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# Optimization of Lead Ion Removal Using Sugarcane Bagasse as an Adsorbent

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This study examined the use of sugarcane bagasse-based adsorbents as an effective way to remove lead (Pb) ions from contaminated water. Response surface methodology (RSM) was used to investigate the effect of contact time, adsorbent dosage, and Pb ion concentration on the percentage of lead removal. The optimum conditions for contact time, adsorbent dosage, and Pb ion concentration were determined to be 4.5 h, 8 g, and 100 ppm, respectively, with a percentage removal of 99.2%. Results from this study suggest that bagasse-based adsorbents can be utilized to effectively remove Pb from contaminated water. However, further studies should be conducted with actual contaminated water from different sources to confirm its efficacy.

**Keywords:** Optimization, Central composite design, Sugarcane bagasse, Adsorption

## INTRODUCTION

Water contamination is a global problem that is caused by dyes, bacteria, cloudiness, oil and its derivatives, and heavy metals from industrial and agricultural waste [1]. Lead is the most common of all heavy metals, and it is particularly pernicious in the environment as it persists indefinitely and poses a significant threat to public health [2]. Lead poisoning can have serious health effects on both children and adults, including lower intelligence, concentration difficulties, and behavioral changes in kids, as well as axonal degeneration, cerebral edema, infertility, menstrual disorders, miscarriages, and stillbirths in adults [3]. Exposure to lead ions for an extended period of time can cause damage to the kidneys, lungs, and liver, as well as increase the risk of cancer [4,5].

Adsorption is an effective method for purifying water and wastewater prior to disposal. Coal-based activated carbon is the most commonly used adsorbent for wastewater treatment, but it is quite costly, particularly for small-scale applications [6]. The research focus is shifting towards the use of cheaper, more sustainable adsorbents which are sourced from local materials and waste, and are better for the environment [7,8]. Agricultural wastes, including bagasse, banana and orange peels, rice and coffee husks, and maize cobs, can be put to effective use in cleaning wastewater before disposal and in water treatment plants, thereby reducing the problem of disposal [9].

Chlorination, reverse osmosis, electrochemical methods, chemical precipitation, biological processes, ion exchange, floatation, and membrane processes are all methods used to clean water for human consumption. These processes help to eradicate pathogens and remove heavy metals from the water [10].

Nevertheless, certain techniques result in the formation of toxic sludge that presents difficulties when it comes to disposal and are also relatively costly [11]. The necessity of further research into cost-effective methods or technologies for water treatment is clear. Adsorption is one possible option

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for eliminating heavy metal ions from water [12]. As the coal sources used to produce granular activated carbon for water treatment become depleted and expensive, alternative adsorbents have become necessary for small-scale industries and point-of-use water applications. With the need for a cost-effective and renewable resource, researchers have been searching for other suitable absorbents to replace granular activated carbon [13]. Attempts have been undertaken to create cost-effective adsorbents by utilizing various agro-industrial and municipal wastes. Sugarcane bagasse is one of the agricultural waste materials that has demonstrated the capacity to absorb heavy metals from polluted water [14]. The surplus of bagasse, however, is not biodegradable, which could create disposal issues for mill owners. As a result, bagasse can be used to create adsorbents for water remediation, providing a useful solution to this problem [3]. The goal of this study is to investigate the removal of lead ions from aqueous matrices under different contact times, initial metal ion concentrations, and adsorbent dosages.

## METHODS/EXPERIMENT

### Preparation of Adsorbate Solution

Three stock solutions of lead chloride were prepared by dissolving 0.453 g, 0.745 g, and 1.025 g of the compound in distilled water in a volumetric flask. This yielded a lead solution with concentrations of 50 ppm, 100 ppm, and 150 ppm, respectively.

