

## Valorization of Peanut Shell (*Arachis hypogaea*) Compositied Humic Acid as Bioadsorbent of Heavy Metal Contamination

### Valorisasi Limbah Kulit Kacang Tanah (*Arachis hypogaea*) Terkomposit Asam Humat sebagai Bioadsorben Cemaran Logam Berat

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#### ABSTRACT

*The problem of organic and industrial waste still be the focus problems in Indonesia, especially in Cilegon city. In this research, organic waste and peanut shells were valorized into composite biosorbent to treat heavy metal contamination. The aim of this research was to create a biosorbent composite that is applied to adsorb heavy metals in industrial waste. The composite bioadsorbent was used as a source of functional groups such as –COOH and –OH phenolics and –OH alcoholates that can absorb the heavy metals. The adsorption process was carried out by mixing the biosorbent with the metal sample solution for 2h. Characterization was carried out using Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), Inductively Coupled Plasma (ICP) and Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX). Based on the experimental results, the composite bioadsorbent had macro-sized pores which were efficient in adsorbing Pb, Al, and Fe adsorption efficiencies of 66.50%, 36.16%, and 50.96% respectively. The developed composite bioadsorbent has good application prospects in sustainable environmental processing.*

**Keywords:** Biosorbent; Environment; Heavy Metals; Organic Waste; Peanut Shells.

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#### 1. INTRODUCTION

The Cilegon city, which is located in Banten Province, is one of the industrial centers in Indonesia. The waste from several industrial sectors in Cilegon, including the petrochemical and steel coating industries, is highly contaminated with heavy metals, including arsenic (As), chromium (Cr), copper (Cu), lead (Pb), mercury (Hg), and cadmium

(Cd). Based on data from the Public Disclosure for Environmental Compliance (PROPER) of the Ministry of the Environment of the Republic of Indonesia in 2022, there are 21.35% of industries in Cilegon and its surroundings in the red ranking position out of a total of 103 industries [1]. So, Industrial waste that is not properly treated will cause serious environmental problems, especially waste containing heavy metals because they

are carcinogenic and difficult to degrade in the environment [2].

One way to address pollution originating from industrial waste is through adsorption methods using natural materials, commonly referred to as biosorbents [3]. Peanuts are an abundant agricultural commodity in Cilegon. Based on data from the Banten Province Central Statistics Agency in 2021 [4], peanut production reached 4054 tons, but only the seeds were used, while the shells were discarded as agricultural waste. The high cellulose content in peanut shells can be used as a natural source for producing activated carbon for biosorbent. However, the use of activated carbon as an adsorbent heavily depends on its porosity, so other materials are needed to increase binding affinity. In this study, we chose humic acid as an environmentally friendly composite material. humic acid has functional groups such as  $\text{COOH}$ ,  $\text{-OH}$  phenolates and  $\text{-OH}$  alcoholates which will experience deprotonation at alkaline pH and are able to chelate metal. The presence of this group allows humic acid to form stable complexes with heavy metal ions [5,6]. Humic acid is obtained from compost extracts made from organic waste such as vegetables obtained from traditional markets in Cilegon. This is done simultaneously to reduce the accumulation of untreated domestic waste.

Activated carbon has good stability as an adsorbent, while humic acid has a high adsorption capacity [7], so this research focuses on the use of peanut shell activated carbon composited with humic acid from organic waste extract to increase stability and efficiency of metals by the biosorbent composite chelation of metal ions. Some existing research on humic acid focuses on its function as fertilizer and plant fertilizer [8, 9, 10]. Other research on increasing the adsorption capacity of activated carbon was only carried out using the activation method [11,12,13]. Research using peanut shells as an active carbon absorbent has been studied, but there has been no research combining them with humic acid as a biosorbent. While several studies have used carbon in the form of multiwalled carbon nanotubes or graphene,

this study focuses on turning organic waste, specifically, peanut shells and organic waste which are an issue in the city of Cilegon into useful materials. It is hoped that using waste-based biomaterials to deal with heavy metal contamination from industrial waste can become a strategic environmental control effort.

