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PHOSPHATE REMOVAL FROM FERTILIZER INDUSTRY WASTEWATER USING IRON-FUNCTIONALIZED ACTIVATED CARBON

BY

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Abstract. Phosphorus-rich effluents generated during fertilizer production require efficient treatment technologies to minimize their environmental impact. This study evaluates the adsorption of phosphate ions using a carbonaceous material before and after surface modification with iron under both batch and continuous-flow operating conditions. The modified adsorbent was prepared by a simple wet impregnation method to introduce iron active sites capable of enhancing phosphate uptake. The influence of operating mode and solid–liquid contact time on adsorption performance was investigated. Surface modification improved phosphate uptake through electrostatic attraction, ligand exchange, and surface complexation mechanisms. Under continuous-flow conditions, extending the contact time by solution recirculation increased adsorption efficiency, demonstrating the importance of residence time in fixed-bed systems. The results indicate that the proposed material is suitable for the treatment of highly concentrated phosphorus-containing wastewaters and that continuous adsorption

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can provide performance comparable to, or slightly better than, batch operation when sufficient contact time is ensured.

Keywords: fixed-bed adsorption, surface functionalization, phosphorus recovery, mass transfer, wastewater treatment.

1. Introduction

Phosphate pollution - one of the major environmental issues - is associated with industrial wastewater, particularly from the NPK fertilizer industry, where phosphorus compounds are substantially used in technological processes. As a result, a part of the phosphorus is inevitably discharged into wastewater streams, increasing their pollutant load (Azalia and Hendrasarie, 2022).

One of the primary sources of phosphate pollution is the wash water generated during the cleaning of NPK production facilities, including equipment, pipelines, and processing surfaces. During these operations, residues of raw materials and intermediate products are washed into the water, leading to the presence of phosphate ions in the effluent. Another source of phosphate-containing wastewater is the granulation stage of fertilizer production. During granulation, raw materials are converted into granules through humidification and drying processes. Fine phosphate particles may be entrained into process water or captured by gas-cleaning systems such as wet scrubbers.

The production of phosphoric acid represents another significant source of phosphate-rich wastewater. The widely applied wet-process route involves the reaction of phosphate rock with sulfuric acid, producing phosphoric acid and phosphogypsum. During this process, part of the phosphorus remains in the liquid phase, and the resulting wastewater may contain high concentrations of phosphates, sulphates, fluorides, and heavy metals. These effluents are considered particularly challenging because of their complex composition and their potential environmental impact (Azalia and Hendrasarie, 2022; Bhandari *et al.*, 2016).

An important characteristic of fertilizer industry wastewater is that these different process streams are not always treated separately. Instead, they are frequently collected into a common wastewater system, resulting in a complex effluent with highly variable composition. This variability complicates the application of conventional treatment technologies and highlights the need for tailored treatment strategies adapted to the specific characteristics of each wastewater stream.

Depending on the operating conditions, phosphorus concentrations in these wastewaters may range from several tens to several hundreds of mg/L. Wastewater generated by the NPK fertilizer industry generally contains higher concentrations of phosphorous than those typically found in municipal

wastewater. While the total phosphorus concentration in domestic wastewater is generally below 10 mg/L, industrial effluents from fertilizer production frequently contain phosphorus concentrations ranging from 100 to 300 mg/L and, in some cases, exceeding 500 mg/L (Bhandari *et al.*, 2016).

Compared to other water pollutants, phosphates exhibit direct toxicity with significant impact on humans and animals only at very high concentrations. For this reason, no specific limits have been established for phosphorus content in drinking water. Exposure to elevated concentrations is associated with the occurrence of transient digestive disorders. The main issue related to the presence of phosphorus in the aquatic environment is the essential role it plays as a limiting nutrient in triggering and accelerating the eutrophication process. By stimulating the excessive development of algae and cyanobacteria, excess phosphorus promotes the occurrence of harmful algal blooms and cyanotoxins, with potentially negative effects on both water quality and public health (Nieder *et al.*, 2018). Excessive phosphorus input into aquatic ecosystems, particularly coastal and marine ones, stimulates *Harmful Algal Blooms - HABs*. This phenomenon is associated with the production of toxins that can affect human health. Exposure occurs mainly through the consumption of contaminated shellfish, with reported cases of neurological, amnesic, paralytic, and diarrheic intoxications. Concentrations exceeding 0.10 mg P/L (≈0.31 mg PO_4^{3-} /L) are frequently associated with the excessive development of algae and cyanobacteria, while values above 0.20 mg P/L (≈0.61 PO_4^{3-} mg /L) indicate a high risk of harmful algal bloom occurrence.

