SYNERGY OF CARBON NANOFIBER INNOVATION FROM ZINC OXIDE MODIFIED SUGAR PALM FIBER AS ADVANCED MATERIALS FOR ARSENIC HEAVY METAL WASTE REMEDIATION

Elisabeth Nugrahini, Ida Setiarini, Wieke Helma Anjelina, and Lina Mahardiani*

Chemistry Education Study Program, Faculty of Teacher Training and Education, Universitas Sebelas Maret
Jl. Ir. Sutami 36A, Jebres, Kentingan, Surakarta, Central Java, Indonesia

* Correspondence, email: mahardiani.lina@staff.uns.ac.id

ABSTRACT

Heavy metal wastes cause water pollution. One of them is very toxic arsenic heavy metal waste; thus, treating them before they are released freely into the waters is necessary. In this case, the manufacture of carbon nanofibers (CNF) from sugar palm fiber waste is an innovation that can produce adsorbents to remediate heavy metals, thereby increasing the use value of sugar palm fiber waste. Carbon nanofiber from palm fiber (*Arenga pinnata*) was modified with ZnO metal, with varying concentrations of 0.1%, 1%, and 10%, in 1 gram of carbon nanofiber. Carbon nanofibers were made by carbonizing sugar palm fibers at 300°C for two hours. Furthermore, wet impregnation was carried out with $Zn(CH_3COO)_2.2H_2O$ and ended with calcination at 250°C for an hour, resulting in black nanofiber powder. This research was conducted to determine the effect of ZnO concentration on the characteristics of the carbon nanofiber produced and its effectiveness in remediating heavy metal arsenic. The ZnO/carbon nanofiber adsorbent material was then characterized using FTIR, XRD, and SEM analyses. Determination of the absorption of ZnO/carbon nanofibers on heavy metal arsenic was also assessed employing AAS analysis.

Keywords: Sugar palm fiber, Carbon nanofibers, ZnO, Heavy metal adsorption, and arsenic

INTRODUCTION

Water contaminated with heavy metals cause it not to be used properly and can impact life. In Indonesia, based on Government Regulation No. 82 of 2001, water pollution is the entry or inclusion of living things, substances, energy, and/or other components into the water by human activities, causing a decrease in water quality to a certain level, which leads the water to be unable to function according to its designation. The certain level in question is the water quality standard set and serves as a benchmark to determine whether water pollution has occurred [1]. In addition, one of the causes of heavy metals in waters is waste from industrial processes directly discharged into the environment.

Karanganyar is an industrial center area with various types of industry, one of which is the gilding of *keris* in Padepokan Keris Brojobuwono, Wonosari, Gondangrejo, which uses As_2S_3 (arsenic trisulfide) compounds mixed with citric acid ($C_6H_8O_7$) as anti-corrosion on iron [2]. In this case, the absence of waste treatment, or improper waste treatment, can negatively impact human life. Lower levels of arsenic exposure can cause nausea and vomiting, reduced erythrocyte and leukocyte production, abnormal heartbeats, stabbing sensations in the hands and feet, and damage to blood vessels. Meanwhile, long-term exposure can lead to skin lesions, internal cancer, neurological problems, lung disease, peripheral vascular disease, hypertension and cardiovascular disease, and diabetes mellitus [3]. Based on Government Regulation No. 28 of 2001 on Management of Water Quality and Control over Water Pollution, the quality standard for heavy metal arsenic (As) in water is 0.05 mg/L [4]. To avoid the negative impact of the heavy metal Arsenic (As), it is necessary to treat the waste before being discharged into the waters.

The most effective and efficient method for absorbing heavy arsenic metal (As) is adsorption. Adsorption is a process that occurs when a fluid (liquid or gas) is bound to a solid and eventually forms a thin layer on the surface, or it can also be interpreted as a result of the force field on the surface of the solid (adsorbent), which attracts gas or liquid molecules (adsorbate). Adsorption is also a chemical equilibrium event; therefore, the reduction in the level of the adsorbed substance (adsorbate) by the adsorbent material (adsorbent) occurs in equilibrium so that,

theoretically, there can be no complete absorption of the adsorbate by the adsorbent. The adsorbed material is called the adsorbate or solute, whereas the absorbent material is called the adsorbent. This adsorbent is specific and is made of a porous material [5].

