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RESEARCH ARTICLE

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Adsorptive removal of phosphate from aqueous solutions using $FeCl_3$-immobilized melamine-formaldehyde-urea resin

Fatos Ayca Ozdemir Olgun[1] | **Pınar Çetin**[2]

[1]Department of Chemical Engineering, Faculty of Engineering and Natural Sciences, Istanbul Health and Technology University, Istanbul, Turkey

[2]Department of Chemistry, Faculty of Science and Letters, Istanbul Technical University, Istanbul, Turkey

Correspondence
Fatos Ayca Ozdemir Olgun, Department of Chemical Engineering, Faculty of Engineering and Natural Sciences, Istanbul Health and Technology University, Istanbul, Turkey.
Email: fatos.olgun@istun.edu.tr

Abstract

The surplus phosphorus in aquatic environments trigger eutrophication, an irreversible pollution stage marked by the rapid growth of algae and water plants, a decline in dissolved oxygen levels, and the emergence of dead zones. To address this critical issue, this study focuses on the removal of phosphorus in form of phosphate from aqueous solutions. The research involves the synthesis and characterization of $FeCl_3$-immobilized melamine-formaldehyde-urea resin, an innovative adsorbent, its characterization through Fourier transform infrared spectroscopy, and the evaluation of its adsorption capacity based on Langmuir and Freundlich equilibrium models. The experimental adsorption data fits well with pseudo-second order kinetic model. Optimal adsorption parameters are carried with batch adsorption technique for 60 min using 0.5 g of adsorbent at $pH = 2$, achieving 15.55 mg g^{-1} adsorption capacity, and conducting experiments with tap-water samples to replicate real-world scenarios. The research findings have the potential to advance sustainable wastewater treatment technologies and contribute to the mitigation of nutrient pollution in aquatic ecosystems, addressing a pressing environmental concern.

KEYWORDS

adsorption, adsoption isotherm, Fe(III) chloride, phosphate removal, water treatment

1 | INTRODUCTION

Phosphorus, the most found 11th element in nature, was first invented by Henning Brandt in 1669, through distillation of urine. After further studies, it was found that it constitutes 1% of the human body weight, taking part in the substances of life (DNA and ATP), in nucleic acids, responsible of cell development and functioning; in nerves, signaling intracellular molecules and playing role as phospholipids in cellular organizations.[1,2] In industry, 85% of phosphorus is utilized as artificial fertilizer when 15% is divided among industries including food, detergent, paper, military, and so forth. Chemical evaluation of phosphorus is a complex issue, as it is difficult to decide whether it is a hero or a villain. It may be found in inorganic and organic forms, showing diversions in both forms with the aid of plants and other organisms.

In recent years, with the growth of population and economy, in combination with pesticide and artificial fertilizer utilization, phosphorus content of wastewaters and sewage has showed a dramatic increasement.[3] Unfortunately, this excess amount of phosphorus in aquatic environment has resulted with "eutrophication," accelerated growth of algae, and water plants in the fertility level of natural waters.[4] Eutrophication is the irreversible stage of pollution, which cannot be corrected naturally. Furthermore, even at concentration values, equal or lower than 0.1 mg L^{-1}, phosphorus can still stimulate the algae reproduction leading eutrophication, and decreases the level of dissolved oxygen, which results a reduction at the number of aquatic species, constructing a "dead zone."[5–8]

However, there are many methods in literature to prevent eutrophication. As it is known, phosphate is the main form of phosphorus in the environment and with the aid of microorganisms, all forms of phosphorus (even organophosphorus) will be converted to inorganic phosphate, eventually. The methodology of phosphorus removal from wastewaters found in literature mainly includes chemical precipitation, coagulation, biological process, adsorption, ion exchange, and electrocoagulation.[9–19] Each method has its own advantage and disadvantage when compared according to their operation costs, effectiveness, concentration range, and control systems (pH, doses of chemicals etc.). Among them, adsorption process and membrane technologies should be evaluated separately, since phosphorus is the rare resource, and needs to be recovered. For this purpose, many novel adsorbents were introduced to the literature in order to remove and recover phosphorus from wastewaters,[19–21] but very few studies were published with iron-based materials.[3] Iron salts including ferrous and ferric salts are considerable with their low cost, easy dissolution, environment friendly properties, and simple operation, especially in case of wastewater treatment systems.

