

(Research Article)

# The Effect of Contact Time and Composition of Activated Carbon-Humic Acid Composite on the Efficiency of Lead (Pb) Reduction

Ninik Triayu Susparini<sup>1\*</sup>, Fita Retnoningtyas<sup>2</sup>, Muhammad Irfan Junaedi<sup>3</sup>, Micha Mahardika<sup>1</sup>, Isna Lailatusholihah<sup>1</sup>, Holishah Widiyanto<sup>1</sup>

<sup>1</sup> Chemical Analyst, Sekolah Tinggi Analisis Kimia Cilegon

<sup>2</sup> Chemistry, Sekolah Tinggi Analisis Kimia Cilegon

<sup>3</sup> PT. Lotte Chemical Titan Nusantara

\* Correspondence author: [alkikaliks4resorsinarena@gmail.com](mailto:alkikaliks4resorsinarena@gmail.com)

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**Abstract:** Liquid waste from industries containing lead (Pb) has the potential to pollute the environment if not treated properly. This study utilized activated carbon-humic acid composites as adsorbents for lead (Pb) metal ions. The research aimed to examine the effects of contact time and the composition of activated carbon-humic acid composites on reducing lead (Pb) levels. The composite was synthesized through a physical stirring method without the addition of binding agents. Characterization using FTIR revealed the presence of functional groups such as OH, C-O, C-H, and C=O, which are responsible for metal adsorption. Adsorption efficiency was evaluated using AAS to measure the reduction of lead (Pb) content. The results showed a lead (Pb) content reduction of 9.406 ppm at a composite concentration of 10 mg and a contact time of 3 hours, achieving an efficiency of 93.58%. The produced adsorbent followed the Freundlich isotherm adsorption equation, as indicated by a regression coefficient value of 0.8763, which is close to 1

## 1. INTRODUCTION

Industrial wastewater in Cilegon City is a significant environmental concern, contributing heavily to water pollution. It contains organic and inorganic compounds, including heavy metals, sulfur, sulfates, ammonia, nitrates, and nitrites. Key indicators of water pollution include changes in temperature, pH, odor, color, taste, microbial presence, and increased radioactivity. Pollution sources are classified into point and nonpoint sources. Point sources, like factory discharges, are easily identifiable, while nonpoint sources, such as pollutants carried by rainwater or river runoff, are more diffuse and challenging to trace [1]. Approximately 2.8 million locations in Europe are thought to be at risk of heavy metal contamination (FAO and UNEP, 2021). Significant environmental issues like water pollution, polluted vegetation, and a decrease in biodiversity are brought on by exposure to heavy metals in natural soils [22]. Lead contamination of water is another significant problem in China. For instance, the World Health Organization's (WHO) drinking water quality standard of 10 µg/L is greatly exceeded by the detected lead concentration of 150 µg/L at the Pearl River estuary [23]. Heavy metals are one of the main causes of environmental pollution because they are not biodegradable in nature and create harmful and deadly health issues for people when they enter the food chain.

Lead is a toxic heavy metal that poses significant health risks to humans when its concentration exceeds safe limits, causing conditions such as anemia, central nervous system damage, and kidney failure. It is a bluish-gray metal that is easily refined during mining processes. In aquatic environments, lead contamination primarily results from the discharge of domestic and industrial waste into rivers. In stagnant water, lead settles as sediment on the riverbed, while in flowing waters, it accumulates in the tissues of aquatic organisms, including fish and plants [2]. Adsorption, which involves using adsorbent materials to remove contaminants, is an effective method for addressing industrial wastewater pollution.

An adsorbent is a solid material with a large internal surface area, created by numerous fine pores on its surface, which allows it to effectively capture contaminants. Activated carbon, a widely used adsorbent in water treatment, is derived from carbon-rich materials such as coconut shells, bamboo, charcoal from wood burning, and lignite (brown coal). These materials are activated through physical or chemical processes to enhance the

surface area of the adsorbent [3]. In addition to activated carbon, humic acid is also utilized as a chemical adsorbent for water treatment.