The manufacture of adsorbents using carbon nanofibers, particularly, has advantages, including high surface area, high mechanical strength, relatively small pore size, porous structure, and high level of modulus of elasticity [6]. One of the potential raw materials for manufacturing carbon nanofibers is sugar palm fiber due to its rich cellulose. This sugar palm fiber is a waste from making flour from sugar palm stem starch [7]. The potential of sugar palm fiber that can be used as an adsorbent because it has a cellulose content of 40.6% [8].

In addition, Zinc oxide is a white solid substance that has molecular formula ZnO with molecular weight of 81.408 g/mol, innert, abundant in nature, environmentally friendly, and non-toxic [9]. ZnO is relatively safe and biocompatible and can be used for biomedical application with minimal toxicity [10]. ZnO is one of the non-toxic inorganic semiconductors which can provide high mobility and good thermal stability. Many studies have been conducted using ZnO as a catalyst, either ZnO as a mono catalyst or ZnO doped with fellow metals (metal-metal) or non-metallic compounds (metal-non-metal) because of its versatility, ease of manufacture, and relatively low cost. In this case, carbon nanofibers modified with ZnO material become a composite material with a dual function, as an adsorbent of the porous nature of carbon nanofibers and as a photocatalyst derived from

ZnO [11]. In this study, it will be a different study from the previous one by trying to make an adsorbent using carbon nano fiber from palm fiber modified with ZnO as an innovation to remediate heavy metals and increase the use value of palm fiber waste. In addition, the effect of ZnO concentration on the characteristics of the resulting carbon nanofibers and their effectiveness in remediating heavy metals were also studied.

METHODS

Material

The materials used included sugar palm fiber (*Arenga Pinnata*), $Zn(CH_3COO)_2.2H_2O$ (Merck), ethanol (p.a), 37.8% HCl (Merck), 99% As_2O_3 (Merck), Whatman No. 41 filter paper, and distilled water.

Procedure

Carbon Nanofiber Manufacturing

This process began with manufacturing carbon nanofibers by washing the sugar palm fiber with distilled water, drying and cutting it into small pieces with a length of 0.5 cm, calcining it at a temperature of 300°C, and smoothing the calcined carbon with a mortar.

Carbon Nanofiber Activation

The mashed carbon nanofibers were soaked in 0.1 M HCl solution for 24 hours at room temperature and then washed with distilled water until neutral and dried in an oven.

Zinc Oxide Manufacturing

The next process was the manufacture of zinc oxide (ZnO), carried out by dissolving 0.1%, 1%, and 10% Zn or 0.003716 g, 0.033716 g, and 0.33716 g $Zn(CH_3COO)_2.2H_2O$ in 42 mL of ethanol solvent while stirring utilizing a magnetic stirrer at 70°C for two hours, and then the solution obtained was cooled to room temperature.

ZnO-Modified Carbon Nanofiber Synthesis

Then, the process of coating carbon nanofibers with zinc oxide (ZnO) was carried out, i.e., by adding a solution of zinc oxide (ZnO) with 1 g of activated carbon nanofiber while stirring vigorously. The resulting mixture was dried at 80°C for 24 hours to remove the solvent, and then the obtained sample was calcined in a furnace by raising the temperature from room temperature to 250°C for an hour. Furthermore, the sample characterization test was conducted utilizing X-Ray Diffraction (XRD), Fourier Transform Infra-red (FTIR), and Scanning Electron Microscope (SEM) instruments. FTIR was employed to determine the functional groups contained in the sample. XRD was used to determine the location of the main peak at 2Ө and the sample's size and degree of crystallinity. Meanwhile, SEM was to determine the morphology and elements contained in the sample's surface.

