Response Surface Modeling of Arsenic Adsorption by Modified Spent Tea Leaves

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Abstract. Arsenic (V) is represented as a very toxic compound, hence the importance of this research is concerned to reduce AS (V) from contaminated water before emitting it into river water by using modified spent tea leaves MSTL. The effect of arsenic initial concentration (10-100) mg.L-1, the mass of MSTL (0.1-0.6) g.L-1, pH (2-10), and mixing time (10-110) min were studies to obtain a mathematical model which describes the arsenic adsorption on MSTL using response surface methodology. The higher adsorption capacity has been conducted at 40 ppm of initial concentration, 0.5 g.L-1 of MSTL, pH=4, and 70 min of mixing time. The maximum removal efficiency and adsorption capacity were 87% and 69.8 mg/g, respectively. The optimum experimental results were analyzed isothermally and they were found fitted in high accuracy with Langmuir isotherm models.

Keywords: adsorption, arsenic, RSM, removal, heavy metals.

1. Introduction
Globally, groundwater contamination by toxic arsenic is an environmental and health concern for its carcinogenic properties which threatens millions of people to rely on As-contaminated drinking water. Arsenic is one of the most harmful heavy metal pollutants since it is emitted to the environment by many industrial factories such as fertilizer, weathering, painting, metal ore, and battery factories[1]. It is existing in two inorganic state formulas either As (III) or As (V) and its severity depends on the quantity of effluent wastewater by anthropogenic activities that emitted to the lake and river as a result of factories actions [2]. The long time exposure to trace levels of arsenic concentrations leads to fatal health problems including cardiovascular, skin, and cancer diseases. Arsenic ion standard concentrations in rivers and lakes in the range of 10 μg.L-1 has been reported [3, 4].

In recent decades, many researchers reported different methods and techniques to remove and eliminate heavy metal ions from wastewater which can be achieved by chemical and biological treatments such as photocatalytic [5, 6], electrochemical [7], advance oxidation [8], adsorption [9] and chemical precipitation [10]. The cost of the removal process should be considered an important factor to choose an appropriate treatment method as well as an efficiency of the considered process. The reasonable process should be to remove the heavy metal ions without producing secondary pollutants to convert them from one area to another [11].

The adsorption processes in form of batch or continuous have been proven as an excellent technique for wastewater treatment since it can be considered an eco-friendly process for the treatment
of heavy metals from industrial wastewater [12]. The success of the adsorption process depends on the efficiency to eliminate pollutants in addition to the availability of adsorbents used for the treatment. For that reason, many researchers focused on the finding of available and cheap materials that may be used as an adsorbent. Several studies have been used agricultural wastes as adsorbents for the removal of heavy pollutants from wastewater. However, few studies have shown the role of chemically modified agricultural wastes in removing heavy compounds from contaminated water.

This study is aimed to establish a mathematical model which is described as the adsorption of AS (V) from simulated wastewater using response surface methodology (RSM). To optimize the operating factors, statistical analysis was used to obtain the maximum removal efficiency and adsorption at optimum operating conditions. The mathematical model was analyzed using Langmuir and Freundlich isotherm to show its consist.

2. Materials and Chemical

2.1 Preparation of adsorbent

Spent tea leaves (STL) were collected from agricultural fields. The leaves were initially washed several times in tap water then it dried under sunlight for 24 hr, after that, it was dried in an electric oven at 85°C for 1 day, the leaves were cut to a small size and ground sieve to size 3-6 mm. The next step has represented the carbonization of biomass in an oven at 400 °C for 2 hr with the passing of nitrogen gas. The final step was chemically modified for the spent tea leaves by impregnation by using KOH solution with a ratio of (2:1) (KOH/STL). The modified spent tea leaves (MSTL) produced were cooled at room temperature and washed in double distillate water until the acidity of the effluent solution became neutral then the MSTL dry and store in a plastic container for experimental work. This procedure had stated in the study of Dakhil [12] for chemical activation which has been proven an excellent way in literature studies for the modification of biomass to produce activated carbon. Table 1 shows the physical properties of MSTL produced after chemical activation.

2.2 Preparation of simulated wastewater

All chemicals used in the experimental work were analytical grade. The simulated wastewater with AS (V) was prepared by dissolving an accurate amount of (Na2HAsO4.7H2O) in double distillate water to obtain an initial concentration of arsenic. The desired initial concentrations used in the experimental work were got by diluting the stock solution. The pH of the solution was measured using a digital pH meter and its value adjusted using drops of 0.01 N HCl or 0.01 N NaOH.