The high phosphorus concentrations in these industrial effluents have important implications for wastewater treatment. Conventional treatment methods may be insufficient or require substantial amounts of chemical reagents, resulting in increased operational costs. At the same time, these phosphorus-rich effluents represent a valuable opportunity for phosphorus recovery, supporting resource conservation and circular economy principles (Peeva, 2026).

Phosphorus is an essential resource for agriculture and food production; however, its availability is limited because global phosphate rock reserves are concentrated in only a few regions of the world, creating economic dependencies and geopolitical risks. Moreover, a significant fraction of the phosphorus used in human activities is lost to the environment, particularly through wastewater discharges. This loss represents not only an environmental concern, but the inefficient use of a valuable non-renewable resource. Previous studies have shown that anthropogenic phosphorus flows have increased considerably over recent decades, leading to nutrient imbalances and adverse impacts on aquatic ecosystems (Peeva, 2026).

In this context, phosphorus recovery plays a pivotal role in sustainable resource management. It has been estimated that recovering phosphorus from secondary sources, such as wastewater and sewage sludge, could supply approximately 15–20% of the global phosphorus demand, thereby reducing the

pressure on natural phosphate rock reserves and supporting the transition toward a circular economy.

2. Phosphate Adsorption from Fertilizer Industry Wastewater

During the production of compound NPK fertilizers, wastewater generated from equipment and process line cleaning operations, as well as from other technological stages, contains not only phosphate ions but also nitrogen compounds, organic matter (expressed as chemical oxygen demand (COD), and suspended solids (Azalia and Hendrasarie, 2022). Conventional treatment methods, such as chemical precipitation, coagulation, and membrane separation, are often insufficient to achieve the required reduction of dissolved phosphorus. Adsorption using materials with a high affinity for phosphate ions has proven to be an effective polishing step for enhancing phosphorus removal from treated effluents.

Beyond its high removal efficiency, adsorption enables phosphorus recovery and reuse, making it an attractive approach for sustainable phosphorus management. Its operational flexibility, the wide range of available adsorbent materials, and the possibility of adsorbent regeneration have established adsorption as one of the most promising technologies for phosphorus removal and resource recovery. Research interest in this approach has increased considerably in recent years, reflecting a shift in the perception of phosphorus—from an environmental pollutant to a valuable secondary resource that can be recovered and reintegrated into the circular economy (Jalilian *et al.*, 2024; Rashid *et al.*, 2021).

A wide variety of adsorbent materials with high affinity for phosphate ions has been investigated, including modified activated carbons, functionalized biochars, metal oxides, zeolites, iron- and aluminum-based materials, and various composite adsorbents (Crovella *et al.*, 2024; Recepoglu *et al*., 2022; Usman *et al.*, 2022). Following phosphorus recovery through desorption enables its reuse as a raw material for fertilizer production, thereby supporting the implementation of circular economy principles and promoting more sustainable phosphorus management (Kasprzyk and Gajewska, 2019).

Among the adsorbent materials reported in the literature, iron- and aluminum-based oxides exhibit the highest phosphate removal efficiencies, typically reaching 85–95% (Mengxue *et al.*, 2016; Zhang *et al.*, 2016). Their superior performance is attributed to the strong affinity of phosphate ions for metal hydroxyl groups on the adsorbent surface, where stable inner-sphere complexes are formed through ligand exchange and surface complexation mechanisms.

Zeolites and modified clays are attractive alternatives among conventional adsorbents because of their wide availability, physicochemical properties and low cost. Reported phosphate removal efficiencies range from 70–90% for zeolites and 70–85% for modified clays. The adsorption performance of

these materials depends on their mineralogical composition, specific surface area, and the presence of metal elements such as iron, aluminium, and calcium, which contribute to phosphate removal through adsorption and precipitation mechanisms (Bouanga Boudiombo *et al.*, 2023).

