aktif dari tempurung kelapa telah diteliti dengan menggunakan metode eksperimental. Penelitian ini bertujuan untuk menghilangkan kadar logam merkuri dalam air dengan menggunakan variasi suhu kalsinasi. Tempurung kelapa dibakar menggunakan furnace pada suhu 300 °C dan 450 °C. Bubuk arang tersebut kemudian diaktivasi secara kimia menggunakan $ZnCl_2$ selama 24 jam. Material dikarakterisasi menggunakan XRD dan FTIR untuk mengetahui fasa kristal dan gugus fungsi yang terkandung dalam material. Material diuji dalam simulasi larutan merkuri, dan konsentrasi merkuri yang tersisa diukur menggunakan AAS. Berdasarkan hasil karakterisasi XRD, mineral grafit ditemukan pada material yang dikalsinasi pada suhu 300 °C dan 450 °C. Sementara itu, beberapa gugus fungsi mempunyai muatan positif dan negatif pada permukaan material. Hasil penelitian menunjukkan bahwa material hasil kalsinasi suhu 300 °C menghasilkan %R dan Qe tertinggi, yaitu masing-masing sebesar 99,91 mg/L dan 9,991 mg/g, secara berurutan. Penelitian ini membuktikan bahwa limbah tempurung kelapa mampu menurunkan kadar logam merkuri dalam air. Tempurung kelapa dapat dimanfaatkan sebagai adsorben pada proses pengolahan limbah hasil proses industri.

Kata kunci: Adsorpsi, Arang aktif, Tempurung kelapa, Merkuri

ABSTRACT

Mercury contamination in water poses severe risks to both living organisms and ecosystems. Removing mercury from contaminated water is crucial for enhancing water quality. The adsorption method, particularly using adsorbents derived from biomass waste, has shown promising results in this purification process. The adsorption of mercury metal using activated charcoal from coconut shells has been investigated using experimental methods. This research aimed to remove mercury metal levels in water by varying the calcination temperature. Coconut shells were calcinated using a furnace at 300°C and 450°C. Afterward, the charcoal powder was chemically activated using $ZnCl_2$ for 24 hours. The material was characterized using XRD and FTIR to determine the crystal phase and functional groups contained in the material. The material was tested in a simulated mercury solution, and the remaining mercury concentration was measured using AAS. Based on the XRD characterization results, graphite minerals were found in the material calcined at 300°C and 450°C. Moreover, several functional groups had positive and negative charges on the material's surface. The finding showed that the material from calcination at 300°C yielded the highest %R and Qe, namely 99.91 mg/L and 9.991 mg/g, respectively. This research proved that coconut shell waste can reduce mercury metal levels in water. The coconut shell is able to act as an adsorbent in the waste processing process resulting from industrial processes.

Keywords: Adsorption, Activated charcoal, Coconut shell, mercury

INTRODUCTION

Environmental pollution is one of the most significant changes in the ecosystem arising from industrial and technical progress that increases contaminants and pollutants. One of the sectors impacted by the phenomenon is water resources. Water pollution may be defined as a negative change in conditions in water reservoirs such as lakes, rivers, seas, and groundwater caused by human action or natural activity. These changes lead to the deterioration of water quality, so the water has unsafe levels to accommodate the requirement of organism. According to World Health Organization (WHO) heavy metals are hazardous to children, so they have harmed the development of the brain and neurological system. Heavy metals also have long-term consequences for humans, such as an increased risk of high blood pressure, cardiovascular problems, and kidney damage. Pregnants who are exposed to high levels of heavy metals may have a miscarriage, stillbirth, premature birth, and low birth weight of infant. Heavy metals in water stock are becoming a dangerous problem when preserving clean water, including iron (Fe), nickel, and mercury (Hg) (Hu et al., 2024; Mohan et al., 2024; Rahman et al., 2024). Indrawan et al. (2018) stated that heavy metal contamination can disrupt ecosystems and aquatic environments, serving as an indicator of water quality. This fact is further supported by the findings of Indrawan & Putra (2021), who revealed that shellfish from one of the Serangan Islands, located in South Denpasar District, Bali, were found to be contaminated with lead-based heavy metals.