### Preparation of Adsorbent Material

Sugarcane bagasse from a custom market in Maiduguri, Nigeria was collected and subjected to a rigorous treatment process to obtain the raw sugarcane bagasse adsorbent. This included multiple washings with tap water to remove ligneous and trapped impurities, followed by drying under the Sun for 24 h. Finally, the bagasse was powdered and sieved to obtain the desired adsorbent. Sugarcane bagasse is a readily available natural waste in the region and thus presents a suitable adsorption material for potential wastewater treatment applications.

### Experimentations

A series of 250 ml conical flasks containing the adsorbate solution of predetermined concentrations (8, 5, 2, 2, and 8 g adsorbent doses and 8, 4.5, 1, 8, and 1 h contact time for 100 ppm, and 5, 5, 8 and 2 g with 1, 8, 4.5 and 4.5 h contact time for 150 ppm) were prepared to carry out the adsorption process. For each of the predetermined concentrations, the adsorbent dose was weighed and added with the corresponding contact time as specified.

The experiment was conducted to investigate the impact of contact time, adsorbent mass, and initial metal ion concentration on the adsorption of Pb ions. After the adsorption process, the samples were filtered and the filtrate was subjected to atomic absorption spectroscopy (AAS) for metal ion concentration analysis.

### Instrumentation

The AAS used in the study is the PerkinElmer analyst 600 atomic absorption spectrometer model from a multi-user research laboratory, Ahmadu Bello University Zaria. It is a highly sensitive instrument for the detection of trace levels of metals and metalloids. It uses high-temperature light sources, optical filters, and monochromators to provide rapid, accurate, and reliable analysis of trace metals.

These are some of the analytical performances of the PerkinElmer analyst 600 atomic absorption spectrometer model used in this study:

i. Linear range: 0-3 absorbance units
ii. Correlation coefficient: > 0.99
iii. Limit of detection (LOD): 0.001 ppm
iv. Limit of quantification (LOQ): 0.002 ppm
v. Relative standard deviation (RSD): < 2%

### Model Evaluation

The analysis of variance (ANOVA) was used to confirm the model fitness, with its results presented in Table 2. The R (%) response regression was statistically significant, with an F-value of 19.93 and a p-value of 0.0023. The p-value for the quadratic model was also less than 0.0023, indicating its significance at a 95% confidence interval. This showed that the model adequately explained the relationship between the variables and response. Figures 1 to 6 illustrate the relationship between the actual and predicted values of the R (%) response. The actual values were the original measurements of the percentage removal % calculated from the experimental data

$$\%\text{Removed} = A - X/A \times 100$$

Where A = Total amount of concentration of a metal solution prepared.

X = Total amount of concentration of metal ion absorbed by the adsorbent.

And the predicted values were generated by model design. The findings suggest that sugarcane bagasse can be used as raw material for synthesizing adsorbent materials for the removal of lead from contaminated water.

## RESULTS

The results of the optimization of sugarcane bagasse for the removal of lead ions from a simulated contaminated solution are presented in Table 1 below. The table highlights the varying conditions or factors used in the optimization process, while the result of the metal (Pb) removed in terms of percentage was presented in a column at the end of the table.

To optimize the variables, response surface methodology (RSM) with central composite design (CCD) was employed and 13 runs of experiments were conducted. A quadratic model was constructed and the multiple regression analysis was performed. The model was used to predict the