Humic acid contains functional groups such as  $-\text{COOH}$ , phenolic  $-\text{OH}$ , and  $-\text{OH}$ , which make it effective in adsorbing heavy metals from contaminated soils or water. Humic acid can adsorb heavy metals in basic conditions, as it dissolves in media with a pH of around 6 or higher, with a solubility of up to 95%. This characteristic makes humic acid a useful adsorbent for treating industrial wastewater and reducing environmental contamination from heavy metals [4].

Several studies have explored the use of humic acid as an adsorbent. For example, Maimunawaro et al [4] reported an adsorption capacity of 52%. Other research, such as that by Sari et al [5], investigated the enhancement of activated carbon's adsorption capacity under varying pH levels and contact times, achieving an efficiency of 70%. However, limited research has focused on specific factors, such as the effects of contact time and the composition of activated carbon-humic acid composite adsorbents on lead (Pb) reduction efficiency. This study aims to address these gaps by investigating these parameters.

## 2. EXPERIMENTAL METHOD

### 2.1 Tools & Materials

The instruments used in this study include an oven, pH meter, and analytical instruments such as a Fourier Transform Infrared Spectrophotometer (FTIR), Atomic Absorption Spectrophotometer (AAS, Shimadzu Model FLAME AA-7000, Japan), Whatman filter paper, and a set of glassware. The materials used in this study include activated carbon from peanut shells, aquades (distilled water), Tris buffer, humic acid, and  $\text{Pb}(\text{NO}_3)_2$  (lead nitrate). All chemicals were sourced from Sigma-Aldrich, USA.

### 2.2 Procedur

All experiments are conducted according to a standardized procedure. The solution is prepared fresh prior to each experimental treatment. Two adsorption methods are used: the first involves a preliminary test, where the adsorbent mass and contact time remain constant. The second method uses an experimental design with independent variables, including adsorbent mass and contact time. Throughout the adsorption process, all experiments are maintained at a neutral pH and room temperature, with stirring at a constant speed of 200 rpm.

#### 2.2.1 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 (KA) 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. The functional group of activated carbon characterized by Fourier transform infrared spectroscopy (FTIR).

#### 2.2.2 Preparation of Activated Carbon-Humic Acid Composite

The solution containing 0.3 g of humic acid was stirred in 50 mL of distilled water with a magnetic stirrer for 6 hours to obtain a saturated solution. It was then filtered using Whatman filter paper. Next, 50 mL of a mixture containing 0.5 g of activated carbon was added, and the pH was adjusted to 8.5 using a 0.1 M Tris buffer solution. The resulting mixture was stirred for 12 hours, followed by filtration. The bioadsorbent composite was dried at  $60^\circ\text{C}$  for 24 hours. Finally, the bioadsorbent composite was characterized using FTIR and Scanning Microscope Elektron (SEM).

#### 2.2.3 Preparation of 10 ppm Pb Standard Solution

As much as 0.08 grams of  $\text{Pb}(\text{NO}_3)_2$  was dissolved in 100 mL of distilled water, then transferred to a 500 mL volumetric flask and diluted to the mark to obtain a 100 ppm lead solution. Next, 5 mL of the 100 ppm lead solution was pipetted into a 50 mL volumetric flask and diluted to the mark to obtain a 10 ppm lead solution.

#### 2.2.4 Lead (Pb) Metal Adsorption with Activated Carbon

The concentration of the Pb mixture was first analyzed using AAS to obtain the initial metal content data. Then, 20 mg of activated carbon adsorbent was added to 20 mL of a 10 ppm Pb standard solution and stirred at 200 rpm for 2 hours. The solution was filtered using Whatman filter paper. The filtrate obtained was then analyzed using AAS.

