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Optimized Zinc Uptake from the Aquatic Environment Using Biomass Derived from *Lantana Camara* L. Stem

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ABSTRACT

Biomass extracted from different plant parts can play a role as a cheap, efficient and eco-friendly adsorbent. In this research, *Lantana Camara* L. Stem biomass (LSB), a low-cost and useless material, was introduced as efficient biomass for divalent zinc biosorption from aqueous environments. For achieving optimal conditions in the zinc biosorption process, the experimental design was applied by the response surface methodology (RSM) based on a *Box-Behnken* design (BBD) model. Based on the comparison between the measured and predicted amounts, the values of R^2, R^2_{adj}, and R^2_{pred} in the Zn(II) biosorption model were 0.9960, 0.9887 and 0.9441. The Zn(II) uptake in the experiments, BBD model-based (p-value of *Lack-of-Fit* term = 0.228 > 0.05), varied from 15.19% to 81.11%. The maximum analyte uptake at a LSB-to-Zn(II) ratio of 8:1, synthetic solution pH of 6.5 and residence time of 75 min was predicted at 97.12%. The maximum Z.R.% based on the validation test performed based on the optimal predicted conditions was also obtained at 94.65%, which is 2.5% different from the model's data amount, confirming the acceptable accuracy of the quadratic model. The LSB, in optimized conditions, as a low-cost biosorbent, can be a suitable candidate with acceptable potential for heavy metals biosorption from aquatic solutions.

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INTRODUCTION

One of the challenges facing humanity is finding a practical and economical solution to remove various water and wastewater pollution, such as pesticides (Ehzari, Safari, et al., 2022; Mensah et al., 2021), dyes (Manjarrez Paba et al., 2021; Samimi & Safari, 2022), microplastics (Takarina et al., 2022), and other contaminates (Hidayah et al., 2022; Sivakumar et al., 2022; Torres-Bejarano et al., 2022). Heavy metals have a wide application (Chatterjee et al., 2021; Ehzari, Amiri, et al., 2022; Nuryadin & Imai, 2021; Samimi, Zakeri, et al., 2023), but if they enter more than the allowed amount in the human body, these materials cause many diseases for humans and organisms, such as disturbances in the immune system, digestive system, central and peripheral nerves and urinary tracts; therefore, one of the major pollutants of the environment is industrial wastewater containing heavy metals (Alprol et al., 2023; Astuti et al., 2021; Feijoo et al., 2021; Labidi & Mechati, 2023; Marefat et al., 2023; Nimesha et al., 2022).

Divalent zinc (Zn(II)) is one of the heavy metals found in the effluents of automotive, electrical appliances, paint, ship-building, textile, papermaking and casting industries (Kumar et al., 2023; Zwain et al., 2014). This heavy metal at low concentration, with antifungal, antimicrobial, anti-inflammatory, and antioxidant properties (Abendrot et al., 2020), is considered one of

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the essential elements for the body due to its role in insulin production and enzyme catalysis (Molnar-Nagy et al., 2022). However, its large amounts cause side effects such as heartache, skin irritation, vomiting, nausea and anemia (Tang et al., 2022).

Among the various physical, chemical and biological methods to remove pollutants, using biomass and biochar emanating from plant parts is one of the biocompatible and economical methods (Manatura & Samaksaman, 2021; Piri & Sepehr, 2022; Samimi & Shahriari Moghadam, 2020). In recent decades, the biomass of microorganisms has been used as cheap and renewable adsorbents to remove heavy metals (Sahli & Belhiouani, 2022; Soltani-Gishini et al., 2022; Song et al., 2022). The cellular structure of these biosorbents, as well as the functional groups in their structure, play an important role in creating a link between the active sites of biosorbents and heavy metals (Gupta et al., 2021; Sud et al., 2008).

The *Lantana camara* L., as an ornamental plant, is readily found in most countries, such as the temperate regions of Iran. This plant until now has been applied for the uptake of Pb(II) (Alaribe & Agamuthu, 2015) and Cd(II) (Liu et al., 2019) from aquatic environments. Recently the biomass derived from the stem of *Lantana camara* L. has been used for the adsorptive removal of malachite green (Samimi & Shahriari-Moghadam, 2023). According to the background literature review, no research has been done on the stem biomass derived from this plant to remove heavy metals. In this study, the performance evaluation of *Lantana camara* L. stem biomass (LSB) in Zn(II) uptake from a synthetic aqueous solution, as well as biosorption kinetics were investigated. In addition, the selected factors that affect adsorbate removal were optimized by response surface methodology (RSM).

