

ORIGINAL ARTICLE

Development of Chitosan–Activated Carbon Composite Adsorbent Films for Fe²⁺ Removal: A Full Factorial Design Approach

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ABSTRACT – Iron (Fe)-contaminated wastewater from industrial activities poses significant environmental challenges, motivating the development of efficient and sustainable treatment technologies. This study investigated a chitosan–activated carbon composite adsorbent film for Fe²⁺ removal using a 2³ full factorial design. The material was prepared by solvent casting, with polyethylene glycol (PEG) used as a pore-forming agent and activated carbon as the principal adsorbent. The effects of PEG concentration, activated carbon loading, and stirring time on residual Fe concentration were evaluated. The term “membrane” in the original study is used here only in the fabrication sense; the material was evaluated as an adsorptive film rather than as a size-selective nanofiltration or microfiltration membrane. Direct porosity or pore-size characterization was not performed, so the proposed roles of PEG in pore formation and of chitosan/activated carbon in Fe²⁺ uptake are interpreted from the fabrication procedure, adsorption trends, and FTIR observations rather than from direct pore-structure measurements. FTIR spectra were consistent with retention of characteristic hydroxyl (–OH), amino (–NH₂), C–N, and C–O–C functional groups, although baseline drift and spectral noise limit the conclusiveness of the FTIR evidence. Among the eight experimental runs, the lowest residual Fe concentration observed was 7.750 mg L^{–1} at 750 mg PEG, 40 wt% activated carbon, and 4 h stirring. The unreplicated factorial model gave R² = 0.924, but the overall model and individual factor effects were not statistically significant at the 95% confidence level (*p* > 0.05), with only one residual degree of freedom. Accordingly, the reported formulation is the best-performing experimental run within the tested design space, not a statistically validated optimum. Replicated experiments, porosity characterization, and microscopy are recommended to establish the controlling factors and adsorption mechanism more rigorously.

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INTRODUCTION

Industrial development, particularly in mining, metallurgy, and mineral processing, has significantly increased the generation of wastewater containing dissolved contaminants, including metal ions [1], [2]. Among these contaminants, iron (Fe) is frequently encountered in industrial aqueous streams and is widely targeted in water/wastewater treatment research involving Fe-bearing materials and Fe-associated interactions [3]. Although iron chemistry in water commonly involves pH-dependent hydrolysis/precipitation behavior (e.g., precipitation of iron hydroxides above mildly acidic pH) [4], excessive dissolved iron is generally treated prior to discharge because it can degrade water quality and produce undesirable solids [4].

Various treatment technologies have been developed for removal of aqueous contaminants, and adsorption-based processes and membrane-based separations are widely investigated for wastewater purification [5], [6], including systems designed specifically for heavy-metal-contaminated water [7], [2]. Adsorptive membranes and mixed-matrix/porous membranes are often highlighted because they can couple separation and sorption while enabling process optimization and reusability studies [3], [5], [6]. At the same time, fabrication can introduce practical limitations such as particle agglomeration, surface defects/ruptures, and performance variability, which can complicate recovery and repeated use [5]; additionally, maintaining structural stability while tuning pore formation is emphasized as a critical design balance in porous adsorbent development [3]. These limitations have encouraged the development of composite membrane adsorbents that integrate adsorption functionality into a membrane structure [5], [6].

Chitosan is widely used as a biopolymer platform in composite adsorbents and related functional materials because it is biocompatible/biodegradable and provides readily modifiable chemical functionality [8], with demonstrated applicability in wastewater-treatment composites (including PEG-modified chitosan-based systems) [1]. To enhance performance, porous or mixed-matrix approaches commonly incorporate additives: activated carbon particles are used as

fillers in mixed-matrix membranes and can measurably affect permeability/flux and separation performance [5], while polyethylene glycol (PEG) is frequently used as a pore-forming or morphology-tuning additive in membrane fabrication to increase flux/permeability and mitigate agglomeration tendencies [5] and to generate porous membrane substructures [9], consistent with its use as a pore former to improve membrane porosity in adsorptive membrane preparation [6].

Previous studies have demonstrated the potential of composite membranes and statistically designed experiments to optimize performance-relevant properties (e.g., flux/permeability, rejection/removal) by varying formulation and fabrication factors such as PEG and activated carbon content [5], [6]. Full factorial design and related design-of-experiments (DoE) approaches are specifically used to quantify not only main effects but also interaction effects among variables, which is an advantage over one-factor-at-a-time experimentation [10], [11].

To address these limitations, this study investigates the fabrication of chitosan-activated carbon composite adsorbent films for Fe^{2+} adsorption by simultaneously evaluating the effects of activated carbon loading, PEG concentration, and stirring time using a full factorial experimental design. Such factorial approaches are widely described as systematic methods that enable estimation of main and interaction effects among multiple factors [10], [11], and have been applied to membrane/material optimization problems involving PEG and carbonaceous fillers [5]. The findings are expected to contribute to the development of efficient composite membrane adsorbents for wastewater treatment by linking fabrication variables to membrane structure and performance, consistent with broader efforts to rationally tune pore structure, stability, and adsorption-relevant functionality [3], [5].

EXPERIMENTAL METHOD

Materials and Instruments

Chitosan (4 wt%) was used as the membrane matrix, while activated carbon served as the adsorbent filler. Polyethylene glycol (PEG) was employed as a pore-forming agent, and 2 wt% acetic acid was used as the solvent for chitosan dissolution. Synthetic Fe^{2+} solution with an initial concentration of $26.0646 \text{ mg L}^{-1}$ was prepared using analytical-grade reagents and deionized water.