The primary objective of this study was to investigate and optimize the adsorptive behavior of a novel adsorbent for the removal of phosphate from tap-water samples. The study focused on the synthesis and characterization of $FeCl_3$-immobilized melamine-formaldehyde-urea (MFU) resin, an innovative adsorbent with the potential for enhanced phosphate adsorption. Experimental procedures included the preparation of the resin, its characterization through techniques such as Fourier transform infrared spectroscopy, and the assessment of its adsorption capacity within the outlined adsorption equilibrium models. Optimum adsorption parameters were also discussed within the proposed study. The adsorption experiments were conducted using actual tap-water samples to simulate real-world conditions. The study aimed to determine the effectiveness of the developed adsorbent in removing phosphate from tap water, with an emphasis on sustainable and environmentally friendly treatment methods. The research outcomes will contribute to the advancement of sustainable wastewater treatment technologies and the mitigation of nutrient pollution in aquatic ecosystems.

2 | EXPERIMENTAL METHODS

2.1 | Reagents and instrumentation

All reagents used during the experiments were of analytical grade (E. Merck). Standard and sample solutions were prepared with distilled-deionized water. Calibration standard solutions of P were prepared by diluting the commercial P stock solution (Merck, 1000 mg L^{-1}). Nickel in nitrate form was used as matrix modifier with a concentration 10 μg mL^{-1}. The plastic and glass-ware installed in all experiments were rinsed with distilled water following a cleaning procedure with dilute HNO_3 (10%). pH measurements and adjustments were performed using 766 Knick pH-meter. Edmind Buhler 7400 Tubingen shaker was installed for shaking step in batch adsorption process.

2.2 | Preparation of $FeCl_3$-immobilized MFU resin

Melamine, formaldehyde and urea solutions were mixed with a mole ratio of 1:1:3 under heat reflux at 80°C 10% (w/v) to synthesize MFU resin according to the method found in literature.[22]

At the second step of the adsorbent preparation, Fe (III)-immobilized MFU resin was obtained by agitating a certain amount of resin with 0.5 mol L^{-1} $FeCl_3$ solution at 300 rpm for 2 h and then heated to110°C until they become dry. After they were let to cool at room temperature, they were rinsed to remove impurities, dried, and sieved to work with a certain particle size.

2.3 | Characterization of the resin

2.3.1 | FTIR analysis

The surface functional groups of samples were enlightened by transmission infrared spectra obtained from a Perkin–Elmer 1310 IR spectrometer, using pellets of KBr containing about 0.5% finely ground MFU resin samples. These pellets were dried overnight at 110°C before the spectra were recorded, and the spectra were the result of averaging 60 scans with the range of 650–4.000 cm^{-1}.

TABLE 1 Graphite furnace Atomic Absorption Spectrometer conditions for phosphorus analysis.

Light source	Phosphorus lamp (wavelength: 213.16 nm, band width: 0.8 nm)
Makeup solution	Ultra-pure water
Modifier	Nickel
Pyrolysis temperature	800°C
Atomization temperature	2700°C
Inert gas/flow	Argon/0.3 mL s^{-1}

2.4 | Phosphate analysis

Graphite furnace Varian AA 280Z Zeeman Atomic Absorption Spectrometer was used to measure the absorbance values of phosphorus in standard and sample solutions. Phosphorus standard solutions were prepared at required concentration levels by dilution of 100 mg L^{-1} stock solution of KH_2PO_4 and atomized at 2700°C. Interference effects were minimized using nickel modifier (Table 1).

