LEAD REMOVAL FROM SIMULATED WASTEWATER USING MAGNETITE AS ADSORBENT WITH BOX–BEHNKEN DESIGN

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Abstract
The current education explores the magnetite aptitude by way of adsorbent in eliminating lead metal from simulated wastewater (SWW). The effect of magnetite dose, initial lead concentration, pH solution, and Adsorption time on the elimination procedure remained explored. The adsorbent remained considered through different instrumental methods (FTIR, SEM, and surface area analyzer) and was rummage-sale aimed at the elimination of Pb2+ metals from SWW. It remained initiate that the lead elimination touched 88.9% through 0.3 g magnetite dose, 75 min, 2 ppm original concentration of lead thru pH = 6 at room temperature. The limits of adsorption were strong-minded aimed at heavy metals adsorption utilizing Langmuir and Freundlich isotherms. The consequences deliver robust evidence to support the adsorption mechanism hypothesis.

Keywords: Heavy metal; wastewater treatment; adsorbents; adsorption; DOP optimization.

1.Introduction:
At the moment, the heavy metals pollution in consumption water remains existence explored through numerous experts in cooperation the usual and anthropogenic sources [1]. Heavy metals pollute in cooperation surface and ground water affecting the water quality by way of these metals remain tenacious, poisonous, and hazardous toward persons [2]. The big problematic remains that metals resist squalor and accrue in alive creatures. Owing to their poisonousness toward numerous lifetime forms, the heavy metal existence remains an important problem [3]. Conservative approaches aimed at eliminating poisonous metal ions as of wastewater mainly comprise chemical treatment, Advanced oxidation processes, biological and membrane action. Though the efficiency of these procedures has remained adequately established, they current certain embarrassments and limits because of high energy supplies, incomplete metal ion elimination, then the related cohort of great quantities of sludge [4], [5],[6],[7],[8],[9]. Lately, novel scientific investigation connecting the elimination of poisonous metal ions as of wastewaters has directed care to adsorbents founded on their huge specific surface areas and chemical functional collections. Elimination of poisonous metal ions through Magnetite might principal to important discounts in cost, time and work toward manufacturing, and consequence in better ecological stewardship [10].In the direction of evade and prevent the opposing belongings of heavy metals, a numeral of skills remained industrialized aimed at their elimination as of the environment [11],[12]. Capital and working costs regularly boundary ability and the competence of these approaches, primarily, when great capacities of wastes include moderately squat concentrations [13],[14]. Adsorption developments remain universal adopted in the ecological defense field, thanks to the aptitude of certain solids toward particularly concentrate on their surface specific materials, such by way of organics and heavy metals [15],[16].The adsorption comprises the materials separation from one phase toward the another surface. The adsorbing phase remains the adsorbent, then the material
absorbed or adsorbed at the surface of that phase remains the adsorbate [17]. Recently, assortment of low-cost materials has remained examined by way of adsorbents aimed at the elimination of metal ions in wastewater toward learn inexpensive substitutes aimed at conservative sorbent resources such by way of activated carbon which remains an luxurious adsorbent. sum of the squat cost adsorbents include sawdust [18], corn silk [19], moringa seeds [20], waste lyocell fiber [21], kiwi peels [22], agricultural waste [23], cassia surattensis seeds [24], modified pine bark [25], and treated waste [26]. This paper goals to address the issue of the presentation optimization of SWW action through magnetite adsorbent. The magnetite dose, pH and the adsorption time are chief influences to influence the overall action competence and these were deliberate in this effort.

2. Materials and methods:

2.1 Bio sorbent characterization:

2.1.1 Morphology analysis: A Scanning Electron Microscopy (SEM) examination remained achieved to detect the morphology of the adsorbent superficial prepared. In the current work, the magnetite remained examined through this method by means of JSM-6100 (JEOL).

2.1.2 FTIR analysis: Fourier transform infrared (FTIR) examination remained useful toward determine the surface, functional groups, by means of FTIR spectroscope (FTIR-2000, Bruker), anywhere the bands remained recorded as of 4000 to 500 cm⁻¹.

2.1.3 Specific surface area: The magnetite specific surface \(BET_S\) remains strong-minded finished a Micro metrics device (ASAP 2010) via nitrogen adsorption at 77 K version in the direction of the old-style method of BET.

2.1.4 Metal analysis: lead was strong-minded via Varian Spectra AA 220 FS atomic absorption spectrophotometer. Mercury content was examined spectro-photometrically (Varian Cary 50 Conc UV/Vis Spectrophotometer) at 283 nm. Trials and examination have remained approved out three times and all-out analytical error was originated to be a smaller amount than 5%.