A polluted environment is greatly influenced by surrounding environmental activities, such as mercury accumulation from mining sector (Abed et al., 2024). Arthur et al. (2023) reported that Artisanal Small-scale Mining (ASM) activities in Ghana have destroyed the Ankobra River until 60% of body water. Crespo-Lopez et al. (2021) concluded that mercury pollution in Amazon reached 78.5%, becoming the worst global contaminant. The source of mercury is originated from gold mining, beer, and soy industries. Gold mining also negatively impacted Senegal (Chen et al., 2023) and Northern Palatinate Germany (Franzaring et al., 2024). The negative impact of mercury occurs in the environment around mining and can also affect the health of humans and other living creatures (Sitarska et al., 2023). WHO also stated that mercury exposure's destructive impact is impairing the baby's brain and nervous system, including cognitive thinking, attention, language, and fine motor skills. WHO also recommended measures to prevent exposure to mercury in water resourches. One way to prevent mercury exposure is to use adsorbents derived from natural materials, such as rice husks, pineapple peels, and coconut shells.

Coconut shells (Cocos nucifera L.) are leftover waste from the agriculture industry, causing abundant solid waste in surroundings (Al-sareji et al., 2023). Due to tropical weather, Indonesia is a potential place to grow coconut plants, but coconut shell utilization has not been well-performed (Mariwy et al., 2023). They contain various beneficial chemical substances, such as filtration and adsorption. Coconut shells include several chemical compositions, namely lignin 29.40%, pentosan 27.70%, cellulose 26.69%, hemicellulose 20%, water 8.01%, extractive solvent 4.20%, uronic anhydride 3.50%, ash 0.62%, and nitrogen 0.11% (Lago et al., 2024). These chemical compounds are a carbon source of activated charcoal production (Baharum et al., 2020). These days, activated charcoal is employed for purifying water due to its non-toxic, renewable, eco-friendly, readily accessible, and inexpensive.

Activated charcoal can adsorb substances or minerals that pollute water on its internal and external surfaces. Activated charcoal is also widely used to adsorb odors, colors, chlorine, pollutants, and heavy metals (Kulkarni et al., 2013). Al-sareji et al. (2023) reported that activated carbon from coconut shells can effectively remove pharmaceutical waste. This result is also reinforced by research from Prasoetsoph et al. (2023) that activated charcoal with a high surface area of 65.95 m2/g has a maximum adsorption capacity of iron ions, and it means the activated carbon from coconut shells can be modified to improve their characteristic. The performance of adsorbents derived from coconut shells in adsorbing mercury metals provides efficiency results that reach 90%.

Isa et al. (2022) used coconut shells as a basic adsorbent to remove mercury in illegal artisanal gold mining in Ghana, and this treatment resulted in 90% efficiency. Mohamad Yusop et al. (2022). also used coconut shell adsorbent to adsorb zinc chloride with an adsorption capacity of 77%. The potency of coconut shells as adsorbent for heavy metal removal may be developed for better environmental quality.

This research explored the characteristics of adsorbent from coconut shells, such as functional groups on the surface's adsorbent and crystal phase. Furthermore, the adsorbent has been implemented to remove mercury from the solution (Rizwan et al., 2024). Adsorption is the method used in this research using coconut shell adsorbent. In this research, the calcination temperature was varied at 300 °C and 450 °C and activated chemically using $ZnCl_2$. The calcination temperature is a critical factor in the chemical activation process of adsorbent materials. For optimal results, the temperature range typically falls between 150 °C and 700 °C. At temperatures of 300 °C to 450 °C, complete combustion occurs, leading to the formation of amorphous material. This process produces adsorbents with high absorption capacity due to the development of a porous structure. However, calcination at temperatures exceeding this range can have detrimental effects. Higher temperatures promote ash formation, which leads to the generation of inorganic compounds. These compounds can subsequently coat and block the pores of the activated charcoal, significantly reducing its absorption capacity. Therefore, careful control of the calcination temperature is essential to maximize the adsorbent's effectiveness and prevent the deterioration of its adsorptive properties (Baharum et al., 2020). In theory, the findings contribute to developing adsorbents from coconut shells to decrease heavy metals in water. Practically, the results can be a reference for adsorbing mercury in polluted environments such as mining areas.