### 2.2.5 Lead (Pb) Adsorption with Humic Acid

The concentration of the Pb mixture was first analyzed using AAS to obtain the initial metal content data. Next, 20 mg of humic acid adsorbent was added to 20 mL of a 10 ppm Pb standard solution and stirred at 200 rpm for 2 hours. The solution was then filtered using Whatman filter paper, and the obtained filtrate was analyzed using AAS

### 2.2.6 Lead (Pb) Adsorption with Activated Carbon-Humic Acid Composite

The activated carbon-humic acid composite mixture was prepared with composition ratios of 10, 20, and 40 mg, each with variation times of 2 and 3 hours. The composites were added to 20 mL of a 10 ppm Pb solution and stirred at 200 rpm. The solution was then filtered using Whatman filter paper, and the obtained filtrate was analyzed using AAS. This experiment was conducted in duplicate, resulting in 12 variations of experimental conditions, with no treatment serving as the negative control. The experimental design is shown in Table 1.

**Table 1. Experimental Design**

Factor A:	Factor B (hours)	Ulangan	Factor A (Composition)			Composite
			A1	A2	A3	
	B1	I	A1B1	A2B1	A3B1	
		II	A1B1	A2B1	A3B1	
	B2	I	A1B2	A2B2	A3B2	
		II	A1B2	A2B2	A3B2	

Composition

A1: Composite Composition 10 mg

A2: Composite Composition 20 mg

A3: Composite Composition 40 mg

Factor B: Contact Time

B1: Contact Time 2 hours

B2: Contact Time 3 hours

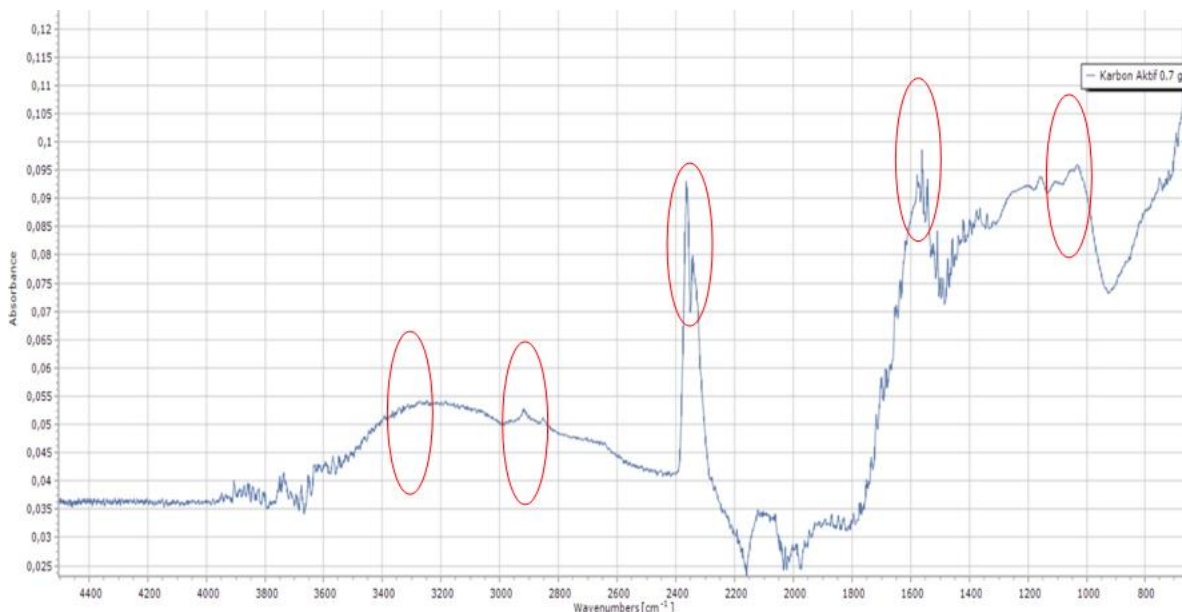
## 3. RESULT AND DISCUSSION

### 3.1 FTIR Characterization of Activated Carbon

The FTIR spectrum of activated carbon displayed an absorption band at 3300 cm<sup>-1</sup>, attributed to the OH functional group, supported by the C-O vibration of secondary OH at 1050 cm<sup>-1</sup>. An absorption at 2930 cm<sup>-1</sup> indicated the presence of C-H stretching, which was further confirmed by the absorption at 1500 cm<sup>-1</sup>, corresponding to acetylene vibrations. Another C-H stretching was observed at 2350 cm<sup>-1</sup>. The characteristic C=O group of activated carbon was identified at 1570 cm<sup>-1</sup> [6-7]. FTIR Analysis of Activated Carbon The analysis is presented in Table 2 and Figure 1.