2.5 | Adsorption studies

In batch studies, standard solutions of phosphate were prepared at pH = 2.0 and allowed to contact with 0.5 g of resin at a particle size of 16–18 mesh for 60 min at 200 rpm. The absorbance values were measured prior and latter to the adsorption procedure, and concentration values were calculated with the aid of the calibration graph, in terms of phosphate. Amount of adsorbed phosphate was calculated via equation:

$$q_{\mathrm{e}} = \frac{V\left(c_0 - C_{\mathrm{eq}}\right)}{m}, \tag{1}$$

where q_{e} is the adsorption capacity at equilibrium, V is the volume of solution (L), C_0 (mg L^{-1}) and C_{eq} (mg L^{-1}) are the concentration values of standard or sample solutions prior to the adsorption and at equilibrium, and m (g) is the amount of resin used. Langmuir and Freundlich adsorption isotherms were studied in order to define the adsorption mechanism of phosphate on the proposed resin.

Pseudo-first order and pseudo-second order models were used to enlighten the adsorption kinetics of phosphate.[23,24] The required equations for the evaluation of kinetic models are given in Equations 2 and 3, respectively, for pseudo-first order and pseudo-second order models:

$$q_t = q_1(1 - \exp(-k_1 t)), \tag{2}$$

$$q_t = \frac{t}{{}^{t}\!/_{q_2} + {}^{1}\!/_{k_2 q_2^2}}, \tag{3}$$

where k_1 (min^{-1}) and k_2 (g mg^{-1} min^{-1}) are the rate constants of pseudo-first order and pseudo-second order models, q_1 and q_2 are the theoretical values for the adsorption capacity (mg g^{-1}), and q_{t} (mg g^{-1}) is the adsorption capacity at time t (min).

Prior to the actual adsorption experiments, parameters such as pH, amount of adsorbent, and contact time were optimized by working with one parameter at a time method.

2.6 | Adsorption studies with real samples

Batch adsorption method was applied to tap-water samples collected from different regions of Istanbul, Turkiye. Optimum adsorption parameters of the proposed method were installed for 5 mL of tap-water samples, directly. The analyses were repeated three times, and the final results were given as average values.

3 | RESULTS AND DISCUSSIONS

3.1 | Characterization of the resin

The IR spectrum of the synthesized MFU resin[22] showed bands at 3275.94, 1625.84, and 1358.01 cm^{-1} that attribute to NH, Cl_4O, and CN group vibrations, respectively. The novel version of the previous resin (synthesized by the author previously) was characterized to check and evaluate the differences as a result of the immobilization procedure. Figure 1 enlightens the bonding structure of the novel material.

As it is displayed in Figure 1, new bands corresponding to the vibrational modes of the bonds were formed around 812 cm^{-1} due to C—H bending, which may be explained with a substitution reaction. Also, when analyzed in detail, a general shift at particular wavenumbers (from 1544 to 1539 cm^{-1}) is observed, which may be called as "blue shift" underlying a molecular change.

3.2 | The immobilization of $FeCl_3$

For the determination of optimum $FeCl_3$ concentration that can remove the maximum amount of phosphate,