Table 1. Physical properties of modified spend tea leaves

| Property           | Unit   | Value |
|--------------------|--------|-------|
| Surface area       | m².g⁻¹ | 82.7  |
| Pore volume        | ml.g⁻¹ | 0.85  |
| Bulk density       | g.cm⁻³ | 3.7   |
| Moisture content   | %      | 12.3  |
| Dry content        | %      | 93.6  |
| Ash content        | %      | 18.2  |
3. Experimental Batch adsorption

The experimental work was conducted to investigate the ability of modified spent tea leaves for the adsorption of arsenic (V) ions from simulated wastewater and to establish a mathematical model that describes the arsenic adsorption on the surface of the adsorbent. The experiments were designed using a central composite conducted in 500 ml volume of a conical flask that contains 100 ml of simulated wastewater. The operating factors selected to establish the mathematical model were varied over the selected ranges according to the experiment design as shown in Table 2. The batch experiments were accomplished under a constant temperature of the laboratory with ±2 °C and shacked under the constant speed of mixing at 200 r.p.m using a magnetic stirrer. Samples were withdrawn according to design time interval using a syringe then filtrated using filter paper. The final concentration for each experiment was determined using Perkin-Elmer atomic absorption spectrophotometer (Model AA200). The removal efficiency of arsenic can be calculated using equation (1) and the adsorption capacity was calculated using equation (2):

\[
\text{\% Removal of Arsenic} = \frac{(C_o - C_f)}{C_o} \times 100
\]  
(1)

\[
q_e = \frac{(C_o - C_f)}{m} \times \frac{V}{m}
\]  
(2)

The initial concentration of arsenic (mg.L\textsuperscript{-1}) was represented by \(C_o\) whereas \(C_f\) was represented the final concentration of arsenic at the end of each experiment. The volume (L) of simulated wastewater for batch experiment and the mass (g) of MSTL were represented by (V) and (m), respectively.

Design of Experiments

To a comprehensive understanding of arsenic adsorption on the surface of MSTL and to establish a mathematical model that describes the removal efficiency of arsenic, the central composite design (CCD) statistical analysed based on the response surface methodology (RSM) was used for this mission. There were four operating factors selected to study its effect on the removal efficiency. (A) is the initial arsenic concentration, (B) is the adsorbent dose, (C) is the pH of the solution and (D) is the mixing time. Table 2 represented the range of operating factors.

| Factors            | Symbol | Unit     | -2  | -1  | 0   | +1  | +2  |
|--------------------|--------|----------|-----|-----|-----|-----|-----|
| Initial concentration | A      | mg.L\textsuperscript{-1} | 10  | 32.5| 55  | 77.5| 100 |
| Adsorbent dosage   | B      | g.L\textsuperscript{-1}  | 0.1 | 0.225| 0.35 | 0.475| 0.6 |
| pH                 | C      | ---      | 2   | 4   | 6   | 8   | 10  |
| Mixing time        | D      | min      | 10  | 35  | 60  | 85  | 110 |

The analyzing of variance (ANOVA) and optimizing of experimental results were statistically analyzed using Minitab-19 and Statistica-10 software. The polynomial equation that describes the removal efficiency can be written as in equation 3:

\[
\% \text{ Removal of AS} = a_0 + a_1 A + a_2 B + a_3 C + a_4 D + a_5 A^2 + a_6 B^2 + a_7 C^2 + a_8 D^2 + a_{10} AB + a_{11} AC + a_{12} AD + a_{13} BC + a_{14} BD + a_{15} CD
\]  
(3)

Where \(a_0\) to \(a_{15}\) have represented parameters of the polynomial model which can be estimated by regression analysis. Table (3) represented the experimental design of the full factorial design with four levels of coded parameters (X1, X2, X3, and X4) which are used to reduce the number of experiments for a good description of removal efficiency (%Removal of AS).
4. Results and Discussion
To assess the influence of operating factors on the adsorption of arsenic on modified spent tea leaves, the experiments were designed to obtain a comprehensive understanding of adsorption arsenic ions on the surface of the adsorbents.