MATERIALS AND METHODS

Biosorption preparation and used devices

In this work, the biomass derived from the *Lantana camara* L. stem was prepared according to the procedure presented in previous study (Samimi & Shahriari-Moghadam, 2023). Briefly, the collected plant stem was washed, dried, pulverized and sieved by a vibrating sieve shaker (SD8-12, the United States) to achieve a sieved biosorbent with sizes less than 45 μm. Zinc ions measurement from Zn(II) and Zn(II)/LSB solutions was accomplished by a flame-type atomic absorption spectrometer (SavantAA, Australia). The morphology of LSB/Zn(II) was analyzed using Scanning Electron Microscopy (SEM).

Batch biosorption experiments for Zn^{+2} removal

To prepare synthetic wastewater containing Zn(II), a certain amount of zinc salt was dissolved in double distilled water, filtered (using paper filter) and stirred for 10 min. The different concentrations of solutions, based on experimental conditions, were obtained by diluting the stock solution (800 mg/L). The LSB biosorbent was added to various concentrations of zinc solution (50 to 200 ppm) at optimal pH and then stirred at different times based on the design of the experiments (DOE). After the LSB-Zn(II) solution centrifugation, the zinc biosorption was calculated by determining the residual zinc ions in the supernatant. Analyte removal efficiency and the LSB capacity in zinc biosorption were determined as follows:

$$\text{Zn}\left(\text{II}\right)\text{removal efficiency}\left(\%\right)=\left(C_0-C_e\right)\times 100/C_0 \tag{1}$$

Where, C_0 (mg/L) and C_e (mg/L) are the initial and final concentrations of Zn(II) in synthetic solutions.

Uptake optimization based on design of experiments

Experimental design was applied to reduce, optimize and estimate experimental conditions, variables interactions and the relative significance of parameters in biosorption processes

(Pournamdari, 2023; Tabatabaei et al., 2020). To study the experimental variables that affect Zn(II) uptake, factors and their levels were considered based on a *Box-Behnken* design (BBD) model. The Minitab software (V-18) was applied to RSM analysis to achieve desired conditions for the Zn(II) sorption. Before the selecting levels, pre-tests were performed to remove out of effective ranges. The effect of the LSB-to-Zn(II) ratio (LZ), the pH value of the synesthetic environment containing metal ions (pH), and residence time (t) were investigated in three coded levels which are presented in Table 1.

The experimental factors were studied in biosorbent-to-sorbate ratio of 2:1, 5:1, and 8:1, the pH value of 2.5, 4.5, and 6.5, and the residence times of 15, 40, and 75 min. Briefly, based on RSM-BBD, 13 tests with two duplicate tests (to determine the errors) were performed. The experimental responses which carried out based on DOE are summarized in Table 2. Analysis of variance (ANOVA) also was carried out for the statistical analysis of the mathematical model at a significance level of 5% (Samimi & Mansouri, 2023).

The relationship between the Zn(II) sorption and the coded response variables is illustrated by the following “full quadratic model” Equation (Mukhopadhyay et al., 2022; Oliver Paul Nayagam & Prasanna, 2023):

$$Z.R.\% = A_0 + \sum_{i=1}^{3} A_i X_i + \sum_{i=1}^{3} A_{ii} X_i^2 + \sum_{i=1}^{2}\sum_{j=i+1}^{3} A_{ij} X_{ij} \tag{2}$$

where;

$ZR\%$: Experimental response

A_0 : Offset term (intercept)

A_i : Linear term

A_{ii} : Quadratic term

A_{ij} : Interaction term

Table 1. The operational parameters with experimental levels

Variables	Symbol	Rang of levels		
		-1	0	+1
LSB-to-Zn(II) ratio	LZ	2:1	5:1	8:1
pH	pH	2.5	4.5	6.5
Residence time (min)	t	15	40	75

