**Research Article**

# Remediation of lead-contaminated sludge for soil conditioning using waste lemon peel

Chinonye Duru[1],* (iD) , Fatima Badiru Ibrahim[1,2] (iD) , Aliyu Adamu Dandajeh[1] (iD)

**ABSTRACT:** Enhancing soil fertility, organic matter, and nutrient content through sludge application to agricultural land is a promising approach to improve crop efficiency. Waste lemon peel, considered agricultural waste, has potential as a remediation agent for lead-contaminated sewage. This study examines the physical and chemical properties of lemon peel extract to evaluate its potential as a substitute for commercial fertilizer. The response surface methodology was employed to investigate the factors influencing the process. A multi-objective numerical optimization technique based on the desirability function was used to identify the optimal conditions for lead removal efficiency in contaminated wastewater. The study focuses on parameters such as pH, extractant concentration, and contact time. The optimization analysis achieved a maximum removal efficiency ($E_R$) of 94.42% at a pH of 5.0, an extractant concentration of 20 g/L, and a contact time of 135 minutes. Under optimal conditions, the highest lead removal efficiency was 90.50%, while the lowest efficiency observed was 47.0%. The findings of this study underscore the significant issue of environmental contamination, particularly with heavy metals like lead, and highlight the necessity for sustainable and eco-friendly solutions. The effective use of lemon peel extract demonstrates its potential as a viable alternative for remediating lead-contaminated wastewater.

**Keywords:** *Sludge, Agricultural land, Lemon peel, Lead removal, Heavy metals*

## 1. INTRODUCTION

Environmental contamination, especially with heavy metals like lead, has become a critical concern in recent years. Lead contamination in soil and sediments poses serious health risks to both humans and ecosystems. Traditional remediation methods often involve the costly and environmentally detrimental removal and disposal of contaminated materials. Various techniques, including ion exchange technologies [1], precipitation [2], ultrafiltration [3], reverse osmosis, and electrodialysis [4, 5], have been developed to extract dissolved heavy metals from wastewater. However, these methods are typically expensive, require high levels of expertise, and often involve secondary waste generation, limiting their accessibility and sustainability.

Recent research explores alternative, eco-friendly remediation techniques, particularly those utilizing agricultural waste materials as biosorbents. Bioadsorption, which uses plant-based materials to bind heavy metals, has emerged as a cost-effective and sustainable method for environmental clean-up. However, many studies have focused on materials such as sawdust, algae, and banana peels, with limited emphasis on citrus peels, particularly lemon peels, for lead remediation. This study aims to address this gap by evaluating the efficacy of waste lemon peel extract as a remediation agent for lead-contaminated sludge. Lemon peel is a rich source of organic acids, including citric and ascorbic acids, which contribute to its natural acidity. These acids, along with functional groups such as hydroxyl (-OH) and carboxyl (-COOH), play a crucial role in binding and chelating heavy metals, thereby

facilitating their removal from contaminated media. FTIR analysis confirms the presence of these functional groups, demonstrating the lemon peel extract's potential for lead remediation. Additionally, unlike synthetic chemical extractants, lemon peel is widely available, biodegradable, and non-toxic, making it an attractive alternative for sustainable remediation.

Recycling valuable components such as organic matter and plant nutrients has generated significant interest in using sludge on agricultural land [6]. However, the presence of harmful elements like heavy metals restricts its application, as these contaminants can accumulate in soils and potentially leach into groundwater [7, 8]. Thus, developing a natural, effective, and low-cost remediation method is essential to enable the safe reuse of sludge while minimizing environmental risks.

In Heavy metals, defined as chemical elements with high density that are toxic or hazardous even at low concentrations, are naturally occurring in the earth and cannot be broken down or removed [9]. Despite their low abundance, heavy metals like lead and mercury are widely used in technology, and even small amounts can be fatal to living organisms. The concern for heavy metals stems from their non-biodegradability, toxicity, and persistence [10]. Research has long indicated that heavy metals can cause serious illness and unexpected death in humans. They are currently considered one of the main sources of environmental contamination. For instance, over 500 children died from lead poisoning in Zamfara within seven months in 2010-the deadliest heavy metal incident ever documented in Nigerian history [11].

One effective approach for mitigating environmental contamination by radionuclides is the adsorption method, which demonstrates excellent performance in the selective and safe removal of radionuclides from contaminated water. This method is characterized by operational simplicity, adaptability to low-contamination effluents, and the availability of various low-cost adsorbents, making it suitable for both continuous and batch processes [8].

Strict regulations have been developed to limit sludge application in agricultural soils due to the detrimental effects of heavy metals on the ecosystem. [12].

Achieving greater sustainability in sludge utilization requires extracting heavy metals from the sludge before application. Once its heavy metal content is reduced, sludge can be used as an organic soil conditioner. Therefore, efforts are being made to find environmentally friendly extractants that are non-toxic, widely accessible, and reasonably priced to effectively remove heavy metals from the environment [13, 14].

## 2. MATERIALS AND METHODS

**2.1. Materials.** The materials used in this study included fresh lemons (Citrus limon 'Eureka') sourced from the ABU Faculty of Agriculture orchard, and sludge collected from the sedimentation tanks of the Ahmadu Bello University water treatment plant. Equipment used included an atomic absorption spectrophotometer, pH meter (PHS-3C, Hanna USA), FTIR spectrophotometer, mechanical shaker, and various laboratory glassware and tools. Reagents such as sulfuric acid, nitric acid, potassium bromide (KBr), and distilled water were employed. The lemon peels were processed by washing, oven-drying, milling, and sieving to prepare extracts at 20 g/L, 60 g/L, and 100 g/L concentrations.