2.2 Materials: Magnetite from Merck (United Kingdom), Sodium chloride, sulfuric acid and sodium hydroxide from Merck (India), and distilled water remained rummage-sale in this research work. Solution’s pH remained attuned with 1N NaOH or 1N H₂SO₄.

2.3 Magnetite adsorption technique: All adsorption trials were appropriate out at the arranged magnetite mass with 100 ml SWW in a 250 ml glass flask. A simulated solution of 1000 ppm of lead metal was prepared through dissolving 1.61g of Pb(NO₃)₂ in 200 ml of distilled water additional by two drops of concentrated nitric acid. After the closure, it remained diluted toward 1000 ml with distilled water. The adsorption remained anxious at 200 rpm by Rushton disc six-blade shaker at 25°C and normal pH. The consequence of the adsorbent quantity remained recognized out from 0.1 to 0.3 g of amount at intermission of 0.15 g with 5 ppm SWW. The pH jested in the variety (3–9) and adsorption time (30–120 min) was approved out through the planned magnetite particle size and adsorbent dose. The quantity of SWW at equilibrium \(q_e\) was envisioned from the mass balance comparison expected by way of underneath [27]:

\[
q_e = \frac{V(C_i - C_f)}{W}
\]
Anywhere \( q_e \) (mg/g) is the whole of Pb\(^{2+}\) ion in SWW per mass unit of watermelon adsorbent at convinced time \( t \), \( V \) (ml) is the SWW volume, \( W \) is the weight of magnetite (g), and \( C_e \), \( C_0 \) (ppm) are the unique and at time \( t \) concentration of wastewater correspondingly. The lead elimination through magnetite remained envisioned meant at all equilibration finished the appearance available by way of:

\[
\text{Adsorption (\%) = } \frac{C_0 - C_e}{C_0} \times 100 \tag{2}
\]

2.4 Experimental design: In this work, untried conditions aimed at SWW mineralization via the procedure of magnetite remained improved by means of the method of central composite design (CCD) underneath RSM. Minitab-17 remained rummage-sale by way of experimental design software, analysis of data, extraction of the quadratic model, and plotting of graphs. The independent variables aimed at instance the adsorption time \( (X_1) \), magnetite dose \( (X_2) \), pH \( (X_3) \), and lead concentration \( (X_4) \) remained coded with their high and low levels in the CCD by way of registered in Table 1. Table 2 lists the obligatory operational variables (i.e. coded and natural) with the Minitab secondhand aimed at the new systems.

| Parameters | levels |
|------------|--------|
| \( X_1 \): Adsorption time (min) | 30-120 |
| \( X_2 \): Magnetite dose (gm) | 0.05-0.3 |
| \( X_3 \): pH | 3-9 |
| \( X_4 \): Lead concentration (ppm) | 2-10 |

| Natural Variables (Xi) | Coded Variables |
|-----------------------|----------------|
|                       | -2  | -1  | 0   | 1   | 2   |
| \( X_1 \): Adsorption time (min.) | 30  | 60  | 75  | 90  | 120 |
| \( X_2 \): Magnetite dose (gm) | 0.05| 0.1125| 0.175| 0.2375| 0.3 |
| \( X_3 \): pH | 3   | 4.5 | 6   | 7.5 | 9   |
| \( X_4 \): Lead concentration (ppm) | 2   | 4   | 6   | 8   | 10  |

2.5 Atomic absorption spectroscopy: Logical Jena (ZEEnit 700P, Germany) Atomic Absorption Spectrometer remained rummage-sale toward determine the Pb\(^{2+}\) concentration in SWW. Air-acetylene with a flow rate of 55, slot burner 100 mm of 6 mm height for Pb\(^{2+}\) ion remained rummage-sale to determine the metal ion concentrations. Interpretations occur in the peak area mode by 283.3 nm aimed at Pb\(^{2+}\)metal.

3. Results and discussion:

3.1 Bio-sorbent characterization:

3.1.1 FTIR Analysis: FTIR spectra in the variety of 4000–500 cm\(^{-1}\) on behalf of the rare magnetite are revealed in Fig. 1. FTIR spectral education recognized the being destructively charged functional groups (hydroxyl, carboxyl, and amine) on the surface of magnetite. The complete band at 3317.67 cm\(^{-1}\) in unadulterated bark powder remains credited owing to hydroxyl (\( ^{-}OH \)) widening or amine (\( ^{-}NH_2 \))
stretching of polymeric complexes. The \( \delta(CH_2) \) symmetric extending vibrations strength be credited to the bands that seemed in the area 2476.21–1743.15\( cm^{-1} \). The peaks of 1658.91 and 1383.01 \( cm^{-1} \) remain mentioning in the carbonyl group \( \sim C=O \), 1271.13 \( cm^{-1} \) signifies the aromatic rings, though 1064.74 \( cm^{-1} \) remains associated through the \( C–O \) in phenols and \( \delta(CH_3) \) and the bands current underneath 827.49 \( cm^{-1} \) remain fingerprint region of sulfur and phosphate functional collections. It careful adequate toward give an imprint around the presence of functional groups on the magnetite adsorbent [28].