**Table 1.** The Results of Factorial Experimental Design for Lead Removal Using Bagasse as an Adsorbent

| Std | Run | Factor 1 A:Adsorbent dose g | Factor 2 B:Time hrs | Factor 3 C:Concentration ppm | Response 1 percentage removal % |
|---|---|---|---|---|---|
| 11 | 1 | 5 | 1 | 100 | 97.7 |
| 4 | 2 | 8 | 8 | 50 | 95.8 |
| 10 | 3 | 5 | 8 | 150 | 98.7 |
| 13 | 4 | 5 | 4.5 | 50 | 97.8 |
| 12 | 5 | 5 | 8 | 100 | 97.7 |
| 5 | 6 | 2 | 4.5 | 150 | 98.5 |
| 1 | 7 | 2 | 1 | 50 | 95.8 |
| 3 | 8 | 2 | 8 | 50 | 95.9 |
| 6 | 9 | 8 | 4.5 | 150 | 99 |
| 8 | 10 | 8 | 4.5 | 100 | 99.2 |
| 9 | 11 | 5 | 1 | 150 | 98.4 |
| 7 | 12 | 2 | 4.5 | 100 | 97.8 |
| 2 | 13 | 8 | 1 | 50 | 98 |

**Table 2.** ANOVA for Reduced Quadratic Model

| Source | Sum of squares | Df | Mean square | F-value | p-value |
|---|---|---|---|---|---|
| Model | 16.06 | 7 | 2.29 | 19.93 | 0.0023 |
| A-Adsorbent dose | 2.00 | 1 | 2.00 | 17.38 | 0.0088 |
| B-Time | 0.1364 | 1 | 0.1364 | 1.18 | 0.3261 |
| C-Concentration | 4.26 | 1 | 4.26 | 36.98 | 0.0017 |
| AB | 1.32 | 1 | 1.32 | 11.49 | 0.0195 |
| BC | 0.6914 | 1 | 0.6914 | 6.01 | 0.0579 |
| $A^2$ | 0.6466 | 1 | 0.6466 | 5.62 | 0.0640 |
| $B^2$ | 2.50 | 1 | 2.50 | 21.69 | 0.0055 |
| Residual | 0.5755 | 5 | 0.1151 | | |
| Cor total | 16.64 | 12 | | | |