**2.2. Methods.** *2.2.1. Preparation of the sludge sample.* The sludge was sampled from six sedimentation tanks to ensure a homogeneous and representative sample. In the laboratory, the sludge was prepared through a series of steps. First, it was allowed to settle for 24 hours to separate the solids from excess water, after which the excess water was carefully decanted. The sludge was then centrifuged in 45 mL tubes at 2,000 rpm to further reduce moisture. Next, the dewatered sludge was oven-dried at 100°C for one hour to ensure complete dryness. Finally, the dried sludge was crushed using a laboratory mortar and pestle and sieved through a 212-micron mesh to achieve a uniform particle size.

*2.2.2. Preparation of the lemon peel extract.* The absorbent was prepared using fresh lemon peels following a series of systematic steps designed to ensure efficiency and reliability. Fresh lemon peels were collected and thoroughly washed to remove any dirt or contaminants. The peels were then dried in two stages: first at 50°C for 12 hours to reduce moisture content and then at 100°C for an additional 2 hours to ensure complete dehydration. The dried peels were ground into a fine powder using a milling machine and then sieved through a 212-micron mesh to achieve uniform particle size. The resulting powder was mixed with distilled water in specific ratios to prepare the extract solution. This extract served as the active agent in the remediation process. The schematic diagram in Figure 1 below provides a visual representation of the step-by-step preparation process.

*2.2.3. Characterization of sludge.* The sludge's physical and chemical properties, including lead content, total nitrogen, available phosphate, pH, and total potassium, were analyzed to evaluate its potential as a fertilizer substitute.

**pH measurement:** 10 g of sludge was mixed with 20 mL distilled water, stirred for 30 minutes, and measured using a pH meter.

**Sludge digestion:** 0.5 g sludge was digested with nitric, perchloric, and sulfuric acids at 150°C, diluted to 50 mL, and analyzed for lead and potassium using Atomic Absorption Spectroscopy (AAS).

**Nitrogen analysis:** The Kjeldahl method was used, and total nitrogen was calculated.

**Phosphate and potassium analysis:** Available phosphate was determined by the Bray and Kurtz method, while potassium was measured using a flame photometer.

*2.2.4. Lemon peel extract characterization.* Lemon peel extract's pH, lead content, nitrogen, phosphate, and potassium levels were analyzed using methods outlined for sludge characterization. FTIR spectroscopy was used to identify functional groups in lemon peel extract, using KBr discs for spectra recorded between 4000 and 600 cm$^{-1}$.

*2.2.5. Response Surface Methodology (RSM).* A Central Composite Design (CCD) with 20 runs optimized lead extraction. Parameters included:pH: 5.8–9.2, contact time: 30–240 min, extractant concentration: 20–100 g/L. The response variable, removal efficiency, was modeled and analyzed for interactions and optimal conditions.

## 3. RESULTS

**3.1. Physicochemical properties of sludge**. The physicochemical properties of the sludge obtained from Ahmadu Bello University waterworks are summarized in Table 1. The sludge had a pH of 6.2, which is slightly acidic and falls within the acceptable
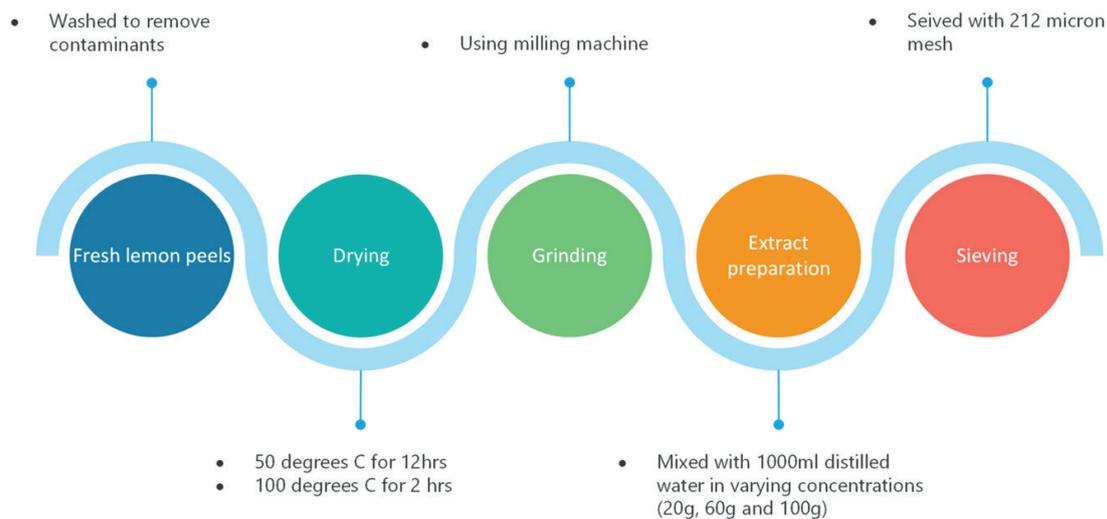
**Figure 1**. Schematic diagram of the preparation of lemon peel extract.

range for agricultural soils (5.5–7.5) [15]. Soil pH is a critical factor as it influences chemical and biochemical processes, regulates plant nutrient availability, and affects crop productivity. A pH range of 6 to 7 is generally optimal for plant growth as most nutrients are readily available within this range. The initial lead concentration in the sludge was 95 mg/L, and its nutrient content, including nitrogen, phosphorus, potassium, organic carbon, and organic matter, is also reported in the table.

