

Analysis and Reduction Efforts of Heavy Metal Pb in Water from Tofu Processing Using Coconut Shell Activated Charcoal with Atomic Absorption Spectrophotometry

Analisis dan Upaya Pengurangan Logam Berat Pb Pada Air Dalam Pengolahan Tahu Dengan Arang Aktif Tempurung Kelapa Menggunakan Spektrofotometri Serapan Atom

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Abstract

Background: Lead (Pb) is a toxic heavy metal that can contaminate water, including wastewater from tofu processing. Untreated sewage may adversely affect the environment and human health. One effective method for Pb reduction is adsorption using activated coconut shell charcoal, which possesses high porosity and surface area. **Objective:** This study aimed to analyze the adsorption efficiency of Pb in tofu wastewater using activated coconut shell charcoal and determine the optimal adsorption conditions based on adsorbent dosage, contact time, and pH. **Methods:** Water samples were collected from a tofu factory in Banjarsari, Solo. Coconut shell charcoal was activated with NaOH and used as an adsorbent. Optimization was performed by varying the adsorbent dosage (6, 8, 10 g), contact time (30, 60, 90 minutes), and pH (6, 7, 8). Lead (Pb) levels were quantified using Atomic Absorption Spectrophotometry (AAS). **Results:** The highest adsorption efficiency was 85.54% under optimal conditions: 8 g adsorbent dosage, 90 minutes contact time, and pH 8. Statistical analysis using ANOVA confirmed that all three factors significantly influenced Pb adsorption. **Conclusion:** Activated coconut shell charcoal effectively reduces Pb levels in tofu wastewater. Prolonged contact time, moderate adsorbent dosage, and alkaline pH enhance adsorption efficiency. This study proposes an environmentally friendly solution for treating wastewater from the tofu industry.

Keywords: Coconut shell adsorbent, lead (Pb), tofu wastewater, Atomic Absorption Spectrophotometry (AAS), adsorption optimization.

Abstrak

Latar Belakang: Timbal (Pb) merupakan logam berat beracun yang dapat mencemari air, termasuk air limbah industri pengolahan tahu. Air limbah yang tidak diolah dapat berdampak buruk bagi lingkungan dan kesehatan manusia. Salah satu metode yang efektif untuk mengurangi kadar Pb adalah adsorpsi menggunakan arang aktif tempurung kelapa, yang memiliki porositas dan luas permukaan tinggi. **Tujuan:** Penelitian ini bertujuan untuk menganalisis efisiensi adsorpsi logam Pb dalam air limbah pengolahan tahu menggunakan arang aktif tempurung kelapa serta menentukan kondisi optimal adsorpsi berdasarkan dosis adsorben, waktu kontak, dan pH. **Metode:** Sampel air diambil dari pabrik tahu di Banjarsari, Solo. Arang tempurung kelapa diaktivasi dengan NaOH dan digunakan sebagai adsorben. Optimasi dilakukan dengan variasi dosis adsorben (6, 8, 10 gram), waktu kontak (30, 60, 90 menit), dan pH (6, 7, 8). Kadar Pb dianalisis menggunakan Spektrofotometri Serapan Atom (SSA). **Hasil:** Hasil penelitian menunjukkan bahwa efisiensi adsorpsi tertinggi mencapai 85,54% pada kondisi optimal, yaitu dosis adsorben 8 gram, waktu kontak 90 menit, dan pH 8. Analisis statistik dengan ANOVA menunjukkan bahwa ketiga faktor tersebut berpengaruh signifikan terhadap adsorpsi Pb. **Kesimpulan:** Arang aktif tempurung kelapa terbukti efektif dalam menurunkan kadar Pb dalam air limbah tahu. Kombinasi waktu kontak yang lama, dosis adsorben moderat,

dan pH basa meningkatkan efisiensi adsorpsi. Penelitian ini memberikan solusi potensial untuk pengolahan limbah industri tahu secara ramah lingkungan.

Kata Kunci: Adsorben tempurung kelapa, logam Pb, air limbah tahu, Spektrofotometri Serapan Atom (SSA), optimasi adsorpsi.