**Table 2. Analysis of Activated Carbon**

No	Fuctional Group	Wavenumber (cm <sup>-1</sup> ) Activated Carbon
1	OH	3300 cm <sup>-1</sup>
2	C-O	1050 cm <sup>-1</sup>
3	C-H	2930 cm <sup>-1</sup> , 2350 cm <sup>-1</sup>
4	C=O	1570 cm <sup>-1</sup>



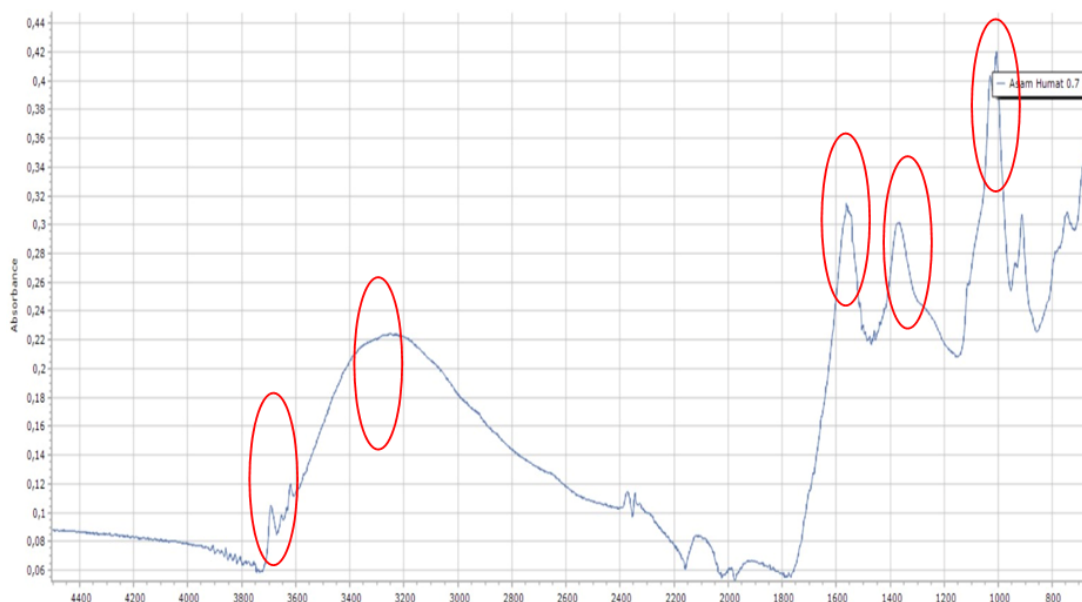
**Figure 1. Infrared Spectrophotometry of Activated Carbon**

### 3.2 FTIR Characterization of Humic Acid

Based on the FTIR data in Figure 2, the characterization results of humic acid are presented. The FTIR spectrum of humic acid shows an absorption band at 3300  $\text{cm}^{-1}$ , corresponding to the OH functional group, supported by the C-O vibration of secondary OH at 1130  $\text{cm}^{-1}$ . The C=O group is observed at 1570  $\text{cm}^{-1}$ , while an absorption at 1390  $\text{cm}^{-1}$  indicates the presence of an  $\text{NO}_2$  compound. The  $\text{NH}_2$  group, a characteristic functional group of humic acid, is identified at 3695  $\text{cm}^{-1}$ . The detailed FTIR analysis of humic acid is summarized in Table 3 and Figure 2 [4,8-9].