Activated carbon also exhibits good adsorption performance, although its phosphate removal efficiency is generally limited to 20–35% due to the relatively low affinity of its surface toward phosphate ions. However, surface modification with metal species such as Fe, Al, La, or Zr significantly enhances its adsorption capacity. The high specific surface area and well-developed porous structure of activated carbon provide a good support for metal functionalization, while the introduced metal species create additional active sites and increase the surface affinity toward phosphate ions through specific adsorption mechanisms. As a result, both the adsorption capacity and selectivity of the material are improved, resulting in more efficient phosphate uptake and retention on the modified adsorbent surface (Azalia and Hendrasarie, 2022; Ganjoo *et al.*, 2023).

When activated carbon is functionalized with iron compounds, such as $FeCl_3$, iron oxides, iron hydroxides, or $Fe_2(SO_4)_3$, active surface groups (e.g. Fe–OH, FeOOH, and Fe_2O_3) are formed on the external surface and within the pore structure of the carbon. These iron-containing functional groups significantly modify the physicochemical properties of the adsorbent and enhance its affinity for phosphate ions (Strawn *et al*., 2023).

The enhanced phosphate adsorption is mainly attributed to the following mechanisms (Nadagouda *et al.*, 2024):

- electrostatic attraction: iron functionalization imparts a more positively charged surface, promoting the attraction of negatively charged phosphate ions.
- ligand exchange: this is considered the dominant mechanism for phosphate adsorption onto iron-functionalized materials. Phosphate ions replace hydroxyl groups bound to surface iron sites, forming stable inner-sphere surface complexes according to the reaction:
  $\equiv FeOH + H_2PO_4^- \rightarrow \equiv FeHPO_4 + H_2O$
- surface complexation: phosphate ions can form mono- or bidentate complexes with surface iron atoms. These complexes remain stable over a wide pH range and contribute to the high selectivity of the adsorbent toward phosphate ions.
- surface precipitation: at elevated phosphate concentrations or in the presence of abundant iron species, insoluble iron phosphate compounds may precipitate on the adsorbent surface. This process complements adsorption and further increases the overall phosphorus retention capacity.

Recent studies have reported that iron functionalization can nearly double the phosphate removal efficiency compared with unmodified activated carbon. Iron impregnation modifies the electrochemical properties of activated carbon, increasing the number of active adsorption sites, and enhances its adsorption

capacity, resulting in significantly greater selectivity toward phosphate ions (Bezza and Chirwa, 2021; Seo *et al.*, 2025).

For wastewater generated by the fertilizer industry, iron-functionalized activated carbon represents a promising adsorbent because it not only reduces phosphate concentrations in industrial effluents but also enables the recovery of phosphorus as a valuable secondary resource, thereby supporting sustainable wastewater treatment and circular economy strategies.

3. Materials and method

Granular activated carbon (GAC, 8–20 mesh, CAS 7440-44-0) supplied by Sigma-Aldrich was used as the base adsorbent in this study. The material consists primarily of elemental carbon and exhibits a highly developed porous structure, making it suitable for adsorption and purification processes. The morphological characteristics of the activated carbon particles were examined by optical microscopy before and after impregnation with $FeCl_3$ using an integrated image acquisition and measurement system. The characteristic particle dimensions were determined by direct measurements on the acquired micrographs using the calibrated reference scale (Fig. 1) with HiView free soft. The unmodified material exhibits irregular, millimeter-sized granules with rough external surfaces and longitudinal striations, indicative of heterogeneous carbonization and surface abrasion. At higher magnification, the granules display a relatively uniform distribution of pores, characterized by near-circular morphologies and diameters ranging from approximately 0.07 to 0.24 mm. This pore size interval places the material within the mesoporous–macroporous domain, a structural regime known to facilitate rapid mass transfer and efficient diffusion of adsorbates. The coexistence of mesopores and macropores is particularly advantageous for adsorption applications, as macropores act as transport channels while mesopores provide active sites for retention.