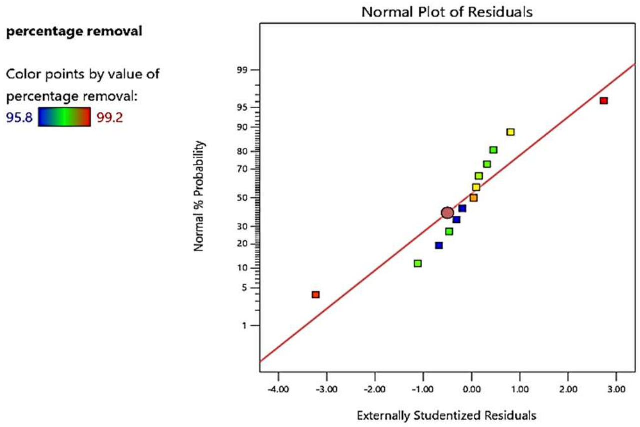


**Fig. 1.** A plot of externally student zed residuals *versus* predicted values of percentage removal (%).

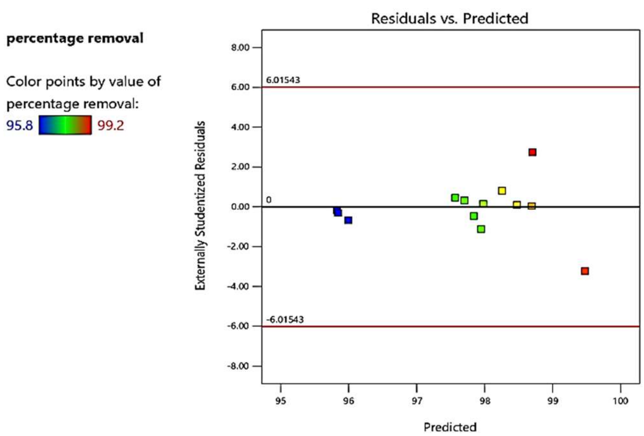


**Fig. 2.** A plot of residuals *versus* predicted values of percentage removal (%).

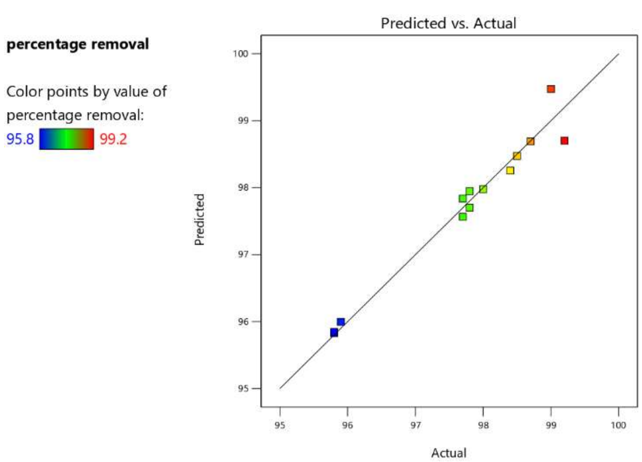


**Fig. 3.** A plot of predicted *versus* actual values of percentage removal (%).

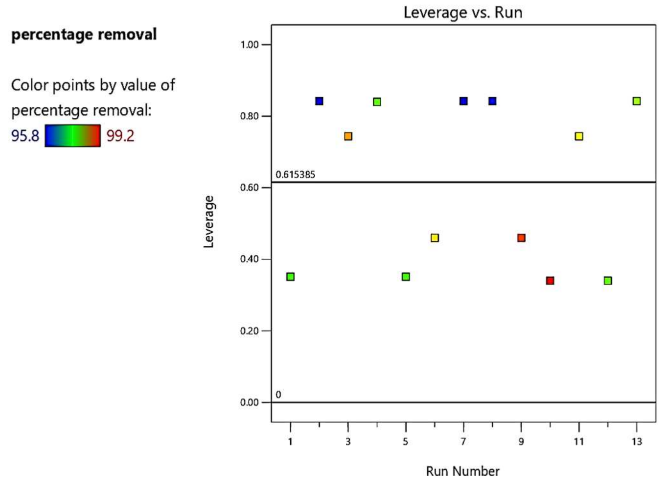


**Fig. 4.** A plot leverage *versus* run number values of percentage removal%.

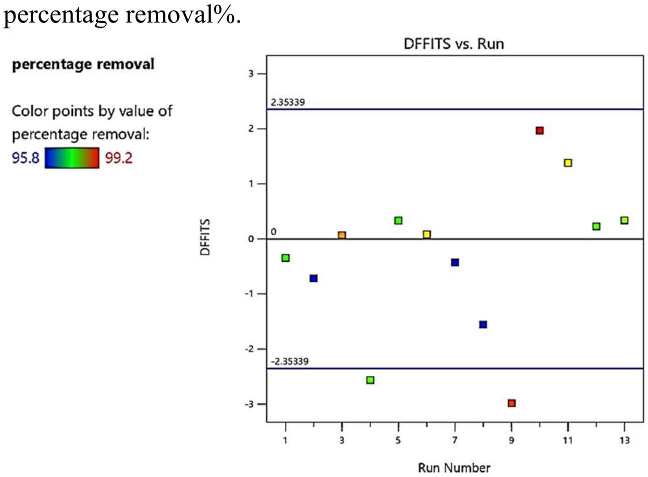


**Fig. 5.** A plot of DFFITS *versus* run number values of percentage removal (%).

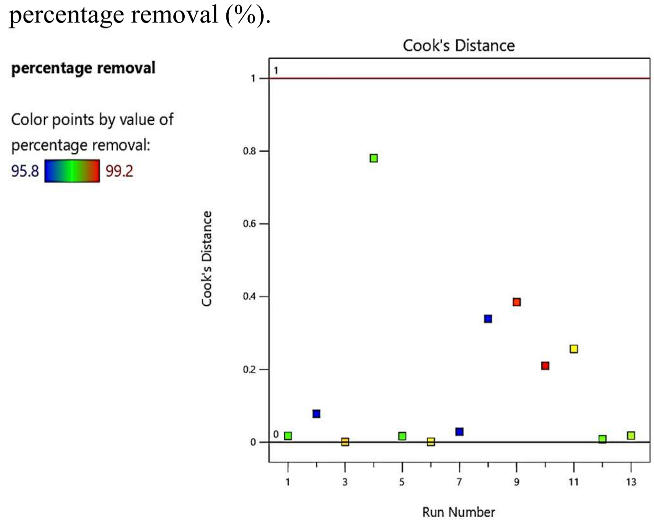


**Fig. 6.** A plot of cook's distance *versus* run number of percentage removal (%).

(percentage removal) response of the test variables.

The F-value of 19.93 and p-value of 0.0023 indicated that the model was significant and a good predictor of the yield. The predicted $R^2$ of 0.8116 was in line with the adjusted $R^2$ of 0.9170 (Table 3), with a difference of less than 0.2 confirming the accuracy of the model. The Adeq precision ratio of 13.699 was greater than 4, which demonstrated an adequate signal and the model could be utilized to explore the design space.

The coefficient estimate in an orthogonal design indicates how much the response value is expected to change when a factor is adjusted while all other factors remain constant. The intercept, which is the average response from all the runs, is then adjusted by the coefficients (Table 4). If the factors are orthogonal, then the variance inflation factors (VIFs) will be equal to 1; if the VIFs are higher than 1, it implies that there is a multi-collinearity issue between the factors, and the higher the VIFs, the more severe the correlation. Generally, VIFs values less than 10 are acceptable.