METHODS

Sample Preparation

Five kg of coconut shells were cleaned using flow water from husks and dirt. The coconut shell was dried under the sun for three days to remove the water content in the leftover coconut shell. After drying under the sun, the coconut shell was crushed into smaller sizes.

Adsorbent Preparation

Coconut shell fragments were subjected to a dehydration process in an oven at 105 °C for 4 hours to remove moisture. The dried fragments were then pulverized using a ring mill to increase surface area and facilitate uniform processing. To ensure consistent particle size, the resulting powder was sieved through a 100-mesh sieve. The sieved powder underwent thermal treatment at two distinct temperatures: 300 °C and 450 °C. This carbonization process converted the coconut shell powder into charcoal, enhancing its porosity and adsorptive properties.

Adsorbent Activation

The charcoal was then chemically activated using a 0.1 M $ZnCl_2$ solution for 24 hours, allowing the $ZnCl_2$ to penetrate the charcoal structure and further develop its porous network. Following chemical activation, the adsorbent underwent a final drying step. It was heated in an oven at 105 °C for 3 hours to eliminate any residual moisture and stabilize the activated material (Kulkarni et al., 2013). Next, the coconut shell fragments were heated using an oven at 105 °C for 4 hours. Heating with an oven removes the shell's water content. This condition makes grinding easier using a ring mill (Lladó et al., 2024). After that, the activated charcoal was shifted using the 100 mesh sieve. Fine coconut shell powder was burned using a furnace to become activated charcoal. The material was immersed using 0.1 M $ZnCl_2$ for 24 hours. This solution was used to chemically activate the surface material. The coconut shell charcoal was rinsed using distilled water until the pH was neutral. The next step was to heat the coconut shell charcoal using an oven at 105 °C for 180 minutes. The purpose of heating with this oven is to remove the water content in the adsorbent after soaking (Lladó et al., 2024).

Characterization of Activated Charcoal from Coconut Shells

Before the adsorption procedure, the powder adsorbent was examined to ensure its purity. The adsorbent from coconut shells was characterized using XRD and FTIR instruments. XRD testing was carried out at Padang State University. The brand used in the characterization test using XRD at Padang State University was XRD XPERT PRO PANalytical PW3040/60, 2013 production from the Netherlands, wavelength 0.154 Armstrong, Copper anode, voltage 40 kV, current 30 mA, scan step 0.02 degree angle diffraction 10 to 100 degrees. Inorganic Crystal Structure Database (ICSD) standard with reference code (01-089-8495) was obtained also from The Integrated Laboratory of Padang State University. Otherwise, the functional group of surface material was analyzed using FTIR (Alpha FT-IR Spectrometer Bruker) (Alomar et al., 2023).

Adsorption Test

Each adsorbent resulting from calcination at 300 °C and 450 °C, which had been activated using $ZnCl_2$ for 24 hours, was contacted for 30 minutes with a 100 ppm mercury solution. The 100 mL mixture was stirred using a magnetic stirrer at a speed of 150 rpm (Tamjidi & Esmaeili, 2019). After the stirring process, the mixture was left for 2 hours to increase the mercury adsorbed level on the adsorbent's internal and external surfaces. After that, it was indispensable to filter the mixture to separate the adsorbent and solution. The mercury levels remaining in the solution were measured using an IKM JLI-74 Atomic Absorption Spectrophotometry. The method used was the Mercury Vapor Unit (MVU). In MVU, Hg ions were converted into atomic form by reduction. These atoms were given additional chemical reactions. Aeration was done to produce steam. The steam formed was channelled into the absorption cell, which was passed by light from the Hg lamp beam so that absorption produced an absorbance that was linear with the Hg level being tested Data obtained from measurements using AAS are calculated using equations 1 and 2. R is used to determine the adsorption efficiency, expressed as a percentage, while Qe is employed to determine the adsorbent's ability to adsorb mercury levels, measured in milligrams per gram. The variable M represents the mass of the adsorbent used in adsorbing mercury metal, measured in grams. V denotes the volume of the mercury metal solution measured in liters (Tamjidi & Esmaeili, 2019).