Adsorption Test

The absorptivity test procedure was carried out with 100 mL of 10 ppb arsenic solution pipetted into a 250 mL Erlenmeyer and then added carbon nanofiber with a mass of 0.1 grams, then shaken for 90 minutes. Then, it was filtered with Whatman filter paper, and the filtrate was taken to measure its absorbance by employing Atomic Absorption Spectroscopy (AAS) [12].
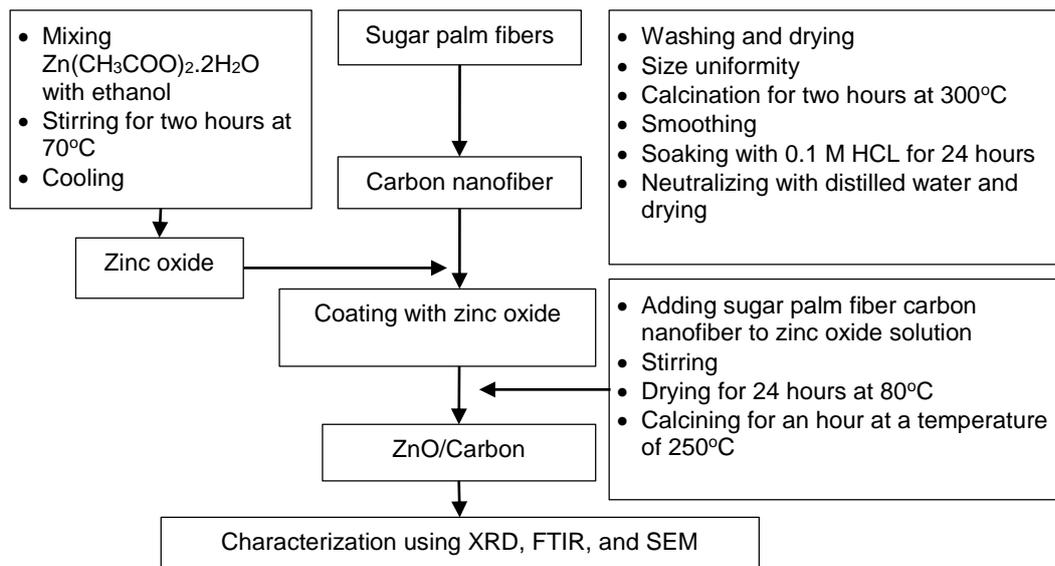
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┌────────────────────────┐   ┌────────────────────┐   ┌──────────────────────────────────┐
│ • Mixing               │   │ Sugar palm fibers  │   │ • Washing and drying             │
│   Zn(CH₃COO)₂.2H₂O     │   └────────────────────┘   │ • Size uniformity                │
│   with ethanol         │            │               │ • Calcination for two hours at   │
│ • Stirring for two     │            ▼               │   300°C                          │
│   hours at 70°C        │   ┌────────────────────┐   │ • Smoothing                      │
│ • Cooling              │   │ Carbon nanofiber   │   │ • Soaking with 0.1 M HCL for     │
└────────────────────────┘   └────────────────────┘   │   24 hours                       │
            │                         │               │ • Neutralizing with distilled    │
            ▼                         │               │   water and drying               │
   ┌──────────────┐                   │               └──────────────────────────────────┘
   │ Zinc oxide   │────────┐          │
   └──────────────┘        ▼          ▼
                    ┌──────────────────────────┐   ┌──────────────────────────────────┐
                    │ Coating with zinc oxide  │   │ • Adding sugar palm fiber carbon │
                    └──────────────────────────┘   │   nanofiber to zinc oxide solution│
                               │                    │ • Stirring                       │
                               ▼                    │ • Drying for 24 hours at 80°C    │
                    ┌──────────────────────────┐    │ • Calcining for an hour at a     │
                    │ ZnO/Carbon               │    │   temperature of 250°C           │
                    └──────────────────────────┘    └──────────────────────────────────┘
                               │
                               ▼
        ┌──────────────────────────────────────────────┐
        │ Characterization using XRD, FTIR, and SEM    │
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Figure 1. Flowchart of carbon nanofiber synthesis from ZnO modified sugar palm fiber