The term “membrane” is used here to describe the solvent-cast geometry of the composite material; the material was evaluated primarily as an adsorptive film rather than as a size-selective nanofiltration or microfiltration membrane. Accordingly, pore-size-dependent separation metrics were outside the scope of the present work. Direct porosity characterization (e.g., BET surface-area analysis or mercury intrusion porosimetry) was not performed and is acknowledged as a limitation. The roles of PEG as a pore-forming additive and activated carbon as an adsorbent are therefore inferred from the fabrication procedure and observed adsorption trends rather than established by direct pore-structure measurements.

The functional groups of chitosan, PEG, and the fabricated composite membranes were characterized using Fourier Transform Infrared Spectroscopy (FTIR). Iron concentrations before and after adsorption were determined using Atomic Absorption Spectroscopy (AAS) at a wavelength of 248.3 nm following SNI 6989.4:2009. Calibration was performed using standard Fe solutions before sample analysis.

Method and Procedure

Composite membranes were prepared using the solvent casting method. Chitosan was dissolved in 2 wt% acetic acid under continuous stirring at 60°C until a homogeneous solution was obtained. Activated carbon was gradually added at concentrations of 20 and 40 wt% relative to the chitosan mass, followed by the addition of PEG at 650 or 750 mg. The suspension was continuously stirred for either 2 or 4 h according to the experimental design to ensure uniform dispersion of the activated carbon within the polymer matrix.

The resulting casting solution was poured onto Petri dishes and dried at 30°C until constant weight. The dried membranes were subsequently washed with distilled water at 85°C to remove PEG and generate a porous membrane structure. Finally, the membranes were rinsed thoroughly with distilled water and dried prior to characterization and adsorption experiments.

Before adsorption testing, the membranes were immersed in distilled water for 24 h to stabilize their structure. Adsorption experiments were conducted using a vacuum filtration system consisting of a Büchner funnel, vacuum flask, and vacuum pump. A total of 100 mL of Fe^{2+} solution ($26.0646 \text{ mg L}^{-1}$) was filtered through the membrane under constant vacuum pressure for 60 min. The permeate was collected and analyzed to determine the residual Fe concentration.

The effects of PEG concentration (X_1), activated carbon loading (X_2), and stirring time (X_3) on Fe removal were evaluated using a 2^3 full factorial design. Each factor was investigated at two levels, resulting in eight experimental combinations as presented in Table 1.

Table 1. Experimental design matrix and factor levels used in the 2^3 full factorial design for the fabrication of chitosan-activated carbon composite membranes.

Factor	Symbol	Low (-1)	High (+1)
PEG concentration (mg)	X1	650	750
Activated carbon (wt%)	X2	20	40
Stirring time (h)	X3	2	4

The experimental response was the residual Fe concentration after adsorption, measured by AAS. Statistical analysis, including ANOVA, parameter estimation, effect tests, residual analysis, prediction profiling, and optimization, was performed using JMP software.

The factorial design generated eight experimental runs, as presented in Table 2.

Table 2. Experimental matrix of factorial design.

Run	X1 (mg)	X2 (wt%)	X3 (h)
1	750	40	4
2	650	40	4
3	750	40	2
4	750	20	2
5	650	40	2
6	650	20	2
7	750	20	4
8	650	20	4

RESULT AND DISCUSSION

Visual Characteristics

The macroscopic appearance of the fabricated membrane is presented in Figure 1. Visual inspection provides a preliminary assessment of membrane quality before detailed physicochemical characterization because it enables rapid identification of gross non-uniformities arising from fabrication and mixing (e.g., visible inhomogeneity that can reflect filler dispersion issues) that may influence subsequent membrane performance in separation/adsorptive applications [12], [13]. Although this evaluation cannot reveal pore size distribution or internal morphology—which are typically resolved by structural/morphological characterization (e.g., microscopy) and are central to membrane selectivity and transport—macroscopic observations (e.g., apparent homogeneity and absence of obvious discontinuities) can still provide an initial indication that membrane preparation and casting produced a continuous film suitable for subsequent characterization [12], [14].

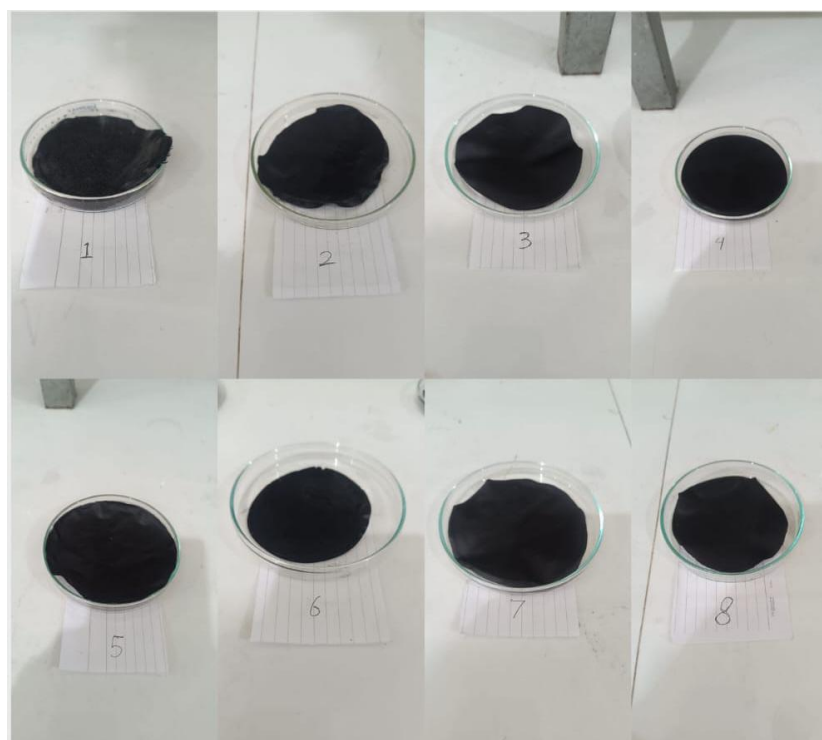


Figure 1. Macroscopic appearance of the fabricated chitosan/activated carbon membrane.