Model for percentage removal is given as:

$$\begin{aligned}\%Removal = {} & 0.989\ (\text{Adsorbent dose}) + 0.780\ (\text{Time}) \\ & + 0.006\ (\text{Concentration}) \\ & - 0.055\ (\text{Adsorbent dose} * \text{Time}) \\ & + 0.002\ (\text{Time} * \text{Concentration}) \\ & - 0.058\ (\text{Adsorbent dose}^2) \\ & - 0.083\ (\text{Time}^2) + 93.075\end{aligned}$$

The equation can be used to predict the response for given levels of each factor in their original units. However, the coefficients in the equation are not indicative of the relative importance of each factor, as they are scaled to fit the units of each factor and the intercept is not located at the center of the design space.

**Table 3.** Fit Statistics

| | |
|---|---|
| $R^2$ | 0.9654 |
| Adjusted $R^2$ | 0.9170 |
| Predicted $R^2$ | 0.8116 |
| Adeq precision | 13.6994 |

The figure presented in Fig. 7 illustrates 2-D plots depicting the percentage of heavy metal removal (%) in relation to the various process variables, including adsorbent dose, time, and concentration. The results indicate a consistent rise in the percentage of heavy metal removal as the adsorbent dose and concentration of the metal ion in the solution are increased. However, it is worth noting that with respect to time, there appears to be an optimal duration required for the effective removal of metals. Beyond this optimum time, there is a risk of metal ions being desorbed back into the solution by the adsorbent.

## DISCUSSION

Tables 2 and 5 of the study presented the levels and ranges of the studied process parameters (A- adsorbent dosage, B- time, C- concentration) and the observed and predicted % of Pb removal by Bagasse, respectively. The results were then analyzed using Minitab 18 to determine the main effect and interaction of different factors.

