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Statistical Optimization of Experimental Conditions for Enhanced Removal of Heavy Metals from Wastewater

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Abstract

Increasing concentrations of heavy metals in industrial liquid waste demand the development of more efficient treatment strategies to minimize the impact on the environment and public health. This study aims to statistically optimize experimental conditions to improve the efficiency of heavy metal removal (especially Pb^{2+}, Cd^{2+}, and Cr^{6+}) from synthetic liquid waste through the batch adsorption process. A well-planned experimental design was implemented using the Response Surface Methodology (RSM) approach with Central Composite Design (CCD) to evaluate the individual influences as well as interactions of four key variables: the initial concentration of the metal, the pH of the solution, the adsorbent dose, and the contact time. The results of the experiment were modeled in the form of second-order polynomial regression, and model validation was carried out strictly through variety analysis (ANOVA), determination coefficients (adjusted R^2 and R^2), and lack-of-fit tests. The optimization process successfully identified a combination of operating parameters that significantly improved the elimination efficiency, reaching a level above 95% at the optimized conditions that had been validated. The residue analysis showed the fulfillment of the assumptions of normality, homogeneity of variance, and error independence, thus confirming the predictive reliability of the model. These findings confirm the effectiveness of RSM-based optimization approaches in wastewater treatment research, as well as the importance of statistical-based experimental planning in maximizing process efficiency. This approach provides a robust framework for advanced applications in industrial waste management and sustainable environmental engineering.

Keywords: Heavy metal removal, Response Surface Methodology, Wastewater treatment, Statistical optimization, Adsorption.
MSC2020: 62K20, 62J10, 62P30

Abstrak

Meningkatnya konsentrasi logam berat dalam limbah cair industri menuntut pengembangan strategi pengolahan yang lebih efisien untuk meminimalkan dampaknya terhadap lingkungan dan kesehatan masyarakat. Penelitian ini bertujuan untuk mengoptimalkan secara statistik kondisi eksperimental guna meningkatkan efisiensi penghilangan logam berat (khususnya Pb^{2+}, Cd^{2+}, dan Cr^{6+}) dari limbah cair sintetis melalui proses adsorpsi batch. Rancangan percobaan yang terstruktur dengan baik diterapkan menggunakan pendekatan Response Surface Methodology (RSM) dengan Central Composite Design (CCD) untuk mengevaluasi pengaruh individual serta interaksi dari empat variabel utama: konsentrasi awal logam, pH larutan, dosis adsorben, dan waktu kontak. Hasil percobaan kemudian dimodelkan dalam bentuk regresi polinomial orde dua, dan validasi model dilakukan secara ketat melalui analisis ragam (ANOVA), koefisien determinasi (R^2 dan R^2 terkoreksi), serta uji lack-of-fit.

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Proses optimasi berhasil mengidentifikasi kombinasi parameter operasi yang secara signifikan meningkatkan efisiensi penghilangan, dengan tingkat pencapaian di atas 95% pada kondisi optimal yang telah divalidasi. Analisis residu menunjukkan terpenuhinya asumsi normalitas, homogenitas varians, dan independensi galat, sehingga mengonfirmasi reliabilitas prediktif model. Temuan ini menegaskan efektivitas pendekatan optimasi berbasis RSM dalam penelitian pengolahan limbah cair, serta pentingnya perencanaan eksperimen berbasis statistik dalam memaksimalkan efisiensi proses. Pendekatan ini memberikan kerangka kerja yang kokoh untuk aplikasi lanjutan dalam manajemen limbah industri dan rekayasa lingkungan berkelanjutan.

Kata kunci: Penghilangan logam berat, Metodologi Permukaan Respon, Pengolahan limbah cair, Optimasi statistik, Adsorpsi
MSC2020: 62K20, 62J10, 62P30

Introduction

Water pollution caused by heavy metals has become an increasingly critical and challenging environmental issue in various countries, both developed and developed. Heavy metals such as lead (Pb^{2+}), cadmium (Cd^{2+}), and hexavalent chromium (Cr^{6+}) are classified as priority pollutants because they are toxic, biodegradable, and have the ability to bioaccumulate in the food chain [1]; [2]. Long-term exposure to heavy metals is known to cause a variety of serious health problems, including cancer, kidney disorders, nerve damage, as well as developmental disorders in children [3]; [4]. The World Water Development Report by UNESCO (2020) notes that about 80% of global liquid waste—particularly from low- and middle-income countries—is dumped into the environment without adequate treatment, exacerbating the risk of contamination of water resources [5].