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Introduction

The tofu industry is one of Indonesia's most prevalent small-scale food industries. Its production process generates large amounts of solid and liquid waste, with the latter containing high concentrations of organic matter such as proteins, fats, and carbohydrates. These compounds contribute to elevated levels of Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand (COD), significantly exceeding permissible limits. [1] Most tofu producers lack adequate wastewater treatment facilities, which results in the direct discharge of liquid waste into the environment [2–4]. Untreated disposal contributes to organic pollution in water bodies and may also carry inorganic contaminants such as heavy metals. Haryati et al. (2012) reported that industrial wastewater contained various heavy metals, with lead (Pb) levels reaching up to 2.3458 ppm [5]. Similarly, Novita et al. (2012) found Pb concentrations of 1.040 mg/L in wastewater discharged into a class II river. [6]. These findings reflect the weakness of waste management systems, particularly in small-scale and household-based industries, including tofu production. Considering that industrial effluents can contain hazardous heavy metals, it is essential to evaluate the potential presence of contaminants, especially lead, in wastewater from tofu industries.

The lead (Pb) content in tofu industry wastewater is likely not solely derived from the production process but may also originate from the healthy water used. Well water, especially in rural areas, is susceptible to heavy metal contamination due to geological factors, industrial activities, and environmental pollution. Such contamination can pose serious health risks, including neurological disorders and kidney damage. Therefore, the presence of Pb in tofu industry wastewater should be examined for potential initial contamination from the water source [7].

Heavy metals are compounds that can pollute water bodies and are toxic. Industrial and agricultural activities can cause heavy metal pollution in aquatic environments. Heavy metals that exceed the quality standard limits set by the Indonesian National Standard (SNI) 7387:2009, which states that the maximum contamination limit for lead in food is 0.5 mg/kg, pose a potential health hazard. Lead content exceeding this limit can endanger public health due to the ability of heavy metals to accumulate, a process known as bioaccumulation and biomagnification [8,9].

To address this issue, various methods for heavy metal adsorption have been developed, one of which involves the use of coconut shell charcoal. This charcoal is a product of coconut shell combustion, rich in carbon content, and has high levels of cellulose and lignin—27.7% and 29.4% respectively, along with 26.6% hemicellulose. These components form numerous pores and cavities after carbonization, enhancing the charcoal's ability to adsorb heavy metals. Studies have shown that activated coconut shell charcoal can reduce lead (Pb) content in pesticide solutions by an average of 27.26% [10], making it an environmentally friendly alternative for treating heavy metal-contaminated waste.

This research is essential to finding an environmentally friendly solution to reduce lead (Pb) contamination in wastewater from tofu processing and to contributing to the application of activated carbon with optimized variations that are more efficient and effective in wastewater treatment.

Experimental Section

Materials and Instruments

The materials used in this study include wastewater from the tofu production process, coconut shell charcoal, sodium hydroxide p.a (NaOH 30%) (Merck, Germany), hydrochloric acid p.a (HCl 37%) (Merck, Germany), nitric acid (HNO₃) (Merck, Germany), distilled water, and standard Pb solution (Merck, Germany).

The instruments used in this study include an atomic absorption Spectrophotometer (AAS), pH meter, oven, analytical balance, porcelain dish, mortar and pestle, furnace, sieve (30–40 mesh), porcelain crucible, desiccator, watch glass, dropper pipette, volumetric pipette, Erlenmeyer flask, beaker, volumetric flask, graduated cylinder, glass funnel, filter paper, and baking tray.

Procedure

The methodology in this study includes method validation, which involves testing for linearity, accuracy, precision, limits of detection (LOD), and quantification (LOQ). It also includes the preparation of coconut shell adsorbent and the reduction of lead (Pb) content in water used in the tofu production process.

Validasi metode

a. Linearity

The linearity test is conducted using a standard solution of five concentrations: 2 ppm, 4 ppm, 6 ppm, 8 ppm, and 10 ppm. Prepare standard solutions of 2 ppm, 4 ppm, 6 ppm, 8 ppm, and 10 ppm. Measure the absorbance of each standard solution using AAS. Plot the absorbance against the standard concentration to create a calibration curve. Calculate the regression coefficient (R²) to assess linearity. An R² value close to 1 indicates a good linear relationship.

b. Accuracy.