## 2. MATERIALS AND METHODS

### 2.1. Materials

The tools used in this research are centrifuges, ovens, analytical instruments consisting of a Fourier Transform Infrared (FTIR) Spectrophotometer, X-ray Ray Diffraction (XRD), Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX), Inductively Coupled Plasma (ICP), and a set of glassware. The materials used in this research where organic waste was obtained from Kranggut market, with the selected waste consisting of vegetable residues, peanut shell waste was obtained from the Mancak area, horse dung was obtained from the Gegeneng area. All samples were sourced from the Cilegon city region. Phosphoric acid, NaOH, HCl 37%, HF, standard humic acid,  $\text{Pb}(\text{NO}_3)_2$ ,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , and  $\text{Al}_2(\text{SO}_4)_3 \cdot 7\text{H}_2\text{O}$ . All the methods following by Chkraborty, et al. (2020); H. Li et al. (2021); Z. Li et al. (2020) and Zingaretti et al. (2020) modified procedure and by our experimental methods.

### 2.2. Peanut Shell Activated Carbon Preparation

Peanut shells are washed and dried for 12 hours in the sun. Once dry, the peanut shells are roasted until they become charcoal and then crushed. The finely ground peanut shell charcoal was sieved using a 100-mesh sieve and soaked for 7 hours in a 10%  $\text{H}_3\text{PO}_4$  solution. The soaked samples were then filtered using Whatman 42 paper and dried in an oven at  $105^\circ\text{C}$  until a constant weight was reached. The activated carbon (AC) that has been produced is then neutralized with distilled water to pH 7. After being dried in an oven at  $110^\circ\text{C}$ , the neutral activated carbon is examined for water content and iodine absorption capability to assess its adsorbent quality. To describe functional group in

activated carbon, used Fourier transform infrared spectroscopy (FTIR).

### 2.3. Compost Production

The production of compost from organic waste aims to serve as the primary material for generating humic acid. Organic waste obtained from Kranggol Market, Cilegon-Banten, is mixed with dried horse manure as a starter in a ratio of 2:1. The mixture is anaerobically placed in a 20 L composting tank. The compost is irrigated once a week using 500 mL of EM4 and a sugar solution. The composting process takes place for around one month. After one month, the compost was dried naturally in the sun for 2 days. Drying continued in an oven for 36 hours. The dry compost is then crushed and uniform in size using a 100-mesh sieve.

### 2.4. Humic Acid Extraction

A total of 250 g of dry compost was added to 3 L of 0.1 M NaOH solution and shaken for 2 hours, then left for 24 hours. The next day, the liquid was centrifuged at 3950 rpm for 15 minutes. Filter the filtrate and acidify with concentrated HCl, shake for 2 hours and leave for 24 hours. The crude extract was centrifuged at 3950 rpm for 15 minutes, and then the sediment was separated from the filtrate. The precipitation obtained is crude humic acid. Crude humic acid was purified with a solution of 0.1 M Hydrochloric Acid (HCl) and 0.3 M Hydrofluoric Acid HF at 1:1 four times. The purification results are then washed with distilled water. Pure humic acid was then oven-treated at 40 °C and characterized by Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD).

### 2.5. Synthesis Activated Carbon Composites - Humic Acid

Synthesis of activated carbon composite with humic acid aimed at a high metal adsorption capacity biosorbent. The solution containing 0.3 g humic acid in 50 mL of distilled water was stirred with a magnetic stirrer for 6 hours to obtain a saturated solution. Then it was filtered using Whatman 42 filter paper, followed by the addition of 50

mL of the mixture containing 0.5 g activated carbon adjusted to pH 8.5 with a 0.1 M Tris buffer solution. The resulting mixture was stirred for 12 hours with a magnetic stirrer and then filtered. The filtered result obtained is called a biosorbent composite. The biosorbent composite formed was washed three times with distilled water and dried at 60 °C for 24 hours.