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┌────────────────────────┐         ┌────────────────────────┐
│ 100 ml arsenic solution│────────▶│ Clean water free of    │
│ of 10 ppb              │    ┆     │ heavy metals           │
└────────────────────────┘    ┆     └────────────────────────┘
            ▲                  ┆
            │                  ▼
┌────────────────────────┐    ┌────────────────────────┐
│ Adsorption using       │    │ Sample testing using   │
│ ZnO/carbon nanofiber   │    │ AAS                    │
└────────────────────────┘    └────────────────────────┘
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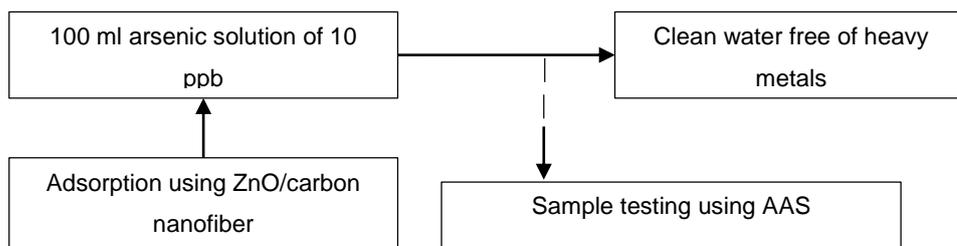
Figure 2. Flowchart of the adsorbent adsorption test on an arsenic solution of 10 ppb

RESULTS AND DISCUSSION

The synthesis of carbon nanofibers in this study used palm fiber washed and dried first to remove impurities and reduce water content, that is done to increase the surface area of the carbon nanofiber and free from impurities that can interfere the adsorption process [13]. Making the size uniform has aim to facilitate the carbonization process, and carbonization oxidized organic compounds to carbon.

Meanwhile, the activation aims to clean and open the carbon pores. Furthermore, the carbon was coated with ZnO at various concentrations to increase its adsorption capacity.

The reactions that occurred in the carbonization process are: Cellulose decomposition reaction (270-310°C)

$(C_6H_{10}O_5)n \rightarrow CH_3COOH + 3CO_2 + 2H_2O + CH_3OH + 5H_2 + 3CO$ ....(1)
Lignin decomposition reaction (310-500°C)
$[(C_9H_{10}O_3)(CH_3O)]n \rightarrow C_{18}H_{11}CH_3 (ter) + C_6H_5OH + CO + CO_2 + CH_4 + H_2$ ....(2)
General reaction for carbon formation (500-1000°C)
$(C_xH_yO_z)n + O_2 \rightarrow C (graphite) + CO_{(g)} + H_2O_{(g)}$ ....(3)
[14].

Characterization with FTIR
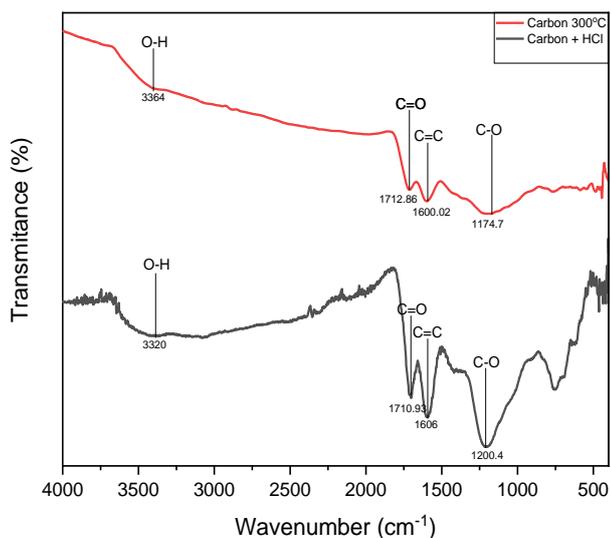
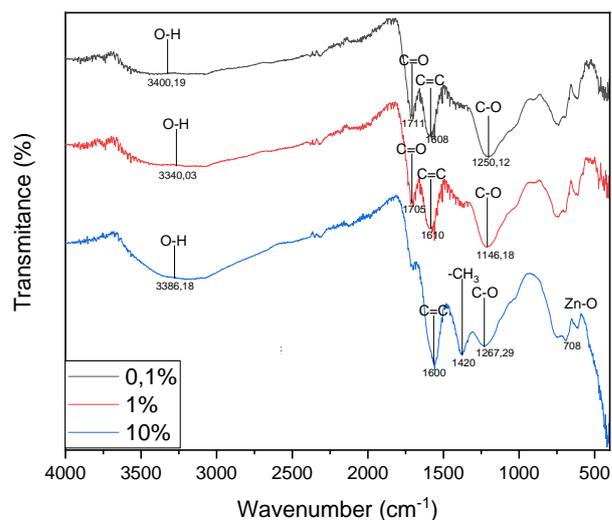**Figure 3.** FTIR results of carbon from sugar palm fiber before and after activation