4.1 Regression and Optimization Analysis
The relationship of percent removal to four independent factors (initial concentration (A), adsorbent dosage (B), pH (C), and mixing time (D)) was investigated with twenty-nine runs specified by response surface methodology (RSM). Table 3 displays the coded and real values of the independent factors, and the experimental results obtained from this design. To obtain the regression equation, experimental findings were fitted to a quadratic model. As regards the coded variables, the quadratic polynomial model is as follows:

\[
\% \text{ Removal} = -7.35 + 0.564 A + 213.9 B + 5.37 C + 0.5851 D - 0.004239 A^2 - 185.3 B^2 - 0.3802 C^2 - 0.003433 D^2 - 0.133 AB - 0.01111 AC - 0.001778 AD + 0.120 BD + 0.00500 CD
\]  

(4)

The mathematical model has correlation coefficient \(R^2 = 97.55\%\) and variance \((S) = 0.98\). This work established an experimental design matrix to perform optimization protocols, covering four variables: initial concentration of arsenic \((10-100 \text{ mg.L}^{-1})\), MSTL dosage \((0.1-0.6 \text{ g.L}^{-1})\), pH \((2-10)\), and mixing time \((10-110)\) min as shown in Table 3. The respective RSM software predicted values were calculated based on these actual results to determine the model's compatibility with current tests using ANOVA data. The ANOVA for the arsenic adsorption process was used to ensure a good model. Table 4 shows the results of the complete factor design model fitting in the form of ANOVA.

Table 4 indicates a number of different ANOVA table coefficients (i.e. F-values, P-values). A proposed model is statistically significant so that optimized parameters can achieve a confidence level of 95% if a group of coefficients fulfills certain acquisitions simultaneously, the P-value is less than 0.05, and \(R^2\) close to one. Also, the main and interacting factors are demonstrated in horizontal columns of the Pareto chart as shown in figure 1. The significant factor can be determined by comparing the magnitude of the standard parameter effect with the Pareto vertical line that represented 5% risk which is equal to 2.14 in this model. According to ANOVA and Pareto chart, it seems the four interacting factors are insignificant which are \((AC, AB, BD, \text{and CD})\).

In order to graphically check the assumption of normality for experimental data obtained, a normal probability plot was carried out to examine the residual values, defined as the difference between the predicted (model) and observed values (experimental values). The normal plot shown in figure 2a has illustrated that the predicted arsenic removal values and the actual experimental data are well accepted and proof that the regression model is valid.

In addition, figure 2 b also states that the experiments have been used randomly and the values can be linearized. It is therefore suggested that the given model was well compatible to explain the results.
Table 3. Design experiments in their coded and real values

| No. | X1 | Conc. | X2 | Dose | X3 | pH | X4 | Time | %R |
|-----|----|-------|----|------|----|----|----|------|----|
| 1   | 1  | 77.5  | 1  | 0.475| 1  | 8  | 1  | 85   | 72 |
| 2   | -1 | 32.5  | 1  | 0.475| 1  | 8  | 1  | 85   | 81 |
| 3   | 1  | 77.5  | -1 | 0.225| 1  | 8  | -1 | 35   | 69 |
| 4   | 1  | 77.5  | -1 | 0.475| -1 | 4  | 1  | 85   | 79 |
| 5   | 1  | 77.5  | 1  | 0.475| 1  | 8  | -1 | 35   | 69 |
| 6   | -1 | 32.5  | -1 | 0.225| -1 | 4  | -1 | 35   | 68 |
| 7   | 1  | 77.5  | -1 | 0.225| -1 | 4  | -1 | 35   | 65 |
| 8   | -1 | 32.5  | 1  | 0.475| -1 | 4  | 1  | 85   | 74 |
| 9   | -1 | 32.5  | -1 | 0.225| 1  | 8  | -1 | 35   | 65 |
| 10  | -1 | 32.5  | -1 | 0.225| -1 | 4  | 1  | 85   | 74 |
| 11  | 1  | 77.5  | 1  | 0.475| -1 | 4  | -1 | 35   | 81 |
| 12  | 1  | 77.5  | -1 | 0.225| 1  | 8  | -1 | 35   | 64 |
| 13  | 1  | 77.5  | -1 | 0.225| -1 | 4  | 1  | 85   | 67 |
| 14  | -1 | 32.5  | -1 | 0.225| 1  | 8  | 1  | 85   | 71 |
| 15  | -1 | 32.5  | 1  | 0.475| -1 | 4  | 1  | 85   | 85 |
| 16  | -1 | 32.5  | 1  | 0.475| 1  | 8  | -1 | 35   | 77 |
| 17  | 2  | 100.0 | 0  | 0.350| 0  | 6  | 0  | 60   | 68 |
| 18  | 0  | 55.0  | 2  | 0.600| 0  | 6  | 0  | 60   | 82 |
| 19  | 0  | 55.0  | 0  | 0.350| 2  | 10 | 0  | 60   | 73 |
| 20  | 0  | 55.0  | 0  | 0.350| 0  | 6  | 2  | 110  | 78 |
| 21  | -2 | 10.0  | 0  | 0.350| 0  | 6  | 0  | 60   | 81 |
| 22  | 0  | 55.0  | -2 | 0.100| 0  | 6  | 0  | 60   | 61 |
| 23  | 0  | 55.0  | 0  | 0.350| -2 | 2  | 0  | 60   | 81 |
| 24  | 0  | 55.0  | 0  | 0.350| 0  | 6  | -2 | 10   | 71 |
| 25  | 0  | 55.0  | 0  | 0.350| 0  | 6  | 0  | 60   | 82 |
| 26  | 0  | 55.0  | 0  | 0.350| 0  | 6  | 0  | 60   | 82 |
| 27  | 0  | 55.0  | 0  | 0.350| 0  | 6  | 0  | 60   | 82 |
| 28  | 0  | 55.0  | 0  | 0.350| 0  | 6  | 0  | 60   | 82 |
| 29  | 0  | 55.0  | 0  | 0.350| 0  | 6  | 0  | 60   | 82 |
Table (4). ANOVA for arsenic adsorption model