As shown in Figure 1, the eight fabricated samples were consistently black, consistent with the incorporation of a carbonaceous filler (activated carbon) into the polymer matrix. However, the samples were not uniform in shape or edge integrity: sample 1 shows a frayed, torn edge on one side, and several samples (e.g., 3, 7, 8) show visible folding or creasing rather than a flat, taut film, with noticeable variation in size and edge regularity across the set. This indicates that while color distribution appears consistent, the casting and drying process did not reliably produce a dimensionally uniform film across replicates, which we address directly rather than describe as uniform. More broadly, polymer nanocomposite/mixed-matrix membrane performance is commonly linked to achieving good filler distribution and minimizing aggregation/defect formation during preparation [12], [13], and the observed shape variability suggests this process still needs optimization.

Most of the eight samples exhibited a paper-like texture, though as noted above, folding and creasing were visible in several samples (Figure 1, samples 3, 7, and 8), which points to warping or handling deformation rather than a fully flat, dimensionally intact film in every case. A dimensionally intact, continuous film was achieved for at least some of the samples, consistent with successful membrane formation via solution processing routes used for polymeric and biopolymer-based membranes [18], [19], but the batch-to-batch shape variability visible in Figure 1 indicates the casting and drying conditions were not fully controlled across replicates.

The surface of individual samples appeared reasonably smooth by eye, but given the shape and edge inconsistencies noted above, we no longer describe the batch as uniform at the macroscopic scale; this is a fabrication-repeatability issue future work should address, for example by standardizing casting volume, dish geometry, and drying conditions across replicates. Macroscopic homogeneity within a given sample remains a reasonable proxy for the absence of gross casting defects, and controlling surface characteristics and processing-induced heterogeneity are recurrent design considerations in polymer composite membranes [13], [18], but we do not claim this was achieved consistently across all eight samples here.

No visible cracks, tears, air bubbles, pinholes, or other macroscopic defects were observed across the membrane surface. While the functional consequence of sub-macroscopic defects must be verified using appropriate characterization, membrane separation principles emphasize that selectivity and sieving performance depend on a continuous selective medium with controlled, appropriately distributed transport pathways rather than uncontrolled large-scale discontinuities [12], [14]. In this context, a defect-free macroscopic appearance provides preliminary support that the casting/formation procedure produced a continuous composite membrane appropriate for subsequent physicochemical and performance testing [13], [18].

Effect of PEG Concentration on Fe Adsorption Performance

The effect of polyethylene glycol (PEG) concentration on the adsorption performance of the fabricated membranes is summarized in Table 3, while the corresponding trends are illustrated in Figure 2. PEG was incorporated as a pore-forming additive to modify the membrane microstructure and thereby influence the accessibility of adsorption sites, consistent with the widely reported use of PEG as a pore former (and related pore-forming polymeric additives) in phase-inversion membrane fabrication to increase porosity and alter transport properties [21], [22]. Notably, PEG is also used in other membrane-modification modes, so the specific mechanism depends on whether PEG is blended as a sacrificial additive or immobilized at the surface [23].

Both the tabular (Table 3) and graphical (Figure 2) presentations of these data are retained deliberately: the table reports the exact numerical values needed for reproducibility and further data re-use, while the figure facilitates rapid visual comparison of trends across factor levels, which is a common and accepted presentation style in factorial-design and DoE studies; the same rationale applies to Table 4/Figure 3 and Table 5/Figure 4.

As presented in Table 3, increasing the PEG content from 650 to 750 mg did not produce a uniform improvement in Fe removal under all experimental conditions. For membranes containing 20 wt% activated carbon, only minor differences in the residual Fe concentration were observed regardless of the stirring time, indicating that increasing PEG concentration alone had a limited effect on adsorption performance. This behavior suggests that the relatively low amount of activated carbon restricted the availability of active adsorption sites, thereby limiting the influence of PEG-driven structural changes; this interpretation is consistent with the general principle that pore-former-driven increases in porosity primarily improve performance when they translate into effective access to functional surfaces [21], [24].

A different trend was observed for membranes containing 40 wt% activated carbon. Under these conditions, increasing the PEG concentration from 650 to 750 mg resulted in a noticeable reduction in the residual Fe concentration at both stirring times. After 2 h of adsorption, the Fe concentration decreased from 11.302 to 7.913 mg L⁻¹, whereas after 4 h it decreased from 9.621 to 7.750 mg L⁻¹. These results indicate that the beneficial effect of PEG became more pronounced when a greater amount of activated carbon was available, suggesting a coupled contribution of membrane porosity (mass-transfer pathways) and adsorbent loading (site availability), which aligns with literature showing that pore-forming additives can increase porosity and thereby enhance transport through more open structures and higher pore density [21], [22].