**Table 1**. Sludge sample characterization

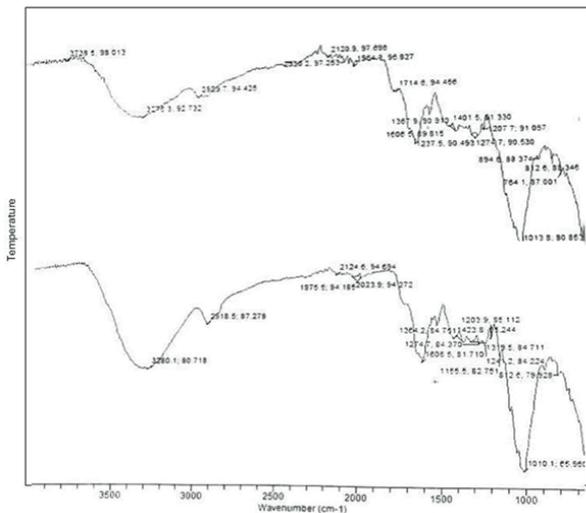| Parameter | Unit | Value |
|---|---|---|
| Pb | mg/kg | 95.0 |
| P | mg/L | 150.6 |
| K | mg/L | 265.0 |
| N | mg/L | 86.8 |
| Organic carbon | % | 1.6 |
| pH | - | 6.2 |
| Organic matter | % | 2.8 |

**3.2. Physicochemical properties of lemon peel extracte**. Table 2 presents the physicochemical characteristics of lemon peel extracts, revealing a pH indicative of slight acidity and ideal for heavy metal extraction. Mechanisms such as ion exchange, dissolution, and desorption govern the migration and transformation of heavy metals [16], with pH playing a pivotal role in heavy metal speciation, thereby influencing their migration and transformation [17]. pH alterations affect the ion forms of metals adsorbed on soil surfaces, as higher pH levels lead to increased metal cation hydrolysis. Additionally, rising pH enhances the net negative surface charge of soils, augmenting the affinity between soil surfaces

and heavy metal ions [18]. Conversely, when pH drops below 3, active sites like carboxyl groups become protonated, impeding the attraction of positive metal ions from the solution. Moreover, soil pH impacts heavy metal solubility through factors such as surface potential, clay edge charge, ion-pair formation, organic matter solubility, and stability of metal carbonates and phosphates [19]. Consequently, heavy metal extractability is markedly affected by soil pH due to alterations in adsorption, solubility, stability, and other factors, resulting in diminished metal extraction as soil pH rises. Furthermore, the lemon peel was found to contain NPK nutrients, enhancing nutrient availability in the soil.

**3.3. Functional properties of lemon peel extract haracterization.** FTIR analysis is imperative to identify distinctive functional groups in the extract [20]. Figure 2 displays an FTIR study of lemon peel aimed at identifying the functional groups aiding in lead extraction. Notable peaks signify various functional groupings, primarily hydroxyl, carboxyl, ether, and amide groups, suspected sites for heavy metal ion bonding to biomass. OH stretching vibrations exhibit a broad spectrum of frequencies, indicating bonded and free hydroxyl bands in carboxylic acids [21]. A broad absorption band in the 3500-3000 cm$^{-1}$ range signifies distinctive O–H stretching vibration and hydrogen bonds of hydroxyl groups, possibly due to intra- and intermolecular hydrogen bonding in the fibre backbone [22]. Peaks at 2929 cm$^{-1}$ correspond to CH group stretching asymmetric vibration, while peaks around 1636-1617 cm$^{-1}$ denote alkyl carbonate (C-OH) of carboxyl and carboxylic acid, and those at 1415-1406 cm$^{-1}$ suggest carbonate ion vibrations of lemon peel. Every sample analyzed exhibits peaks in the 1069.66-10588.73 cm$^{-1}$ range, attributed to cellulose component stretching [23]. Polysaccharides in lemon peel, containing groups like alcohols, acids, phenolic hydroxides, aldehydes, and ethers, enhance ion exchange capacities due to their polar nature, aiding in lead extraction [24]. The fiber fingerprint region, encompassing CH$_3$ deformation, C–O–C stretching, and O–H bending, ranges from 800 to 1200 cm$^{-1}$ wave numbers in the studied peel

**Table 2**. Characterization of lemon extracts.

| Concentration (g/L) | pH | N (mg/kg) | % N | P (mg/kg) | % P | K (mg/kg) | % K |
|---|---|---|---|---|---|---|---|
| 20 | 5.04 | 14 | 0.0014 | 172.14 | 0.0172 | 31 | 0.0031 |
| 60 | 5.13 | 112 | 0.0112 | 236.69 | 0.0237 | 170 | 0.017 |
| 100 | 5.21 | 478 | 0.0478 | 322.76 | 0.032276 | 210 | 0.021 |