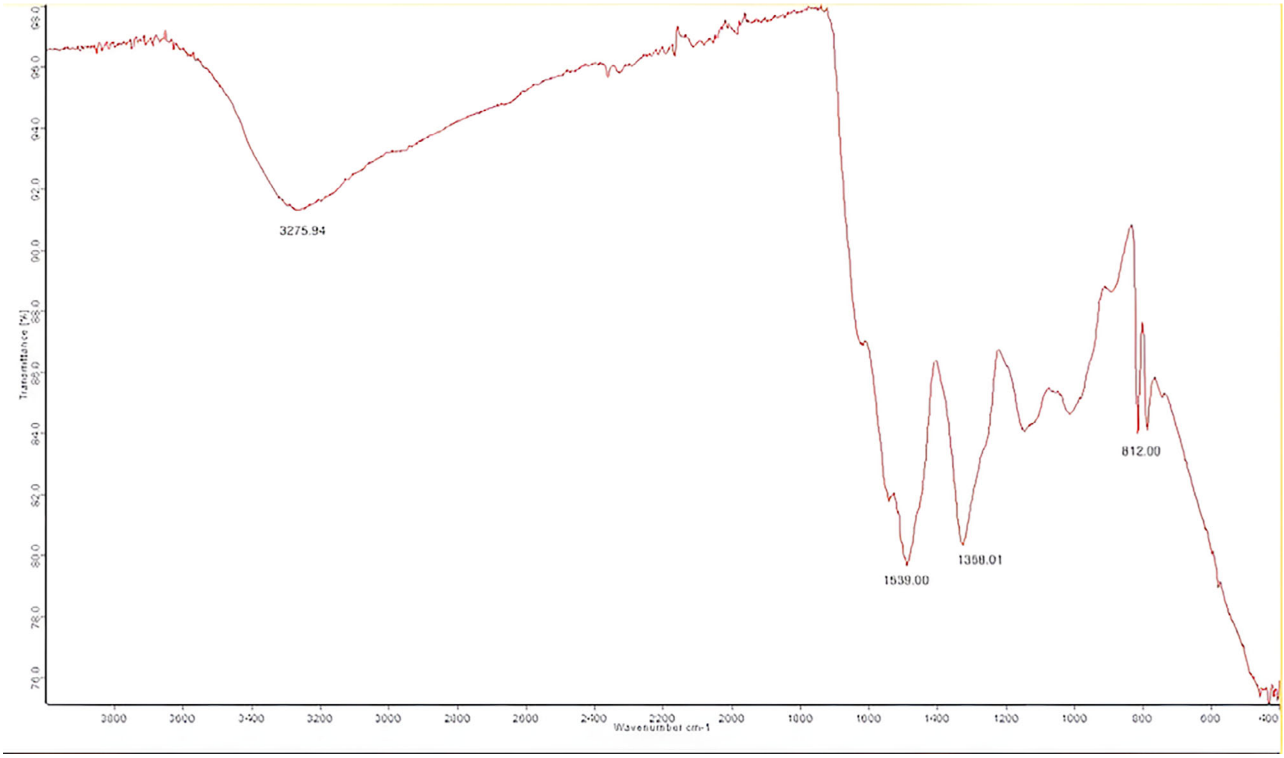

FIGURE 1 Infrared spectrum of $FeCl_3$-immobilized melamine-formaldehyde-urea resin. [Color figure can be viewed at wileyonlinelibrary.com]

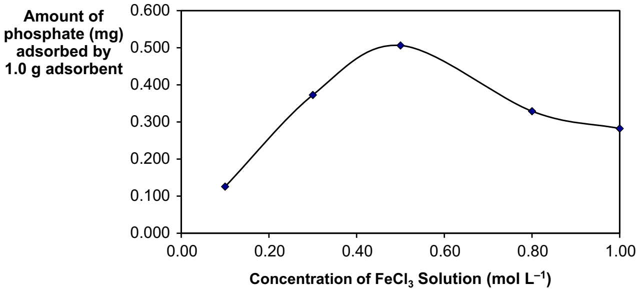

FIGURE 2 Amount of phosphate (mg) adsorbed by 1.0 g adsorbent. [Color figure can be viewed at wileyonlinelibrary.com]

concentration of $FeCl_3$ versus phosphate amount removed in milligrams per 1 g of resin graph was obtained.

Irrelevant of adsorption parameters, as it was displayed in Figure 2, the maximum amount of phosphate was removed by 0.5 mol L^{-1} of $FeCl_3$ installed for the immobilization.