**Table 3. Analysis of Humic Acid**

No	Fuctional Group	Wavenumber ( $\text{cm}^{-1}$ )
		Humic Acid
1	OH	3300 $\text{cm}^{-1}$
2	C-O	1130 $\text{cm}^{-1}$
3	C-H	1570 $\text{cm}^{-1}$
4	$\text{NO}_2$	1390 $\text{cm}^{-1}$
5	$\text{NH}_2$	3695 $\text{cm}^{-1}$



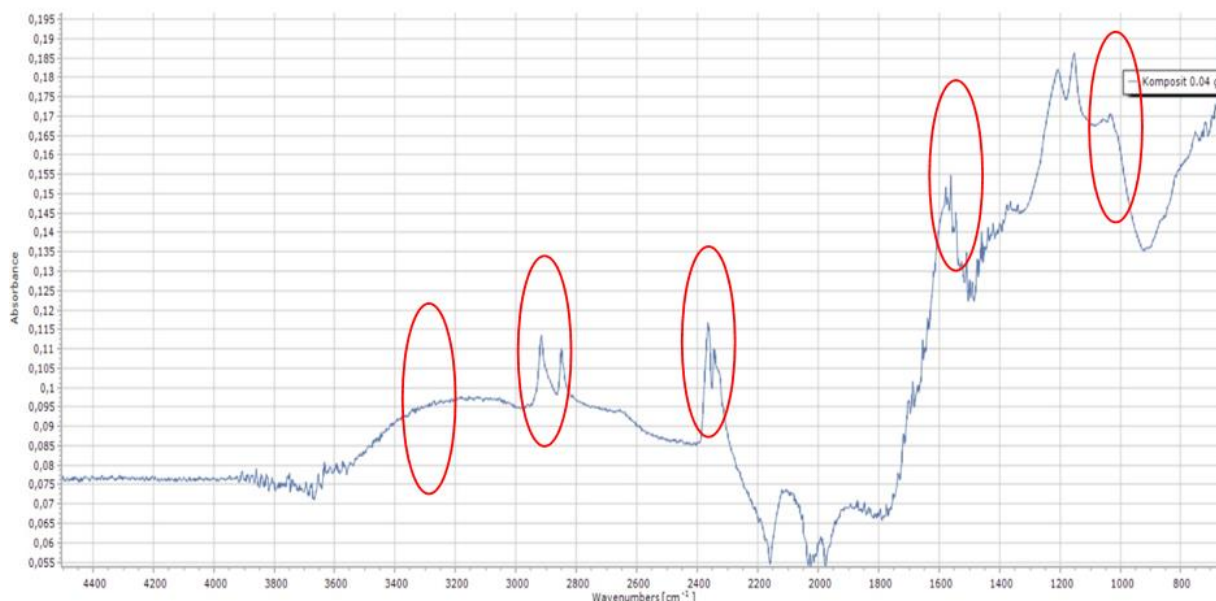
**Figure 2. Infrared Spectrophotometry of Humic Acid**

**3.3 FTIR Characterization of Activated Carbon-Humic Acid Composite**

Based on the FTIR data in Figure 3, the characterization results of the activated carbon-humic acid composite are presented. The FTIR spectrum shows an absorption band at 3300 cm<sup>-1</sup>, corresponding to the OH functional group, supported by the C-O vibration of secondary OH at 1050 cm<sup>-1</sup>. The absorption at 2930 cm<sup>-1</sup> indicates the presence of C-H stretching, further confirmed by the absorption at 1500 cm<sup>-1</sup>, which corresponds to acetylene vibration. Another C-H stretching is observed at 2390 cm<sup>-1</sup>. The C=O group, characteristic of activated carbon, appears at 1570 cm<sup>-1</sup>. However, the FTIR spectrum does not show the NH<sub>2</sub> functional group, which is typical of humic acid. This could be due to the higher proportion of activated carbon compared to humic acid [2, 10-11]. The FTIR analysis of the activated carbon-humic acid composite is summarized in Table 4 and Figure 3.