### 2.6. Batch Adsorption of Heavy Metals and Real Sample Applications

A mixture of the heavy metals Pb, Al, and Fe from the salts of  $\text{Pb}(\text{NO}_3)_2$ ,  $\text{Al}_2(\text{SO}_4)_3 \cdot 7\text{H}_2\text{O}$  and  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , was made with the same concentration ratio, and its concentration was first analyzed using Inductively Coupled Plasma (ICP) as initial metal content data. A total of 20 mg of biosorbent composite was added to 20 mL of synthetic liquid waste, then stirred at a speed of 200 rpm for 2 hours. The solution was filtered, and the filtrate obtained was analyzed by ICP. The analysis results of metal content using ICP before and after the adsorption process will be compared and the efficiency percentage will be calculated. For the application in real samples, the same treatment was carried out but using samples a real sample from industrial liquid waste in Cilegon. While the solid biosorbent that has been used was dried and characterized using EDX to observe the percentage of metals adsorbed onto the biosorbent. The morphology of the biosorbent composite was characterized using Scanning Electron Microscope (SEM). The percentage efficiency of adsorption is obtained by:

$$R = \frac{C_0 - C_1}{C_1} \times 100\%$$

Where  $C_0$  and  $C_1$  is concentration of solution before and after adsorption, respectively.

## 3. RESULTS AND DISCUSSION

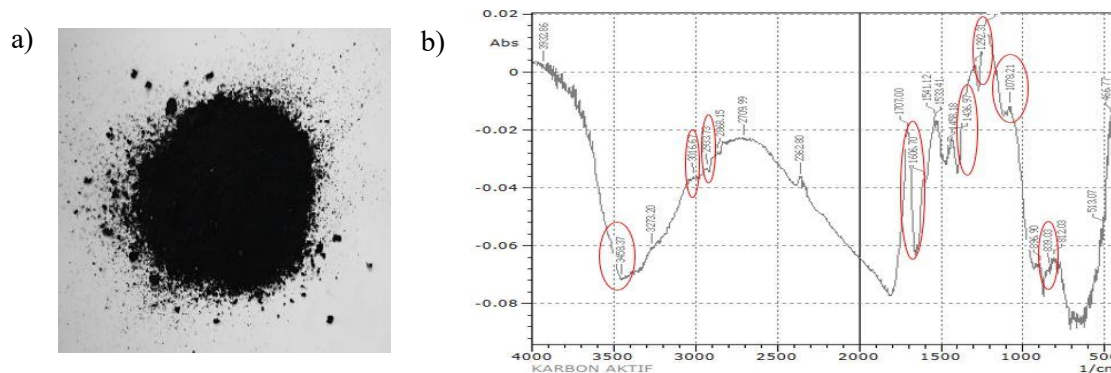
In this research, the author used organic vegetable waste as a source of humic acid and peanut shells which are converted into active carbon to carry out a waste valorization procedure. Organic waste was previously composted for 1 month, before being extracted into humic acid. These two materials are then

composed into a biosorbent which aims to adsorb metal ions. Performance tests were conducted on laboratory samples consisting of a mixture of Pb, Fe, and Al metals prior to testing on actual samples in the form of industrial waste testing with instruments such as FTIR, XRD, SEM-EDX and ICP has been carried out to support data interpretation.