Take 2 ml of the sample and add 2 ml of Pb (NO₃)₂ solution to prepare a spiked solution. Measure the absorbance using the AAS instrument. Compare the measurement results with the expected value. Calculate the recovery percentage. Good accuracy is indicated by a recovery percentage between 95% and 102%.

c. Precision

Measurements are performed on a standard solution with a fixed concentration (e.g., 6 ppm) six times. A 6 ppm standard solution is prepared. The absorbance is measured six times using atomic absorption spectrophotometry. The average, standard deviation (SD), and percent relative standard deviation (%RSD) are calculated, with results not exceeding 2%.

d. Detection Limit and Quantitation Limit

The absorbance of a blank solution without lead is measured to determine the baseline signal. Standard solutions of 2 ppm, 4 ppm, 6 ppm, 8 ppm, and 10 ppm are prepared. The absorbance of each standard solution is measured using atomic absorption spectrophotometry. The LOD (Limit of Detection) and LOQ (Limit of Quantitation) values are calculated.

Preparation of Coconut Shell Charcoal Production

This study carried out three main processes in producing coconut shell charcoal: dehydration, carbonization, and activation. The selected coconut shells were dark brown, almost black, and weighed 3 kg. The shells underwent fiber removal and washing to eliminate impurities [11,12]. Afterward, the cleaned coconut shells were sun-dried before being placed in the furnace. This drying process was intended to prevent smoke formation when the coconut shells were inserted into the furnace at 400°C for 10 minutes. [12,13]. The resulting charcoal was then removed and cooled using a desiccator [14].

Activated Carbon Production

The coconut shell that has been turned into charcoal is then activated. The carbon is chemically activated by soaking 150 grams of carbon in 500 ml of a 30% NaOH solution for 24 hours [15–17]. After washing, the

carbon is drained, filtered, and rinsed repeatedly with distilled water until it reaches the required pH. The activated carbon is then placed in a desiccator for cooling [16,17].

Subtraction of Pb in tofu tofu-making process water

Reduction of Pb Metal in Tofu Production Wastewater Using Coconut Shell Adsorbent. Experiments were conducted to evaluate Pb removal efficiency using a formula developed with Design Expert and considering three parameters: contact time, adsorbent dosage, and pH. The pH of the water sample was measured before adding the adsorbent. The adsorbent was then mixed with the water sample, stirred, and left for a predetermined duration. Afterward, the mixture was filtered to separate the adsorbent from the water. Each water sample was analyzed using an Atomic Absorption Spectrometer (AAS) to determine the amount of adsorbed Pb ions. Additionally, Pb adsorption efficiency was calculated using the % adsorption formula.

Optimasi Run Formula Design Expert

RUN	FACTOR 1	FACTOR 2	FACTOR 3
	A-Adsorbent Concentration (gram)	B-Contact Time (minute)	C: pH
1	6	30	7
2	6	60	6
3	8	60	8
4	10	60	6
5	6	90	8
6	8	90	8
7	8	90	7
8	10	30	8

Results and Discussion

Results of Analytical Method Validation

Linearity is measured by constructing a calibration curve, plotting the measured absorbance values (y-axis) against the concentration of the standard solution (x-axis). The correlation coefficient (r) is used in the linear regression equation ($y = bx + a$). The calibration curve for Pb metal has a linear equation of $y = 0.0395x - 0.1957$ with a correlation coefficient (r) of 0.9995. The value in linear regression indicates the sensitivity of the analysis to the instrument used [18]. This can be seen in Table 1.

Table 1. Linearity Test Results

Pb Concentration (mg/L)	Absorbance
2	0,2714
4	0,3552
6	0,4359
8	0,5125
10	0,5875

Precision testing shows that the standard deviation (SD) of the calculated results from data obtained through six replications is 0.00049295 at an analyte concentration of 6 ppm, with a %RSD (Percent Relative Standard Deviation) value of 0.112919551. The precision test results indicate that the precision value meets the %RSD requirement, which should not exceed 2%. This can be seen in Table 2.

Accuracy is a parameter that indicates the closeness between the analytical result (measured value) and the actual analyte concentration (accepted true value), which is usually expressed as a recovery percentage. A method is still considered accurate if the recovery percentage falls within the required range. The obtained recovery value reflects how close the measurement result is to the actual value. A good recovery value generally falls within the range of 80%-120% with three repetitions. This can be seen in Table 3.