Table 2. experimental responses based on DOE

Run No.	Coded variables			Responses
	X_{LZ}	X_{pH}	X_t	Z. R. %
1	-1	0	-1	17.44
2	1	0	1	81.11
3	1	1	0	74.20
4	-1	-1	0	15.19
5	-1	0	1	35.00
6	0	1	1	68.57
7	0	1	-1	39.27
8	0	0	0	41.22
9	1	0	-1	43.70
10	0	-1	-1	19.34
11	0	0	0	43.10
12	0	0	0	40.60
13	-1	1	0	33.40
14	0	-1	1	38.20
15	1	-1	0	37.86

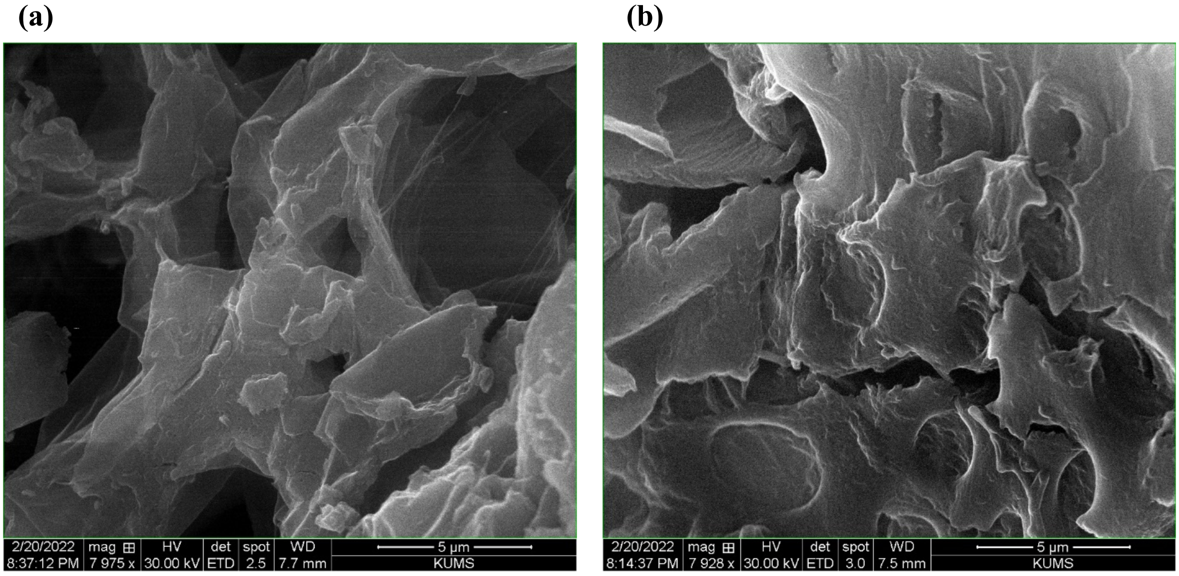

Fig. 1. SEM images of LSB with the resolutions of 5 µm **a)** before and **b)** after zinc biosorption.

X_i and X_{ij} : Independent variables

The F-value also was calculated as used in previous studies (Samimi & Shahriari-Moghadam, 2021) as follows:

$$\text{F} - \text{value} = \frac{\text{Mean Square Regression}\left(\text{MS}_{\text{Reg.}}\right)}{\text{Mean Square Residual}\left(\text{MS}_{\text{Res.}}\right)} \tag{3}$$

RESULTS AND DISCUSSION

Biosorbent characteristics

The surface morphology of the LSB before and after Zn(II) sorption was investigated using SEM images. As shown in Fig 1, the prepared biosorbent and sorbent/sorbate have a porous structure that can be considered suitable for adsorbing and trapping zinc ions. According to the FTIR spectroscopy in previous work (Samimi & Shahriari-Moghadam, 2023) on LSB, this biosorbent have active sites, such as –NH groups, and methyl group stretching vibration, as well as, C – C, C – O and C = O groups, which possibly affected metal uptake.