**Table 4. Analysis of Activated Carbon-Humic Acid Composite**

No	Fuctional Group	Wavenumber (cm <sup>-1</sup> )
		Activated Carbon-Humic Acid Composite
1	OH	3300 cm <sup>-1</sup>
2	C-O	1050 cm <sup>-1</sup>
3	C-H	2930 cm <sup>-1</sup> , 2390 cm <sup>-1</sup>
4	C=O	1570 cm <sup>-1</sup>



**Figure 3. Infrared Spectrophotometry of Activated Carbon-Humic Acid Composite**

### 3.4 Results of Lead (Pb) Adsorption

The mixture of heavy metal Pb was first analyzed for its concentration using AAS as initial metal content data. Then, 20 mg of activated carbon adsorbent was added to 20 mL of 10 ppm standard Pb solution, and stirred at 200 rpm for 2 hours. The solution was filtered using Whatman filter paper. The AAS reading data showed that the initial adsorption concentration of 10.05 ppm decreased to 0.240 ppm, indicating an efficiency of 97.61%. The structure of activated carbon is characterized by the presence of micro, meso, and macro pores, which collectively offer a large surface area for metal adsorption. This interaction primarily occurs through Van der Waals forces or electrostatic attractions. Metal ions, depending on their size, can diffuse into these pores. Moreover,  $Pb^{2+}$  ions can interact with electron-donating groups such as  $-OH$ ,  $-COOH$ , or  $-C=O$ , forming stable chelate complexes. This mechanism accounts for the relatively high adsorption efficiency observed [16].

The adsorption process was also conducted with pure humic acid to evaluate its individual adsorption performance. Then, 20 mg of humic acid adsorbent was added to 20 mL of a 10 ppm Pb standard solution and stirred at 200 rpm for 2 hours. The solution was filtered using Whatman filter paper. The AAS data showed that the initial concentration of 10.05 ppm was reduced to 6.476 ppm, indicating an adsorption efficiency of 35.57%. Humic acid contains various active groups that facilitate interactions with metals, including carboxyl groups ( $-COOH$ ), phenolic groups ( $-OH$ ), alcoholic hydroxyl groups ( $-OH$ ), ketone groups ( $-C=O$ ), quinones, and nitrogen-containing groups ( $-NH_2$  in more complex structures). These active groups in humic acid act as electron donors, forming complexes with  $Pb^{2+}$  ions. In solution, the  $-COOH$  group can release  $H^+$  ions, promoting the exchange of  $Pb^{2+}$  ions. Similarly, the phenolic group often participates in forming stable complexes via coordinate covalent bonding. Other groups, such as quinones and ketones, can engage in redox reactions. However, the effectiveness of adsorption is relatively low, possibly due to the purity of the humic acid. Variations in humic acid quality—based on its source or extraction method—can affect the types and amounts of active groups present. The content of oxygen-containing groups (carboxyl, phenolic) plays a significant role in metal interactions. Additionally, the adsorption process occurs without an increase in temperature, which may limit the diffusion of metal ions to the active sites [17].

The concentration of the heavy metal Pb mixture was first analyzed using AAS to obtain the initial metal content data. A composite of activated carbon and humic acid at a ratio of 20 mg was prepared and added to a 10 ppm Pb solution. The mixture was stirred at 200 rpm for 2 hours and then filtered using Whatman filter paper. The AAS results showed that the initial concentration of 10.05 ppm was reduced to 2.694 ppm, indicating an adsorption efficiency of 73.20%, as shown in Table 5. When activated carbon is composited with humic acid, the adsorption efficiency typically decreases compared to pure activated carbon. This reduction occurs because humic acid contains numerous active groups, such as carboxyl ( $-COOH$ ), phenolic ( $-OH$ ), and ketone ( $-C=O$ ) groups, which can interact with metal ions. In the presence of activated carbon, humic acid competes for binding with the metal ions, rendering them unavailable for adsorption by the carbon and thus lowering its efficiency. Additionally,

humic acid can alter the physical structure of activated carbon, particularly if there is strong interaction between the humic acid and the carbon surface. This interaction may lead to the blockage of activated carbon's pores, further diminishing its ability to adsorb metal ions. Furthermore, humic acid is acidic and can lower the pH of the solution when combined with activated carbon. A reduced pH can protonate the carboxyl groups (-COOH) on activated carbon, diminishing their capacity to interact with metal ions [18].