| Source | Degree of Freedom | Sum of Square | Mean Square | F-Value | P-Value | Effect |
|--------|-------------------|---------------|-------------|---------|---------|--------|
| Model  | 14                | 1426.93       | 101.923     | 39.82   | 0.000   | S      |
| A      | 1                 | 181.50        | 181.500     | 70.91   | 0.000   | S      |
| B      | 1                 | 682.67        | 682.667     | 266.72  | 0.000   | S      |
| C      | 1                 | 112.67        | 112.667     | 44.02   | 0.000   | S      |
| D      | 1                 | 60.17         | 60.167      | 23.51   | 0.000   | S      |
| A²     | 1                 | 119.47        | 119.471     | 46.68   | 0.000   | S      |
| B²     | 1                 | 217.58        | 217.579     | 85.01   | 0.000   | S      |
| C²     | 1                 | 60.01         | 60.011      | 23.45   | 0.000   | S      |
| D²     | 1                 | 119.47        | 119.471     | 46.68   | 0.000   | S      |
| AB     | 1                 | 2.25          | 2.250       | 0.88    | 0.364   | N.S    |
| AC     | 1                 | 4.00          | 4.000       | 1.56    | 0.232   | N.S    |
| AD     | 1                 | 16.00         | 16.000      | 6.25    | 0.025   | N.S    |
| BC     | 1                 | 20.25         | 20.250      | 7.91    | 0.014   | N.S    |
| BD     | 1                 | 2.25          | 2.250       | 0.88    | 0.364   | N.S    |
| CD     | 1                 | 1.00          | 1.000       | 0.39    | 0.542   | N.S    |
| Error  | 14                | 35.83         | 2.560       |         |         |        |
| Lack of Fit | 10               | 35.83         | 3.583       |         |         |        |
| Pure Error | 4               | 0.00          | 0.000       |         |         |        |

Where (S) means significant factor, whereas, (NS) means insignificant factor. To optimization the adsorption process of arsenic, the experimental results were analyzed using RSM to evaluate the best value of parameters that give the maximum arsenic removal efficiency with the composite desirability equal to unity. After optimization analyzing, the optimum values were found (initial arsenic concentration = 40 mg.L⁻¹, adsorbent dose = 0.5 g.L⁻¹, pH = 4, and mixing time = 70 minutes). Under these optimum conditions, the overall removal efficiency was 87% as illustrated in figure 3.

Figure 1. Pareto chart
Effect of Variables on Adsorption Process
The effects of interacting of the variables in the adsorption process were shown by the threedimensional surface response plots and double-dimensional contour plots, depending on two factors with constant the other factor on the optimum value as shown in figures 4.

Figure 4a shows the interacting effect at different values of initial concentration and different adsorbent dose and a constant optimum value of pH at 4 and mixing time at 70 min. According to this figure, the removal efficiency increases with the increasing of the adsorbent dose until reaches the optimum value of 0.5 g. After optimum amount of dosage, the arsenic removal efficiency was slightly decreased. This decrease can be attributed to the accumulation of adsorbents on each other, which leads to a decrease in removal efficiency. This result is in agreement with almost all research findings [13, 14].