Industrial sectors such as mining, electroplating, tanning, textiles, and battery production are major contributors to heavy metal pollution [6]. Because they cannot undergo biological degradation, heavy metals require an effective processing process to prevent long-term contamination. Several conventional methods such as chemical precipitation, ion exchange, membrane filtration, and electrochemical reduction have been widely applied. However, these methods generally have limitations in the form of high operating costs, secondary sludge production, and low effectiveness at small concentrations of metals [7]; [8].

Among the various treatment approaches, adsorption has gained widespread attention due to its high efficiency, relatively low cost, ease of operation, as well as its ability to remove various types of heavy metals [9]; [10]. The effectiveness of the adsorption process is greatly influenced by a number of operating parameters such as pH, contact time, initial concentration of metals, adsorbent dosage, and temperature. The complexity of the interaction between these parameters causes traditional methods such as the One Factor at a Time (OFAT) approach to be inadequate, as they are unable to capture the effects of interactions and require a large number of experiments to produce accurate conclusions [11]; [12].

To overcome these limitations, statistical optimization approaches based on Design of Experiments (DoE) and Response Surface Methodology (RSM) are increasingly used in environmental and chemical engineering research. DoE allows for efficient experimental design by considering the simultaneous influence of several independent variables on the response variables [13]. RSM, especially with the Central Composite Design (CCD) approach, allows the development of empirical models in the form of quadratic regression, analysis of interactions between variables, and visualization of optimal conditions through contours and response surfaces [14]; [15]. This approach significantly reduces the number of experiments required while increasing the strength of statistical analysis and the validity of the results.

Although RSM has been widely applied in heavy metal removal studies, there are still shortcomings in terms of in-depth statistical validation of the resulting model. Many studies do not perform comprehensive residual diagnostic tests or neglect testing for basic statistical assumptions such as normality, homogeneity of variance, and error independence. This inaccuracy can reduce the predictive validity of the model and limit its application in highly variable real systems.

Based on this background, this study aims to develop a statistically robust and experimentally validated optimization model for the process of removing heavy metals (Pb^{2+}, Cd^{2+}, and Cr^{6+}) from synthetic wastewater through a batch adsorption system. This study uses the Central Composite Design in the framework of RSM to evaluate the main influences and interactions of four important variables: solution pH, initial concentration of metals, adsorbent dose, and contact time. The second-order regression model was constructed and validated through variance analysis (ANOVA), determination coefficients (adjusted R^2 and R^2), lack of fit test, and residual diagnostic analysis. This validation is intended to ensure that the model not only accurately describes the data, but also has high predictive power for real applications [16]; [17].

Through a data-driven experimental approach and robust statistical modeling, this research contributes to the development of a reliable and replicable waste treatment optimization framework. In addition, the results of this study support the urgent need for sustainable, affordable, and scientifically tested wastewater management solutions, in line with Sustainable Development Goals (SDGs) Number 6: "Ensure the availability and sustainable management of clean water and sanitation for all."

Methods

Research Design

This study employed an experimental quantitative design using the Response Surface Methodology (RSM) to optimize the efficiency of heavy metal removal from synthetic wastewater. RSM is a statistical and mathematical approach used to model and analyze processes influenced by several variables, aiming to determine optimal conditions through polynomial regression and interaction analysis [18].

Within RSM, the Central Composite Design (CCD) was selected because it efficiently estimates both linear and quadratic effects and detects curvature in the response surface. CCD consists of three types of points: factorial points (high and low levels of each variable), axial points ($\pm\alpha$ from the center to capture quadratic effects), and center points (replicates used to estimate experimental error) [19]. This balanced design allows accurate modeling with a minimal number of experimental runs.

This research follows the general framework of the Design of Experiments (DoE), a systematic strategy for planning and conducting experiments to understand factor influences and optimize responses. Common DoE types include factorial, fractional factorial, randomized block, and response surface designs. In this study, RSM–CCD was chosen for its capability to evaluate factor interactions and predict the optimal combination of parameters for maximum metal removal efficiency [20]; [21].

Materials and Chemicals

Synthetic liquid waste is made by dissolving compounds $Pb(NO_3)_2$, $CdCl_2 \cdot H_2O$, and $K_2Cr_2O_7$ into distilled water to represent Pb^{2+}, Cd^{2+}, and Cr^{6+} ions in an initial concentration of 100 mg/L. The adsorbent used is commercial granular activated carbon with a particle size of 250–500 µm, which has previously been washed, dried, and stored in a closed condition. All chemicals have an analytical purity level and are obtained from standardized laboratory distributors [22].