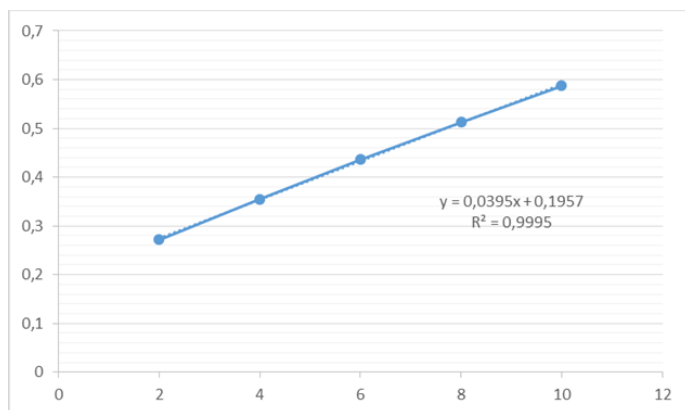


Figure 1. Linearity Test Results Graph

Table 2. Precision test

Replicate	Pb Concentration (mg/L)
1	0,4362
2	0,4363
3	0,4375
4	0,4365
5	0,4366
6	0,4362
Mean	0,43655
Standard Deviation	0,00049295
%RSD	0,112919551

Table 3. Accuracy test

Accuracy	Concentration (ppm)			Recovery	Mean %	Mean %
	Sample + standard	Sample	standard			
80%	0,7622	0,3221	0,4517	96,40	96,34	98,85
	0,7620	0,3225	0,4519	96,31		
	0,7615	0,3220	0,4514	96,30		
100%	0,8405	0,3758	0,4721	98,05	98,32	
	0,8417	0,3751	0,4725	98,47		
	0,8421	0,3754	0,4727	98,40		
120%	0,8260	0,3591	0,468	99,69	101,90	
	0,8263	0,3470	0,4688	103,06		
	0,8265	0,3455	0,4687	102,96		

The recovery results in the table above satisfy the required recovery criteria, as the obtained values fall within the acceptable 95%–102 % range. The average percentage recovery in this study is 98.85%.

LOD and LOQ. The limit of detection (LOD) is the smallest amount of analyte in a sample that can still be detected by the analytical method. The limit of quantification (LOQ) and the limit of detection are parameters used to determine the concentration threshold at which an instrument can quantify and detect an analyte. Afterward, absorbance measurements are taken. Before calculating LOD and LOQ, a linear relationship test between concentration and absorbance needs to be conducted. Atomic Absorption Spectrophotometry measurements produce concentration and absorbance values, which are then used to determine the LOD and LOQ parameters. The results can be seen in Table 4.

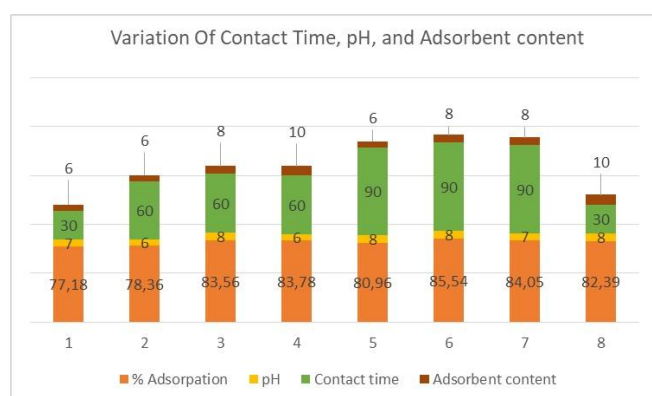
Table 4. LOD & LOQ test

Concentration (mg/L)	Absorbance	y ¹	y-y ¹	(y-y ¹)*2
2	0,2714	0,2747	-0,0033	0,00001089
4	0,3552	0,3537	0,0015	0,00000225
6	0,4359	0,4327	0,0032	0,00001024
8	0,5125	0,5117	0,0008	0,00000064
10	0,5875	0,5907	-0,0032	0,00001024
			Total	0,00003426
Total/n-2	3			
A	0,1957			
B	0,0395			
R	0,9995			
Standard Deviation	0,003379			
LOD	0,256659			
LOQ	0,855531			

In the table above, the calibration curve meets the requirements for further analysis. The data is then processed to determine the limit of detection (LOD) and the limit of quantification (LOQ). In this study, the LOD value is 0.256659 mg/L, while the LOQ value is 0.855531 mg/L, representing the lowest concentration limit that can be used for quantification while still ensuring analytical accuracy.