**Figure 2**. FTIR analysis of lemon peel before and after the extraction process.

**Table 3**. Central Composite Design (CCD) matrix and output response for lead removal from contaminated sludges.

| Run | Factor 1<br>A: pH | Factor 2<br>B: Extract concentration (g/L) | Factor 3<br>C: Contact time (min) | Respone<br>Removal efficiency (%) |
|---|---|---|---|---|
| **1** | **10** | **20** | **240** | **47.0** |
| 2 | 5 | 60 | 135 | 71.25 |
| 3 | 5 | 100 | 240 | 79.0 |
| 4 | 7.5 | 60 | 135 | 69.61 |
| 5 | 7.5 | 60 | 135 | 70.86 |
| 6 | 7.5 | 60 | 240 | 58.44 |
| 7 | 5 | 20 | 240 | 50.6 |
| 8 | 7.5 | 60 | 135 | 68.30 |
| 9 | 10 | 100 | 30 | 83.62 |
| **10** | **5** | **100** | **30** | **90.50** |
| 11 | 7.5 | 100 | 135 | 88.86 |
| 12 | 5 | 20 | 30 | 52.0 |
| 13 | 7.5 | 60 | 135 | 66.0 |
| 14 | 7.5 | 60 | 135 | 66.0 |
| 15 | 10 | 100 | 135 | 81.54 |
| 16 | 10 | 20 | 135 | 50.9 |
| 17 | 7.5 | 60 | 30 | 64.0 |
| 18 | 7.5 | 20 | 135 | 51.0 |
| 19 | 7.5 | 60 | 30 | 65.33 |
| 20 | 10 | 60 | 240 | 56.0 |

samples. Following extraction, the FTIR spectrum of the lemon peel is shown in the lower image and reveals changes in peak position, strength, and number compared to that of the original lemon peel, suggesting extraction of phytochemical components. This alteration may result from sludge colloid containing heavy metal ions exchanged and combined by phytochemical constituent functional groups.

**3.4. Responses from the removal efficiency of lead from the sludge after the experiment**. The sludge containing 95.8 mg/kg of lead was washed using waste lemon extract and the lead concentration after the extraction process for samples 1 to 20, subjected to different conditions was observed. The design of the experiment layout with responses is given in Table 3. The laboratory experiment, conducted using the lead-contaminated sludge experiment design outlined in Table 3, yielded the removal efficiency response for further analysis via ANOVA. The results indicated a maximum lead removal efficiency of 90.50% at pH 5.0, a contact time of 30 min, and an extractant concentration of 100 g/L. Conversely, the minimum removal efficiency recorded was 47.0% at pH = 10, contact time = 240 min, and extractant concentration = 20 g/L. These findings suggest that increasing the extractant concentration, coupled with moderate contact time and an acidic environment, enhances removal efficiency from the sludge

**3.4.1. Effect of extractant concentration on lead extraction from sludge.** The concentration of the extractant plays a significant role in lead extraction efficiency and can impact heavy metal removal [17]. Lead removal notably increased with the highest extractant concentration of 100g/L, as this concentration determines the availability of functional groups in the reaction, potentially supplying more complex sites for heavy metal binding [17]. [25] Observed that the highest lead removal efficiency was achieved at a concentration of 100g/L. Additionally, the identification of phenol and alkene, indicated by wave bands at 1237.5 cm$^{-1}$ and 1606.5 cm$^{-1}$, respectively, aids in lead extraction from the sludge, as also noted by [26]. Furthermore, other functional groups present in lemon peel, such as carboxylic acids and amines, which exchange hydrogen ions for metal cations or enhance electronic

**Table 4**. Fit summary for removal efficiency

| Source | Sum of squares | Df | Mean square | R² value | F value | p-value Prob > F | |
|---|---|---|---|---|---|---|---|
| Mean | 146092.16 | 1 | 146092.16 | | | | |
| Block vs Mean | 32.59 | 2 | 16.30 | | | | |
| **Linear** | **1047.94** | **3** | **349.31** | | **4.62** | **0.0290** | **Suggested** |
| Suggested | | | | | | | |
| 2FI vs Linear | 391.92 | 3 | 130.64 | | 2.16 | 0.1511 | |
| **Quadratic** | **450.25** | **3** | **150.08** | **0.90** | **5.55** | **0.0235** | **Suggested** |
| Cubic vs Quadratic | 193.27 | 4 | 48.32 | | 4.32 | 0.0321 | **Aliased** |
| Residual | 23.23 | 4 | 5.81 | | | | |
| Total | 148231.40 | 20 | 7411.56808 | | | | |

donating ability, thereby promoting lead removal from sludge, were identified. This finding aligns with the work conducted by [27], who also confirmed the presence of these functional groups.

***3.4.2. Effect of pH on lead extraction from sludge.*** The pH of the sludge significantly influences the adsorption-desorption behavior of heavy metals and the ionization degree of functional groups in the extracts, consequently affecting the removal efficiency of heavy metals from the sludge. The research findings revealed that the highest removal efficiency of 90.50% was achieved at a pH of 5. This aligns with the research conducted by [28], who suggested that an acidic pH range of 3.5-5.5 is crucial for heavy metal removal. Similarly, [25] noted in their study that heavy metal removal is highly pH dependent, with maximum removal observed at acidic levels. This trend is corroborated by the present research, where the highest removal efficiency was attained at a slightly acidic pH of 5. The lemon peel extractant, aided by the pH, mitigates the negative surface charge of sludge particles and organic matter, thereby facilitating the dissolution of oxides and the formation of soluble metal-organic chelates, ultimately leading to the removal of associated metals.

***3.4.3. Effect of contact time on lead extraction from sludge.*** Contact time also plays a significant role in the adsorption-desorption behavior of heavy metals in sludge, as noted by [17]. In our experiment, it was observed that the lead removal efficiency improved between 30 and 135 minutes of contact time, indicating enhanced extraction of lead from the sludge. This can be attributed to the extraction of more lead from the sludge within this timeframe. However, it is expected that electrostatic repulsion between metal cations on the extractant may impede the adsorption of subsequent metal cations. This observation is consistent with the findings of [29], who reported that the contact time for heavy metal removal varied from 30 to 100 minutes, aligning with the contact time range utilized in our study. On the contrary, the removal efficiency of lead decreased with a contact time of 240 minutes, possibly due to the stability of soluble metal-organic polymers [30]. Additionally, metal removal may be affected by resorption and reprecipitation phenomena, leading to decreased efficiency with increased contact time.