The enhanced adsorption performance can be explained by the role of PEG as a sacrificial pore-forming agent. During fabrication, water-soluble PEG can be incorporated into the polymer dope and subsequently removed during post-treatment (e.g., washing), leaving voids and interconnected pores; for example, PEG removal by washing is explicitly used to generate porosity in cellulose acetate/activated carbon composite mats [25], and additive leaching is a known concern/feature for conventional hydrophilic polymeric additives used in nonsolvent-induced phase separation (NIPS)

membranes [26]. An appropriate PEG concentration can therefore increase membrane porosity and accessible internal surface area, facilitating solute diffusion into the membrane interior and improving access to adsorption sites—consistent with evidence that PEG can create porous structures (via removal/combustion) and substantially increase specific surface area and pore volume in porous solids [24], and with membrane literature linking pore-former addition to increased porosity and altered permeation/transport behavior [21], [22].

We did not run this experiment to a defined target concentration or verify equilibrium/saturation directly: each condition was tested at a single, fixed adsorption time, so we cannot state whether any of the eight runs had reached saturated Fe uptake or would have continued to improve with longer contact time. Reporting adsorption performance against a specific discharge or regulatory target, and running a proper contact-time series to establish equilibrium, are both needed to make this comparison meaningful in a future revision.

Table 3. Effect of PEG Concentration on the Residual Fe Concentration after Adsorption (mg L^{-1})

PEG (mg)	20 wt% AC (2 h)	20 wt% AC (4 h)	40 wt% AC (2 h)	40 wt% AC (4 h)
650	9.858	11.313	11.302	9.621
750	10.411	10.654	7.913	7.750

However, the results also demonstrate that increasing PEG concentration does not necessarily lead to proportional improvements in adsorption performance. This nonlinearity is consistent with broader membrane-additive studies reporting an optimum additive content beyond which further increases may yield diminishing returns or introduce countervailing effects on overall separation performance (e.g., trade-offs among permeability, selectivity, and susceptibility to surface-related losses), rather than monotonic improvement with additive concentration [21]. Thus, the influence of PEG depends not only on its concentration but also on its interaction with other membrane components—particularly the activated carbon content—and whether increased porosity translates into additional utilizable adsorption capacity [21], [24].

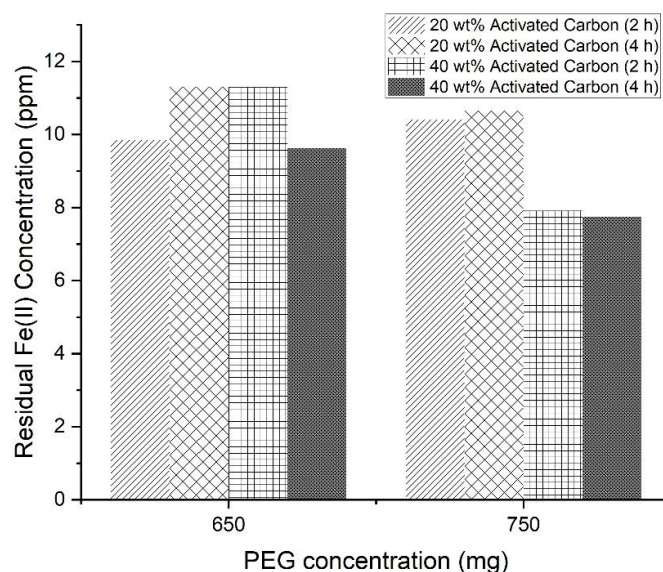


Figure 2. Effect of PEG Concentration on the Residual Fe Concentration

The adsorption trends shown in Figure 2 further support this interpretation. The lowest residual Fe concentration (7.750 mg L^{-1}) was obtained using the membrane containing 750 mg PEG, 40 wt% activated carbon, and a 4 h stirring time, whereas the highest residual concentration (11.313 mg L^{-1}) was observed for the membrane containing 650 mg PEG, 20 wt% activated carbon, after 4 h of adsorption. These findings demonstrate that adsorption performance was governed by the combined effects of membrane porosity and adsorbent loading rather than PEG concentration alone—consistent with the general understanding that pore-former-induced porosity improves mass transfer/penetration only insofar as it increases access to available internal surfaces and functional sites [21], [24].

Effect of Activated Carbon Content on Fe Adsorption Performance

The effect of activated carbon loading on the adsorption performance of the fabricated membranes is presented in Table 4, while the corresponding trends are illustrated in Figure 3. Activated carbon serves as the primary adsorbent in

the membrane owing to its high specific surface area and porous structure, which together increase the accessible adsorption sites for aqueous contaminants and promote adsorption performance [27]–[29]. In addition, oxygen-containing surface functionalities commonly present on activated-carbon-based materials (e.g., C–O containing groups) are frequently implicated in the adsorption of dissolved species via surface interactions in water treatment contexts, including for inorganic contaminants [27], [29].

Overall, increasing the activated carbon content from 20 wt% to 40 wt% improved the adsorption performance under most experimental conditions. The improvement was particularly evident for membranes containing 750 mg PEG, where the residual Fe concentration decreased from 10.411 to 7.913 mg L⁻¹ after 2 h of stirring and from 10.654 to 7.750 mg L⁻¹ after 4 h. These results indicate that increasing the amount of activated carbon provided a greater number of accessible adsorption sites, thereby enhancing Fe removal efficiency; this interpretation is consistent with the general adsorption principle that increasing accessible surface area/active sites can increase uptake when mass transfer and site accessibility are not limiting [27], [28].

Table 1 shows the relationship between operating pressure (2–30 kPa) and flux values for styrofoam membranes with thicknesses of 0.35 mm, 0.75 mm, and 1.20 mm, as well as the control glass fiber membrane (GF/b) with a thickness of 0.67 mm. In general, all membrane variations exhibit an increase in flux with increasing pressure. This behavior is consistent with the basic principle of pressure-driven filtration, where a higher driving force enhances the rate of water permeation through membrane pores.