When iron salts dissolve in aquatic environment including phosphate, several reactions may occur. The possible removal pathways of phosphate were summarized in the reactions given below.

$$Fe^{3+} + PO_4{}^{3-} \rightarrow FePO_4 \downarrow$$

$$Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3 + 3H^+$$

$$Fe(OH)_3 \rightarrow FeOOH + H_2O$$

$$2Fe(OH)_3 \rightarrow Fe_2O_3 + 3H_2O$$

$FeCl_2$ and $FeCl_3$ hydrates, the respective precursors of iron oxides were the results of the modifications. As a result of heating, water and other compounds leave the structure constructing a porous pattern in the modified materials enhancing adsorption capacity by increasing surface area.[12,19]

3.3 | Adsorption studies

3.3.1 | Effect of pH on removal percentage of phosphate

Standard solutions with adjusted pH values of 2.0, 4.0, 6.0, and 8.0 were prepared for the investigation of pH effect on adsorption. Each solution was allowed to contact with 0.1 g adsorbent for 120 min. The absorbance values of solutions before and after adsorption procedure was recorded and evaluated at the column graph displayed in Figure 3.

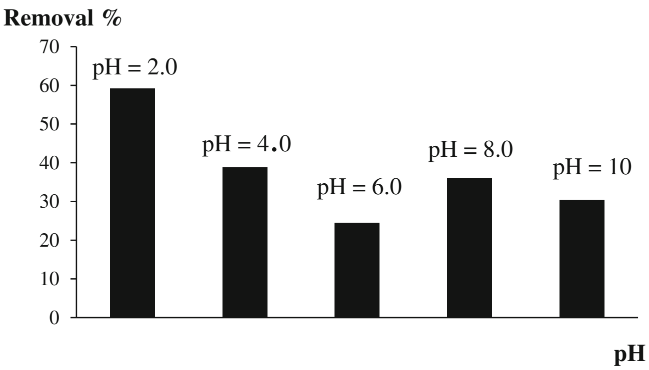

FIGURE 3 Optimization of pH value ($T = 25°C$).

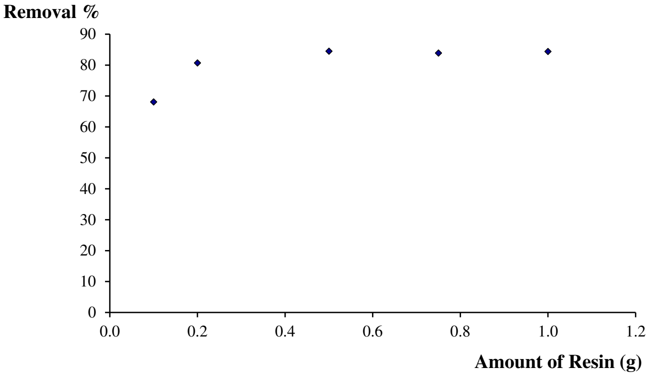

FIGURE 4 Optimization of resin dose. [Color figure can be viewed at wileyonlinelibrary.com]

Maximum removal percentage was obtained at experimental medium with pH = 2. The result is considerable, whereas at relatively high pH values, positively charged surface alters as a result of deprotonation leading electrostatic interactions toward phosphate.[25]

3.3.2 | Effect of adsorbent dose

The phosphate adsorption onto the $FeCl_3$-immobilized MFU adsorbent is studied by varying adsorbent dose (0.1, 0.2, 0.5, 0.75, and 1.0 g) in 50 mL phosphate solution with an initial phosphate concentration of 3.0 mg L^{-1}, keeping the initial pH, sorbent grain size, and temperature constant (Figure 4).

Removal percentage of phosphate by the suggested adsorbent reaches a plateau at adsorbent dosage of 0.5 g for 50 mL, due to the increasement at phosphate species in the solution.

3.3.3 | Effect of contact time

Final parameter to be optimized is contact time, as it is important to gain the maximum efficiency at minimum period of time. For this purpose, keeping all the previous parameters constant with their optimum values, removal percentage of phosphate by $FeCl_3$-immobilized MFU resin was calculated.