The BBD model & analysis of response variance

The surface Based on RSM and the BBD model, a regression equation in uncoded units was obtained for the Zn(II) removal, presented in Eq. 4:

$$\begin{aligned} Z.R.\ \% &= 41.64 + 16.980\,X_{LZ} + 13.106\,X_{pH} + 12.891\,X_t + 0.75\,X_{LZ}^2 - \\ &2.22\,X_{pH}^2 + 1.93\,X_t^2 + 4.53\,X_{LZ}.X_{pH} + 4.96\,X_{LZ}.X_t + 2.61\,X_{pH}.X_t \end{aligned} \tag{4}$$

The Zn(II) biosorption measured from the supernatant of a synthetic aqueous solution and the values predicted by the full quadratic model, fitted as a normal probability plot, are displayed in Fig. 2. The values of R^2, R^2_{adj}, and R^2_{pred} in the Zn(II) uptake model, based on the comparison between the measured and predicted amounts, were 0.9960, 0.9887 and 0.9441, respectively. Table 3 reported the ANOVA of the selected model for metal biosorption. As can be seen in

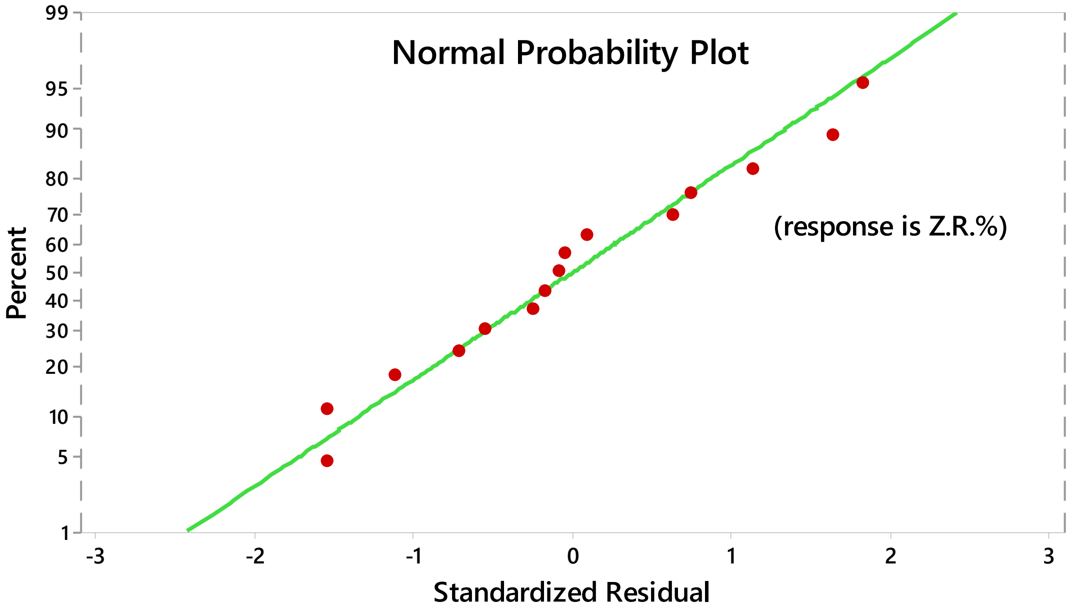

Fig. 2. Normplot of residuals for Zn(II) uptake

Table 3. The ANOVA of the quadratic model for Zn(II) biosorption

Source	DF	SS$_{adj}$	MS$_{adj}$	F-Value	P-Value	Degree of significance
Regression	9	5254.94	583.88	136.77	< 0.001	Significant
X_{LZ}	1	2306.56	2306.56	540.29	< 0.001	Significant
X_{pH}	1	1374.19	1374.19	321.89	< 0.001	Significant
X_t	1	1329.47	1329.47	311.42	< 0.001	Significant
X_{LZ}^2	1	2.05	2.05	0.48	0.519	Not significant
X_{pH}^2	1	18.24	18.24	4.27	0.094	Not significant
X_t^2	1	13.72	13.72	3.21	0.133	Not significant
$X_{LZ}.X_{pH}$	1	82.17	82.17	19.25	0.007	Significant
$X_{LZ}.X_t$	1	98.51	98.51	23.07	0.005	Significant
$X_{pH}.X_t$	1	27.25	27.25	6.38	0.053	Not significant
Residual error	5	21.35	4.27	-	-	-
Lack-of-Fit	3	17.96	5.99	3.53	0.228	Not significant
Pure Error	2	3.39	1.69	-	-	-
Total	14	5276.29	-	-	-	-

Table 3, the coded factors of X_{LZ}, X_{pH}, X_t, $X_{LZ}.X_{pH}$ and $X_{LZ}.X_t$ have significant regression conditions (p-value < 0.05). The p-value of the $X_{pH}.X_t$ term was 0.053, which is close to 0.05; therefore, there is a possibility that this 2-way interaction term is also significant. However, the square-coded terms were not significant.