$$\%R = \frac{(C_I - C_0)}{C_I} \times 100 \quad (1)$$

$$q_e = \frac{(C_I - C_0)}{M} \times V \quad (2)$$

RESULT AND DISCUSSION

The cellulose, lignin, and hemicellulose content in coconut shells has the potential to be a carbon source for making adsorbents. Physical and chemical activation treatments were applied to coconut shells to increase fruitful pores on the surface of the adsorbent. Physical activation was carried out by varying the calcination temperature of 300 ºC and 450 ºC, and the chemical activation process was applied by immersion in a $ZnCl_2$ solution. The treatment results can be seen in Figures 1 (A) and (B).

Figure 1 A is the result of the calcination at a temperature of 450 °C, and it has a dark black color compared to the black color at 300 °C (Figure 1 B). Temperature differences in the heating process lead to this color difference. Previous research also stated that variations in calcination temperature have caused mass reduction of the produced adsorbent (Ugwu & Conradie, 2024). The decrease in adsorbent mass at 300 °C and 450 °C is caused by the release of organic matter, CO2, and water molecules from adsorbent. The mass reduction of adsorbent is listed in Table 1. Table 1 shows the mass reduction of the adsorbent at calcination of 300 °C (52.11%) and 450 °C (68.74%), respectively.

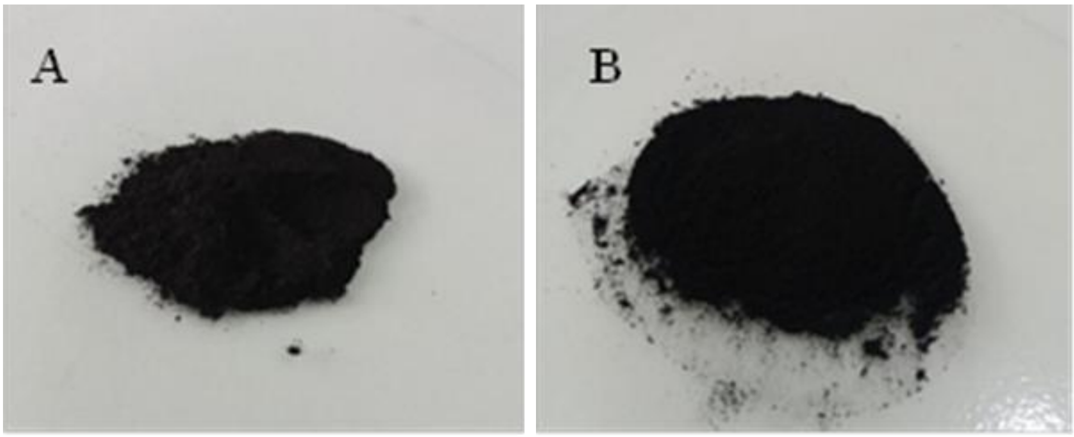

Figure 1. Calcination of coconut shell at 300°C (A) and 450°C (B)

Table 1. Mass diminution of adsorbent from coconut shells

Temperature (°C)	Initial	Mass (g) Final	Loss	% Mass
300	56,0624	26,8462	29,2162	52,11
450	30,0475	9,3967	20,6508	68,74

X-ray diffraction

The XRD diffractogram shows graphite as a mineral identified in activated charcoal. Honorisal et al. (2020) stated that the mineral graphite is an allotrope of carbon with a honeycomb-like and layered structure. The graphite structure forms covalent bonds with three carbon atoms forming a hexagonal arrangement. The X-axis in the diffractogram shows the movement of the goniometer (2Θ), while the Y-axis shows the peak intensity of X-rays. The diffractogram peak shows the adsorbent's different structures due to the variations in calcination temperatures. The diffractogram of adsorbent from coconut shell from calcination at 450 °C is shown in Figure 2.