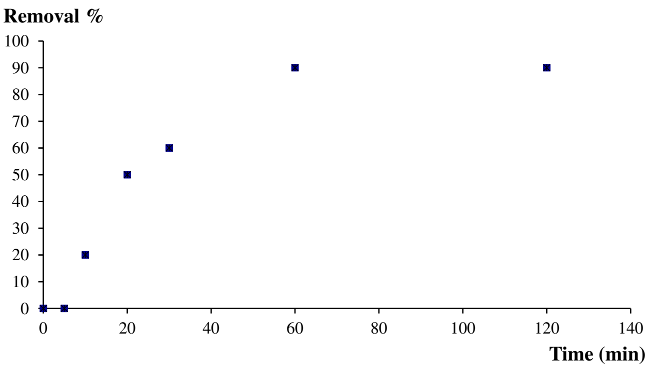

FIGURE 5 Optimization of contact time. [Color figure can be viewed at wileyonlinelibrary.com]

As it is displayed on Figure 5, 3.0 mg L^{-1} phosphate solution was allowed to contact with the adsorbent at increasing time intervals as 5, 10, 20, 30, 60, and 120 min. At each time interval, portion of solution was taken, and absorbance values were recorded. Rest of the batch adsorption experiments were performed for 60 min, as any dramatic change at absorbance value did not occur.

3.4 | Kinetic models of adsorption

Pseudo-first order and pseudo-second order kinetics models are terms often used to describe the adsorption of solutes onto a solid surface and help to analyze the rate at which solute molecules or ions are adsorbed onto a surface. The pseudo-first order model is commonly employed during the initial minutes of the adsorption process, while the pseudo-second order model is well-suited for the entire range of contact times.[26]

The fitting parameters for the kinetic studies of adsorption were tabulated in Table 2. According to the comparison between the regression coefficients of the kinetic models, it may be concluded that adsorption data are more compatible with pseudo-second order kinetics model ($R^2 = 0.9137$).

Adsorption of phosphate on other adsorbents such as hydrous zirconium oxide[27] agreed with the similar phenomena. The well-fitting of the pseudo-second order kinetic model suggests that the adsorption of phosphate on $FeCl_3$-immobilized MFU resin involves strong chemical interactions where multilayer adsorption is significant (Figure 6).

TABLE 2 Kinetic parameters for phosphate adsorption on $FeCl_3$-immobilized melamine-formaldehyde-urea resin.

Pseudo-first order			Pseudo-second order		
q_1 (mg g^{-1})	k_1 (min^{-1})	R^2	q_2 (mg g^{-1})	k_2 (min^{-1})	R^2
15.51	0.1195	0.8872	6.340	0.0039	0.9137

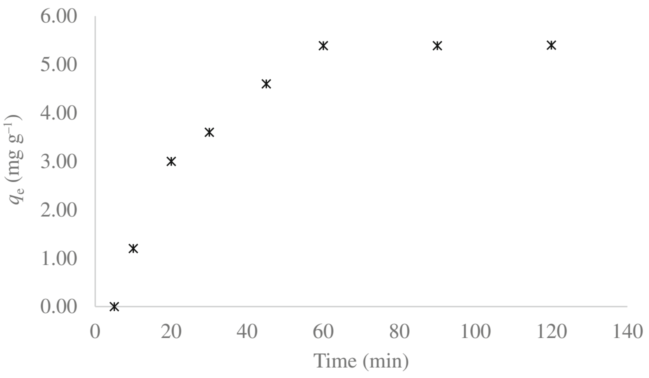

FIGURE 6 Adsorption kinetics of phosphate on $FeCl_3$-immobilized melamine-formaldehyde-urea resin.

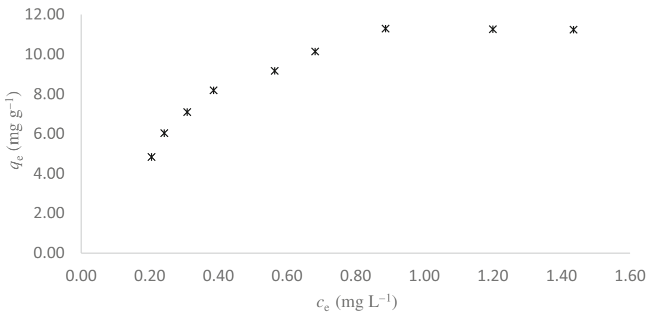

FIGURE 7 Adsorption isotherm for $FeCl_3$-immobilized melamine-formaldehyde-urea resin.