**Table 5. AAS Data Results for Activated Carbon, Humic Acid, and Composite Adsorbents**

Pb Concentration Before Adsorption (ppm)	Contact Time (Hours)	Adsorbent Composition	Pb Concentration After Adsorption (ppm)		Reduction of Pb Metal Content (ppm)		Efficiency (%)	
			Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
			10,05	2	Activated Carbon	0,240	0,240	9,79
10,05	2	Humic Acid	6,476	6,476	3,575	3,575	35,57	35,57
10,05	2	Carbon-Humic Acid	2,694	2,694	7,358	7,358	73,20	73,20

**3.5 Results of Lead (Pb) Adsorption with the Influence of Contact Time and Composite Composition of Activated Carbon-Humic Acid**

The activated carbon-humic acid composite was prepared with composition ratios of 10, 20, and 40 mg, each subjected to contact time variations of 2 and 3 hours. The composites were added to 20 mL of a 10 ppm Pb solution and stirred at 200 rpm. The solution was then filtered using Whatman filter paper. The AAS results showed the highest adsorption efficiency of lead (Pb) with the activated carbon-humic acid composite at a contact time of 3 hours and a composition of 10 mg. The initial concentration of 10.05 ppm was reduced to 0.645 ppm, achieving an efficiency of 93.58%. In 2021, Ghahremani et al. impregnated carbon nanoparticles utilizing lightweight clay aggregates as adsorbents for Pb<sup>2+</sup> elimination. The highest capacity for adsorption was 22.8 mg/g, 99% of Pb<sup>2+</sup> was eliminated. from a focus of 10 g/L for the adsorbent and 100 mg/L [19]. According to Pfeifer et al. (2020), the maximal zeolite and bentonite adsorption capabilities were 137.0 and 119.7 mg/g, respectively. While bentonite worked best at pH values between 7 and 8.5, zeolite worked best at pH values between 3 and 7. The initial Pb<sup>2+</sup> values ranged from 362.6 to 2693.6 mg/L [20]. An inexpensive adsorbent is probably the ZnOnp-CSD matrix. Nonetheless, the CSD and the adsorption capacity were almost same. The results showed that Pb<sup>2+</sup> was removed from aqueous solution by more than 90% in a number of experiments on industrial wastes [21]. Conversely, the lowest adsorption efficiency was observed at a contact time of 3 hours with a composition of 40 mg, where the concentration was reduced from 10.05 ppm to 3.861 ppm, yielding an efficiency of 61.59% [13]. The detailed AAS data are presented in Table 6.

**Table 6. AAS Data Results for Composite with Variations in Contact Time and Composition**

Pb Concentration Before Adsorption (ppm)	Contact Time (Hours)	Adsorbent Composition	Pb Concentration After Adsorption (ppm)		Reduction of Pb Metal Content (ppm)		Efficiency (%)	
			Replicate 1	Replicate 2	Replicate 1	Replicate 2	Replicate 1	Replicate 2
			10,05	2	10	1,027	1,027	9,024
20	2,694	2,694			7,358	7,358	73,20	73,20
40	3,218	3,218			6,834	6,834	67,99	67,99
3	10	0,645		0,645	9,406	9,406	<b>93,58</b>	<b>93,58</b>
	20	1,169		1,169	8,883	8,8825	88,37	88,37
	40	3,861		3,861	6,191	6,1905	61,59	61,59

To assess the effectiveness of reducing metal content, a prerequisite test must first be conducted. This test determines the appropriate statistical method to be used, whether parametric or non-parametric statistical analysis. Based on the normality test results, a value of 0.017 was obtained, indicating that the data were not normally distributed. Additionally, the homogeneity test yielded a value of 0.00, showing that the data were not homogeneous. Since these two prerequisites were not met, the efficiency analysis could not be performed using parametric tests. Instead, a non-parametric test was employed. For the non-parametric analysis, the Kruskal-Wallis test was used. The Kruskal-Wallis test is a rank-based non-parametric test designed to determine whether there are statistically significant differences between two or more independent groups on a dependent variable measured on a numeric (interval/ratio) or ordinal scale. It serves as an alternative to one-way ANOVA. Based on the results of the Kruskal-Wallis test, a value of 0.052 was obtained, indicating no statistically significant differences. This suggests that the treatments applied in the study did not have a substantial impact [14].