### 3.1. Peanut Shell Activated Carbon Preparation

The process of making activated carbon goes through two stages, namely carbonization and activation. Since carbonization produces carbon has a low absorption capacity, activation is necessary to expand the carbon pores surface area. For the lignocellulosic materials, activation process used  $H_3PO_4$  is better compared to alkaline activators because acid activators can form functional groups that contain oxygen and provide hydrophilic properties to activated carbon [14, 15]. After the activation process, the carbon is then neutralized and dried.

reinforced by the absorption at  $1436.97\text{ cm}^{-1}$  which is an acetylene vibration. The aromatic C–H strain is found at wave numbers  $839.03\text{ cm}^{-1}$  and  $3016.67\text{ cm}^{-1}$ . The C=O group is a typical group found in active carbon, shown at wave number  $1606.70\text{ cm}^{-1}$ . Carbon bound to organic phosphate because of  $H_3PO_4$  activation is shown at a wave number of  $1292.31\text{ cm}^{-1}$  [16,17]. This research result is confirmed by research from Mohtashami et al. who conducted carbon activation of sugarcane bagasse with  $H_3PO_4$ . This research shows the peak spectra of FTIR in nearly the same region, In Mohtashami's et al. (2021) research, the broad intense peak at  $3400\text{ cm}^{-1}$  represents the stretching vibration of O–H bonds in phenolic functional groups and carboxylates, meanwhile OH– bond in our research appeared in  $3458.37\text{ cm}^{-1}$ . The stretching vibration of C–H bonds cause vibrations around  $2900\text{ cm}^{-1}$  and  $1450\text{ cm}^{-1}$ . And we obtained the bond of aliphatic C–H at  $2933.73\text{ cm}^{-1}$ .



**Figure 1.** (a) Activated Carbon from Peanut Shell (b) FTIR Characterization of Activated Carbon

The identification of active carbon functional groups in peanuts was analyzed using FTIR. With this instrument, the chemical structure of a activated carbon can be analyzed by examining its molecular bonds and composition.

The FTIR spectrum of activated carbon has an absorption band at a wavelength of  $3458.37\text{ cm}^{-1}$  which is the OH functional group, strengthened by the presence of C–O vibrations from secondary OH at  $1078.21\text{ cm}^{-1}$ . The absorption at  $2933.73\text{ cm}^{-1}$  shows the presence of aliphatic C–H stretching,

The C=O bond is present in the structures of ketones, aldehydes, lactones, or carboxyl groups, resulting in a small peak around  $1700\text{ cm}^{-1}$ . In our research, the C=O appears in the region of  $1606.70\text{ cm}^{-1}$ . Thus, the FTIR results of the activated carbon we produced show the appropriate characteristic peaks [30]. Activated carbon resulting from roasting peanut shells and FTIR characterization is shown in Figure 1.

The quality of activated carbon as an adsorbent was carried out on two parameters, namely water content and iodine absorption

capacity, which referred to SNI 06-3730-1995, can be seen in Table 1.

**Table 1.** Result of Activated Carbon Quality

| Parameter                         | Result | SNI 06-3760-1995<br>Specification |
|-----------------------------------|--------|-----------------------------------|
| Water content (%)                 | 5      | Maks. 15                          |
| Iodine absorption capacity (mg/g) | 24.890 | Min. 750                          |

### 3.2. Result of Compost Production

Samples were made using composted organic waste and horse dung through a humification method. This compost will be used as a source of humic acid. The characteristics of the compost produced meet SNI 19-7030-2004 with test parameters for texture, odor, and color which can be seen in Table 2.

**Table 2.** Characterization of Compost

| Parameter | Result           | SNI 19-7030-2004<br>Specification |
|-----------|------------------|-----------------------------------|
| Texture   | Not Fibrous      | Not Fibrous                       |
| Odor      | Smell like earth | Smell like earth                  |
| Color     | Blackish Brown   | Black                             |

Based on these three parameters, it can be concluded that the results of the humification of organic waste and horse manure have successfully transformed into compost. This compost will then be extracted into humic acid.