Fig.1: FTIR analysis of magnetite before adsorption.

3.1.2 SEM analysis and Specific surface area: The SEM picture demonstrations the surface texture and the adsorbent morphology. The morphology of the surface of the magnetite remains considered through a filamentous structure (Fig. 2), it is originate that it remains a material by a spongy and irregular structure with a high pores number that can remain work as active adsorption centers. This feature brands the surface of magnetite a appropriate adsorbent aimed at eliminating lead element. The magnetite invention has a surface area of around 69.742 m\(^2\)/g.
Fig. 2: Scanning electron microscope micrograph of magnetite.

3.2 Regression models: The association between responses against self-governing variables might be erudite from by the second-order model with a least-squares method [29]:

\[ Y = B_0 + \sum_{i=1}^{q} B_i X_i + \sum_{i=1}^{q} B_i^2 X_i^2 + \sum_{i<j} B_{ij} X_i X_j + \varepsilon \]  

(3)

Anywhere the representation Y means the deliberate responses. Symbols X₁, X₂, … Xq indicate the working variables. Representation B₀ mentions to the constant of regression and Bᵢ is the constant of linear regression. Though, Bᵢᵢ and Bᵢⱼ are the squared regression and the cross-product regression coefficients, congruently. Symbol ε references the random error. Table 3 lists the working variables values, studied replies’ percentage elimination (Pb²⁺ removal).

Table 3: Deliberate variables consequences

| Run | StdOrder | RunOrder | PtType | Blocks | X₁: Adsorption time (min) | X₂: Magnetite dose (gm) | X₃: pH | X₄: Lead concentration (ppm) | Pb removal (%) |
|-----|----------|----------|--------|--------|--------------------------|------------------------|-------|-----------------------------|----------------|
| 1   | 1        | 1        | 2      | 1      | 30                       | 0.05                   | 6     | 6                           | 37             |
| 2   | 2        | 2        | 2      | 1      | 120                      | 0.05                   | 6     | 6                           | 45.2           |
| 3   | 3        | 3        | 2      | 1      | 30                       | 0.3                    | 6     | 6                           | 62.2           |
| 4   | 4        | 4        | 2      | 1      | 120                      | 0.3                    | 6     | 6                           | 68.4           |
| 5   | 5        | 5        | 2      | 1      | 75                       | 0.175                  | 3     | 2                           | 62.8           |
| 6   | 6        | 6        | 2      | 1      | 75                       | 0.175                  | 9     | 2                           | 67.9           |
| 7   | 7        | 7        | 2      | 1      | 75                       | 0.175                  | 3     | 10                          | 25.4           |
| 8   | 8        | 8        | 2      | 1      | 75                       | 0.175                  | 9     | 10                          | 42.1           |
| 9   | 9        | 9        | 2      | 1      | 30                       | 0.175                  | 6     | 2                           | 79.1           |
| 10  | 10       | 10       | 2      | 1      | 120                      | 0.175                  | 6     | 2                           | 86.4           |
| 11  | 11       | 11       | 2      | 1      | 30                       | 0.175                  | 6     | 10                          | 32.5           |
| 12  | 12       | 12       | 2      | 1      | 120                      | 0.175                  | 6     | 10                          | 38.9           |
| 13  | 13       | 13       | 2      | 1      | 75                       | 0.05                   | 3     | 6                           | 42.5           |
| 14  | 14       | 14       | 2      | 1      | 75                       | 0.3                    | 3     | 6                           | 53.8           |
| 15  | 15       | 15       | 2      | 1      | 75                       | 0.05                   | 9     | 6                           | 51.1           |
| 16  | 16       | 16       | 2      | 1      | 75                       | 0.3                    | 9     | 6                           | 62.8           |
| 17  | 17       | 17       | 2      | 1      | 30                       | 0.175                  | 3     | 6                           | 46.2           |
| 18  | 18       | 18       | 2      | 1      | 120                      | 0.175                  | 3     | 6                           | 48.3           |
| 19  | 19       | 19       | 2      | 1      | 30                       | 0.175                  | 9     | 6                           | 51.2           |
| 20  | 20       | 20       | 2      | 1      | 120                      | 0.175                  | 9     | 6                           | 53.42          |
| 21  | 21       | 21       | 2      | 1      | 75                       | 0.05                   | 6     | 2                           | 75.8           |
| 22  | 22       | 22       | 2      | 1      | 75                       | 0.3                    | 6     | 2                           | 88.9           |
| 23  | 23       | 23       | 2      | 1      | 75                       | 0.05                   | 6     | 10                          | 43.2           |
| 24  | 24       | 24       | 2      | 1      | 75                       | 0.3                    | 6     | 10                          | 49.6           |
| 25  | 25       | 25       | 0      | 1      | 75                       | 0.175                  | 6     | 6                           | 58.44          |
Founded on untried consequences, the mathematical equation (Eq. 4) remained industrialized in terms of real issues connecting the lead element response toward the working variables revealing the interactions between these variables:

\[
Pb \text{ Removal} = 34.4 + 0.303 \times X_1 + 63.9 \times X_2 + 10.72 \times X_3 - 0.000147 \times X_1^2 + 62 \times X_2^2 - 0.905 \times X_3^2 + 0.156 \times X_4^2 - 0.089 \times X_1 \times X_2 + 0.0002 \times X_1 \times X_3 - 0.0013 \times X_1 \times X_4 + 0.27 \times X_2 \times X_3 - 3.35 \times X_2^2 \times X_3 + 0.242 \times X_3 \times X_4
\]  

(4)

BDD competence remained recognized through usage alteration parts completed through specific sources of difference. The competence of the model in ANOVA examination remains documented founded on Fisher F-test and P-test. Numerous of the alteration in response can remain exposed through the regression equation if the value of Fisher remains higher. P-value test is rummage-sale to regulate whether F is high sufficient to suggest statistical meaning (90%). The percent of variability of the model might remain elucidated if the P-value was fewer than 0.05[30]. Table 4 exemplifies ANOVA aimed at the RSM model. In this table, degree of freedom (DOF), sum of square (Seq. SS) for all parameter, adjusted sum of the square (Adj. SS), adjusted mean of the square (Adj. MS), Fisher-value, and P-test value were assessed. Fisher-value of (9.22) and P-test value of (0.0001) were got, which exemplified the high meaning of the regression model. The multiple correlation constant of the model was 93.15% compliant that the regression was statistically important and only (6.35%) of the full variations are not reinforced through the education. The attuned manifold correlation coefficient (adj. R^2 = 85.15%) was well-matched with R^2 in this model.

### TABLE 4: Analysis of variance aimed at heavy metal removal

| Source | DOF | Seq. SS   | Adj. MS   | Fisher Value | P-test Value |
|--------|-----|----------|-----------|--------------|--------------|
| 1-Model| 20  | 11201.3  | 560.07    | 13.73        | 0.000        |
| Linear | 5   | 9210.5   | 1842.09   | 45.15        | 0.000        |
| X1     | 1   | 87.3     | 87.33     | 2.14         | 0.156        |
| X2     | 1   | 3683.6   | 3683.58   | 90.29        | 0.000        |
| X3     | 1   | 4984.0   | 4984.01   | 122.17       | 0.000        |
| X4     | 1   | 179.2    | 179.16    | 4.39         | 0.046        |
| Square | 5   | 1865.6   | 373.11    | 9.15         | 0.000        |
| X1^2   | 1   | 77.8     | 77.82     | 1.91         | 0.179        |
3.2.1 Effect of magnetite solution: The adsorption of the lead metal on magnetite experimental by way of a function of the adsorbent quantity remains exposed in Figure 3. The exclusion efficiency of lead metal remained increased with the cumulative amount of sorbent pending accomplishment (64.2%). The upsurge in metal retrieval with the amount of magnetite can remain credited toward the greater number of available adsorption places or high superficial areas [30], [31]. It is experiential from Fig. 3 that increasing the magnetite dose, ms, augmented the lead elimination from SWW, from this ostensible that through cumulative the dose of the magnetite powder, the number of sorption sites obtainable aimed at sorbent–solute communication remains augmented, thus subsequent in the augmented elimination of lead metal from SWW. A similar observation has been stated in the literature [32].