Based on the XRD diffractogram pattern in Figure 2, the diffractogram shows a sloping and widening peak and displays 2θ= 23.5°; 26.5°; 30°; 32.5°; 36.5°; 40°; 44°; 45.5°; 48°; 49°; 59.2°. Based on the diffractogram, the mineral contained in activated charcoal is graphite. This result is similar to previous study research, which stated that at 400 °C calcination of coconut shell charcoal with mineral detected in the XRD diffractogram was graphite oxide at peak 2θ= 23, 57° and 2θ= 43.41° (Putri & Supardi, 2023). The sloping and broad peak of the diffractogram at 450°C indicates that the peak is categorized as amorphous. At the same time, small intensity exhibits amorphous Honorisal et al. (2020). The XRD results of calcination at 300 °C can be seen in Figure 3.

Figure 3 shows an adsorbent from a coconut shell that calcined at 300 °C. Broad and sloping peaks dominate the diffractogram, indicating amorphous (He & Zong, 2019). Based on the ICSD standard, the 2θ= 44° peak indicates the mineral phase formed is graphite with a hexagonal crystal system. The other peaks also emerge at 2θ= 26,5° and 33°. A previous study reported that graphite appeared in adsorbent from coconut shell calcined at 600 °C with impurities such as H, O, N, K, Na, P, and Ca (Sujiono et al., 2022). Adsorbents calcined at 300 °C and 450 °C have amorphous properties with different minerals. The temperature and immersion in the activation liquid have produced the peak difference. At 300 °C, the material experiences oxidation, so the oxygen compound groups are released (Putri & Supardi, 2023).