**3.5. Fit summary statistics.** The fit summary provides a comprehensive overview of the test results and computed statistics for each distribution utilized to fit the model. Regression analysis examines the relationship between independent variables and the response, fitting models to the chosen response [31, 32]. These models encompass first-order or linear, second-order or linear with interaction, and cubic equations [35]. Table 4 displays the highest-order polynomial selected from the fit summary analysis, suggesting both linear and quadratic models with significant terms. A model with a P-value less than 0.05 is deemed significant, providing results with a confidence level exceeding 99.5%. The quadratic model demonstrates significance with an F-value of 5.55 at P < 0.0235 [33, 34].

**3.6. Model selection, removal efficiency and analysis of variance.** The analysis utilized Central Composite Design (CCD) for Regression and ANOVA in Design-Expert version 12 software to assess the interaction between variables and responses, aiming to generate the best-fit equations for the model. The validity of the model was confirmed through correlation coefficient and standard deviation assessment [35].

Model creation relied on analyzing variance findings, evaluated against the model's suitability (lack of fit), determined by ANOVA creation. Table 5 presents the ANOVA for removal efficiency across all potential models. Notably, the model with all terms included showed no significant difference from the lack of fit. The low likelihood of noise explaining such variance (0.42 percent) is indicated by the model's F-value of 7.76, affirming its significance. Furthermore, significant model terms are identified by "Prob > F" values less than 0.05. In this case, the significant models include C, BC, and C², as their p-values fall below 0.05. Conversely, non-significant terms such as A, B, AB, AC, A², and B², which did not contribute to the model's hierarchy, were eliminated to enhance it. The refined model is presented in Table 5. Consequently, Equations 2 and 3 provide the model equations derived from the updated and comprehensive model.

**3.7. Model analysis for removal efficiency ($E_R$).** Equations (1) and (2) describe the removal efficiency models in terms of both coded and real components. The coded equation (1) is used to predict removal efficiency by analyzing the relative influence of each factor, while the real equation (2) directly represents experimental conditions and outcomes.

$$E_R = 86.86 + 1.42A + 0.85B + 8.60C + 0.52AB - 1.10AC + 6.89BC + 3.00A^2 - 0.51B^2 - 4.38C^2 \qquad (1)$$

where: A is pH, B is extractant concentration (g/L) and C is con-

**Table 5**. Analysis of Variance for Lead Removal Efficiency (Complete Model Terms)

| Source | Sum of squares | Df | Mean square | F value | $p$-value Prob > F | |
|---|---|---|---|---|---|---|
| Model | 1890.11 | 9 | 210.01 | 7.76 | **0.0042** | **Significant** |
| A-pH | 27.41 | 1 | 27.41 | 1.01 | 0.3437 | |
| B-Extractant concentration | 9.92 | 1 | 9.92 | 0.367 | 0.5616 | |
| C-Contact time | 1010.60 | 1 | 1010.60 | 37.35 | 0.0003 | |
| AB | 2.14 | 1 | 2.14 | 0.08 | 0.7856 | |
| AC | 9.72 | 1 | 9.72 | 0.36 | 0.5655 | |
| BC | 380.05 | 1 | 380.05 | 14.04 | 0.0056 | |
| A$^2$ | 129.91 | 1 | 129.91 | 4.80 | 0.0598 | |
| B$^2$ | 3.70 | 1 | 3.70 | 0.14 | 0.7210 | |
| C$^2$ | 275.85 | 1 | 275.85 | 10.19 | 0.0128 | |
| Residual | 216.50 | 8 | 27.062 | | | |
| Lack of Fit | 201.40 | 5 | 40.28 | 8.00 | **0.0587** | **not significant** |
| Pure Error | 15.10 | 3 | 5.03 | | | |
| Cor Total | 2139.20 | 19 | | | | |

tact time (min)

$$-E_R = 76.47434 - 42.39988A + 21.70612B + 20.20056C$$
$$+ 0.51750AB - 0.31500AC - 1.96929BC + 3.00361A^2$$
$$- 0.50718B^2 - 0.35729C^2 \tag{2}$$

Analysis of the coded equation reveals that contact time (C) and extractant concentration (B) had the most significant effects on removal efficiency, with contact time being the most dominant factor. The model's F-value of 14.08 indicates statistical significance, with a 0.01% probability that the observed effects are due to noise. The significance of model terms is further confirmed by Prob > F values less than 0.05, where C, BC, and C$^2$ are identified as significant contributors. Additionally, the Lack of Fit F-value of 7.55, with a probability of 6.15%, suggests that the model reasonably fits the data.

Equation (3) refines the removal efficiency model by focusing on the most influential variables:

$$-64.64722 + 21.52996B + 18.22712C - 1.96929BC$$
$$- 0.37582C^2 \tag{3}$$

where: B - Extractant concentration (g/L), C - Contact time (min). The perturbation plot (Figure 3) visually represents how removal efficiency responds to variations in key parameters:

• Contact time (C): Removal efficiency increases significantly with contact time up to a certain point, beyond which further increases yield minimal improvements. This is indicated by the steep slope.

• pH (A): A rise in pH enhances removal efficiency, supporting its role in optimizing extraction.