However, significant differences are observed among the membrane thicknesses. The 0.35 mm membrane exhibits the highest flux values across the entire pressure range. At 30 kPa, the flux reaches 55.27×10^{-3} L/h·m². The thinner membrane results in lower flow resistance, allowing water to pass more easily through the membrane structure. This indicates that the diffusion distance and flow path length are shorter compared to thicker membranes. For the 0.75 mm membrane, the flux increases linearly with pressure but remains consistently lower than that of the 0.35 mm membrane. At 30 kPa, the flux reaches 14.33×10^{-3} L/h·m². This decrease is attributed to the increased membrane thickness, which leads to greater hydraulic resistance. The 1.20 mm membrane, being the thickest, shows the lowest flux at low pressures, even approaching zero at 2–4 kPa. This indicates that at low pressures, the driving force is insufficient to overcome the structural resistance of the membrane. At 30 kPa, the flux increases to 12.60×10^{-3} L/h·m², but it remains significantly lower than that of the thinner membrane. The control GF/b membrane (0.67 mm) shows a significant increase in flux with increasing pressure, reaching 37.42×10^{-3} L/h·m² at 30 kPa. Although this value is still lower than that of the 0.35 mm styrofoam membrane, it is higher than those of the 0.75 mm and 1.20 mm membranes. This indicates that pore structure and material characteristics strongly influence permeability, not merely membrane thickness.

The reduced rate of flux increase within the pressure range of 22–24 kPa is associated with the initial membrane compaction phenomenon under moderate pressure, which temporarily increases hydraulic resistance. Once the pressure exceeds 24 kPa, the higher driving force overcomes this resistance, causing the flux to increase significantly again. This phenomenon indicates that the membrane response to pressure is not entirely linear and is influenced by pore structure dynamics during the filtration process. Such behavior is normal in fiber-based filtration systems and does not indicate system failure, but rather a structural response to applied pressure. The efficiency value of the membrane can be seen in the following table.

Table 4. Effect of activated carbon loading on the residual Fe concentration following adsorption under different PEG concentrations and stirring times (mg L⁻¹).

Activated Carbon (wt%)	PEG 650 mg, 2 h	PEG 650 mg, 4 h	PEG 750 mg, 2 h	PEG 750 mg, 4 h
20	9.858	11.313	10.411	10.654
40	11.302	9.621	7.913	7.750

The enhanced adsorption can be attributed to the larger surface area available for adsorption as the activated carbon loading increased, consistent with the established role of carbonaceous adsorbents in water treatment [27], [28]. More broadly, adsorption performance typically depends on (i) the number of accessible adsorption sites and (ii) the ability of the adsorbate to reach them through the pore network or membrane morphology; thus, increases in filler loading may enhance adsorption when they effectively increase accessible area and do not impede transport [28], [30]. In parallel, the use of polymeric additives such as PEG is widely reported to influence membrane pore formation and hydrophilicity, which can facilitate solute transport to embedded sorbent sites and thereby improve overall removal performance under suitable formulations [31], [32], [30].

However, the relationship between activated carbon loading and adsorption performance was not entirely linear. Under the condition of 650 mg PEG and 2 h stirring, increasing the activated carbon content unexpectedly increased the residual Fe concentration from 9.858 to 11.302 mg L⁻¹, indicating lower adsorption efficiency. This behavior suggests that simply increasing the amount of activated carbon does not necessarily improve membrane performance, because higher carbonaceous-filler loadings in polymer matrices can introduce processing–structure limitations (e.g., reduced effective accessibility of adsorption sites) when dispersion and morphology are not optimized [33], [30]. In particular, inadequate dispersion and/or particle–particle association at elevated carbonaceous filler fractions can reduce the effective surface area available for adsorption and constrain pore accessibility within composites, thereby diminishing the benefits expected from higher nominal adsorbent loading [33], [30]. More generally, nanocomposite membrane performance is

frequently governed by trade-offs among additive/filler loading, morphology development, and transport resistances, rather than by filler fraction alone [32], [30].

The remaining experimental conditions exhibited the expected trend. At 650 mg PEG with 4 h stirring, increasing the activated carbon content reduced the residual Fe concentration from 11.313 to 9.621 mg L⁻¹. Similar improvements were observed for both adsorption times when 750 mg PEG was used, demonstrating that the beneficial effect of activated carbon became more pronounced when the membrane possessed a more porous structure. This observation is consistent with literature describing PEG as an additive that can alter membrane formation and hydrophilicity and, in turn, affect pore development and transport behavior relevant to separation and adsorption [31], [32], [30]. In this sense, PEG and activated carbon can function synergistically: PEG-mediated morphology/hydrophilicity improvements can enhance Fe-ion diffusion and access into/through the membrane structure, while activated carbon provides the principal adsorption sites responsible for contaminant uptake [27], [28], [30].

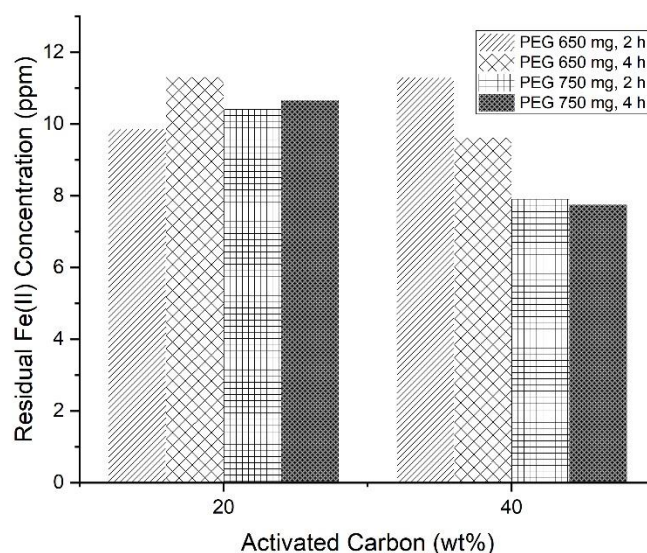


Figure 3. Effect of activated carbon loading on the residual Fe concentration.