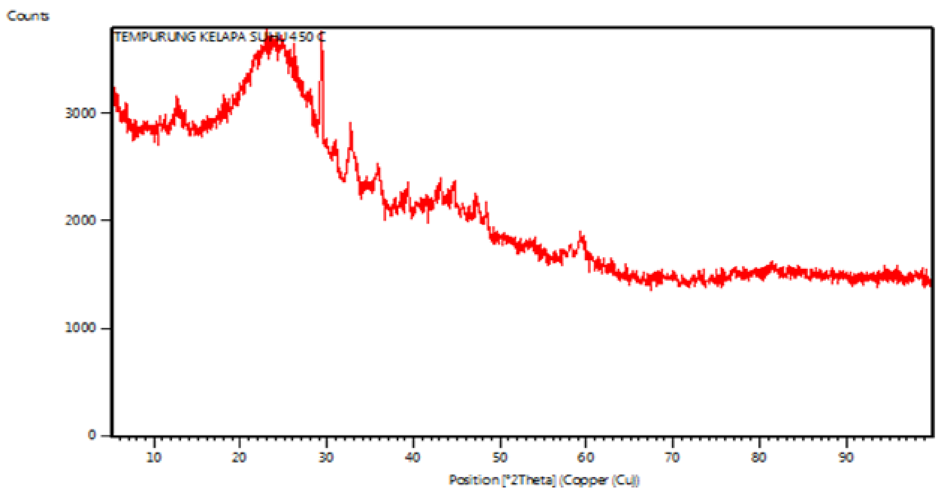

Figure 2. Diffractogram of adsorbent from coconut shells 450 °C

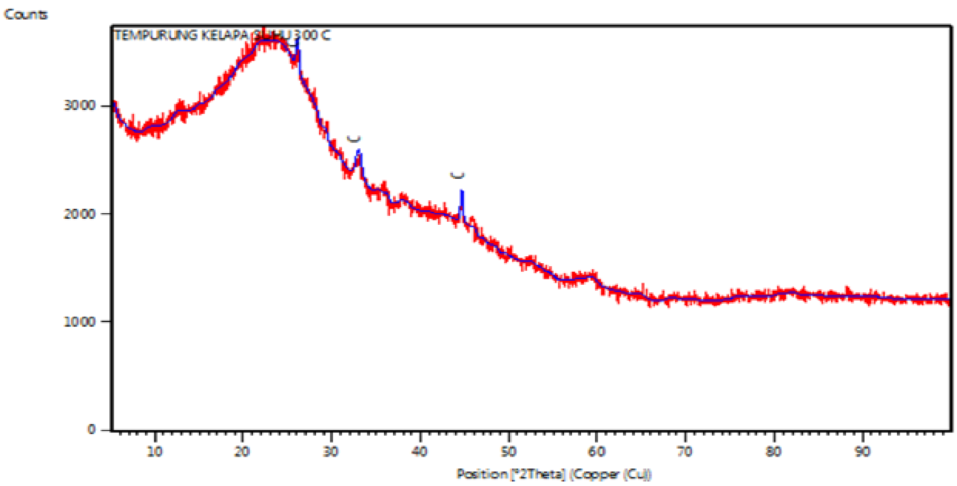

Figure 3. XRD diffractogram of coconut shell activated charcoal adsorbent at 300 °C

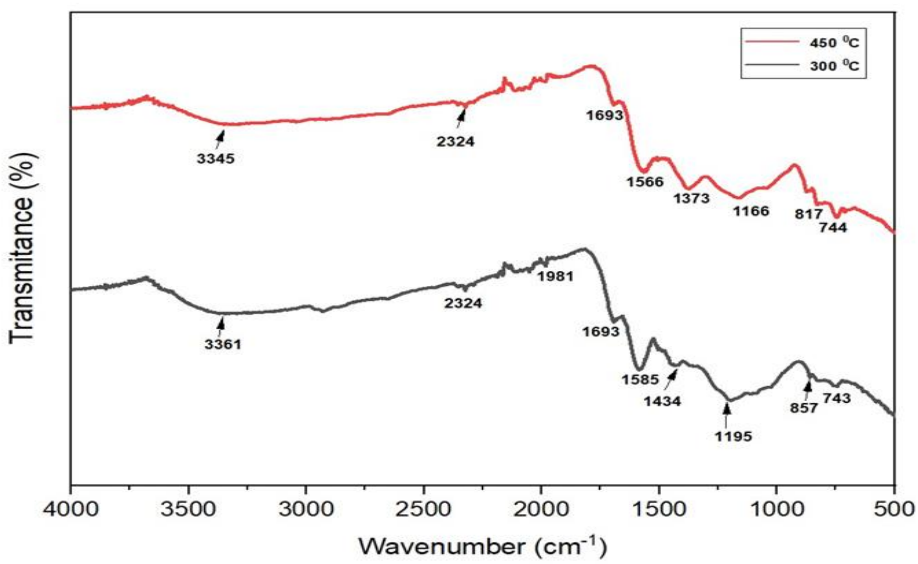

Figure 4. Adsorbent Adsorbent spectrum of coconut shell activated charcoal at 450 °C (red) and 300°C (black)

Fourier Transform Infra-Red

The FTIR test results from this research were used to determine the various functional groups found on the surface of the coconut shell adsorbent. In this study, the FTIR wavelength was read in the range 3500-500 cm^{-1}. Each peak in the adsorbent compound indicates a functional group. The results of the FTIR spectrum of the adsorbent with calcination of 450 °C can be seen in Figure 4.

According to Kosela (2010), the FTIR spectrum results in Figure 4 (red line) with a wavelength of 3345.73 cm^{-1} indicate the functional group (O-H), then the wave number 2324.53 cm^{-1} shows the function (C≡C), respectively. At the peak wave number of 1693.93 cm^{-1}, there is a functional group (C=C). Furthermore, the peak number 1566.66 cm^{-1} exhibits a functional group (C=C). The wave number 1373.13 cm^{-1} corresponds to the presence of a functional group (C-C), whereas the wave number 1157.23 cm^{-1} indicates the presence of a functional group (C-O).

