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# Optimization Study for Desorption of Arsenic and Regeneration Performance on Magnetic Carbon Xerogels for Environmental Sustainability †

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**Abstract:** Magnetic carbon xerogels were synthesized via direct sonication to load magnetite nanoparticles in sol–gel polycondensation onto resorcinol-formaldehyde gels. The resulting organic gels were carbonized and subjected to surface modification using $H_2O_2$ and characterized using scanning electron microscopy and energy-dispersive X-ray spectroscopy. The desorption capacity was optimized using response surface methodology, with the adsorbent dose identified as the most significant quantitative factor. The kinetic adsorption was well described using the Elovich and Power equations. The regeneration capacity was evaluated over four sequential adsorption–desorption cycles, demonstrating the possibility of reusing the adsorbent and reducing the environmental impact.

**Keywords:** adsorption–desorption; arsenic; kinetic model; response surface methodology



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## 1. Introduction

The presence of arsenic in the aquatic environment has caused significant exposure and health problems in many countries worldwide [1,2]. Adsorption is considered an effective technology for removing arsenic from water and wastewater treatment [3], but investigating various factors that influence the desorption and the reuse of adsorbents is necessary to develop an economically and environmentally acceptable approach.

Numerous methods for preparing magnetite nanoparticles have been reported, with conventional co-precipitation using the $Fe^{3+}/Fe^{2+}$ molar ratio established by the reaction stoichiometry being considered a simple, cheap, and high-yield method, with control over nanoparticle distribution and low-temperature requirements [4].

Gels possess a unique structure of large interconnected pores, mechanical and chemical stability, and feasibility for control and design according to variations in the synthesis and processing conditions. Mesoporous gels and carbon xerogels have been studied for water treatment processes, with success in removing toxic organic compounds [5], organic dyes [6], and heavy metals [7–10].

This study evaluated the desorption capacity of magnetic carbon xerogels and their potential for regeneration. The optimal condition for the arsenate desorption process was studied using a response surface methodology (RSM). Additionally, the kinetics of arsenic

adsorption and desorption were investigated with various models. Finally, the regenerative capacity of the magnetic carbon xerogels were determined to carry out the sequential adsorption–desorption.

## 2. Methods

### *2.1. Synthesis of Magnetic Carbon Xerogels*

Magnetic xerogels were synthesized via sol–gel polymerization of resorcinol (R), formaldehyde (F), water (W), and magnetite nanoparticles (M) using sodium carbonate (C) as a catalyst. The molar ratios were M/R = 0.07, R/C = 100, R/W = 0.04, and R/F = 0.5. For full details on the synthesis procedure of magnetic xerogels, please refer to [11].

### *2.2. The Effect of Carbonization Temperatures to Obtain the Optimum Conditions*

The effect of carbonization temperatures ranging from 600 °C to 850 °C were investigated to determine the optimum condition for preparing magnetic carbon. A tubular furnace was used for carbonization with a 50 mm alumina tube (GSL-1600X-50-UL; MTI Corporation, Richmond, CA, USA).

The XMC10 sample was carbonized using a heating ramp of 2 °C/min and nitrogen flow of 100 mL/min at a pyrolysis temperature of 600 °C for 6 h, resulting in the XMC10-600 sample. Additionally, some samples were pyrolyzed at 350 °C for 1 h and 850 °C for 2 h with a constant heating rate of 10 °C/min and nitrogen flow of 100 mL/min, resulting in the MCXM850 sample. Surface modification using $H_2O_2$ was applied to enhance metal binding on the surface [12], resulting in the XMC10-600M and MCXM850M samples.

### *2.3. Characterization of Materials*

The surface morphology and porous structure of the magnetic carbon xerogels were analyzed using a scanning electron microscope (SEM) with analytical systems of energy-dispersive X-ray spectroscopy (EDS) (Bruker) on an FE-SEM (7800F Prime; JEOL, Peabody, MA, USA).

### *2.4. The Kinetics of Arsenic Adsorption*

The experimental adsorption data for the kinetic study were obtained by varying the contact time at intervals of 3, 6, 10, 30, 60, 120, 240, 360, 1200, 1320, and 1440 min. The magnetic xerogel carbons used for comparison were XMC10-600M and XMC10-850M, which were pyrolyzed at 600 °C and 850 °C, respectively.

### *2.5. The Optimal Condition for the Arsenate Desorption Process Using an RSM*

The optimal conditions for arsenate desorption were determined using RSM. Three variables were considered: desorbing concentration, adsorbent dose, and agitation speed, with low, medium, and high levels for each variable (as shown in Table 1). Two center points were also included. The experimental design and data analysis were carried out using R statistical software version 4.0.3.