Research Variables and Their Limitations

In this study, there are four main independent variables (experimental factors) that were identified to affect the process of removing heavy metals from liquid waste, namely:

1) pH larutan (X_1): 3 – 9
2) Initial concentration of metals (X_2): 25 – 100 mg/L
3) Adsorbent dose (X_3): 0.5 – 2.0 g/100 mL
4) Contact time (X_4): 10 – 120 minutes

These four factors were selected based on the literature and their significance in influencing the performance of the adsorption system. Each variable was tested in a planned experimental design to evaluate both the single effects and the interactions between variables on the system's output. The dependent variable in this study is the % removal efficiency, which is calculated based on the comparison between the concentration of metals before and after the adsorption process. The formula used is:

$$\text{Removal Efficiency (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

where:

- C_0 is the initial concentration (mg/L) and
- C_t is the final concentration after adsorption.

This formula provides a quantitative measure of the effectiveness of the adsorption process, which is used as a basis for the development of statistical models as well as the evaluation of the overall performance of the system. The high efficiency value reflects the success of the operational parameters in maximizing the mass transfer of metal ions from the liquid phase to the adsorbent surface [23].

Experimental Procedure

Experiments were carried out in a batch system at room temperature (±27°C). A total of 100 mL of heavy metal solution is placed in a 250 mL erlenmeyer, then adsorbents are added according to the predetermined dose. The pH is adjusted using a solution of HCl 0.1 M or NaOH 0.1 M. The mixture is stirred using an orbital shaker at a fixed speed (150 rpm), then filtered using Whatman No. 42 paper. The concentration of residual metals was measured using an Atomic Absorption Spectrophotometer (AAS). The total number of experiments was determined based on the CCD design for four variables, which consisted of:

- 16 factorial points
- 8 axial points
- 6 titik pusat (replicates)

  Total: 30 trials

Statistical Analysis and Model Validation

Experimental data were analyzed using Design-Expert v13 to evaluate and optimize process parameters affecting heavy metal removal efficiency. A second-order quadratic model was developed to predict the response as a function of four independent variables. Model adequacy was assessed using Analysis of Variance (ANOVA), which evaluates whether variations in the response are significantly explained by the regression model. ANOVA partitions total variation into regression and residual components, with model significance tested by the F-ratio:

$$F = \frac{MS\ regression}{MS\ error} \tag{2}$$

where MS=SS/df. A high F-value and $p < 0.05$ indicate that the model is statistically significant [24]. The coefficients of determination (R^2 and adjusted R^2) were used to measure model fit, while the lack-of-fit test ensured compatibility between predicted and experimental data. Residual diagnostics verified normality, homoscedasticity, and independence of errors. Optimization was performed using contour and 3D response surface plots, along with the desirability function method to identify optimal parameter combinations. Finally, confirmation experiments were conducted under predicted optimal conditions to validate model accuracy.

Results and Discussion

Removal Efficiency

From 30 experiments designed using Central Composite Design (CCD), the heavy metal removal efficiency range (Pb^{2+}, Cd^{2+}, and Cr^{6+}) was obtained from 68.2% to 97.6%, depending on the combination of pH value, adsorbent dose, contact time, and initial concentration of metal. The highest efficiency values were achieved at pH 6–6.5, contact time >90 minutes, and adsorbent dose >1.5 g/100 mL. Pb^{2+} shows the highest efficiency under these conditions, while Cr^{6+} tends to be more sensitive to the pH of the solution. In contrast, Cd^{2+} has more consistent efficiency across the pH range, but is strongly influenced by the initial concentration and dose of adsorbents.

Development of Second-Order Quadratic Models

The mathematical model used to describe the relationship between the elimination efficiency (Y) and the input variables (pH X_1, initial concentration X_2, adsorbent dose X_3, and contact time X_4) follows the general form of quadratic regression:

$$y = \beta_0 + \sum_{i=1}^{4} \beta\, x_j + \sum_{i=1}^{h} \beta_{ii} x_i^2 + \sum_{i=1}^{3} \sum_{j=i+1}^{4} \beta_{ii} x_1\, x_j + \varepsilon \tag{3}$$

The final model for the metal Pb^{2+} based on the regression results is:

$$\begin{aligned} Y_{Pb} \quad &= 91{,}74 + 4{,}32\, X_1 - 3{,}15\, X_2 + 2{,}86\, X_3 + 3{,}02\, X_4 - 1{,}25\, X_1^2 \\ &- 1{,}12\, X_2^2 - 0{,}98\, X_3^2 - 1{,}45\, X_4^2 \\ &+ 0{,}77\, X_1 X_2 + 0{,}64\, X_3 X_4 \end{aligned}$$

Each coefficient in a quadratic regression model has important statistical significance and physical interpretation in the context of experimental optimization. The linear coefficient (βi) represents the direct effect of each independent variable on the response, indicating how much the response changes if the variable is increased or decreased independently.