![Scatterplot of Pb Removal vs Magnetite Dose (gm)](image)

Fig. 3: The dosage consequence on the removal of Simulated wastewater.
3.2.2 Effect of adsorption time: It remains vital to assess the consequence of adsorption time on the adsorption proceeding to the teaching of the adsorption [28]. The trials are lead once the adsorption time varies from 30 to 120 min at a different magnetite dose from 0.05 to 0.3 gm., and the significances got are showing in Fig.4. Adsorption processes proceed fast and adsorption equilibrium can remain reached within 30 min. Additional upsurge in adsorption time does not improve the adsorption action. Correspondingly, the adsorption conduct upsurge with cumulative the adsorption time. The singularity might remain meanwhile, originally, all active sites on the the adsorbent surface are vacant and the heavy metal concentration is high. Afterward that period, few surface active sites are obtainable, consequently only a very low upsurge in the metal uptake is experiential [19]. So, the finest adsorption time aimed at lead metal was nominated by way of 90 min intended for all the evenness inspections.

![Scatterplot of Pb Removal vs Time (min)](image)

**Fig. 4:** The adsorption time consequence on the elimination of simulated wastewater.

3.2.3 Effect of solution pH: The pH of the SWW in which adsorption remains existence lead is a important supervisory limit in the treatment of adsorption. The magnitude of electrostatic customers connected finished the ionized heavy metal and the functional groups on the magnetite superficial remain primarily skillful through pH of the average [32]. In the pH variety 3–9, sorption of both divalent metal materials remained tremendously low, and with the increase in the initial pH, metal uptake augmented (Fig. 5). Look as if between pH 3.5 and 6, though all-out Pb$^{2+}$ acceptance took place at pH 5–6. Higher pH values were evaded to prevent likely precipitation by way of hydroxides [33]. These explanations can remain clarified through the detail that at lower pH values, the surface charge of the biomass remains positive, which remains not promising to cations biosorption. Meanwhile, hydrogen ions compete strongly with metal ions at the active sites, subsequent in fewer bio-sorptions. With cumulative pH, electrostatic repulsions between cations and surface places and the competing result of hydrogen ions reduction. So, the metal bio-sorption upsurges [34].
3.2.4 Effect of initial Pb\(^{2+}\) concentration: The consequence of the initial concentration on lead remained examined in the variety of 2–10 ppm with a pH value fixed at assumed circumstances. The consequences are exposed in Fig. 6. If the mass of magnetite adsorbent in the interruption remains equal to 0.3 gm., 76 \% of the initial lead with 2 ppm a concentration is detached. At inferior metal concentrations, the proportion of the number of magnetite moles in wastewater to the obtainable surface area remains little and henceforth adsorption is self-governing of the initial concentration. At higher concentrations, the number of obtainable places aimed at adsorption reductions, and, henceforth, metal element elimination is reliant on the initial concentration [35].

3.3 Optimizing the working variables: Finest values of employed variables such by way of adsorption time, magnetite dose, pH and heavy metal concentration were got using Minitab-17. Fig. 7 labels the measurement significances of the D-optimization. Greatest elimination competencies of heavy metal were larger than 88 \%.
3.4 Adsorption Isotherms: Consequences from the bio-sorption process were attuned to linear equations of Langmuir and Freundlich (Figs. 8 & 9) mathematical isotherm models correspondingly aimed at magnetite adsorbent. From the alteration of each adsorption isotherms, isothermal limits remain calculated toward choice the finest model shadowed through untried consequences of bio-sorption tests. These data specify a high adsorption capacity of magnetite associated with other material published educations with bio sorbents. Aimed at the contrast of the data, it must be careful the working circumstances of each of the educations since the setting isothermal experimental consequences depend on them [36]. It seems that the Langmuir model finest fits the experimental consequences over the experimental variety with decent constants of correlation (R^2 > 0.99). Langmuir isotherm intended for heavy metal is defenseless in Fig.8. This tendency is alike to bags specified in the[37].
4. Conclusions:

This effort has deliberate the request of magnetite by way of bio sorbent aimed at the elimination of heavy metal in SWW. it has remained strong-minded the following best circumstances: Firstly the adsorption time between adsorbent and SWW is around 75 min aimed at heavy metal, meanwhile adsorption capacity keeps constant afterward this time, the adsorption capacity remains higher through the lowermost magnetite dose verified, however percentages of elimination upsurge once magnetite amounts are too augmented meanwhile the whole amount of adsorbent additional remains higher. It originated that all-out heavy metal adsorption capacity might be attained to be 23.11 mg/g (88.9%) with an absorbent quantity of 0.3 g and adsorption time of 75 min with the lead metal concentration of 2 ppm and best pH of 6 f. Langmuir isotherm was originate better-quality tailored than Freundlich model. Though magnetite adsorption procedures can remain rummage-sale aimed at the conduct of simulated wastewater, the adsorption treatment is supplementary well-organized.

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