**Table 1.** Configuration of variables for implementation of the RSM method to optimize arsenic adsorption.

| Factors | Coding Factors | Low (−1) | Center (0) | High (1) |
|---|---|---|---|---|
| Concentration of KOH solution (M) | $x_1$ | 0.5 | 1 | 1.5 |
| Orbital shaker speed (rpm) | $x_2$ | 80 | 120 | 160 |
| Spent adsorbent dose (g/L) | $x_3$ | 0.4 | 1.2 | 2 |

### *2.6. Arsenic Adsorbent Regeneration*

The experiment to regenerate the arsenic adsorbent was carried out using XMC10-850M that was previously loaded with arsenic, as obtained from the kinetic adsorption

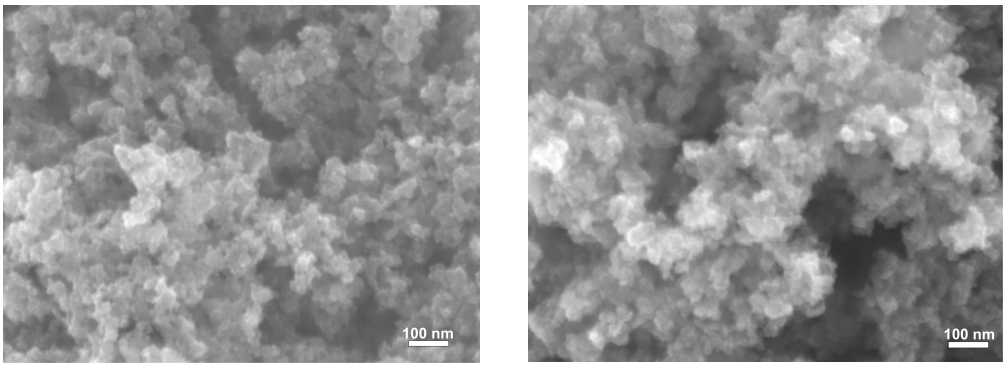
study under conditions of pH 3.0, contact time of 1440 min, and initial concentration of arsenic solution of 1.024 mg/L. The concentration of arsenic was determined using an inductively coupled plasma optical emission spectrometer (ICP-OES), model OPTIMA 8300.

## 3. Results and Discussion

Magnetic xerogel carbon composites loaded with magnetite nanoparticles were obtained to evaluate the effect of carbonization temperatures between 600 °C and 850 °C.

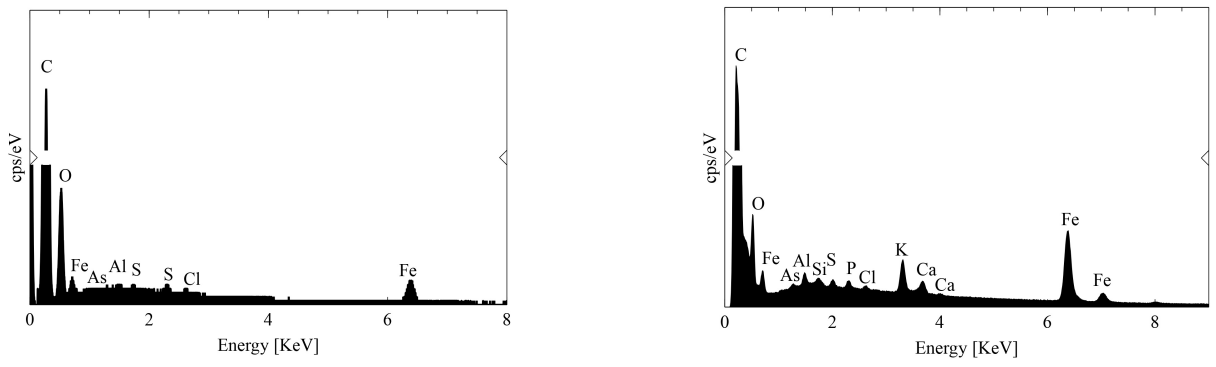
### *3.1. Characterization of Magnetic Carbon Xerogels*

Figure 1 depicts the surface morphology and pore structure of the magnetic carbon xerogels analyzed through SEM. XMC10-600M and XMC10-850M were synthesized with R/W = 0.04, R/C = 100, and M/R = 0.07 at carbonization temperatures of 600 °C and 850 °C, respectively. Both xerogels exhibited similar microstructures that are homogeneous and composed of fine particles. The xerogel structure consists of a continuous solid skeleton, which is composed of chains of monomer particles arranged in a string of pearl-like 3D networks. The SEM images of XMC10-600M and XMC10-850M showed that the magnetite nanoparticles were evenly distributed across the RF gels and adhered to the RF gel surface. The corresponding EDS analysis in Figure 2 confirmed that the magnetic carbon xerogels XMC10-600 and XMC10-850, after they had adsorbed the arsenic, were composed of elements C, O, Al, Si, Fe, and As. The presence of Fe in the RF gels was also confirmed by an atomic absorption spectrometer, with an Fe content of 3.56% by weight. However, the magnetic nature of the magnetite during the sonication process on the synthesis of xerogels resulted in some parts of the RF gel showing agglomeration of magnetite nanoparticles [13].