### 3.6 Adsorption Kinetics

The adsorption isotherm was determined based on the  $R^2$  value and adsorption capacity data. The equations used were the Langmuir and Freundlich isotherm equations. From these equations, the x and y values for each isotherm adsorption equation were calculated and obtained. Based on the x and y values from each equation, the  $R^2$  values and equation results were obtained. These data are presented in the graphs shown in Figures 4 and 5 below

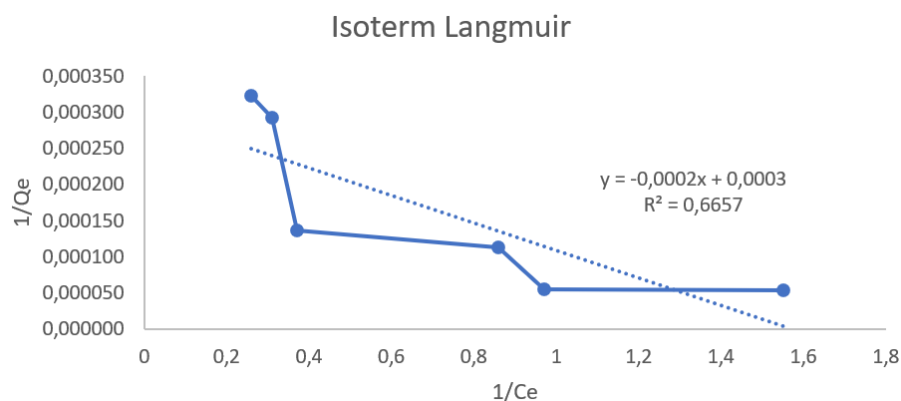


Figure 4. Langmuir Isotherm Results

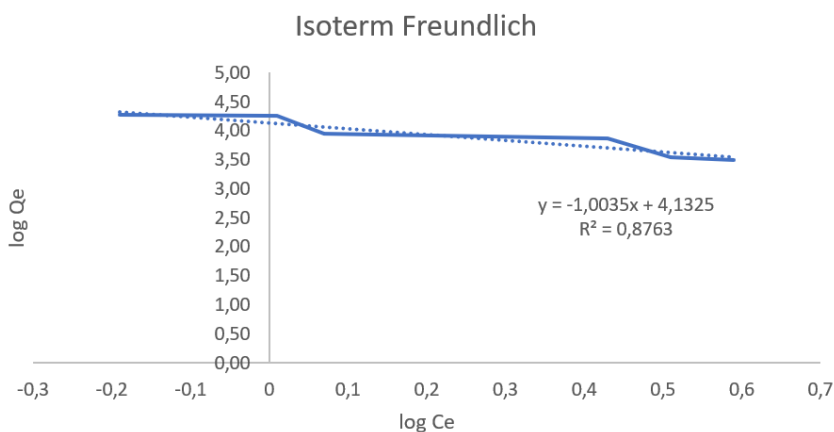


Figure 5. Freundlich Isotherm Results

From both equations, the Freundlich adsorption isotherm equation was determined, which is used as the equation for the effect of contact time and the composition of the Activated Carbon-Humic Acid composite on the reduction of Lead (Pb) concentration. The selection of the Freundlich adsorption isotherm equation is based on the analysis results, which show that the regression coefficient is close to 1 (0.8763) and indicates a higher adsorption capacity compared to the Langmuir isotherm equation. The Freundlich model assumes the presence of multiple surface layers (multilayer adsorption) and heterogeneous sites, suggesting variations in binding energy



at each adsorption site. This contrasts with the Langmuir isotherm, which assumes a monolayer adsorption on a homogeneous surface [15].

#### 4. CONCLUSION

The adsorption efficiencies of activated carbon, humic acid, and the activated carbon-humic acid composite for adsorbing the heavy metal lead (Pb) are 97.61%, 35.57%, and 93.58%, respectively. The test results on the influence of contact time variation and the composition of the activated carbon-humic acid composite in reducing lead (Pb) content indicate that these factors do not have a significant impact. The adsorption behavior of this composite follows the Freundlich isotherm, suggesting that physical adsorption occurs across multiple layers of the adsorbate

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