The internal architecture consists of an extensive network of interconnected cavities, indicating a high specific surface area—a defining characteristic of high-performance activated carbons. Such a hierarchical pore system enhances both the kinetics and capacity of adsorption by increasing the number of accessible active sites and reducing diffusion limitations. The observed morphology therefore aligns with the expected structural features of an efficient adsorbent and supports the material's suitability for applications involving liquid-phase or gas-phase pollutant removal.

Following $FeCl_3$ impregnation, the granular morphology remains largely intact; however, subtle yet discernible modifications in surface texture are observed. These changes are consistent with partial occlusion of surface pores or localized deposition of iron-containing species, suggesting that $FeCl_3$ interacts preferentially with accessible pore domains and external surface irregularities. This behavior indicates an initial redistribution of iron precursors across the

carbon matrix, potentially influencing subsequent adsorption or catalytic performance.

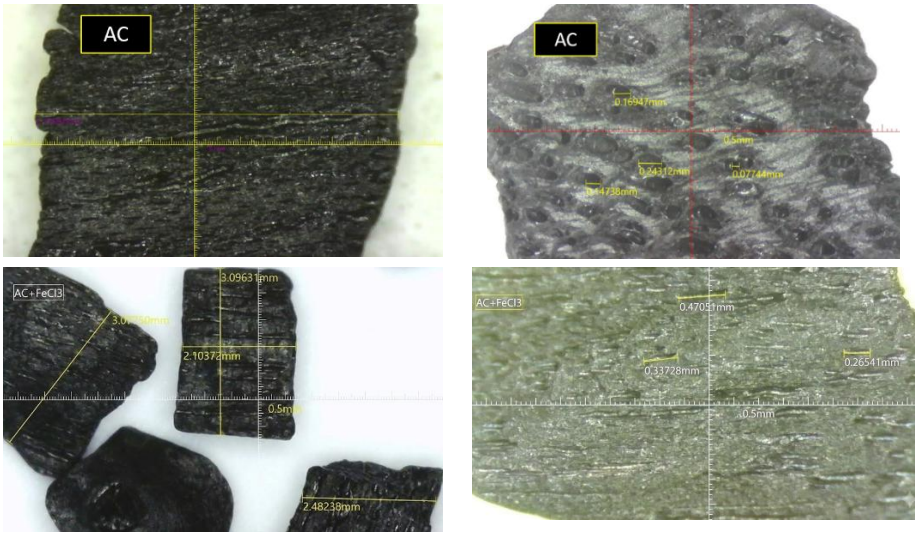

Fig. 1 − Optical microscopy images of activated carbon samples before and after $FeCl_3$ treatment.

Activated carbon was pretreated with 0.1 mol L^{-1} HCl to remove inorganic impurities and improve the accessibility of its porous structure. The material was washed with distilled water until the filtrate reached pH ≈ 6 and dried at 105°C for 6 h to constant weight.

Iron functionalization was performed by the wet impregnation method using a 0.5 mol L^{-1} $FeCl_3$ solution at a solid-to-liquid ratio of 1 g of activated carbon per 15 mL of solution. The suspension was stirred at room temperature for 6 h and aged for 18 h to promote the diffusion and anchoring of Fe^{3+} species within the porous structure. The pH was then adjusted to approximately 9.0 by adding 1 mol L^{-1} NaOH, promoting the n-situ formation of FeOOH on the carbon surface. After stirring for an additional 2 h and aging for 24 h, the material was recovered by filtration, dried at 105–110°C in order to obtain the iron-functionalized activated carbon (AC–$FeCl_3$) used in the adsorption experiments.

A synthetic phosphate solution containing ≈300 mg/L of phosphate ions (PO_4^{3-}) was prepared using sodium dihydrogen phosphate monohydrate ($NaH_2PO_4 \cdot H_2O$) as the phosphorus source.

The initial phosphate concentration was determined by an indirect complexometric titration with EDTA, following the precipitation of phosphate as magnesium ammonium phosphate ($MgNH_4PO_4$).

A 50 mL aliquot of the prepared solution was transferred into a beaker, followed by the addition of 0.25 g NH_4Cl and 10 mL of 0.05 M $MgCl_2$ solution.