The 2-way interaction plot of the coded various factors on the Zn(II) uptake is presented based on BBD in Fig. 3. Type of removal changes and the trend of increasing metal uptake in higher-level factors with an almost identical approach can be seen in all three cases (namely $X_{LZ}.X_{pH}$, $X_{LZ}.X_t$ and $X_{pH}.X_t$). Although, this increasing slope was less evident in the $X_{pH}.X_t$ term in accordance with its lack of fit value. Although this increasing slope was less evident in the $X_{pH}.X_t$ term, this behavior was in accordance with its p-value (0.053).

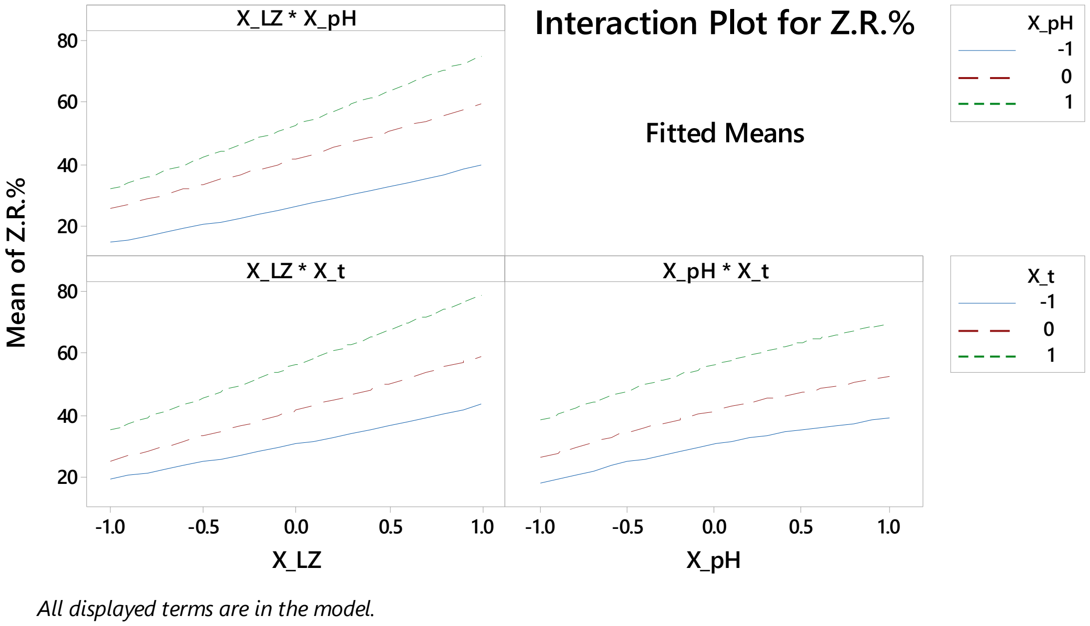

Fig. 3. The 2-way interaction plot of coded parameters on Z.R.%

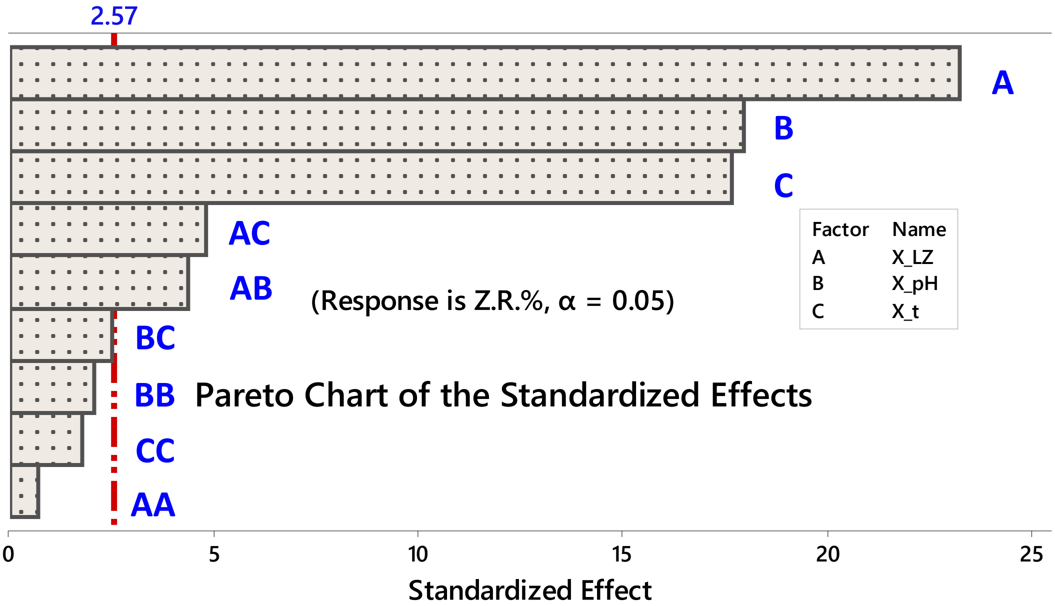

Fig. 4. The *Pareto* graph of the standardized effects for Z.R.%.

As indicated in the *Pareto* graphical analysis (Fig. 4), the terms A, B, C, AC, and AB (namely X_{LZ}, X_{pH}, X_t, $X_{LZ}.X_t$, and $X_{LZ}.X_{pH}$) have crossed the hypothetical area red limit, confirming the significance of these parameters as they are further away from this line (Samimi, Mohammadzadeh, et al., 2023). The closeness of the BC parameter ($X_{pH}.X_t$ term) to the hypothesized point boundary also confirms the significance probability of this interaction (Moghadam & Samimi, 2022). These results were in accordance with the data derived from the regression equation.

The contour plots of selected variables' effect on Z.R.% are shown in Fig. 5. According to the BBD experiments, the contour diagrams of Zn(II) uptake from synthetic aqueous solution were