Meanwhile, the quadratic coefficient (βii) describes the existence of a non-linear relationship and the potential existence of a saturation point or local optimum value of the variable, which is important to know at what point an increase in the variable can actually lead to a decrease in efficiency. The interaction coefficient (βij) indicates the synergy or antagonism between two variables, which means that the influence of a variable on the response can change depending on the value of the other variable. This interpretation is important in understanding the dynamics of multivariate systems and is the basis for the formulation of the most effective operational conditions.

Variety-Analysis (ANOVA) and Model Statistical Validation

To evaluate the significance of the model, an ANOVA test was performed. The ANOVA summary for the Pb^{2+} model shows:

Table 1. Variance Analysis (ANOVA) for Quadratic Regression Models

Source of Variance	df	Sum of Squares	Mean Square	F-Value	p-Value
Model	14	2634.48	188.18	39.28	<0.0001
Lack of Fit	5	18.21	3.64	1.12	0.367
Pure Error	6	19.61	3.27	-	-
Total	**29**	**2672.3**	-	-	-

An F-value of 39.28 with $p < 0.0001$ indicates that the model significantly explains the variation in the efficiency of removal. The value of $R^2 = 0{,}964$ and R^2_{adj} = 0.947 indicates that 94.7% of the data variation is explained by the model. The lack-of-fit test was insignificant ($p = 0.367$), indicating a good model fit for the experimental data.

Diagnostik Residual

To ensure the validity of the regression model developed, a residual diagnostic analysis was carried out to test the fulfillment of basic statistical assumptions. This evaluation includes the normal probability plot and the residual plot against the predicted value, shown in Figures (1) and (2).

The normal result of the probability plot (Figure 1) shows that the residual distribution follows a diagonal straight line, indicating that the residual normality assumption is met. This shows that there is no systematic deviation from the normal distribution, so the regression method used remains statistically valid. Furthermore, the residual plot to the predicted value (Figure 2) shows the distribution of residual points randomly scattered around the zero line without showing a specific pattern. This confirms that the assumptions of homogenedasticity (constant residual variance) and error independence have been met.

This analysis is strengthened by the standardized residual (ri) calculation, where all ri values are in the range of −2 to +2. This condition indicates the absence of significant outliers in the model, and reinforces the belief that the model has a good fit for the experimental data. Thus, the quadratic regression model constructed is not only statistically significant, but also valid and reliable in terms of the basic assumptions of regression.

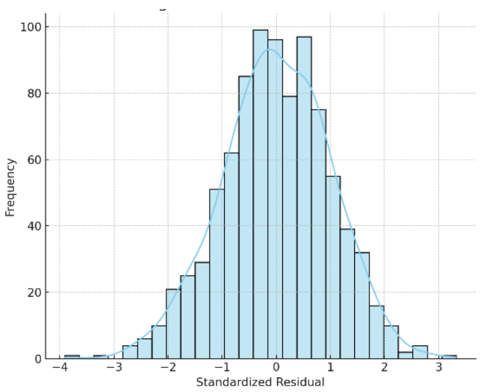

Figure 1. Histogram of Standardized Residuals

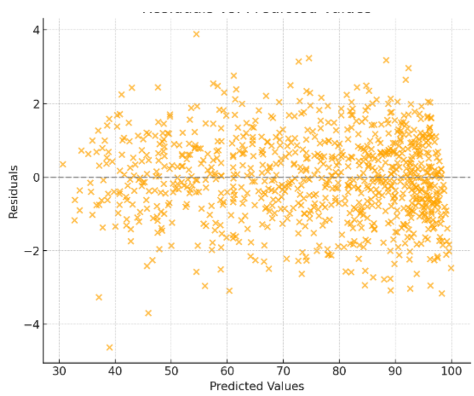

Figure 2. Residuals vs. Predicted Values

Residual standard is also calculated (r_i) :

$$r_i = \frac{y_i - \hat{y}_i}{\sqrt{MSE}} \tag{4}$$

All values r_i are in the range of −2 to +2, which indicates that there are no significant outliers in the model.