The pH was adjusted to 9.2–9.5 using an ammonia buffer to promote the precipitation of phosphate as magnesium ammonium phosphate:

$$Mg^{2+}+NH_4^{+}+PO_4^{3-}\rightarrow MgNH_4PO_4\downarrow$$

The suspension was stirred for approximately 30 min and subsequently allowed to stand to promote precipitate maturation and settling. After filtration, the excess Mg^{2+} remaining in the filtrate was determined by complexometric titration with 0.05 M EDTA at pH≈10, using Eriochrome Black T as the indicator. The endpoint was identified by the color change from wine red to blue.

Considering the 1:1 stoichiometric ratio between Mg^{2+} and phosphate in magnesium ammonium phosphate, phosphate concentration was calculated from the corresponding molar difference and expressed as mg PO_4^{3-}/L.

The repeatability of the analytical procedure was assessed through three independent measurements of the same phosphate solution, expressed as mean ± standard deviation. The resulting relative standard deviation (RSD) of 1.84% demonstrates satisfactory repeatability of the method within the investigated concentration range.

The adsorption behavior of phosphate ions was investigated using three different adsorbent systems: unmodified activated carbon (AC), iron-functionalized activated carbon (AC–$FeCl_3$) under batch (static) conditions (Fig. 2), and iron-functionalized activated carbon (AC–$FeCl_3$) under continuous-flow (dynamic) conditions (Fig. 3).

For each experiment, 5 g of adsorbent was contacted with 50 mL of a ≈300 mg/L phosphate solution (PO_4^{3-}). All experiments were performed at room temperature. The batch experiments were conducted with a contact time of 2 h, while the suspensions were continuously agitated at 150 rpm. The dynamic experiments were carried out either in single-pass mode for 1 h or in double-pass (recirculation) mode for 2 h. This approach allowed the effect of contact time and repeated solution–adsorbent interactions on phosphate removal efficiency to be evaluated.

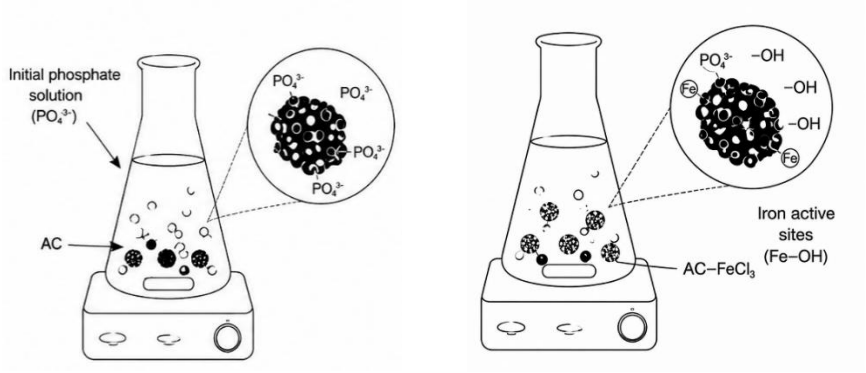

Fig. 2 − Schematic representation of batch phosphate adsorption experiments on unmodified activated carbon (AC) and iron-modified activated carbon (AC–$FeCl_3$).

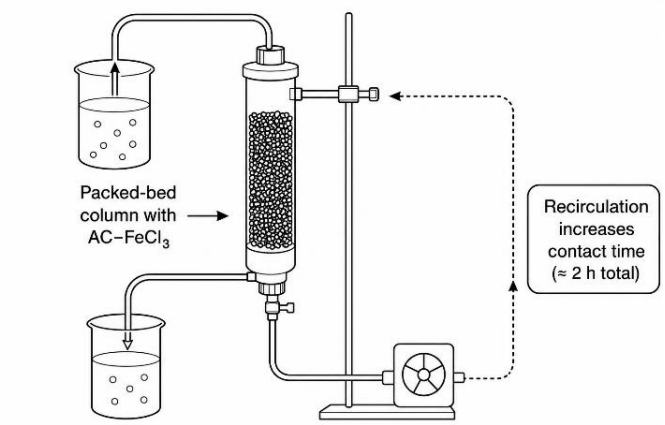

Fig. 3 − Schematic illustration of dynamic phosphate adsorption in a packed-bed column containing iron-modified activated carbon (AC–$FeCl_3$).