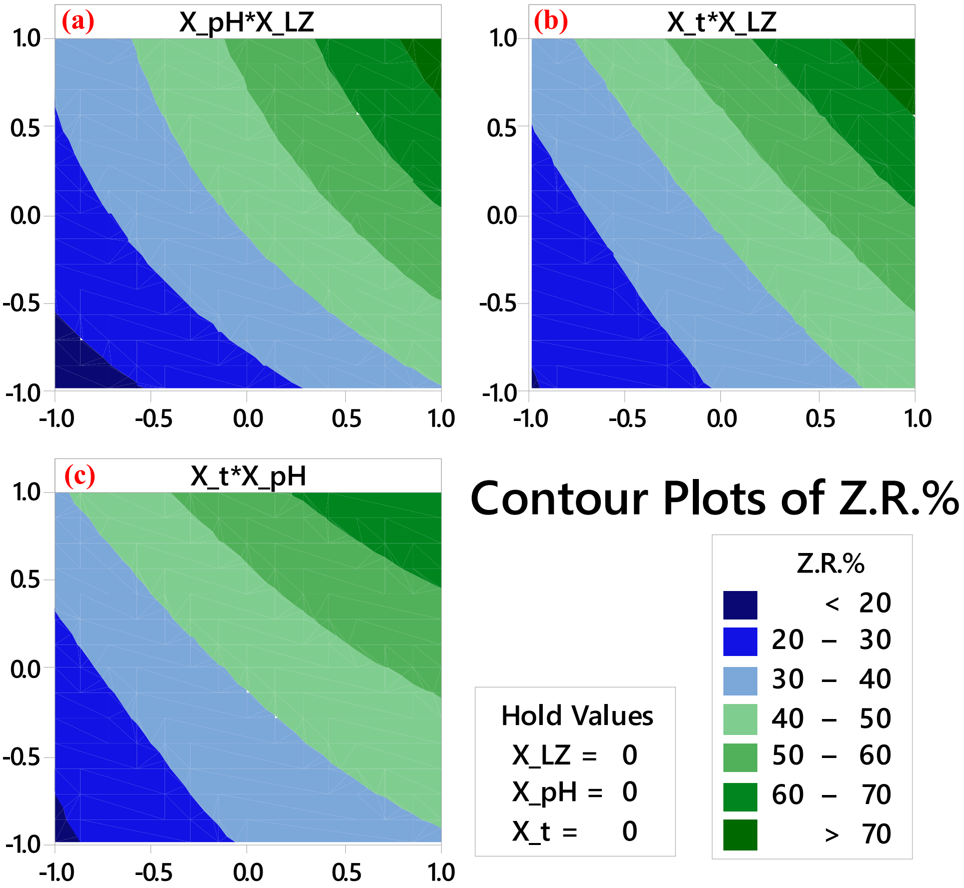

Fig. 5. The contour diagrams of operational variables' effect on Z.R.% for **a)** X_{pH} versus X_{LZ}; **b)** X_{t} versus X_{LZ}; and **c)** X_{t} versus X_{pH}.

Table 4. Multiple response prediction of variables for Z.R.%

Optimization	X_{LZ}	X_{pH}	X_t	Z.R.% Fit	Composite Desirability
Maximize	+1	+1	+1	97.12	1
Minimize	-1	-1	-1	11.22	1

sketched in average levels of X_t (Fig. 5a), X_{pH} (Fig. 5b), and X_{LZ} (Fig. 5c). As shown in Fig. 5a, the simultaneous increase in LSB-to-Zn(II) ratio and synthesis solution pH at the medium level of residence time led to an increase in Z.R.%. However, the biosorbent-to-metal ratio increase at low pH values did not significantly affect Z.R.%. Fig. 5b (plotted at pH = 4.5) shows an increasing trend in the simultaneous increase in residence time and LSB-to-Zn(II) ratio. As can be seen, this increasing trend at higher times has lower dependence on the biosorbent-to-metal ratio. Fig. 5c, plotted at an LSB-to-Zn(II) ratio of 5:1, shows an increasing trend in the simultaneous increase in residence time and solution pH value. Furthermore, at low levels of residence time or solution pH, the changes of other factors did not considerably affect Z.R.%.

Numerical optimization of the used model was done to evaluate the suitable conditions for obtaining the maximum/minimum percentage of Zn(II) uptake (by LSB biosorbent) from a synthetic solution. The optimization results, presented as multiple response prediction of variables for metal, are summarized in Table 4. As can be seen, the maximum Z.R.% was 97.12%, predicted at the LSB-to-Zn(II) ratio of 8:11, the solution pH of 6.5, and the residence time of 75 min. The extremum points of the function were achieved by calculating the derivative root of Eq. 4. Model's validity was confirmed according to high p-value of the *lack-of-fit* (Samimi & Shahriari Moghadam, 2018), presented in Table 3 (p-value = 0.228 > 0.05). The validation test also was performed based on the best conditions predicted in Table 4. The maximum Z.R.% based on the validation test was obtained at 94.65%. The difference between the predicted value and the practical test was about 2.5%, confirming acceptable accuracy of the selected model (Samimi & Moeini, 2020).

CONCLUSION

The advantage of replacing low-cost and biocompatible adsorbents with other current methods to remove pollution, such as heavy metals, is not hidden from anyone. In this regard, the current study was carried out to evaluate the LSB performance in removing zinc ions from a synthetic aqueous solution. The numerical optimization of the quadratic model was investigated to predict the optimal conditions for achieving the maximum amount of Zn(II) uptake by LSB biosorbent using RSM and BBD. Regression equation, *Pareto* graphical analysis, contour and 2-way interaction diagrams show that all used experimental parameters, such as LSB-to-Zn(II) ratio, synthetic solution pH and residence time, significantly affected Zn(II) uptake. The maximum Zn(II) biosorption at LSB-to-Zn(II) ratio of 8:1, pH of 6.5 and residence time of 75 min was predicted based on the BBD model at 97.12. This amount was then determined based on a validation test at 94.65%, confirming the validity and acceptable accuracy of the full quadratic model. This study suggested that LSB powder naturally can be introduced as a suitable candidate for heavy metal adsorption from aqueous environments.

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The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/ or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research

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