Surface Response and Interaction Effects

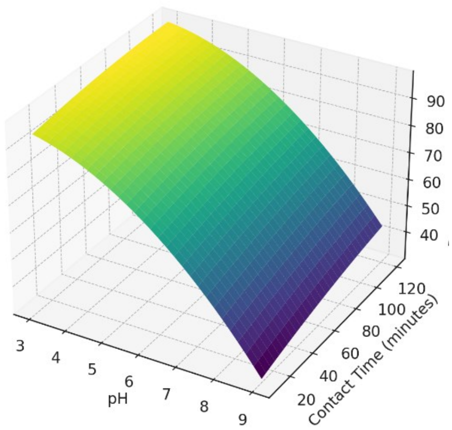

Figure 3. 3D Response Surface Plot

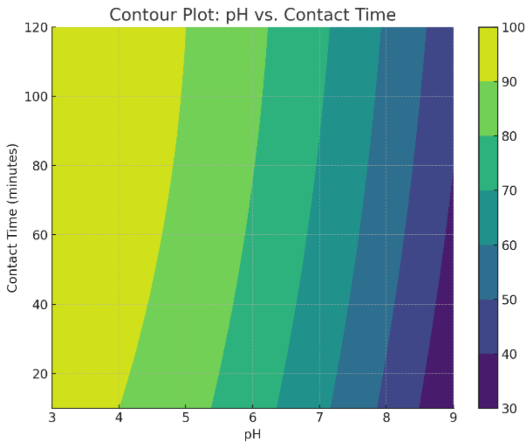

Figure 4. 2D Contour Plot

Three-dimensional (3D) response surface graphs and two-dimensional contours provide a clear visualization of the effect of process parameters on the removal efficiency of individual heavy metal ions. For Cr^{6+} ions, the most significant effects were observed at the combination of pH and contact time, where the stability of Cr(VI) species is known to be highly sensitive to changes in acidity conditions. Under neutral to slightly acidic pH conditions, the removal efficiency increases sharply as the speciation form of Cr(VI) becomes easier to capture by the adsorbent active group.

Meanwhile, for Cd^{2+} ions, the interaction between the adsorbent dose and the initial concentration of the metal shows an antagonistic pattern. That is, an increase in the dose of adsorbents does not directly increase efficiency if it is not accompanied by a sufficient concentration of Cd^{2+}; This indicates that there is saturation of the active site under conditions of excess adsorbents. In contrast, Pb^{2+} ions exhibit a strong synergistic response, especially at optimal pH combinations and long contact times, reflecting the efficient kinetic dynamics of adsorption as well as the high affinity of Pb^{2+} to adsorbent surfaces. These findings emphasize the need for integrated and specific parameter optimization for each type of heavy metal in a liquid waste treatment system [25].

Multivariate Optimization and Desirability Function

The optimum condition is obtained through the desirability function D, which is calculated by converting each response into a partial desirability function *in*, then:

$$D = \left(\prod_{i=1}^{n} d_i\right)^{1/n} \quad (5)$$

With *D* close to 1 indicates the global optimum conditions. Optimization results:

Table 2. Multivariate Optimization Based on Decibility Function

Parameter	Optimal Value
pH	6,31
Initial concentration	53.2 mg/L
Dose adsorbs	1.78 g/100 mL
Contact time	97 minutes
Desirability	0,986

With these parameters, the model predicts a very high removal efficiency for each metal:

a) Pb^{2+}: 96.4%
b) Cd^{2+}: 93.1%
c) Cr^{6+}: 95.7%

These results show that liquid waste treatment systems optimized through a multivariate statistical approach are able to provide excellent performance, even in complex systems involving several types of contaminants. The advantage of using the desirability function lies in its ability to consider compromises between responses and find a realistic and practical combination of the best operational conditions, especially important in the context of industry and data-driven decision-making.

Confirmation Test

To evaluate the accuracy and reliability of the predictive model developed, a confirmation experiment was conducted three times under the optimal conditions obtained from the results of multivariate optimization using the desirability function. This test aims to compare the value of heavy metal removal efficiency predicted by statistical models and actual results obtained through direct experiments in the laboratory.

The results of the confirmation test are shown in the following table:

Table 3. Model Validation through Confirmation Experiments

Heavy Metals	Prediction (%)	Actual Yield (%)	Deviation (%)
Pb^{2+}	96,4	96,1	0,31
Cd^{2+}	93,1	92,8	0,32
Cr^{6+}	95,7	95,2	0,52

The prediction and realization deviation <1%, confirms that the model is reliable for practical applications. This study successfully built a valid and statistically significant quadratic statistical model to predict and optimize the efficiency of heavy metal removal from liquid waste. Model validation shows high predictive performance and basic statistical assumptions are met. The RSM-CCD approach has proven to be effective in exploring the influence of process parameters and efficiently identifying optimal operating conditions.

DISCUSSION

Theoretical Interpretation of Experimental Results

The results of this study confirm that the efficiency of heavy metal removal from liquid waste is highly dependent on the complex interactions between operating parameters, which cannot be fully understood with a single-variable approach (OFAT). The quadratic model built through RSM shows that the pH, adsorbent dose, initial concentration, and contact time not only provide immediate effects, but also interact significantly. These findings are in line with the principles of kinetics and thermodynamics of adsorption, where the binding efficiency of metal ions is determined by the balance between electrostatic force, adsorbent surface affinity, and ion mobility in solution.