(**a**) (**b**)

**Figure 1.** Scanning electron microscopy (SEM) images of magnetic carbon xerogels (**a**) XMC10-600M and (**b**) XMC10-850M.

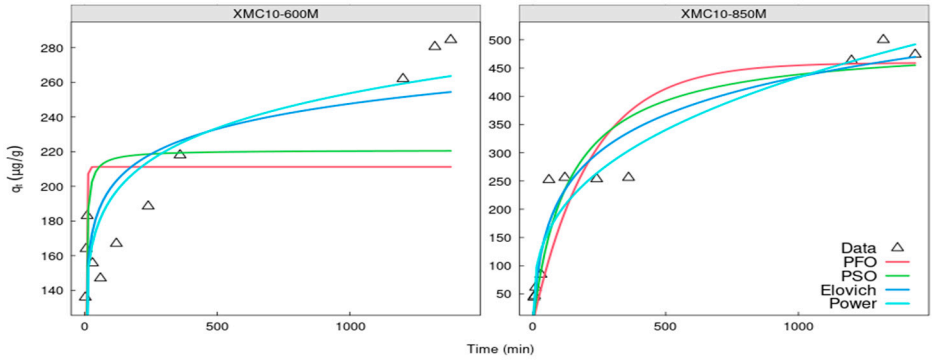



(**a**) (**b**)

**Figure 2.** EDS spectrum of magnetic carbon xerogels (**a**) XMC10-600M and (**b**) XMC10-850M after arsenic adsorption.

### *3.2. The Kinetics of Arsenic Adsorption*

The kinetic adsorption data of arsenate were plotted as the amount of arsenate adsorbed per unit weight of xerogel carbon (qt, mg/g) versus time (min). Nonlinear equations were used to analyze the data, which are presented in Figure 3. The adsorption kinetics of XMC10-600M and XMC10-850M were evaluated using pseudo-first order (PFO), pseudo-second order (PSO), Elovich, and Power models. The kinetic parameters were then calculated. The kinetics of both Elovich and Power models present a correlation coefficient greater than 0.90 for XMC10-850M.




(**a**) (**b**)

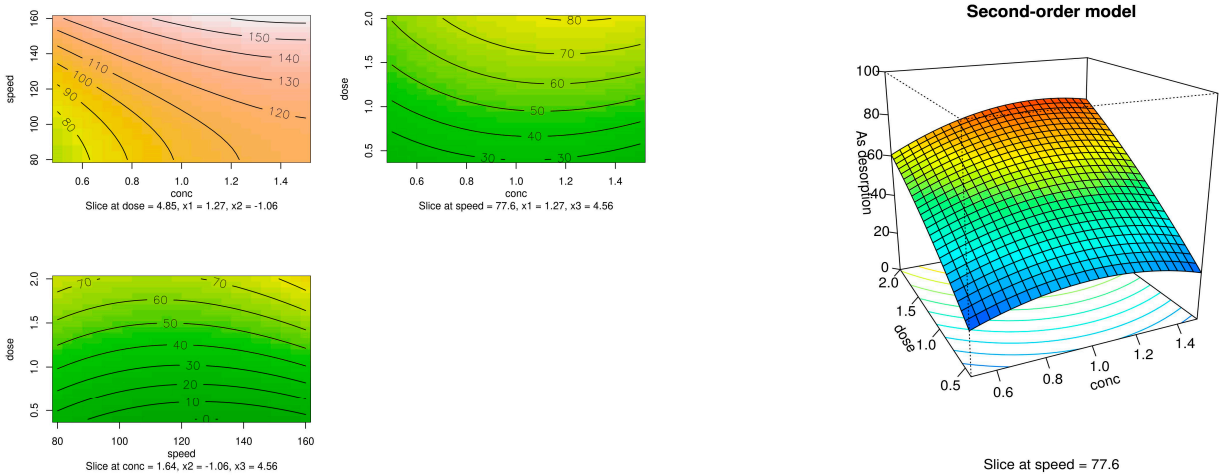
**Figure 3.** Adsorption kinetics of arsenate using magnetic xerogel carbons at carbonization temperatures of (**a**) 600 °C and (**b**) 850 °C (condition: adsorbent dosages of 2 g/L, pH of 3.0, and initial concentration of arsenic solution of 1.024 mg/L).