Figure 4b shows the interacting effect at different values of initial concentration and different pH and a constant optimum value of adsorbent dose at 0.5 g.L⁻¹ and mixing time at 70 min. This figure indicates that the removal efficiency increases with increasing of pH until reach the optimum value of 4. After that, the removal efficiency was decreased. The decreasing in the removal efficiency can be attributed to the protonation of the amino groups on the adsorbent surface was greatly decreased when
the pH of the solution was transferred from acidic to alkaline conditions decrease. This phenomenon is similar to the finding of Hao, Wang, Wang and Li [15] et al. (2018) for removal of arsenic using a combined process of adsorption and membrane process.

Figure 4c shows the effect of adsorbent dose interacting with pH at a constant initial concentration at 40 mg.L⁻¹ and mixing time at 70 min. From this figure, it is clear to notice the optimum values of the operating factor of adsorbent dose at 0.5 gm.L⁻¹ and pH at 4.

Figure 4d illustrated the interaction of adsorbent dose with mixing time. The results show that the arsenic removal efficiency increases with increasing mixing time until the optimum time of 70 min. This result is in agreement with the finding of researchers such as Dakhil [16], Andrade, Pires, Grossi, Aguiar and Amaral [10].

Figure 4a. Initial concentration vs. adsorbent dose

Figure 4b. Initial concentration vs. pH
Adsorption Isotherms

It is important to analyze isothermic data to develop an equation that accurately represents the results and can be used for design purposes. The optimum parameters were analyzed with the most famous two, Langmuir, Freundlich, isotherm models to investigate the adsorption process.

The Langmuir isotherm model is the monolayer adsorption on the adsorbent surface with limited sites on adsorption areas. The model is based on the premise that there is no adsorbed transmigration to the surface plane, and that the adsorbed molecules have homogeneous adsorption energies and no lateral interactions. The Langmuir nonlinear model is given in equation (5) [17]:

\[
\frac{C_e}{q_e} = \frac{C_e}{q_{\text{max}}} + \frac{1}{b q_{\text{max}}} 
\]

Where the equilibrium states at the end of each experiment for arsenic concentration and adsorption capacity are represented by \(C_e\) (mg.L\(^{-1}\)) and \(q_e\) (mg.g\(^{-1}\)), respectively. \(q_{\text{max}}\) is represented the maximum adsorption capacity in the unit (mg.g\(^{-1}\)) and the Langmuir constant, \(b\), is represented the adsorption energy (L.mg\(^{-1}\)). Table 5 demonstrates the values of \(q_{\text{max}}\) and
b which can be estimated through the slopes and plot intercepts of \((C_e/q_e)\) vs. \((C_e)\) as shown in figure 5.

Another famous isotherm model that describes the heterogeneous adsorption process is the Freundlich model. The empirical linear form of this model can be written as shown in equation (6) [18]:

\[
\ln q_e = \ln k_f + \left(\frac{1}{n}\right) \ln C_e
\]  

(6)

Where \(k_f\) has represented a Freundlich constant isotherm model and \(n\) is the linearity adsorption constant. The values of constants can be determined by plotting \(\ln q_e\) versus \(\ln C_e\) as shown in figure 6. The Freundlich constants are tabulated in Table 5. The value of the Freundlich constant, \(k_f\), can be used as a rough measure of arsenic adsorption affinity and is correlated with the distribution of active sites. Besides, the values of \(n\) means strong adsorption to the adsorbent surface because of active forces.

![Figure 5. Langmuir model](image)
![Figure 6. Freundlich model](image)

| Table 5. Constants of adsorption isotherm models |
|-----------------------------------------------|
| **Langmuir Constants** | **Freundlich Constants** |
| \(q_0 (\text{mg.g}^{-1})\) | \(K_l (\text{l.mg}^{-1})\) | \(R^2\) | \(K_f\) | \(1/n\) | \(R^2\) |
| 379 | 0.002849 | 0.988 | 1.5840 | 0.74184 | 0.948 |

5. Conclusions
This work is aimed to establish a mathematical model for the adsorption of arsenic ions from simulated wastewater using MSTL. The experiments were designed using response surface methodology (RSM) and the results were regression analyzed and optimized to obtain the optimum values of operating factors. The experimental results also analyzed using adsorption isotherm models and the equilibrium results were found to fit with the Langmuir model. The maximum removal efficiency and adsorption uptake at the optimum operating factor were 87% and 69.8 mg.g\(^{-1}\), respectively.
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