Figure 4. Carbon FTIR results from zinc oxide (ZnO) modified palm fiber 0.1%, 1%, and 10%

Figure 3. the FTIR spectra, carbon nanofiber samples with a carbonization temperature of 300°C showed an O-H group at the peak of 3364 cm^{-1}. The peak at 1712.86 cm^{-1} indicates the presence of a C=O group. In addition, the peak at 1600.02 cm^{-1} denotes the presence of a C=C bond, and the peak at 1174.70 cm^{-1} signifies the presence of a C-O group. The carbon nanofiber sample after activation with 0.1 M HCl revealed the presence of an O-H group at the peak of 3320 cm^{-1}. The peak at 1710.93 cm^{-1} suggests the presence of a C=O group. Besides, the peak at 1606 cm^{-1} demonstrates the presence of a C=C bond, and the peak at 1200.40 cm^{-1} denotes the presence of a C-O group. It aligns with research Septiani (2014) that activated carbon has functional groups of -OH, -CH, C=C, C-O, and C-O-C [11].

In other words, the carbon from sugar palm fiber had the same functional groups before and after activation, only the intensity differed.

Figure 4. the FTIR spectra of 0.1% ZnO modified carbon nanofiber sample uncovered an O-H group at the peak of 3400.19 cm^{-1}. The peak at 1711 cm^{-1} indicates the presence of a C=O group. The peak at 1608 cm^{-1} signifies the presence of a C=C bond, and the peak at 1250.12 cm^{-1} displays the presence of a C-O group. The 1% ZnO modified carbon nanofiber sample showed an O-H group at the peak of 3386.18 cm^{-1}. The peak at 1705 cm^{-1} denotes the presence of a C=O group. The peak at 1610 cm^{-1} indicates the presence of a C=C bond, and the peak at 1146.18 cm^{-1} signifies the presence of a C-O group.

Figure 4. the FTIR spectra of 10% ZnO modified carbon nanofiber sample revealed an O-H group at the peak of 3386.18 cm^{-1}. The peak at 1600 cm^{-1} indicates the presence of a C=C group. The peak at 1420 cm^{-1} demonstrates the presence of -CH3 bonds. In addition, the peak of 1267.29 cm^{-1} suggests the presence of a C-O group, and the peak of 708 cm^{-1} denotes the presence of a ZnO functional group. Consistent with research [15] stated that metal oxide has a peak in the fingerprint area

below 1000 cm-1. The optimum ZnO absorption peak with FTIR was 745.70 cm-1 [16], Bonding between ZnO is in the range of 400-700cm-1, it means that 708cm-1 clearly represent the ZnO bound [17].

Characterization with XRD

Based on [11] the X-ray diffraction pattern of ZnO, the crystals with the dominant intensity were at a 2θ value of 31.75°; 34.40°; 36.22°. XRD analysis of pure CNF samples showed a crystallinity of 12.18 nm, a degree of crystallinity of 93.66%, and the location of the main peaks at 2θ at 20.6°, 23.5°, and 25.5° indicating the presence of carbon. It is in line with research Ilyas (2019) that the location of the main peak at 2Θ was around 20-25° [18].