### *3.3. The Optimal Condition for the Arsenate Desorption Process Using an RSM*

The RSM was utilized to develop a model for optimizing arsenic desorption by analyzing the effects of various factor combinations through a novel linear model approach. The R v4.0.3 software was used to obtain the results of a second-degree polynomial model, where the variables $x_1$ (KOH concentration), $x_2$ (Agitation speed), and $x_3$ (Adsorbent dose) were encoded. The resulting second-degree model is

$$\text{As desorption} = 49.07 + 2.4x_1 + 3.78x_2 + 29.37x_3 - 2.54x_1x_2 + 3.84x_1x_3 + 4.98x_2x_3 - 8.9x_1^2 + 11.04x_2^2 - 3.17x_3^2 \quad (1)$$

Figure 4a demonstrates the behavior of arsenic desorption with respect to the variables considered, using the stationary points in their original units, namely, a concentration (conc) of 1.64, a speed of 77.79, and a dose of 4.85. It can be observed that as the agitation speed and the concentration of the desorbing agent increase, the arsenic desorption of the material improves. Figure 4b shows a 3D representation of the behavior of the arsenic desorption percentage as a function of desorbing agent concentration (KOH) versus adsorbent dose, with an agitation value of 77.79 rpm as the estimated stationary point, which was obtained by implementing the methodology of response surface. It can be seen that the variable $x_3$ (dose) has a significant influence on the response variable, arsenic desorption, as indicated by the higher number of asterisks. Moreover, the *p*-value of the model is significant as it is less than 0.05.




(**a**) (**b**)

**Figure 4.** The behavior of arsenic desorption (**a**) 2D representation (contour lines) as a function of the different variables (conc, speed and dose) and (**b**) 3D representation as a function of conc vs. dose.

### *3.4. Arsenic Adsorbent Regeneration*

Regarding the results of the experimental design using RSM, the optimal conditions for the regeneration process were found to be 1.0 M KOH, 150 rpm, with a dose of 2 g/L, and a contact time of 180 min. It can be observed from Figure 5 that the removal efficiency of arsenic adsorption decreased from cycle 1 to cycle 4, by approximately 7.83%, and remained at 88.19% at cycle 4. The percentage of arsenic desorption achieved during cycle 1 was 82.75%, but after desorption in cycle 2–4, the percentage of desorption was only 66.16–58.22%, decreasing by around 26.59% from cycle 1 to cycle 4. From the desorption experiment, it can be concluded that XMC10-850M can recover arsenic and be reused for more than four cycles with the arsenic adsorption remaining over 88.19%, without requiring any treatment, and thus ensuring environmental sustainability.

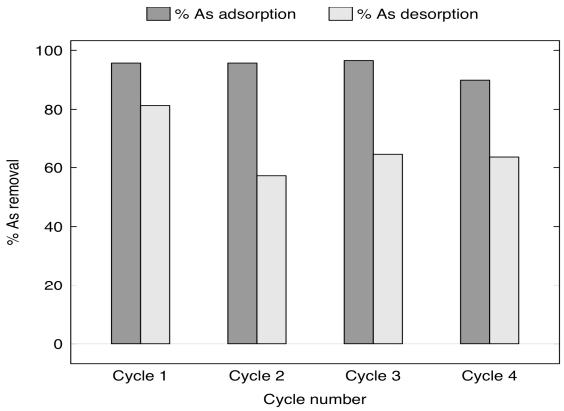



**Figure 5.** Adsorption–desorption of arsenic under optimal conditions given by RSM.

## 4. Conclusions

The use of magnetite nanoparticles, organic gel pyrolysis, and surface modification with $H_2O_2$ had a significant impact on the morphology and surface chemistry of magnetic carbon xerogels, which resulted in improved adsorption capacity for arsenic. The magnetite nanocrystals, which are dispersed throughout the carbon xerogels in a crosslinked nanostructure, were found to range in size from 15 to 20 nm. Furthermore, the pyrolysis treatment at 850 °C improved the material's texture, increasing its porosity and decreasing particle size. By utilizing a response surface methodology to investigate optimal conditions

for the magnetic xerogels in the desorption process, their maximum desorption capacity can be determined, and there is a possibility of regeneration in an aqueous solution.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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