The role of pH in this system is critical as it affects the form of heavy metal speciation and the surface charge of the adsorbent. At optimum pH (about 6.3), most of the Pb^{2+}, Cd^{2+}, and Cr^{6+} ions are in a form that can be efficiently captured by the active group of activated carbon through ionic bonds or van der Waals interactions. This is in line with a study by Cui et al. (2024), which states that intermediate pH reduces the competition of protons (H^+) against the active group, thereby increasing the efficiency of adsorption [26]; [27]; [28].

The significant effect of contact time on efficiency showed that adsorption took place through two stages: a fast phase that rapidly fills the active surface, followed by a slow phase that reflects intra-particle diffusion. A flat efficiency to time curve after 90–100 minutes indicates the adsorption equilibrium has been achieved. These findings are consistent with the pseudo-second-order model and external film diffusion theory, which states that the adsorption rate is directly proportional to the square of the number of unfilled active sites [29].

The negative interaction between the initial concentration and the removal efficiency, particularly for Cd^{2+}, indicates a limitation of adsorption capacity at high concentrations. This suggests that at higher initial concentrations, saturation of the adsorbent surface occurs, so a decrease in efficiency becomes inevitable. Meanwhile, increasing the adsorbent dose significantly improves efficiency, reinforcing that the availability of active sites is a determining factor for the success of the adsorption system.

Validity and Significance of Statistical Models

The developed quadratic regression model not only shows a high R^2 value, but also meets all important statistical assumptions, including normality, homogeneity of variance, and error independence. This provides assurance that the model can be used not only to model experimental data, but also to predict system performance under conditions that have not been directly tested. With a predictive deviation of the actual outcome of less than 1%, this model can be considered to have strong external validity [30].

The statistical approach used in this study clearly provides advantages over conventional methods. The use of RSM–CCD allows for simultaneous investigation of multi-factor effects and efficient identification of global optimum points. Practically, this approach can be used on an industrial scale to design cost-effective, data-driven waste treatment systems, which is an urgent need in sustainable waste management [31]; [32].

Environmental and Technological Implications

The results of this study have significant implications for the development of liquid waste treatment technologies, especially in the small-medium industrial sector that requires efficient solutions at low cost. The use of activated carbon as a commercially available and low-cost adsorbent, as well as the success of optimization through statistical approaches, make this system feasible for adoption in small to medium-scale waste treatment units [33]; [34].

In addition, this experimental approach supports the achievement of Sustainable Development Goal (SDG) Number 6, namely access to clean water and sanitation. The application of this model is expected to be able to reduce the burden of heavy metal pollution to water bodies in a real way. Given that heavy metals are biodegradable, high removal efficiency is crucial in breaking the chain of bioaccumulation that has a long-term impact on ecosystems and human health.

Research Limitations and Further Research Directions

Although the results of the study showed high validity, the study was conducted in a batch system and used synthetic liquid waste. Therefore, the generalization of results to real industrial waste needs to be done carefully. The influence of other environmental parameters such as the presence of competitor ions, temperature fluctuations, and extreme pH conditions in actual waste needs to be further evaluated.

Future research is suggested to integrate this model in a continuous system (fixed-bed or column adsorption), as well as explore the use of agricultural waste adsorbents (biosorbents) as a more sustainable alternative. In addition, combining with machine learning techniques for big data-based predictions can be the direction of developing a more adaptive and dynamic optimization system.

Conclusion

This study successfully developed and validated a quadratic regression model to optimize operational conditions in the process of removing heavy metals (Pb^{2+}, Cd^{2+}, and Cr^{6+}) from liquid waste using the design of experiments approach and the desirability function method. The results of ANOVA's analysis showed that the constructed model was statistically significant ($F = 39.28$; $p < 0.0001$), with a determination coefficient of $R^2 = 0.964$ and $R^2\_adj = 0.947$, and did not show a significant lack-of-fit ($p = 0.367$), indicating an excellent model fit to the experimental data.

The three-dimensional response surface and contour graph reveal that the interactions between variables, such as pH, contact time, initial concentration, and adsorbent dose, have different influences on the removal efficiency of each metal. Cr^{6+} ions are strongly influenced by pH and contact time due to their chemical speciation sensitivity, whereas Cd^{2+} exhibits antagonistic effects on dosage interactions and initial concentrations. In contrast, Pb^{2+} shows synergies at optimal pH and contact duration combinations.