The analysis of variance (ANOVA) was used to determine the interacting factors affecting the removal of Pb

**Table 4.** Coefficients in Terms of Coded Factors

| Factor | Coefficient estimate | df | Standard error | 95% CI Low | 95% CI High | VIF |
|---|---|---|---|---|---|---|
| Intercept | 98.72 | 1 | 0.240 | 98.10 | 99.34 | |
| A-Adsorbent dose | 0.500 | 1 | 0.120 | 0.192 | 0.808 | 1.00 |
| B-Time | -0.136 | 1 | 0.125 | -0.458 | 0.186 | 1.09 |
| C-Concentration | 0.772 | 1 | 0.127 | 0.446 | 1.10 | 1.25 |
| AB | -0.575 | 1 | 0.170 | -1.01 | -0.139 | 1.00 |
| BC | 0.354 | 1 | 0.145 | -0.017 | 0.726 | 1.09 |
| $A^2$ | -0.518 | 1 | 0.219 | -1.08 | 0.044 | 1.28 |
| $B^2$ | -1.02 | 1 | 0.219 | -1.58 | -0.456 | 1.28 |

**Table 5.** Detailed Results for the Predicted, Residual, Leverage, and Outlier Statistical Data Values

| Run order | Actual value | Predicted value | Residual | Leverage | Internally studentized residuals | Externally studentized residuals | Cook's distance | Influence on fitted value DFFITS | Standard order |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 97.70 | 97.84 | -0.1384 | 0.351 | -0.506 | -0.465 | 0.017 | -0.342 | 11 |
| 2 | 95.80 | 95.85 | -0.0461 | 0.842 | -0.342 | -0.310 | 0.078 | -0.715 | 4 |
| 3 | 98.70 | 98.69 | 0.0078 | 0.744 | 0.046 | 0.041 | 0.001 | 0.069 | 10 |
| 4 | 97.80 | 97.95 | -0.1480 | 0.840 | -1.091 | -1.117 | 0.780 | -2.560[(1)] | 13 |
| 5 | 97.70 | 97.57 | 0.1344 | 0.351 | 0.492 | 0.451 | 0.016 | 0.332 | 12 |
| 6 | 98.50 | 98.47 | 0.0260 | 0.460 | 0.104 | 0.093 | 0.001 | 0.086 | 5 |
| 7 | 95.80 | 95.83 | -0.0279 | 0.842 | -0.207 | -0.186 | 0.029 | -0.430 | 1 |
| 8 | 95.90 | 96.00 | -0.0961 | 0.842 | -0.713 | -0.673 | 0.339 | -1.555 | 3 |
| 9 | 99.00 | 99.47 | -0.4740 | 0.460 | -1.901 | -3.231 | 0.385 | -2.982[(1)] | 6 |
| 10 | 99.20 | 98.70 | 0.4980 | 0.340 | 1.807 | 2.743 | 0.210 | 1.969 | 8 |
| 11 | 98.40 | 98.26 | 0.1442 | 0.744 | 0.840 | 0.811 | 0.256 | 1.383 | 9 |
| 12 | 97.80 | 97.70 | 0.0980 | 0.340 | 0.356 | 0.322 | 0.008 | 0.231 | 7 |
| 13 | 98.00 | 97.98 | 0.0221 | 0.842 | 0.164 | 0.147 | 0.018 | 0.340 | 2 |