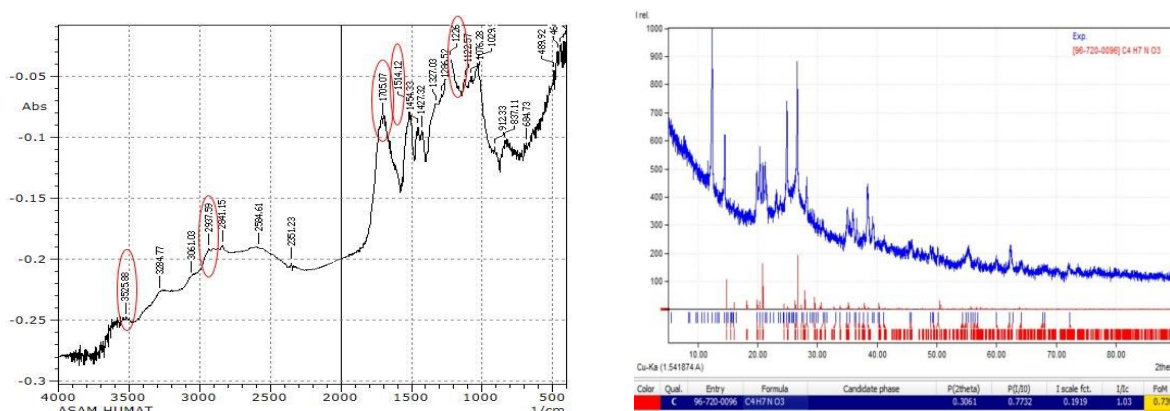
### 3.3. Humic Acid Extraction

The extraction of humic acid is carried out using the compost previously prepared. The extraction is performed using NaOH solution to separate humate and vulvate contained in the compost. Purification process of Crude

humic with a HCl and HF solution. The purification results then washed with distilled water and dried. The extract of humic acid that had been dried was characterized using FTIR and XRD.

This characterization result is confirmed by other research to compare the obtained results. The broad and dull absorption peak occurring at  $3450\text{ cm}^{-1}$  can be associated with the stretching vibration of  $\text{—OH}$  in  $\text{—COOH}$ , phenol, and alcohol. While our FTIR spectrum of humic acid has an OH absorption band at a wavelength of  $3525.88\text{ cm}^{-1}$ , strengthened by the presence of OH bending and stretching vibrations from the carboxylate group at a wavelength of  $1226\text{ cm}^{-1}$ . There is an aromatic component structure for the  $\text{C=O}$  stretching vibration of the  $\text{—COOH}$  group at a wavelength of  $1705.07\text{ cm}^{-1}$ . The strong vibration peak approaching  $1640\text{ cm}^{-1}$  is associated with the stretching vibration of  $\text{C=C}$  in the benzene ring, then our resulted  $\text{C—C}$  from the aromatic ring at  $1514.12\text{ cm}^{-1}$  and aliphatic  $\text{C—H}$  at a wavelength of  $2937.59\text{ cm}^{-1}$ .

Although not identical, the range we obtained is similar. Therefore, to further confirm, we used XRD. XRD used to confirm the structure of humic acid based on their diffraction patterns [18]. XRD analysis is carried out on solid substances. These results were also confirmed through X-ray diffractograms compared with JCPDS. The two theta peaks indicate results that indicate humic acid peaks at  $2\theta = 10, 30, \text{ and } 40$  degrees [19,20]. From the XRD peaks, it appears that the purity of the humic acid is still not sufficiently high. However, with the presence of several matching characteristic peaks, we conclude that the synthesized humic acid has been formed. The results of the humic acid analysis can be seen in Figure 2.



**Figure 2.** The Result of Humic Acid Analysis (a) FTIR Characterization of Humic Acid (b) XRD characterization of Asam Humic Acid

While natural humification is a geochemically protracted process requiring decades to millennia, recent advances in biotechnology and catalytic chemistry have demonstrated that the formation of humic-like substances (HLS) can be substantially accelerated under controlled conditions. Several studies have demonstrated that short-term composting (30–60 days) can yield materials rich in humic-like substances that exhibit similar functional groups and characteristics to naturally formed humic acids. Recent studies demonstrate that microbial-mediated biotransformation can significantly accelerate humic acid (HA) formation under controlled conditions. For instance, Zhang *et al.* (2022) showed that co-inoculation of *Bacillus subtilis* and *Aspergillus fumigatus* during composting enhanced HA production by 53% within 35 days, with  $^{13}\text{C}$ -NMR confirming a 38% increase in aromaticity and FTIR revealing characteristic HA functional groups ( $1725\text{ cm}^{-1}$  carboxyl,  $3400\text{ cm}^{-1}$  phenolic  $-\text{OH}$ ) [31].