Determination of Pb Metal Adsorption with Variations in Contact Time, pH, and Adsorbent Concentration in Water

The contact time indicates that adsorption has not yet occurred optimally within 30 minutes at the initial stage. At 60 minutes, the adsorption percentage of Pb has been detected, showing that the adsorbate moves due to rotational turbulence in search of empty spaces. The fulfillment of pore volume is influenced by contact time. The longer the contact time, the more the adsorbate is pushed to the end of the pores, creating space at the front for new adsorbate to be absorbed. As the contact time increases, the amount of adsorbate adsorbed on the bioadsorbent surface also increases, resulting in a lower Pb(II) concentration in the wastewater sample. This finding aligns with previous studies, which indicate that the longer the contact time between the adsorbent and lead ions, the greater the amount of lead adsorbed [19]. As shown in Figure 2, at a contact time of 90 minutes, using formula 6, Pb metal adsorption with the adsorbent reached 85.54%.

**Figure 2.** Diagram of contact time variation, pH, and adsorbent dosage.

In the research on reducing Pb metal ions using jackfruit peel as a NaOH-activated adsorbent in batik wastewater, the optimum pH for Pb metal absorption was found at pH 8, with an efficiency value of 98.20%. This finding highlights the crucial role of pH in enhancing the adsorption capacity of various NaOH-activated adsorbents to effectively remove lead metal from batik wastewater. [20]. As shown in Figure 2 of this study, the optimum pH for adsorption is pH 8, with an efficiency value of 85.54%.

The amount of adsorbent used in this study was 6 grams, 8 grams, and 10 grams of activated coconut shell charcoal. According to Alfiany & Bahri (2013), the more activated charcoal used, the higher its adsorption capacity for metal ions, which is proportional to the increased number of particles and surface area of the activated charcoal [21]. The increase in adsorption capacity is due to the greater number of active sites available, allowing the adsorbent to capture more adsorbate, leading to an increase in adsorption capacity until the optimum weight is reached. After reaching the optimum condition, no significant adsorption is observed. This is because no more adsorbate is being captured, meaning that adding more adsorbent beyond a certain point will not impact the adsorption capacity. The results, as shown in Figure 2, indicate that an adsorbent amount of 8 grams provides the most optimal adsorption of Pb meta. [21].

ANOVA Test of Adsorption Percentage, Adsorbent Concentration, Contact Time, and pH

In this study, analysis of variance (ANOVA) was conducted to evaluate the effect of several factors on the Pb metal adsorption process. The influence of formula components was assessed using ANOVA drug loading tests with Design Expert, where the P-value must be ≤ 0.05 , indicating that the model is acceptable. The ANOVA test results for % adsorption showed a model P-value of 0.0056 and an F-value of 22.84. These results suggest a significant effect of the independent variables or formula components in Design Expert on Pb metal adsorption. The results are presented in Table 5.

Table 5. ANOVA test of %adsorption

Source	Sum of Squares	df	Mean Square	F-value	P-value	
Model	56.74	3	18.91	22.84	0.0056	Significant
A-Adsorbent Concentration	35.52	1	35.52	42.91	0.0028	
B-Contact Time	22.87	1	22.87	27.62	0.0063	
C-pH	2.82	1	2.82	3.41	0.2384	
Residual	3.31	4	0.8279			
Cor Total	60.05	7				

The ANOVA test results for the adsorbent level showed a model P-value of 0.0289 and an F-value of 9.17. These results indicate a significant effect of the independent variables or formula components in the Design Expert on the adsorbent level, as shown in Table 6.

Table 6. ANOVA test of adsorbent concentration

Source	Sum of Squares	df	Mean Square	F-value	P-value	
Model	20.95	3	6.98	9.17	0.0289	Significant
A-Adsorbent Concentration	19.14	1	19.14	25.14	0.0074	
B-Contact Time	0.0384	1	0.0384	0.0505	0.8333	
C-pH	0.4384	1	0.4326	0.5681	0.4929	
Residual	3.05	4	0.7614			
Cor Total	24.00	7				

Table 7. ANOVA test of contact time

Source	Sum of Squares	df	Mean Square	F-value	P-value	
Model	4073.19	3	1357.73	17.28	0.0094	Significant
A-Adsorbent Concentration	731.40	1	731.40	9.31	0.0380	
B-Contact Time	3559.05	1	3559.05	45.29	0.0025	
C-pH	668.84	1	668.84	8.51	0.0434	
Residual	314.31	4	78.58			
Cor Total	4387.50	7				

ANOVA testing for drug loading was conducted using Design Expert, where a P-value of ≤ 0.05 indicates that the model is acceptable. The ANOVA test results for contact time showed a model P-value of 0.0094 and an F-value of 22.84. These results suggest a significant effect of Design Expert's independent variables or formula components on contact time, as shown in Table 7.