• Extractant concentration (B): Unlike the other factors, extractant concentration has a minimal effect, suggesting that a saturation point is reached.

• Quadratic correlation: A quadratic relationship exists between pH, contact time, and removal efficiency, indicating that their combined effects are nonlinear and require optimization.

This analysis confirms that contact time is the most critical factor in removal efficiency, followed by extractant concentration. The findings demonstrate that while increasing contact time and pH improves performance, extractant concentration has a limited effect. This provides valuable insight into optimizing lemon peel extract as an eco-friendly solution for lead removal.
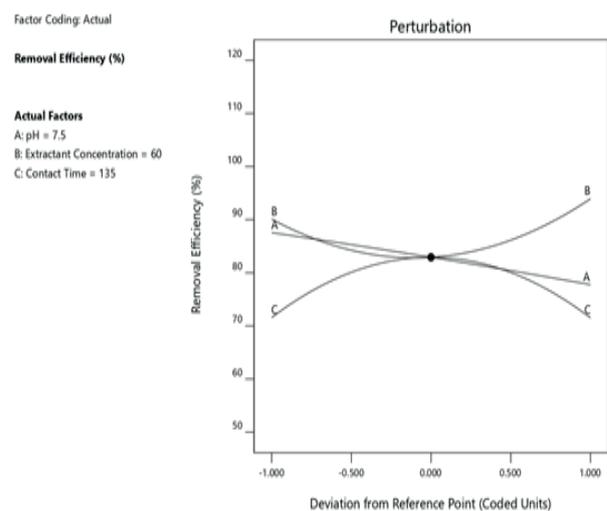


**Figure 3**. Perturbation plot for removal efficiency ($E_R$).

**3.8. Model accuracy and validation.** To verify that the created model is sufficient to support the ANOVA, statistical characteristics including $R^2$, adjusted and anticipated $R^2$, lack of fit, sufficient precision, and residual behaviour are employed. The absence of fit indicates data distributed over the fitted model. Table 6's negligible lack of fit suggests that the $E_R$ model fits the data well enough. The improved and full model terms have $R^2$ values of 0.8124 and 0.8972, respectively. The $R^2$ calculates the percentage (a value between 0 and 1) of the total variance in the data that the

**Table 6**. Analysis of Variance for $E_R$ (Adjusted model terms)

| Source | Sum of squares | Df | Mean square | F value | p-value Prob > F | |
|---|---|---|---|---|---|---|
| Block | 32.59 | 2 | 16.30 | | | |
| Model | 1711.44 | 4 | 427.86 | 14.08 | **0.0001** | **Significant** |
| B-Extractant concentration | 9.92 | 1 | 9.92 | 0.33 | 0.5775 | |
| C-Contact time | 1010.60 | 1 | 1010.60 | 33.25 | < 0.0001 | |
| BC | 380.05 | 1 | 380.05 | 12.50 | 0.0037 | |
| C² | 310.86 | 1 | 310.86 | 10.23 | 0.0070 | |
| Residual | 395.17 | 13 | 30.40 | | | |
| Lack of Fit | 380.07 | 10 | 38.01 | 7.55 | **0.0615** | **not significant** |
| Pure Error | 15.10 | 3 | 5.03 | | | |
| Cor Total | 2139.20 | 19 | | | | |
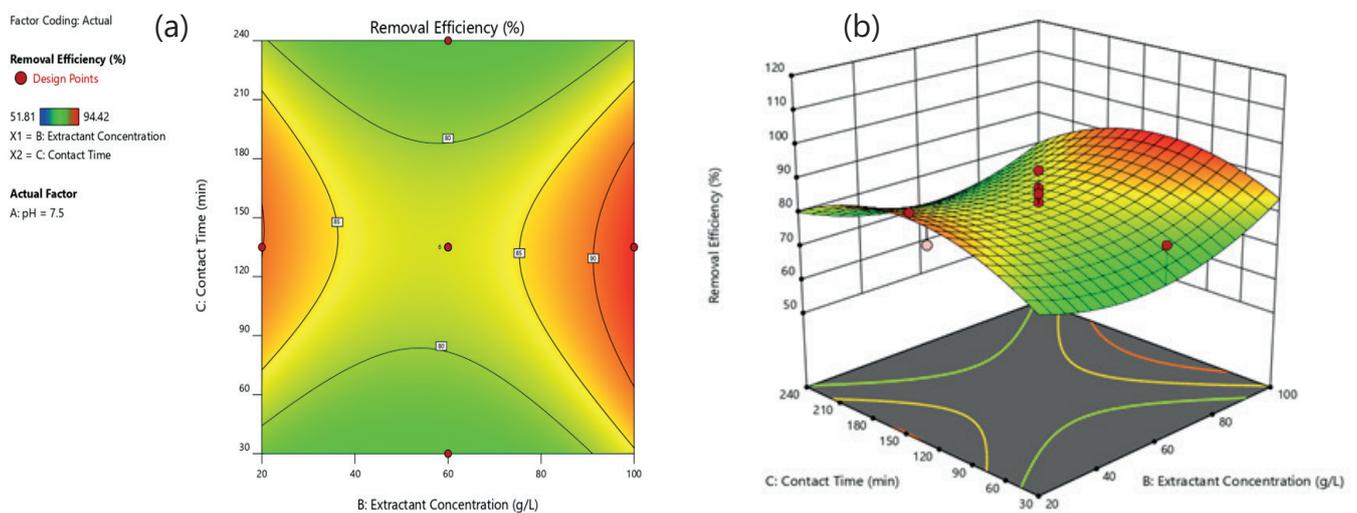