This aligns with earlier work by Jurado *et al.* (2015), where *Trichoderma reesei* and *Bacillus subtilis* increased HA aromaticity by 42% in 40 days, confirming microbial consortia can replicate natural humification processes in weeks rather than years.

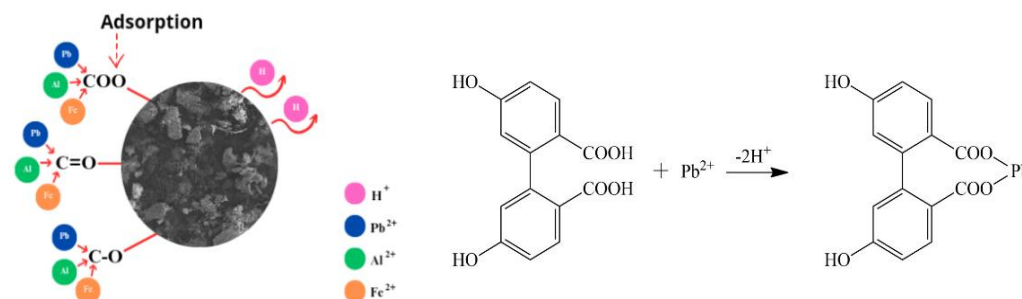
Both studies employed rigorous validation (IHSS protocols, enzymatic activity assays, and spectral analysis) to prove the structural

equivalence of microbially synthesized HA to natural standards, offering scalable strategies for sustainable HA production [32]. In this study, the microbial mediation process used EM4 bacteria. The composting process lasted for 30 days, as described based on the research method by Maimunawaroh [33].

### 3.4. Biosorbent Performance Results

Compositing process of activated carbon and humic by stirring for 12 hours without the use of a cross-linking agent, allowing electrostatic forces to be the only means of interaction between the two. Activated carbon provides adsorption sites that are wide enough to allow the metal to diffuse into the pores of the biosorbent composite, while humic acid has  $-\text{COOH}$ ,  $-\text{OH}$  phenolic, and  $-\text{OH}$  alcoholic groups that act as electron pair donors. The adsorption mechanism occurs when the  $-\text{COOH}$  group experiences  $\text{H}^+$  deprotonation under alkaline conditions adjusted to the Tris buffer pH 8.5 due to the presence of  $\text{OH}^-$  ions [21,22, 23]. That's when ion exchange occurs which causes the carboxylate group to have a negative charge ( $\text{COO}^-$ ) and tend to bind with metal ions [24,25]. The interaction process of metal cations with the active groups of the bioadsorbent composite can be seen in Figure 3.





**Figure 3.** Mechanism of Ion Metal Adsorption

The ability of biosorbents to adsorb metals was measured using ICP. Adsorption efficiency and capacity were tested on the analyte content parameters Pb, Al, and Fe using artificial waste with concentrations containing 0.1 and 1 ppm for each metal and on real samples from Ciwandan, one of subdistrict in Cilegon City, waters in batches. The ICP instrument was chosen due to its accuracy and sensitivity in detecting the presence of metals. Furthermore, in this study, the three metals are mixed into one during the adsorption process, making the use of ICP expedient for the analysis process in terms of time. The determination of adsorbent efficiency states the percent of metal content that can be adsorbed, while adsorption capacity states the ability of an adsorbent to adsorbate. The efficiency value calculation, as mentioned in the method, involves analyzing the effectiveness of the adsorption process in removing pollutants from the solution. It is known that the percentage efficiency of 2 hours of contact time ranges from 19.5019-96.4978%. The best adsorption results were found on Pb metal with a concentration of 10 ppm, namely 96.4978%, which means 96.4978% of the total adsorbed mass was adsorbed or 0.9203 mg/g. The complete results of metal adsorption efficiency can be seen in Table 3.