As shown in Figure 3, the lowest residual Fe concentration (7.750 mg L⁻¹) was achieved using the membrane containing 40 wt% activated carbon, 750 mg PEG, and a 4 h stirring time, whereas the highest residual concentration among the membranes containing 40 wt% activated carbon (11.302 mg L⁻¹) occurred at 650 mg PEG after 2 h of adsorption. These results demonstrate that adsorption efficiency is governed not only by activated carbon loading but also by membrane structure and adsorption time, aligning with broader evidence that transport limitations and morphology can control the extent to which added adsorption capacity is practically utilized in composite membranes [28], [30]. Therefore, higher adsorbent content can only be fully exploited when membrane morphology provides sufficient pore connectivity and accessibility for efficient mass transfer to sorbent surfaces [32], [30].

Compared with the initial Fe concentration of 26.0646 mg L⁻¹, all fabricated membranes exhibited considerable Fe removal, consistent with the contribution of activated carbon as the principal adsorbent component. The superior performance obtained with 40 wt% activated carbon demonstrates that increasing the number of adsorption sites can substantially enhance membrane adsorption capacity when accompanied by an appropriate membrane structure that supports accessibility and transport [28], [30]. Overall, these findings reinforce the need to balance adsorbent loading with pore formation (e.g., via PEG-mediated morphology control) to maximize accessibility of adsorption sites while preserving membrane integrity and enabling efficient diffusion throughout the membrane [31], [32], [30].

On the specific question of why activated carbon loading increased Fe removal in some conditions but decreased it in others (650 mg PEG, 2 h): we interpret this as a dispersion/accessibility effect rather than a straightforward dose-response relationship, but we did not perform a dispersion or porosity measurement (e.g., BET surface area, SEM cross-section) that would let us confirm this mechanistically rather than by inference from the adsorption data alone. This is a gap we flag directly rather than resolve, since resolving it would require additional characterization beyond what was collected for this study.

Effect of Stirring Time on Fe Adsorption Performance

The effect of stirring time during membrane preparation on the adsorption performance is presented in Table 5, while the corresponding trends are shown in Figure 4. Stirring time plays an important role in membrane fabrication because it influences the homogeneity of the casting (dope) solution and the dispersion of additives/fillers within the polymer matrix, which in turn governs membrane structure and performance outcomes [34–36]. In mixed-matrix and additive-containing

membranes, achieving a homogeneous dope and suppressing agglomeration/non-uniformity are repeatedly emphasized as key prerequisites for realizing the intended transport/adsorption functionality of the final membrane [33], [36], [37].

Overall, extending the stirring time from 2 to 4 h produced different effects depending on the membrane composition. This aligns with broader membrane-fabrication evidence that performance is typically controlled by interacting formulation and processing factors (e.g., polymer concentration, pore-former content, and filler loading) rather than a single factor acting independently; such interaction effects are commonly revealed when fabrication variables are varied concurrently (e.g., via design-of-experiments/response-surface approaches) [36], [38]. Moreover, because PEG is widely used as a pore-forming additive and can alter dope viscosity and phase-inversion behavior, its concentration can modulate how changes in stirring translate into morphology and performance [34], [35], [38], [39].

Table 5. Effect of stirring time on the residual Fe concentration after adsorption under different PEG concentrations and activated carbon loadings (mg L^{-1}).

Stirring Time (h)	PEG 650 mg, AC 20 wt%	PEG 650 mg, AC 40 wt%	PEG 750 mg, AC 20 wt%	PEG 750 mg, AC 40 wt%
2	9.858	11.302	10.411	7.913
4	11.313	9.621	10.654	7.750

For membranes containing 20 wt% activated carbon, increasing the stirring time resulted in only minor changes in the residual Fe concentration. In both PEG concentrations, the residual Fe concentration remained approximately $10\text{--}11 \text{ mg L}^{-1}$, suggesting that prolonged stirring provided limited improvement in adsorption performance. A mechanistic interpretation consistent with adsorption-membrane literature is that, when sorbent/filler content is relatively low, the number of accessible adsorption sites can remain the limiting factor, so improvements in dispersion/homogeneity from additional stirring may not translate into substantial gains in overall uptake [33], [37]. More generally, carbonaceous-filler dispersion quality is widely recognized as important to functional performance, but its impact can be constrained when the functional phase fraction is low [33], [37].

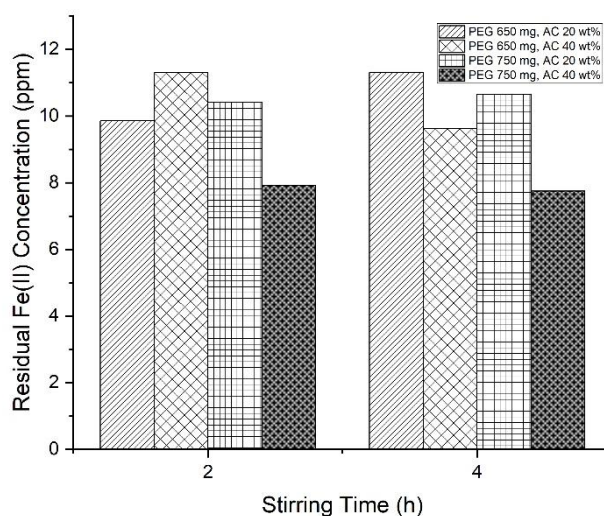


Figure 4. Effect of stirring time on the residual Fe concentration.