3.5 | Adsorption isotherms

Evaluating adsorption equilibria is required to understand the process of adsorption process of a system. Considering this, adsorption experiments were studied measuring the absorbance values of 50 mL phosphorus solutions at increasing concentration levels keeping the adsorption parameters at optimum values (adsorbent dose = 0.5 g, pH = 2, contact time = 60 min).

The simplest theoretical model for the monolayer adsorption is the Langmuir isotherm equation having the following form:

$$q_e = \frac{1}{4} Q\max \times K_L C_e (1 + C_e), \tag{4}$$

$$\frac{C_e}{q_e} = \frac{1}{(Q\max K_L)} + \frac{C_e}{Q\max}, \tag{5}$$

$$q_e = \frac{(C_0 - C_e)V}{m}, \tag{6}$$

where Qmax and K_L are Langmuir isotherm parameters representing the maximum adsorption capacity for the resin and equilibrium constant related to the Gibbs free energy change of adsorption. C_e and q_e represent the equilibrium concentrations of the adsorbed phosphate in solution and on the suggested adsorbent, respectively; V and m denote the solution volume and resin mass, respectively. Figure 7 shows the main adsorption isotherm (represented by Equation 4) and its linearized form (represented by Equation 5), respectively, regarding phosphate adsorption on $FeCl_3$-immobilized MFU resin.

The Freundlich isotherm may be linearized with a similar algorithm: the logarithmic version of the Freundlich equation represents a line with a slope of $1/n$ and an intercept of K_f in accordance with Equation 7.

$$\log q_e = m \log C_e + b. \tag{7}$$

$$\frac{1}{n} = m. \tag{8}$$

$$K_f = \text{antilog}(b). \tag{9}$$

All the parameters calculated for Langmuir and Freundlich adsorption modeling of dye uptake data are collectively summarized in Table 3. Among Freundlich parameters, K_f is related to adsorption capacity and n characterizes the heterogeneity of the system.

One of the important characteristics of Langmuir isotherm can be explained by the separation factor R_L, which is calculated by the following equation:

$$R_L = \frac{1}{(1 + bQ_0)}, \tag{10}$$

For a feasible adsorption process, the calculated value of R_L should be $0 < R_L < 1$. In this study, it was calculated as 0.1 enabling a favorable value of R_L.

In a Freundlich adsorption isotherm, the adsorbate is assumed to form a monomolecular layer on the surface of the adsorbent. If Freundlich model is applied to the

TABLE 3 Parameters of adsorption isotherms.

Adsorption isotherm	Isotherm equation	Slope^{-1}	K_L or K_f	R^2
Langmuir	$q_e = c_e[1/(0.064c_e + 0.018)]$	Qmax = 15.55 mg g^{-1} resin	$K_{L=}$ 3.53 (L mg^{-1})	0.99
Freundlich	$q_e = 2.13\ C_e^{0.88}$	$n = 1.134$	$K_f = 2.13$ (mg g^{-1})	0.99

TABLE 4 Adsorbents and installed experimental conditions for phosphate adsorption.

	Experimental conditions				
Adsorbent	pH	T (°C)	Resin dose (g L^{-1})	Adsorption capacity	Reference
Activated alumina	5.5	20	-	12–14	28
Activated carbon	-	-	-	3.2	29
Activated carbon	6.7	30	1	13.55	30
Anion exchange from soybean hulls	7	25	10	20.0	31
Iron-oxide tailing	6.6	20	-	5–8	32
Mg-Fe-LDH	8.4	25	5	15.5	33
Calcinated Mg-Fe-Cl LDH	7.0	25	2	9.8	34
Iron zirconium binary oxide	4.0	25	-	13.7	28
This study	2.0	22	1	15.55	

adsorption process, the coefficient n should be greater than 1, which was calculated as 1.134 in our study.