Anggriani *et al.* reported that the adsorption efficiency of coconut shell-activated carbon on Pb metal was 85.6% [26], while the adsorption efficiency of humic acid on Pb metal of 54.06% [27]. The results of activated carbon adsorption on Fe metal

showed an efficiency value of 44% [28], while the efficiency of adsorption of Fe metal by humic acid was 34.78%. It can be concluded that the results of the adsorption efficiency of Pb and Fe metals in this research are relatively greater compared to several previous studies. In this study, the three metals are mixed into one solution during the adsorption process.

**Table 3.** Results of Metal Adsorption Efficiency

| Sample<br>Concentration<br>(ppm) | Adsorption Efficiency |        |        |
|----------------------------------|-----------------------|--------|--------|
|                                  | Pb                    | Fe     | Al     |
| 1                                | 74,210                | 58,696 | 41,351 |
| 10                               | 96,498                | 45,216 | 19,502 |

The adsorption capacity of Fe is relatively low, possibly due to the stronger attraction of Pb, leading Pb to occupy the biosorbent pore sites first, while Fe fills the remaining available pores. As for Al metal, the efficiency value is relatively small compared to Pb and Fe metals. This is because the  $\text{Al}^{3+}$  ions react first with phosphate ions which come from the activated carbon activation process to form  $\text{AlPO}_4$  resulting in the number of  $\text{Al}^{3+}$  ions in the solution being reduced so that the adsorption efficiency decreases [29,30]. In this research, real sample testing was carried out in Ciwandan waters, resulting in Pb, Al, and Fe adsorption efficiencies of 66.50%, 36.16%, and 50.96% respectively. The efficiency results of testing on real samples are relatively lower compared to synthetic (laboratory) samples. This is because there are more matrices present in real samples. Since in this

study, the real samples were not destructed beforehand but directly adsorbed with the biosorbent.

### 3.5. Morphology and Composition of Biosorbent

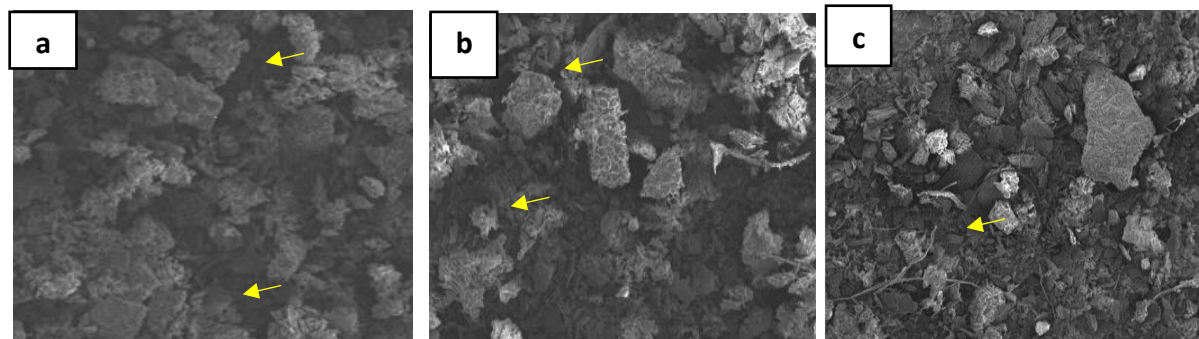
Adsorption ability can be influenced by the average pore diameter of the adsorbent. The larger the average pore diameter of the adsorbent, the better the adsorption efficiency. SEM is used to analyze the surface morphology of a material; in this case, the analysis reveals the pores of the bioadsorbent composite. The pore diameter measured by ImageJ software. Based on the calculation of the average distribution of the adsorbent pore diameter, the result was 4,344  $\mu\text{m}$ .