**Figure 4**. 3-D surface and contour plots of $E_R$ with contact time and extractant concentration: (a) contour map and (b) surface plot.

model is responsible for. If a model term does not significantly improve the model, the adjusted R² falls as the number of terms in the model rises. The predicted R² calculates how much of the variation in fresh data is explained by the model. A ratio larger than 4 is considered ideal, and a ratio of 10.596 and 12.356 for both complete and enhanced model terms, respectively, shows an appropriate model. Appropriate accuracy measures signal due to model ratio. For the modified model, the difference between the corrected R² and the projected R² is 0.2, falling within a fair acceptability range (Design Expert 12). These statistical characteristics show that the model can accurately anticipate the behaviour of $E_R$.

**3.9. Surface and contour plots.** Figures 4 through 6 display the 3-D response surface plots and the matching 2-D contour maps produced by the model for $E_R$. Plots showing the $E_R$ trend together with concurrent changes in the important components are displayed. Given the significance of elements A, B, and C for the $E_R$, the model graph's priority was established using the perturbation plot shown in Figure 4 through 6. The steep slope or curve of a factor indicates that it influences the answer. Insensitivity to changes in that specific factor is indicated by a comparatively flat line. These important variables make excellent choices for the contour plots' axes. Factors B and C have a high slope or curve, according to plotting, suggesting that they are more sensitive and have a bigger impact on the $E_R$'s outcome. It is noteworthy that component B has a linear trend, indicating a constant value at the midway point.

Additionally, it is noted that while increasing factors B and C raises $E_R$ up to a point within the experimental value range, there is a threshold beyond which $E_R$ begins to decline with additional increases in both factors.

**3.10. Optimization and validation.** To achieve optimal lead removal, a multi-objective numerical optimization technique based on the desirability function was employed, setting the goal to maximize lead removal while specifying pH, contact time, and extractant concentration ranges [36]. Utilizing response surface methodology, the optimal conditions for lead removal efficiency in contaminated sludge were determined. The optimization analysis indicated that at a pH of 5.0, an extractant concentration of 20 g/L, and a contact period of 135 minutes, the maximum removal
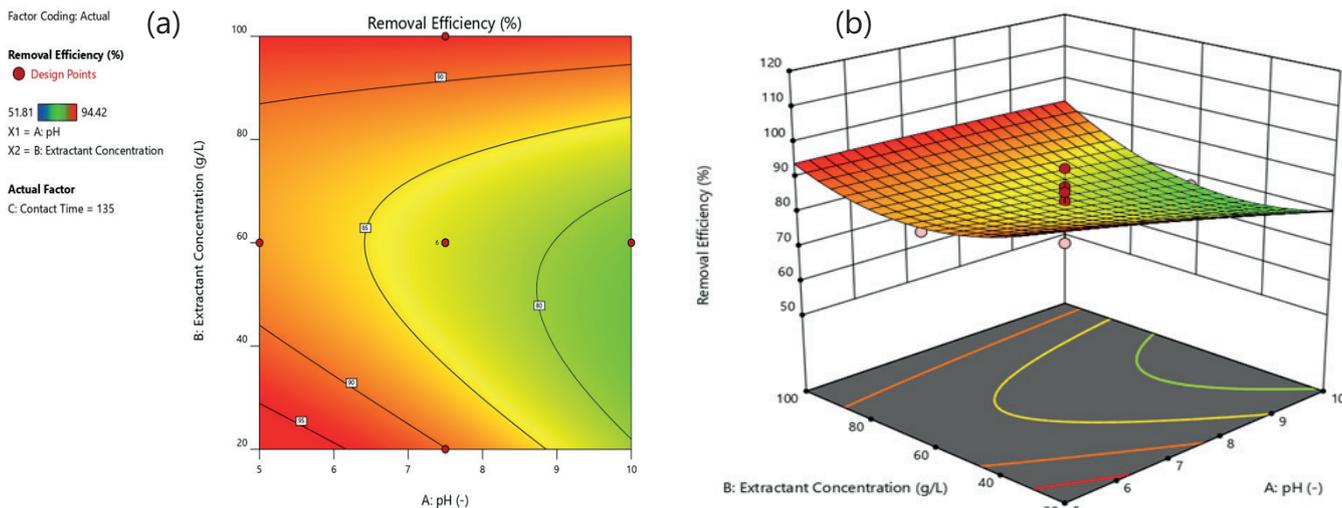
**Figure 5**. 3-D surface and contour plots of $E_R$ with extractant concentration and pH. (a) contour map and (b) surface plot
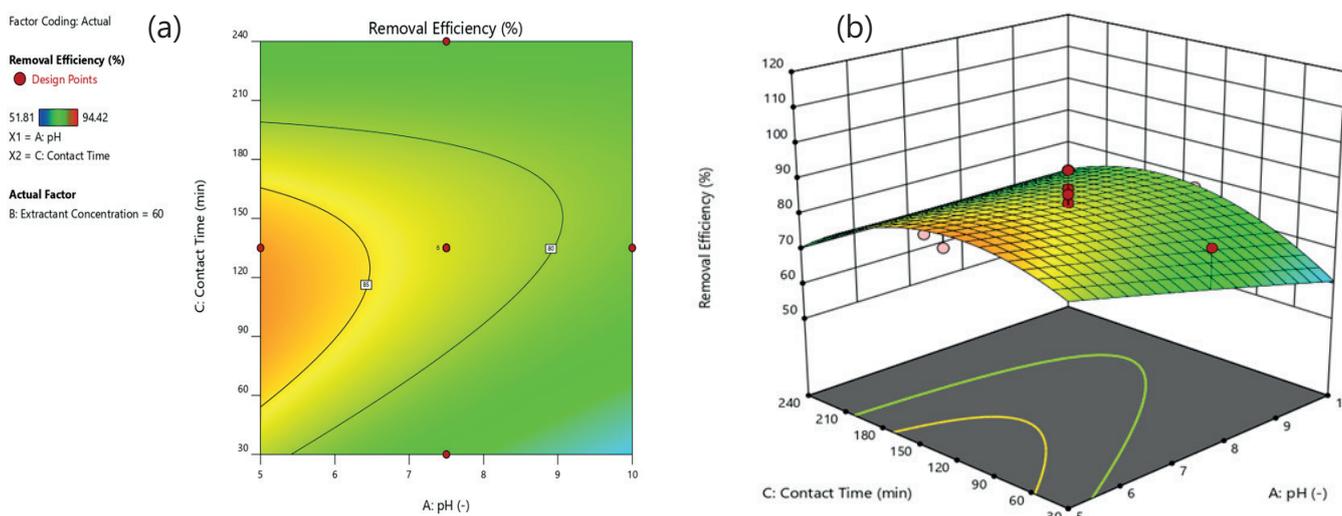