In contrast, membranes containing 40 wt% activated carbon exhibited a more pronounced response to longer stirring times. At 650 mg PEG, increasing the stirring time reduced the residual Fe concentration from 11.302 to 9.621 mg L^{-1} , representing the largest improvement among all experimental conditions. A similar, although less pronounced, reduction was observed for the membrane containing 750 mg PEG, where the residual Fe concentration decreased from 7.913 to 7.750 mg L^{-1} . These results are consistent with the general observation that better mixing/stirring can improve the distribution of a dispersed phase (including carbonaceous fillers/particles) and reduce aggregation, thereby improving accessibility of functional sites and the effectiveness of additive-enabled structures [33], [36], [37], [40]. In related carbon-containing systems, PEG has also been reported to promote dispersion stability of activated-carbon particles (i.e., helping maintain a more stable dispersion), supporting the plausibility that dispersion improvements can be mixing-sensitive and formulation-dependent [40].

The improved adsorption performance associated with longer stirring time can be attributed to more effective mixing of chitosan, PEG, and activated carbon before membrane casting. Adequate mixing promotes more uniform filler distribution and reduces the likelihood of localized particle aggregation, a mechanism that is widely invoked in mixed-matrix membranes to explain improved performance when the filler is well dispersed [33], [36], [37]. Additionally, PEG

is commonly used specifically as a pore-forming/structure-directing additive; after leaching/removal, PEG can contribute to pore development and pore connectivity, which can improve mass transfer to active sites in separation/adsorptive membranes [34], [39], [41]. Nevertheless, the relatively small improvement observed for the membrane containing 750 mg PEG and 40 wt% activated carbon suggests diminishing returns, which is compatible with literature in which an "optimum" region is often observed because multiple factors (pore structure, viscosity, demixing behavior, and filler distribution) must be balanced simultaneously [35], [36], [38], [39].

Conversely, excessively long stirring may increase the viscosity of the casting solution (and/or promote a highly viscous state), which can hinder mixing efficiency and favor particle–particle contacts that precede aggregation, potentially undermining the intended morphology and transport pathways [35], [42]. Independent experimental contexts similarly document that extended reaction/mixing times can be associated with substantial viscosity increases (e.g., "formation of a very viscous phase" as time proceeds), illustrating a general mechanism by which "more time" can become counterproductive beyond an optimum duration [42]. Therefore, while longer stirring can help homogenize the dope solution, the net effect on adsorption is expected to remain contingent on composition-dependent rheology and the coupled roles of PEG (as pore former/viscosity modifier) and activated carbon loading (as the adsorption-site provider) [34], [35], [39], [40].

As illustrated in Figure 4, the lowest residual Fe concentration (7.750 mg L^{-1}) was obtained using the membrane prepared with 750 mg PEG, 40 wt% activated carbon, and 4 h stirring, whereas the highest residual concentration (11.313 mg L^{-1}) was observed for the membrane containing 650 mg PEG, 20 wt% activated carbon, after 4 h of stirring. Taken together, these outcomes support the broader conclusion that adsorption performance is determined by a synergistic interaction between membrane composition (pore-former level and filler loading) and processing conditions (including stirring time), rather than stirring time alone [36], [38]. This perspective is consistent with the way membrane studies commonly optimize performance by jointly tuning additive content (e.g., PEG as pore former) and filler loading, because these variables collectively control dope homogeneity, phase inversion morphology, and functional-site accessibility [34], [36], [38–41].

Fourier Transform Infrared (FTIR) Analysis of Chitosan

The FTIR spectrum of the pristine chitosan used as the membrane matrix is presented in Figure 5. FTIR analysis was performed to confirm the presence of characteristic functional groups responsible for the adsorption properties of chitosan. As a deacetylated derivative of chitin, chitosan contains abundant hydroxyl ($-\text{OH}$) and amino ($-\text{NH}_2$) groups, which are widely reported as key reactive functionalities in chitosan's backbone and are central to its physicochemical behavior in aqueous media [44], [45]. These functionalities act as primary active sites for metal-ion uptake, with adsorption commonly attributed to a combination of electrostatic interactions (especially when amine groups are protonated) and coordination/complexation (chelation) with electron-donating heteroatoms (N and O) on chitosan [46–49]. Consistent mechanistic interpretations further emphasize that the amino and hydroxyl groups provide lone-pair electrons/coordination sites and can participate in ion exchange and binding energetics for metal ions [46, 50–52].

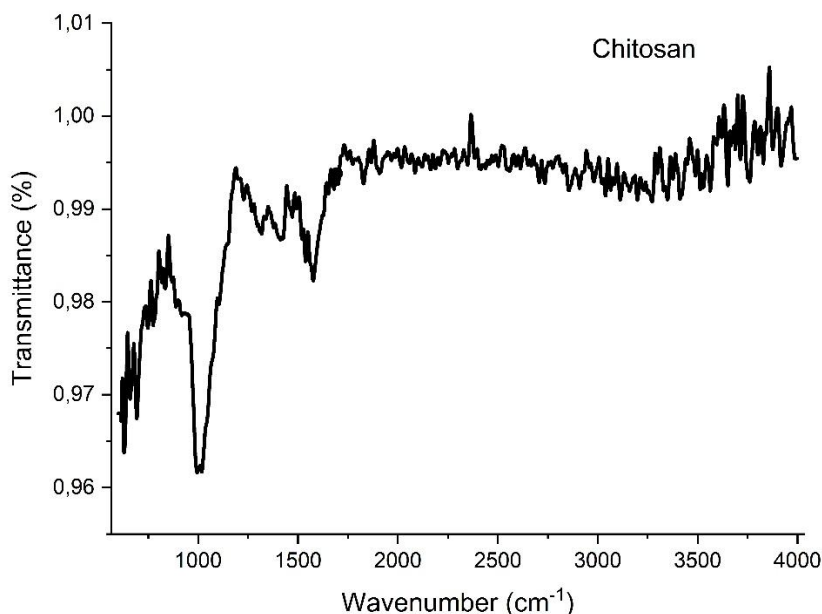


Figure 5. FTIR spectrum of pristine chitosan used as the polymer matrix for composite membrane fabrication.