Figure 4 (black line) displays a spectrum with fewer peaks than the 450 °C calcination. It demonstrates that increasing the temperature during calcination induces the formation of a new peak. Kosela (2010) stated that the wave number 3361.10 cm^{-1} corresponds to the presence of the functional group O-H, while the peak at 2929.48 cm^{-1} also indicates the presence of the functional group C-H. At the maximum point of the wave, which is at a frequency of 2324.45 cm^{-1}, there is a functional group represented by the chemical formula C≡C. The functional group C=C is present at the peak of 1693.83 cm^{-1} and also at 1585.17 cm^{-1}. The peak wave number of 1428.11 cm^{-1} belongs to a functional group C-C. This finding aligns with a previous study, which indicated that a C-C bond existed at the peak wave number of 1445 cm^{-1} (Mastiani et al., 2018). A peak of 1195.29 cm^{-1} is also denoted as C-O (Kosela, 2010), while 743.68 cm^{-1} exhibits C-C. The wave number at 857.83 cm^{-1} corresponds to the presence of a functional group Si-O. In their study, Bakti et al. (2023) reported that the maximum intensity of 830 cm^{-1} presented the Si-O group due to rocks containing silica.

Characterization of functional groups resulting from the FTIR spectrum of adsorbent from coconut shell with varying calcination temperatures of 450 °C and 300 °C has slightly different peaks. The functional groups included in the compound are O-H, C=C, C-H, and C-O. The O-H functional group exhibits robust hydrogen bonding arising from alcohol. The C=C functional group has higher carbon content, but the C-H functional group demonstrates less adsorption capacity. The presence of the C-O and O-H functional groups suggests that the adsorbent has polarity. The discovery indicates that coconut shell adsorbent contains chemicals with positive and negative charges. These charges enable the charcoal to behave as an adsorbent, effectively adsorbing metal substances present in water (Leslie Mendame et al., 2021).

Adsorption Test

The remaining mercury levels after the adsorption test were measured using an AAS instrument. The adsorbent from coconut shells has shown excellent results. The 300 °C calcination adsorbent produces an adsorption efficiency (R) of 99.91%, while the 450 °C calcination adsorbent is 99.58%. Calcined adsorbent 300 shows a higher R than calcined adsorbent 450 °C. The data suggests that about 99.91% of the initial mercury concentration is adsorbed onto the surface of the adsorbent. Furthermore, adsorbent capacity (Qe) indicates the amount of heavy metals that are adsorbed per gram of adsorbent. Calcined adsorbent 300 proved a higher Qe than calcined adsorbent 450 °C, namely 9.991 and 9.958 mg/g, respectively. These findings reveal that each gram of the 300 °C calcination adsorbent can adsorb up to 9,991 milligrams of mercury metal.

CONCLUSION

Coconut shells were effective adsorbents for mercury removal. The qualities of the adsorbent derived from coconut shells were worth considering. The XRD test findings indicated that coconut shell charcoal includeed graphite from 450 °C and 300 °C calcination temperatures, respectively. Furthermore, the adsorbent has several functional groups on its internal and external surfaces, exhibiting positive and negative charges. This functional group facilitated the adsorption of mercury metal on the surface via several interactions. The adsorbent obtained by calcination at 300 °C exhibited an adsorption efficiency of 99.91% and an adsorption capacity of $mg^{-1}.g^{-1}$. According to the findings of this study, adsorbents derived from coconut shells that undergo both physical and chemical activation may be recommended as practical materials for treating water polluted with metals, particularly mercury. Coconut shell has a high adsorption affinity. This high affinity makes the coconut shell adsorbent capable of adsorbing mercury metal with an adsorption efficiency capacity of 99.91 mg/g.

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