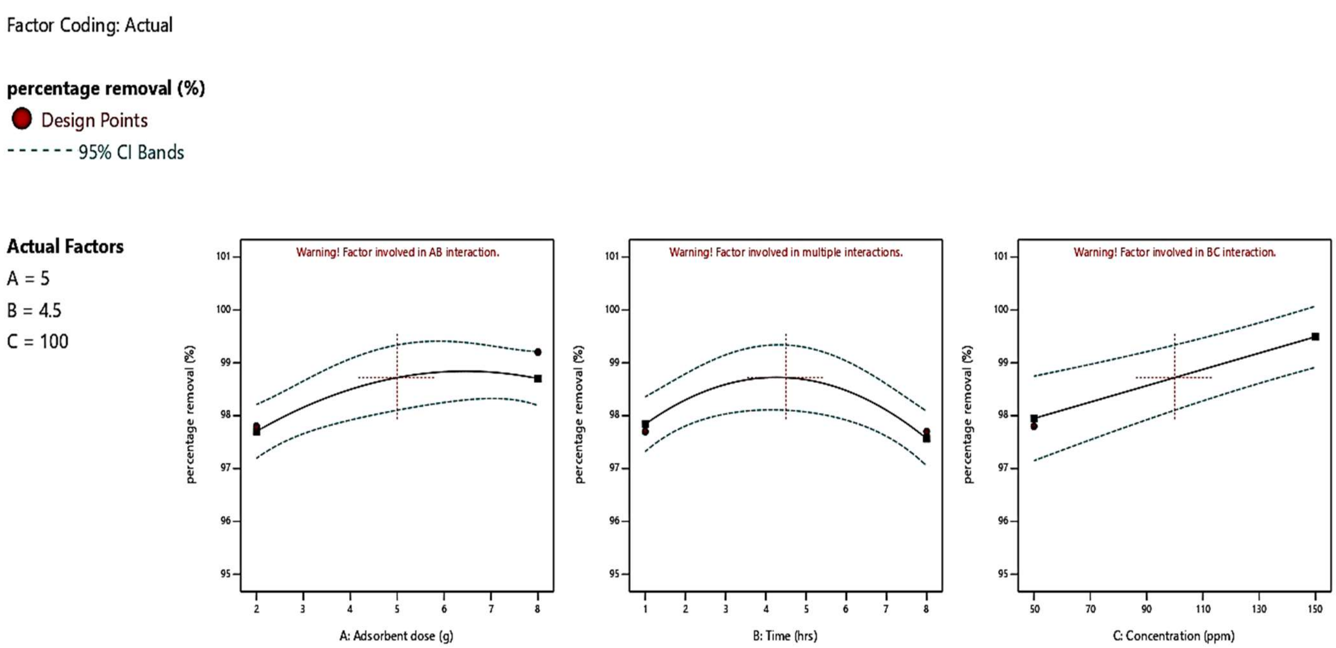


**Fig. 7.** 2-D plots of percentage removed (%) against the process variables.

after estimating the main effect. The sum squares (SS) of each factor were used to evaluate its importance, with higher values indicating greater significance. The main and interaction effects of each factor with a p-value less than 0.0023 were considered to be potentially significant (Table 2).

## Effect of Process Variables

**Effect of contact time.** Figure 8 illustrates the 3D surfaced response of Pb ions uptake by the selected adsorbents, as the contact time was varied from 1 to 8 h in separate experimental runs. The amount of ion absorbed into the adsorbent was seen to increase with increasing contact time, until reaching a point of dynamic equilibrium, where the amount of ion desorbing from the adsorbent is equal to the amount being absorbed. Beyond this point, no more ions were removed from the solution. The 3D plots demonstrate that the percentage removal of 97.8-99.2% is attainable when the concentration is increased from 50-150 ppm. This is further evidenced by Fig. 8, which reveals that the percentage removal increases significantly by 1.4% in the span of 1-8 hours, indicating the high efficiency of the adsorbent.

**Effect of adsorbent dosage.** The interaction between Adsorbent dosage and Pb concentration on the R (%) response was investigated through the use of 3D surface plots, which are shown in Fig. 9. The results demonstrated that increasing the Adsorbent dosage resulted in an increase in the Percentage removal %, from 97.8 to 99.2%. This increase is likely due to the increased number of Adsorbent sites available for Pb binding when the dosage is increased, thus providing more opportunities for Pb to attach to the Adsorbent surface.

**Effect of Pb ion concentration.** Figure 10 illustrates 3D surface plots depicting the interaction between Pb ion concentration and Time (h) on the R (%) response. Increasing the Pb ion concentration from 50 to 150 ppm caused a decrease in percentage removal from 99.2 to 97.8%. This is likely at lower metal ion concentrations, the ratio of available adsorbent surface to the total metal ions in the solution is higher, allowing for all metal ions to interact with the adsorbent and be removed from the solution, thereby increasing the removal efficiency. At high metal ion concentrations, the ratio of adsorbent surface-active sites to the total metal ions in the solution is low, indicating that not all metal ions can interact with the adsorbent and be removed from the solution, resulting in a decrease in the percentage removal%.