At the end of each experiment, the adsorbent was separated from the liquid phase by filtration. The resulting solutions were subsequently analyzed according to the analytical procedure described in the previous section to determine the residual phosphate concentration and evaluate the adsorption performance of each experimental configuration. The calculated RSD values ranged from 2.01 to 2.37%, demonstrating good analytical precision for phosphate concentrations

4. Results and discussion

A decrease in phosphate concentration was observed for all investigated adsorbents, indicating their capacity to remove phosphate ions from aqueous solutions. The adsorption efficiency varied according to the characteristics of the adsorbent and the operating system.

Under batch conditions, unmodified activated carbon (AC) decreased the phosphate concentration from 291.4 mg/L in the initial solution to 253.8 mg/L after adsorption, corresponding to a phosphate removal efficiency of approximately 12.9%. This relatively low removal efficiency indicates that the activated carbon surface exhibits only a limited affinity for anionic phosphate species. Surface functionalization of the activated carbon with iron significantly improved the adsorption performance. Under these conditions, the residual phosphate concentration decreased to 235.0 mg/L, corresponding to a removal efficiency of 19.4% (Fig. 4). The enhanced adsorption performance is attributed to the presence of iron-based active sites on the adsorbent surface, which promote phosphate uptake through electrostatic attraction, ligand exchange, and surface complexation mechanisms.

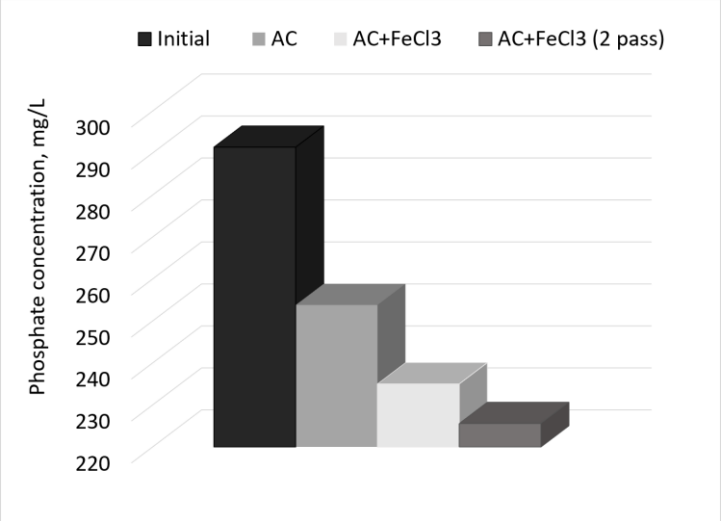

Fig. 4 − Influence of adsorbent modification and operating mode on phosphate adsorption performance.

Under dynamic operating conditions, the adsorption performance was strongly influenced by the contact time between the phosphate solution and the adsorbent. A single pass of the phosphate solution through the packed column, corresponding to a contact time of approximately 1 h, resulted in the lowest phosphate removal efficiency (6.5%), with a residual phosphate concentration of 272.6 mg/L (Fig. 5). The relatively low adsorption efficiency is attributed to the insufficient residence time of the solution in the column, which limited phosphate diffusion to the active adsorption sites.

When the solution was recirculated through the column, increasing the total contact time to 2 h, the adsorption performance improved markedly. Under these conditions, the phosphate solution passed twice through the adsorbent bed, reducing the residual phosphate concentration to 225.6 mg/L and increasing the phosphate removal efficiency to 22.6%. This value is comparable to, and slightly higher than, that obtained under batch conditions using the same iron-functionalized activated carbon. The improved adsorption performance is attributed to the combined effect of the longer contact time and enhanced mass transfer under continuous-flow conditions, which promoted more efficient interaction between phosphate ions and the iron-containing active sites on the adsorbent surface.