XRD analysis on pure CNF + HCl samples revealed a crystallinity size of 17.6 nm, a degree of crystallinity of 98.92%, and the location of the main peaks at 2θ at 22.4°, 23.9°, and 27.80°, suggesting the presence of carbon. XRD analysis on 0.1% ZnO/CNF samples uncovered a crystallinity size of 1.308 nm, a degree of crystallinity of 98.19%, and the location of the main peaks at 2θ at 21.02°, 23.7°, and 25.26°, showing the presence of the element carbon.

XRD analysis on 1% ZnO/CNF samples displayed a crystallinity size of 2.69 nm, a degree of crystallinity of 97.78%, and the location of the main peaks at 2θ at 21.74°, 23.76°, and 27.60° revealing the element carbon.

XRD analysis of 10% ZnO/CNF samples exhibited a crystallinity of 34.5 nm, a degree of crystallinity of 80.72%, and the location of the main peak at 2θ at 31.60°, indicating the presence of ZnO. According to research Alfarisa (2018), the maximum ZnO crystallite size reached 32.23 nm [19]. Sani (2016) shown that peaks of ZnO are obviosly observed at 2θ= 31.5°, 34.8°, 36.5°, 47° and 56.74°, respectively [20].

Thus, the more ZnO is used, the more visible the presence of ZnO at 2θ of 31.60°. In addition, the results of the degree of crystallinity get smaller when more ZnO is added. It is because the crystal size is getting bigger. Factor that influences the enlarging crystallite size after ZnO incorporation is the growth of ZnO, which is affected by precipitation rate, Zn-supporting material, and the calcination temperature [21]. The growth of ZnO in carbon nanofibers is detected by the change of the crystallite size by XRD before and after impregnation data.

Table 1. Carbon XRD results from pure sugar palm fiber and ZnO-modified sugar palm fiber, with various concentrations (above)

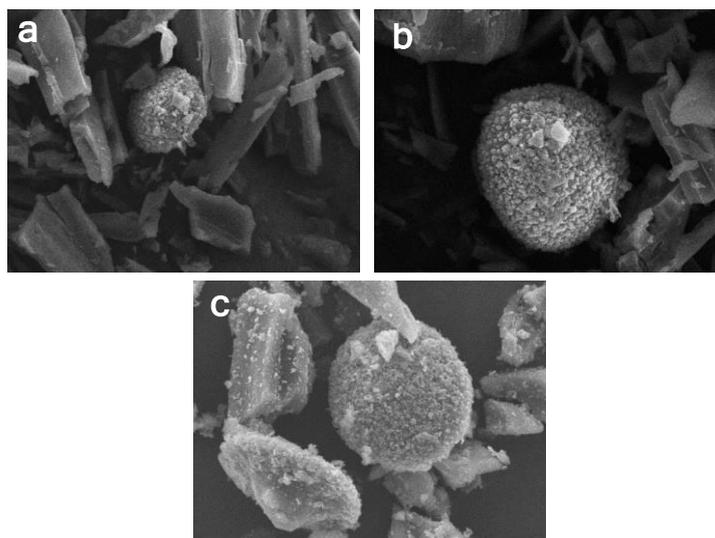| Sample name	Crystallinity size (nm)	Degree of crystallinity (%)	Location of the main peak at 2θ
Pure CNF	12.18	93.66	20.6°, 23.5°, 25.5°
Pure CNF + HCl	17.60	98.92	22.4°, 23.9°, 27.80°
0.1 % ZnO/CNF	1.31	98.19	21.02°, 23.7°, 25.26°
1 % ZnO/CNF	2.69	97.78	21.74°, 23.76°, 27.60°
10 % ZnO/CNF	34.50	80.72	31.60°

Characterization with SEM

Figure 5. Carbon SEM results from a) 0.1%, b) 1%, and c) 10% ZnO modified palm fiber

Figures 5 depict SEM results on carbon coated with 0.1%, 1%, and 10% zinc oxide, where zinc oxide (ZnO) can be seen in the presence of fine white particles on the carbon surface. The greater the concentration of ZnO coated on the carbon, the finer grains spread evenly across the carbon surface (shown in Figure c).