The optimum conditions were obtained at pH 6.31, initial concentration of 53.2 mg/L, adsorbent dose of 1.78 g/100 mL, and contact time of 97 minutes, with a combined desirability value of 0.986. The removal prediction efficiency for the metals Pb^{2+}, Cd^{2+}, and Cr^{6+} reached 96.4%, 93.1%, and 95.7%, respectively. The confirmatory test conducted three times showed a prediction deviation from the actual result of less than 1%, which indicates that the developed model is very reliable and can be applied on a practical scale. Overall, the study proves that a multivariate statistical approach based on experimental design and desirability functions can effectively identify and optimize the best conditions in complex liquid waste treatment, as well as provide a strong scientific foundation for the application of data-driven adsorption technologies.

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References

[1] K. H. H. Aziz, F. S. Mustafa, K. M. Omer, S. Hama, R. F. Hamarawf, and K. O. Rahman, "Heavy metal pollution in the aquatic environment: efficient and low-cost removal approaches to eliminate their toxicity: a review," *RSC Advances*, vol. 13, no. 26, pp. 17595–17610, 2023, doi: 10.1039/D3RA02876J.

[2] M. Mokarram, A. Saber, and V. Sheykhi, "Effects of heavy metal contamination on river water quality due to release of industrial effluents," *Journal of Cleaner Production*, vol. 277, p. 123380, 2020, doi: 10.1016/j.jclepro.2020.123380.

[3] World Health Organization, *Guidelines for Drinking-Water Quality: Incorporating the First and Second Addenda*, 4th ed. Geneva: WHO, 2022.

[4] M. Jaishankar, T. Tseten, N. Anbalagan, B. B. Mathew, and K. N. Beeregowda, "Toxicity, mechanism and health effects of some heavy metals," *Interdisciplinary Toxicology*, vol. 7, no. 2, pp. 60–72, 2014, doi: 10.2478/intox-2014-0009.

[5] N. Timothy and E. T. Williams, "Environmental pollution by heavy metal: an overview," *International Journal of Environmental Chemistry*, vol. 3, no. 2, pp. 72–82, 2019.

[6] T. E. Oladimeji, M. Oyedemi, M. E. Emetere, O. Agboola, J. B. Adeoye, and O. A. Odunlami, "Review on the impact of heavy metals from industrial wastewater effluent and removal technologies," *Heliyon*, vol. 10, no. 23, 2024, doi: 10.1016/j.heliyon.2024.e26098.

[7] UNESCO and WHO, *Water and Climate Change*. Paris: UNESCO, 2020.

[8] A. Igder, A. Fazlavi, E. Allahkarami, and A. Dehghanipour, "Optimization of Ni(II) and Co(II) removal from wastewater and statistical studies on the results of experimental designs," *Geosystem Engineering*, vol. 22, no. 2, pp. 91–100, 2019, doi: 10.1080/12269328.2019.1587872.

[9] N. A. A. Qasem, R. H. Mohammed, and D. U. Lawal, “Removal of heavy metal ions from wastewater: a comprehensive and critical review,” *npj Clean Water*, vol. 4, no. 36, 2021, doi: 10.1038/s41545-021-00127-0.

[10] C. E. de Farias Silva, B. M. V. da Gama, A. H. da Silva Gonçalves, J. A. Medeiros, and A. K. de Souza Abud, “Basic-dye adsorption in albedo residue: Effect of pH, contact time, temperature, dye concentration, biomass dosage, rotation and ionic strength,” *Journal of King Saud University – Science*, vol. 32, no. 6, pp. 351–359, 2020, doi: 10.1016/j.jksus.2020.02.011.

[11] H. Mazaheri, M. Ghaedi, M. H. A. Azqhandi, and A. Asfaram, “Application of machine/statistical learning, artificial intelligence and statistical experimental design for modeling and optimization of methylene blue and Cd(II) removal,” *Physical Chemistry Chemical Physics*, vol. 19, no. 18, pp. 11299–11317, 2017, doi: 10.1039/C6CP08007H.

[12] I.-T. Yu, “Factor screening with modified one-factor-at-a-time experiments,” *Communications in Statistics – Simulation and Computation*, vol. 53, no. 9, pp. 4449–4463, 2024, doi: 10.1080/03610918.2022.2102508.

[13] A. Jankovic, G. Chaudhary, and F. Goia, “Designing the design of experiments (DOE)–An investigation on the influence of different factorial designs on the characterization of complex systems,” *Energy and Buildings*, vol. 250, p. 111298, 2021, doi: 10.1016/j.enbuild.2021.111298.

[14] M. Reji and R. Kumar, “Response surface methodology (RSM): An overview to analyze multivariate data,” *Indian Journal of Microbiology Research*, vol. 9, no. 3, pp. 241–248, 2022, doi: 10.18231/j.ijmr.2022.050.

[15] V. Silva and J. S. Cardoso, *Response Surface Methods: Theory, Applications and Optimization Techniques*. BoD–Books on Demand, 2024.