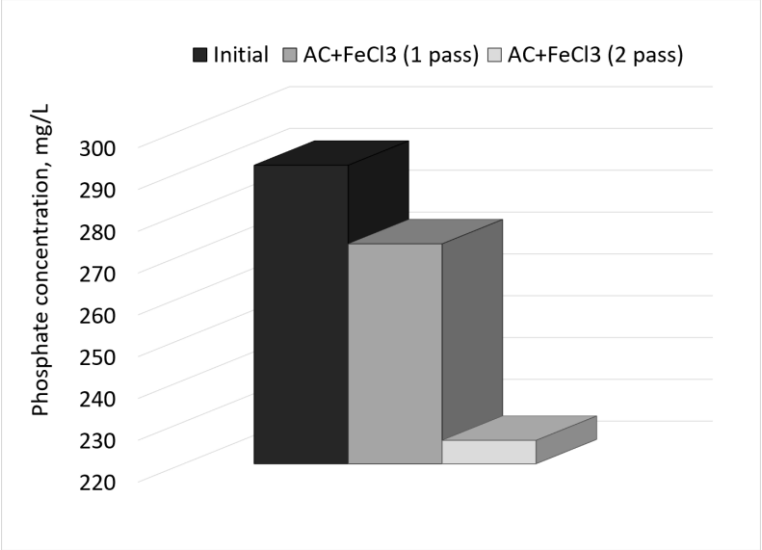

Fig. 5 − Effect of contact time (one-pass vs. two-pass operation) on phosphate removal using iron-modified activated carbon (AC–$FeCl_3$).

5. Conclusions

The results demonstrate that iron functionalization substantially enhances the phosphate adsorption performance of activated carbon by introducing iron-containing active sites that promote phosphate uptake through electrostatic attraction, ligand exchange, and surface complexation. Iron-functionalized activated carbon proved to be more effective than unmodified activated carbon for phosphate removal from aqueous solutions.

Under dynamic operating conditions, the adsorption performance was strongly dependent on the contact time between the phosphate solution and the adsorbent. Extending the contact time by recirculating the solution through the adsorption column resulted in improved phosphate removal efficiency due to enhanced interaction between the solution and the active adsorption sites. A longer contact time promoted mass transfer to the adsorbent surface and enabled more effective utilization of the available active sites, leading to a higher adsorption capacity. These results highlight the importance of solid–liquid contact time as a key operating parameter in fixed-bed adsorption processes.

The experimental results are consistent with the trends reported in the literature (Seo *et al.*, 2025), confirming the beneficial effect of iron functionalization on phosphate adsorption. Although the present study was conducted using highly concentrated phosphate solutions representative of fertilizer-industry wastewaters, the proposed adsorbent showed promising performance under both batch and dynamic operating conditions.

Overall, iron-functionalized activated carbon can be considered a promising material for the treatment of phosphorus-rich wastewaters generated by the fertilizer industry. Future research should focus on optimizing the functionalization procedure, investigating adsorption kinetics and equilibrium under different operating conditions, and evaluating adsorbent regeneration and reuse to assess its long-term practical applicability.

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ÎNDEPĂRTAREA IONILOR FOSFAT DIN APELE UZATE ALE INDUSTRIEI ÎNGRĂȘĂMINTELOR UTILIZÂND CĂRBUNE ACTIV FUNCȚIONALIZAT CU FIER

(Rezumat)

Efluenții cu un conținut ridicat de fosfor generați în procesul de fabricare a îngrășămintelor necesită tehnologii eficiente de tratare pentru a reduce impactul acestora asupra mediului. În acest studiu a fost evaluată adsorbția ionilor fosfat utilizând un material tip cărbune activ înainte și după funcționalizarea suprafeței cu fier, atât în regim static, cât și în regim dinamic cu curgere continuă. Adsorbantul modificat a fost obținut printr-o metodă simplă de impregnare umedă, în vederea introducerii unor centri activi pe bază de fier capabili să îmbunătățească reținerea ionilor fosfat. A fost investigată influența modului de operare și a timpului de contact solid–lichid asupra performanței

procesului de adsorbție. Funcționalizarea suprafeței a îmbunătățit reținerea ionilor fosfat prin mecanisme de atracție electrostatică și complexare la suprafață. În condiții de curgere continuă, prelungirea timpului de contact prin recircularea soluției a condus la creșterea eficienței de adsorbție, evidențiind importanța timpului de rezidență în sistemele cu strat fix. Rezultatele obținute arată că materialul propus este adecvat pentru tratarea apelor uzate cu concentrații ridicate de fosfor și că adsorbția în regim dinamic poate asigura performanțe comparabile sau chiar ușor superioare celor obținute în regim static, atunci când este asigurat un timp de contact suficient.