Table 2. Surface elements of ZnO modified carbon nanofibers based on SEM analysis

CNF/ZnO 0.1%	CNF/ZnO 1%	CNF/ZnO 10%
C	C	C
O	O	O
Si	Si	Si
-	-	Zn

Table 2 shows the elements found on the surface of the modified ZnO carbon, where there were C, O, and Si elements in each concentration variation, while Zn was only detected at 10% ZnO variation.

Moreover, the ZnO modified carbon nanofiber produced based on the XRD analysis results was nano-sized, this is supported by [22] which state that the particle size distribution of ZnO nanoparticles varies from 20 to 90 nm. Due to their very small size, nanomaterials have a large adsorption capacity so that they can be used as adsorbents to remediate heavy metals. In addition, based on the SEM results, the addition of ZnO resulted in increasingly fine granules spreading, so the ZnO activity in absorbing heavy metals was also greater, this is evidenced by the adsorption of ZnO-carbon nanofibers on adsorption of heavy metal arsenic.

Adsorption Test of Adsorbent against Arsenic (As) Wastewater

Adsorption with carbon nanofiber from ZnO-modified sugar palm fiber was carried out to determine the ability of the adsorbent to absorb heavy metal arsenic (As) in wastewater. The determination of the absorptivity in this study used variations in the concentration of ZnO of 0.1%, 1%, and 10% in 1 gram of carbon nanofiber.

Table 3. Test results of carbon nanofiber adsorption from sugar palm fiber against arsenic (As) heavy metal waste

No	Adsorbent	Co (mg/L)	Ce (mg/L)	Ct (mg/L)	Arsenic (As) Adsorbed (%)
1	CNF	10.7149	23.3842	-	-
2	CNF-HCL	10.7149	22.2713	-	-
3	ZnO/CNF 0.1 %	10.7149	1.9260	8.7889	82.0250
4	ZnO/CNF 0.1 %	10.7149	1.7946	8.9203	83.2513
5	ZnO/CNF 0.1 %	10.7149	1.3390	9.3759	87.5033

Based on the Table 3, it can be seen that carbon nanofiber with the addition of 10% ZnO had the largest adsorption capacity, where the concentration of adsorbed arsenic metal was 9.3759 mg/L from the initial concentration of 10.7149 mg/L. The percentage of arsenic metal successfully adsorbed was 87.5033%, with a contact time of 90 minutes. Related to that, CNF/ZnO can absorb arsenic metal due to the adsorbent's increased pore volume and surface area due to the presence of metal oxides [23]. ZnO is also easy to generate electron and hole pairs on the semiconductor catalyst surface; the electrons are then excited to reach the surface of the catalyst so that they can reduce heavy metals [24]. In addition, the ability of ZnO/CNF to adsorb heavy metals is due to the presence of C, H, and O atoms, which are chemically bound to form functional groups [25].

CONCLUSION

Sugar palm fiber waste has been succesfully utilized as carbon nanofiber adsorbent. This is indicated by the presence of cellulose-forming functional groups, namely OH groups, CO groups, and CH groups in each adsorbent sample. In addition, the impregnation of zinc oxide on carbon nanofiber has also been successful, as indicated by the presence of Zn-O functional groups in wavenumber of 708 cm^{-1}. In XRD analysis, the crystal sizes of pure CNF, pure CNF+HCl, 0.1% ZnO/CNF, 1% ZnO/CNF, and 10% ZnO/CNF were 12.18 nm; 17.60 nm; 1.31 nm; 2.69 nm; and 34.50 nm respectively. Meanwhile, the SEM analysis revealed that at 0.1% and 1% ZnO/CNF, there was no ZnO, while at 10% ZnO/CNF, there was ZnO. In addition, based on the adsorption test results, it was found that the 10% ZnO/CNF adsorbent had the largest adsorption capacity at 87.5033%. The addition of zinc oxide (ZnO) can increase the adsorption capacity.

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