Ensuring the conditions of both models, the case of our study is both coherent with Langmuir and Freundlich models, providing high regression coefficient values. It may be concluded as the phosphate adsorption model occurs at particular homogenous sites on the surface of $FeCl_3$-immobilized MFU. In addition, being compatible with Freundlich model, we may infer that the adsorbate forms a monomolecular layer on the surface of the proposed sorbent.

The value of the maximum adsorption capacity was found as 15.55 mg phosphate/g adsorbent with the aid of the adsorption isotherm data, fitting well with Langmuir adsorption model. Phosphate removal percentage was recorded as approximately 90% after 60 min of contact time with 0.5 g of adsorbent. The adsorbents suggested for the adsorption of phosphate were filtered from the literature and listed in Table 4. The adsorption capacity found by this study is reported among the considerable values of the literature.

3.6 | Regeneration studies

The adsorbent regeneration experiments were performed batchwise at 22°C with different solutions as NaOH (0.1, 0.2, and 1.0 mol L^{-1}), HCl (0.1 and 0.5 mol L^{-1}), NaCl and KCl (both at 0.05, 0.1, and 0.5 mol L^{-1}). The initial concentration of phosphate solution was 5 mg L^{-1} at an adjusted pH value of 2.0. Among all the solutions, taking place at regeneration studies, 0.5 mol L^{-1} HCl solution was found to be the most suitable, enabling a regeneration percentage of 90.3%, giving an opportunity to reuse the adsorbent and thus making it environment friendly.

TABLE 5 Total phosphate removal percentages from collected tap-water samples.

Sample	Removal (%)
1	72.0
2	69.6
3	68.2
4	65.6
5	70.3

3.7 | Phosphate removal from tap-water samples (application of the proposed method)

The optimal conditions of adsorption predetermined for phosphate were applied to tap-water samples collected from home faucets of five different regions of Istanbul, Turkiye. Table 5 represents the removal percentage of phosphate from the water samples before and after the batch treatment processes.

As it is seen from Table 3, averagely 70.0% of phosphate removal percentages may be achieved by batch adsorption technique with the aid of $FeCl_3$-immobilized MFU resin within 60 min. The decrease at removal percentages refers to the presence of coexisting anions in solution interfering in the phosphate adsorption process. In literature, similar results were achieved leading 25% and 15% decrease at removal percentages in the presence of sulfate and monovalent anions, respectively.[35]

4 | CONCLUSION

The findings of this study underscore the role of Fe (III)-immobilized MFU resin as an indispensable adsorbent for phosphate removal in the context of sustainable wastewater treatment. Notably, this resin has proven its worth through its cost-effectiveness, substantial adsorption capacity, and the capacity for efficient regeneration under dilute acid conditions, achieving an impressive 90.3% efficiency in regeneration studies and enabling multiple rounds of use. Moreover, the adsorption behavior of phosphate on the resin predominantly adheres to the Langmuir model, emphasizing its effectiveness in this application. Furthermore, the optimal pH for phosphate removal was found to be 2.0, and the resin's adsorption capacity ($Qmax = 15.55$ mg g^{-1} according to Langmuir model) is comparable with the most available sorbents.[28–36] The underlying kinetic adsorption mechanism is fundamentally corroborated with pseudo-second order kinetics. These findings collectively support the potential of $FeCl_3$-immobilized MFU resin as a sustainable and efficient sorbent for enhanced phosphate removal from water samples and contribute to the advancement of environmentally friendly wastewater treatment practices.

AUTHOR CONTRIBUTIONS

F. Ayça Ozdemir Olgun: Conceptualization (lead); data curation (lead); formal analysis (supporting); investigation (lead); methodology (lead); resources (lead); supervision (lead); validation (lead); writing – original draft (lead); writing – review and editing (lead). **Pınar Çetin:** Formal analysis (supporting).

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CONFLICT OF INTEREST STATEMENT

The author declares no conflicts of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author (F. Ayça Ozdemir Olgun), upon reasonable request.

ORCID

Fatos Ayca Ozdemir Olgun https://orcid.org/0000-0003-1077-2621

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