Enough pore diameter exists for effective adsorbate adsorption. After the adsorption process, the pore cavity of the adsorbent appears to be more filled with adsorbate. This is shown by the surface morphology via SEM images at 500x dan 1000x magnification. The average distribution of the adsorbent pore diameter upon adsorption decreased by 1,216  $\mu\text{m}$ , according to the measurement data. The surface morphology before and after the adsorption process can be seen in Figure 4.

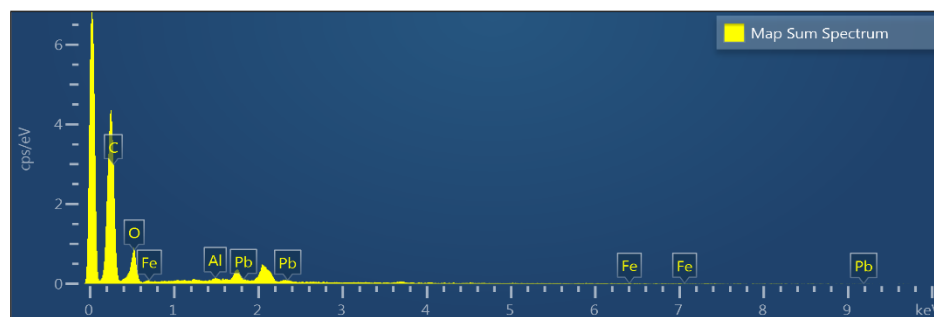
Image 4a shows the morphology before the adsorption process. From the indicated arrows, the pores of the biosorbent are still wide. It is at these sites that metals will be adsorbed. In Figure 4b, after the adsorption process, the pore image appears fuller. This is because the pores are now filled with metals that have been adsorbed at those sites. To ensure that the pores have indeed been filled with metals after the adsorption process, characterization with EDX is conducted.

EDX is utilized to determine the elemental composition of biosorbent composites after they have been used for metal adsorption. Based on measurements with EDX, the elemental composition after the adsorption process can be identified by the appearance of Pb, Al, and Fe spectrum peaks respectively with atomic abundance percentages of 0.39%, 0.47%, and 0.85%. The EDX graph after trapping can be seen in Figure 5.

With the detection of Pb, Al, and Fe metals used EDX, which are the pollutants used in this study, it proves that the material filling the pores of the biosorbent in the SEM image is indeed waste metal. Thus, the biosorbent we have created can effectively absorb these metals.



**Figure 4.** Morphology of Bioadsorbent Composite (a) Before Heavy Metal Adsorption 500x (b) After Heavy Metal Adsorption 500x (c) After Heavy Metal Adsorption 1000x



**Figure 5.** EDX Result After Adsorption



This study is an initial exploration into combining the functions of activated carbon and humic acid as absorbents. The results obtained are quite satisfactory, with a good adsorption performance. However, further research on the formula or ratio comparison of activated carbon and humic acid used would provide more characteristic information. This aims to determine the composition that provides the best efficiency in adsorbing various types of metals.

#### 4. CONCLUSIONS

A biosorbent composite based on peanut shells and organic waste has been synthesized. The synthesized biosorbent composite has a macro pore size that can adsorb  $Pb^{2+}$  and  $Fe^{2+}$  metals efficiently, with adsorption percentages reaching 96.49% and 58.69%, respectively. Meanwhile,  $Al^{3+}$  adsorption has a smaller efficiency value because  $Al^{3+}$  ions react first with phosphate ions to form  $AlPO_4$ . Overall, this research can be applied to reduce industrial waste samples. Since the adsorption efficiency percentage is already more than 50%, with the best result reaching over 90%. To achieve a better efficiency value, more research is necessary to determine the exact amounts of each substance (humic acid and active carbon) and the adsorption experiments were conducted separately for each metal. This biosorbent is very prospective to be developed to overcome environmental problems.

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