The ANOVA test results for the adsorbent levels showed a model P-value of 0.0228. Since the P-value is ≤ 0.05 and the F-value is 10.53, these results indicate a significant influence of the independent variables or formula components in the Design Expert on pH. It can be observed in Table 8.

Table 8. ANOVA Results for pH Effects on Pb Adsorption

Source	Sum of Squares	df	Mean Square	F-value	P-value	
Model	5.33	3	1.78	10.53	0.0228	Significant
A-Adsorbent Concentration	0.7735	1	0.7735	4.59	0.0988	
B-Contact Time	4.35	1	4.35	25.84	0.0071	
C-pH	1.42	1	1.42	8.42	0.0441	
Residual	0.6741	4	0.1685			
Cor Total	6.00	7				

Determination of Overlay Plot for Optimal Conditions: Adsorption Percentage, Adsorbent Concentration, Contact Time, and pH

The optimum region is determined by analyzing the generated overlay plot. The optimum region is the area that meets the response criteria and is indicated by the yellow color. This region represents the values of % adsorption, adsorbent concentration, and contact time that satisfy the criteria. In the overlay plot, flags can be placed at any point to display the % adsorption, adsorbent concentration, contact time, and pH, along with the corresponding response at that point. This can be seen in Figure 3.

Based on the overlay plot analysis (Figure 3), the optimum region in the adsorption process occurs at a combination of high adsorbent concentration (around 10 g/L), long contact time (approximately 90 minutes), and a solution pH that is relatively neutral to slightly acidic (6–7). This region is characterized by a bright yellow color in the upper right part of the graph, representing the area with the highest response value (% Adsorption), reaching up to 81.09%. These results indicate that increased adsorbent dosage significantly improves adsorption efficiency due to the greater availability of active sites for interaction with dissolved. [22]. In addition, a longer contact time allows diffusion and chemical bonding processes to proceed more effectively, thereby increasing contaminant removal efficiency [23]. Although the effect of pH is not as strong as that of the other two factors, a pH range of 6–7 has proven to be the most favorable condition for maximizing adsorption capacity without causing precipitation or altering the adsorbent's surface structure [24]. A clear interaction between adsorbent dosage and contact time is also observed, where their combined use leads to a greater improvement in response compared to optimizing only one factor at a time [25]. The experimental design points within the optimum region show consistent results, proving that the predictive model can accurately represent the relationship between independent variables and the response [26]. Therefore, these findings provide relevant operational guidance for applying adsorption technology in wastewater treatment or water purification, while also opening opportunities for further research on process optimization, adsorption mechanism characterization, and adsorbent regeneration testing at pilot or industrial scales.

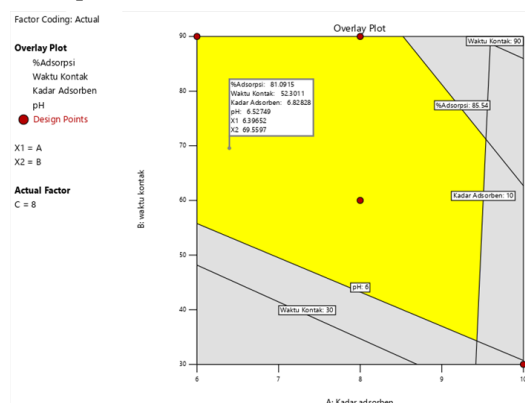


Figure 3. Overlay plot in the optimum region.

Conclusion

This study concludes that activated carbon adsorbent from coconut shell at pH 8, with a contact time of 90 minutes and an adsorbent dosage of 8 grams, could absorb 85.54% of Pb metal. At pH 8, the adsorption capacity was significantly high in this study, and a contact time of 90 minutes proved efficient for Pb metal adsorption. Longer contact times result in greater Pb metal adsorption. An adsorbent dosage of 8 grams demonstrated better metal adsorption than other dosages. The combination of contact time, adsorbent dosage, and pH provides an optimal range for reducing heavy metals. Coconut shell shows excellent potential as a raw material for activated carbon production and can be further explored for other applications.

Conflict of Interest

All authors certify that they have no affiliations with or involvement in any organization or entity with any financial or non-financial interest in the subject matter discussed in this manuscript.

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Supplementary Materials

No supplementary materials are available for this paper.

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