**Figure 6**. 3-D surface and contour plots of $E_R$ with contact time and pH. (a) contour map and (b) surface plot

efficiency $(E_R)$ achieved was 94.42%. Subsequently, a validation experiment was conducted after setting the amendment factors to the ideal values determined using CCD and RSM. Under the optimized conditions, a lead removal efficiency of 94.59% was attained. The calculated percentage error of 0.03 between the anticipated and actual values signifies negligible variation, consistent with the findings of [34, 37]. This suggests that the constructed model is legitimate and suitable for predicting response variables.

**3.11. Control experiment with distilled water treatment.** The control experiment using distilled water as the treatment medium aimed to provide a baseline for assessing the specific contributions of the lemon peel extract treatment on the sludge. The outcomes of the control experiment, in which the lead removal efficiency with distilled water was measured at 20.40%, are examined below in relation to the conclusions of the primary exper-

iment. The control experiment, employing distilled water as the treatment medium, exhibited a lead removal efficiency of 20.40%, serving as the baseline for evaluating the effects of lemon peel extract treatment.

In contrast, the main experiment conducted under optimal conditions yielded a maximum lead removal efficiency of 90.50% (pH = 5.0, contact time = 30 min, and extractant concentration = 100 g/L). This substantial improvement underscores the positive impact of the lemon peel extract treatment compared to distilled water.

Furthermore, under the optimized conditions, lead removal efficiency reached 94.59%, as detailed in the confirmation report. The negligible percentage error between predicted and actual values (0.03) supports the validity of the developed model for predicting response variables, consistent with the findings of [34].

## 4. CONCLUSION

The study characterized the waste lemon peel extract and found a pH of 5.2, indicating slight acidity, which is conducive to heavy metal extraction. The presence of NPK nutrients in the peel suggests its potential to enhance soil nutrient availability. The FTIR analysis identified various functional groups in the lemon peel extract, including hydroxyl, carboxyl, ether, and amide groups. These groups are known to bond with heavy metal ions, thereby aiding in lead extraction. The presence of these groups, along with the ability of polysaccharides to engage in ion exchange due to their polar nature, was confirmed, indicating that lemon peel can effectively interact with lead ions in the sludge. The study successfully demonstrated the effectiveness of waste lemon peel extract in removing lead from water works sludge, achieving a maximum removal efficiency of 90.50% under specific conditions: pH 5.0, contact time of 30 minutes, and extractant concentration of 100 g/L. Using Response Surface Methodology (RSM), the optimal conditions for lead removal were identified as pH 5.0, an extractant concentration of 20 g/L, and a contact period of 135 minutes, leading to a maximum removal efficiency of 94.42%. The validation experiment showed a very close actual removal efficiency of 94.59%, confirming the accuracy of the model predictions. The lemon peel extract significantly altered the sludge properties, enhancing its ability to remove lead. The pH of the sludge, extractant concentration, and contact time were found to be crucial factors influencing the efficiency of lead removal. The control experiment with distilled water yielded a much lower removal efficiency (20.40%), emphasizing the effectiveness of lemon peel extract. Post-treatment, the lead concentration in the sludge was reduced by 94.38%, confirming the treatment's substantial impact. The study not only demonstrates the efficacy of waste lemon peel in removing lead from contaminated sludge but also highlights the broader implications for sustainable waste management and environmental protection. By repurposing an agricultural byproduct, the research offers a cost-effective and eco-friendly alternative to conventional heavy metal remediation techniques. The findings support the viability of integrating waste materials into environmental remediation processes, promoting resource efficiency and reducing the environmental burden of waste disposal. Moreover, the high lead removal efficiency achieved under optimal conditions suggests that this method could be scaled for practical applications in wastewater treatment and soil remediation. Future research could focus on exploring the applicability of this method to other heavy metals and pollutants, as well as assessing the long-term effects of using treated sludge in agricultural or environmental contexts. This approach aligns with global efforts toward achieving a circular economy, where waste is minimized, and resources are continuously cycled back into productive use.

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## ■ CREDIT AUTHOR STATEMENT

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## ■ DECLARATIONS

**Conflict of interest** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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