## CONCLUSION

It was found that Sugarcane Bagasse has the potential to

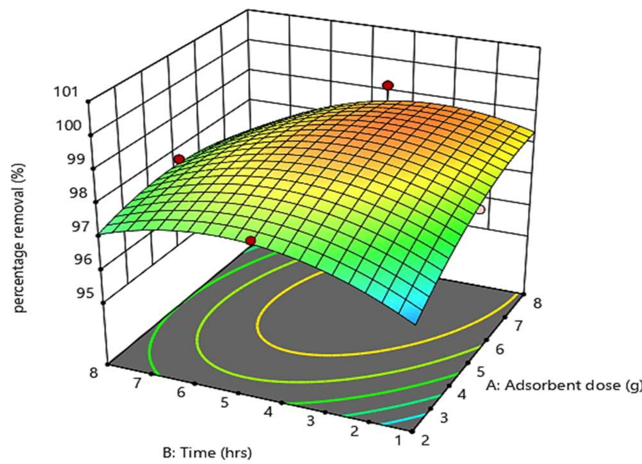


**Fig. 8.** 3D surface plots of percentage removed (%) response adsorbent dose (g) by time (h).

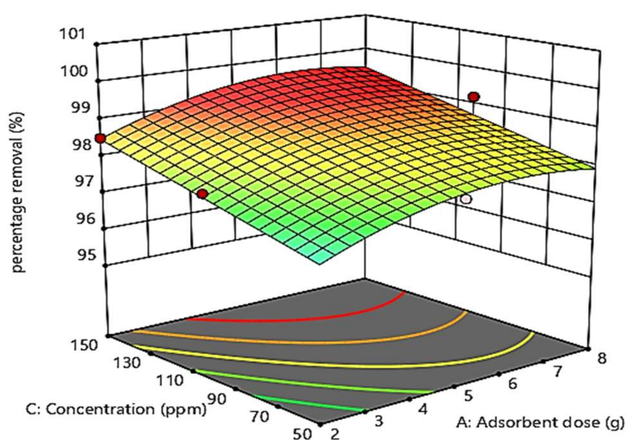


**Fig. 9.** 3D surface plots of percentage removed (%) response concentration (ppm) by adsorbent dose.

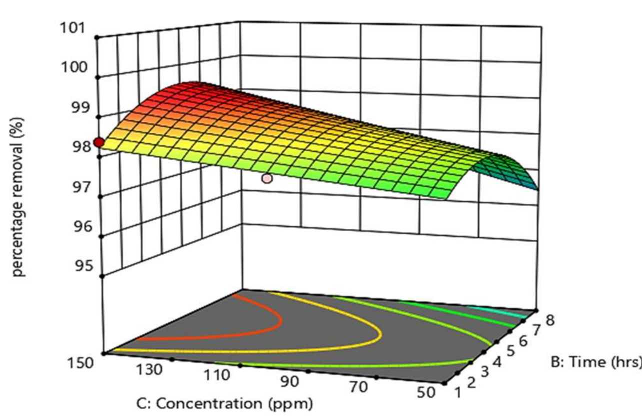


**Fig. 10.** 3d surface plots of percentage removed (%) response for concentration (ppm) by time (h).

effectively remove Lead from contaminated water, with the highest percentage of removal of 99.2% observed at an initial metal ion concentration of 100 ppm. This result was found to be statistically significant at a 95% confidence level, with a p-value of less than 0.0023. Therefore, the hypothesis that bagasse adsorbents are effective in removing Pb from contaminated water at different experimental conditions is upheld. The dosage of 8 g and contact time of 4.5 h was seen to be the most effective in achieving the highest percentage of removal.

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