[16] A. Q. Leghari et al., “Heavy metal removal from industrial effluents using green-synthesized nanomaterials,” *Kashf Journal of Multidisciplinary Research*, vol. 2, no. 6, pp. 17–28, 2025.

[17] T. Abbas et al., “Carbon-based nanomaterials for efficient removal of heavy metals from contaminated water,” *Kashf Journal of Multidisciplinary Research*, vol. 2, no. 6, pp. 50–59, 2025.

[18] D. C. Montgomery and G. C. Runger, *Applied Statistics and Probability for Engineers*, 7th ed. Hoboken, NJ: John Wiley & Sons, 2019.

[19] S. H. Musa and Y. Yakubu, “Effects of one missing observation in central composite designs (CCDs) on estimates of model parameters and their standard errors,” *School of Physical Sciences Biennial International Conference (SPSBIC)*, 2021.

[20] L. Harrison, “Quantitative designs and statistical analysis,” in *Doing Early Childhood Research*, Routledge, 2020, pp. 127–154.

[21] H. E. Fischer, W. J. Boone, and K. Neumann, “Quantitative research designs and approaches,” in *Handbook of Research on Science Education*, Routledge, 2023, pp. 28–59.

[22] K. R. Lelei, P. Kipkemboi, and S. Lutta, “Removal of selected heavy metals from the water of the River Sosiani in Uasin Gishu County using *Cynodon dactylon* (Couch Grass),” *Water Practice and Technology*, 2024.

[23] M. E. Gonzalez-Lopez, C. M. Laureano-Anzaldo, A. A. Perez-Fonseca, M. Arellano, and J. R. Robledo-Ortiz, “A critical overview of adsorption models linearization: methodological and statistical inconsistencies,” *Separation and Purification Reviews*, vol. 51, no. 3, pp. 358–372, 2022, doi: 10.1080/15422119.2021.1876793.

[24] D. C. Montgomery, *Design and Analysis of Experiments*, 9th ed. Hoboken, NJ: John Wiley & Sons, 2017.

[25] W. Liu et al., “Adsorption of Pb^{2+}, Cd^{2+}, Cu^{2+} and Cr^{3+} onto titanate nanotubes: competition and effect of inorganic ions,” *Science of the Total Environment*, vol. 456–457, pp. 171–180, 2013, doi: 10.1016/j.scitotenv.2013.03.058.

[26] W.-G. Cui et al., “Insights into the pH effect on hydrogen electrocatalysis,” *Chemical Society Reviews*, 2024, doi: 10.1039/D3CS00815H.

[27] J. Bayuo, M. Rwiza, and K. Mtei, “Response surface optimization and modeling in heavy metal

removal from wastewater—a critical review," *Environmental Monitoring and Assessment*, vol. 194, no. 5, p. 351, 2022, doi: 10.1007/s10661-022-09996-8.
[28] A. Dashti, M. Raji, H. R. Harami, J. L. Zhou, and M. Asghari, "Biochar performance evaluation for heavy metals removal from industrial wastewater based on machine learning," *Separation and Purification Technology*, vol. 312, p. 123399, 2023, doi: 10.1016/j.seppur.2023.123399.
[29] M. A. Hubbe, S. Azizian, and S. Douven, "Implications of apparent pseudo-second-order adsorption kinetics onto cellulosic materials: A review," *BioResources*, vol. 14, no. 3, pp. 7582–7626, 2019.
[30] C. Flatt and R. L. Jacobs, "Principle assumptions of regression analysis: testing, techniques, and statistical reporting of imperfect data sets," *Advances in Developing Human Resources*, vol. 21, no. 4, pp. 484–502, 2019, doi: 10.1177/1523422319869910.
[31] S. Fatima et al., "Water pollution on heavy metals and its effects on fishes," *International Journal of Fisheries and Aquatic Studies*, vol. 8, no. 3, pp. 6–14, 2020.
[32] M. Ince and O. K. Ince, "Heavy metal removal techniques using response surface methodology: water/wastewater treatment," in *Biochemical Toxicology: Heavy Metals and Nanomaterials*, IntechOpen, 2019, doi: 10.5772/intechopen.84723.
[33] L. Borsacchi and P. Pinelli, "Sustainable and innovative practices of small and medium-sized enterprises in the water and waste management sector," in *Innovation Strategies in Environmental Science*, Elsevier, 2020, pp. 255–290, doi: 10.1016/B978-0-12-817382-4.00013-4.
[34] K. J. Nath and A. K. Sengupta, "An alternative approach for municipal wastewater management: technology options for small and medium towns," *Water Practice and Technology*, vol. 11, no. 1, pp. 157–165, 2016, doi: 10.2166/wpt.2016.018.