The spectrum exhibited a broad absorption band centered at 3272.13 cm^{-1} , corresponding to overlapping stretching vibrations of hydroxyl ($-\text{OH}$) and amino ($-\text{NH}$) groups, a feature repeatedly reported for chitosan and chitosan-based materials in the $\sim 3200\text{--}3500 \text{ cm}^{-1}$ region [44], [47], [50], [53]. The pronounced broadness of this band is characteristic

of intermolecular and intramolecular hydrogen bonding in polysaccharide networks rich in -OH/-NH functionalities, and such hydrogen-bonding-driven broadening is commonly discussed for chitosan-containing systems [45], [53], [54]. The importance of these groups to adsorption is consistent with literature describing -OH/-NH sites as electron-donating/coordination-capable functionalities and/or electrostatically active sites (upon protonation) that contribute directly to binding of ionic species in water treatment applications [44], [48], [50].

A distinct absorption peak at 1575.59 cm^{-1} was assigned to the bending vibration of primary amino (-NH_2) groups (often discussed in the $\sim 1565\text{--}1606\text{ cm}^{-1}$ range for chitosan, depending on formulation and analysis conditions) [50], [53]. The presence of an amine-associated band in this region is also consistent with reports using FTIR to evidence deacetylation-related features in chitosan, including the identification of free amino group contributions (e.g., around $\sim 1598\text{--}1600\text{ cm}^{-1}$) alongside amide-related signals [44], [53], [54]. Since deacetylation increases the availability of amino functionalities, this observation aligns with the broad consensus that -NH_2 groups are principal binding sites for metal ions via coordination/complexation (chelation) and that adsorption frequently involves N/O donor atoms on chitosan [44], [46], [48], [49], [52].

Additional absorption bands at 1412.11 cm^{-1} and 1308.95 cm^{-1} were attributed to vibrations associated with the chitosan backbone (commonly discussed in chitosan-containing spectra as part of bands reflecting polysaccharide structure and C–N/amide-related contributions in the $\sim 1230\text{--}1300\text{ cm}^{-1}$ region) [55], [53], [54]. In particular, amide III/C–N-associated regions are frequently reported around $\sim 1230\text{--}1300\text{ cm}^{-1}$ in biopolymer composites containing chitosan, supporting the assignment of the $\sim 1309\text{ cm}^{-1}$ band to C–N/amide-type vibrations rather than indicating new chemical functionalities [53]. Furthermore, the absorption band observed at 995.35 cm^{-1} corresponds to C–O/C–O–C vibrations associated with glycosidic/polysaccharide linkages, consistent with widely reported chitosan saccharide-region peaks (commonly in the $\sim 1150\text{--}900\text{ cm}^{-1}$ range) attributed to C–O–C and C–O stretching and $\beta\text{-(1}\rightarrow\text{4)}$ glycosidic features in chitosan [50], [53], [56]. (Notably, the precise position of these polysaccharide-region bands can vary across studies and sample preparation routes, with reports commonly emphasizing bands near ~ 1150 , $\sim 1075\text{--}1030$, and $\sim 898\text{--}894\text{ cm}^{-1}$ for glycosidic-related vibrations) [53], [56].

Overall, the FTIR spectrum demonstrates the presence of characteristic chitosan functional groups, including hydroxyl and amino functionalities and polysaccharide (glycosidic) backbone bands, consistent with established FTIR profiles of chitosan-based adsorbent materials [47], [50], [53], [56]. In chitosan systems where processing is primarily physical (or where no new covalent chemistry is introduced), the retention of the main chitosan FTIR features without emergence of new diagnostic bands is commonly interpreted as preservation of the polymer's core chemical structure, as reported in chitosan-based material preparations where characteristic peaks remain largely unchanged aside from intensity shifts or overlaps [50]. More importantly, the abundance of hydroxyl and amino groups provides numerous active binding sites that are repeatedly identified as dominant adsorption/coordination sites for metal ions in aqueous environments, including mechanisms combining electrostatic affinity (via protonated amines under appropriate pH) and coordination/complexation involving N/O donor atoms on chitosan [44], [46]–[48]. This mechanistic framework is also consistent with Fe(III)-relevant discussions in the literature where Fe(III) uptake/coordination is attributed (wholly or partly, depending on the specific chitosan system) to interactions with amino and hydroxyl functionalities as coordination atoms/sites [46], [49].

Fourier Transform Infrared (FTIR) Analysis of Polyethylene Glycol (PEG)

The FTIR spectrum of polyethylene glycol (PEG), used as the pore-forming agent during membrane fabrication, is presented in Figure 6. FTIR analysis was conducted to verify the characteristic functional groups of PEG and to confirm its chemical integrity prior to membrane preparation. Owing to its hydrophilic nature and high water solubility, PEG is widely employed as a pore-forming additive in polymeric membranes. During membrane fabrication, PEG is subsequently leached out during the washing step, generating interconnected pores that enhance membrane porosity and facilitate mass transport [57].

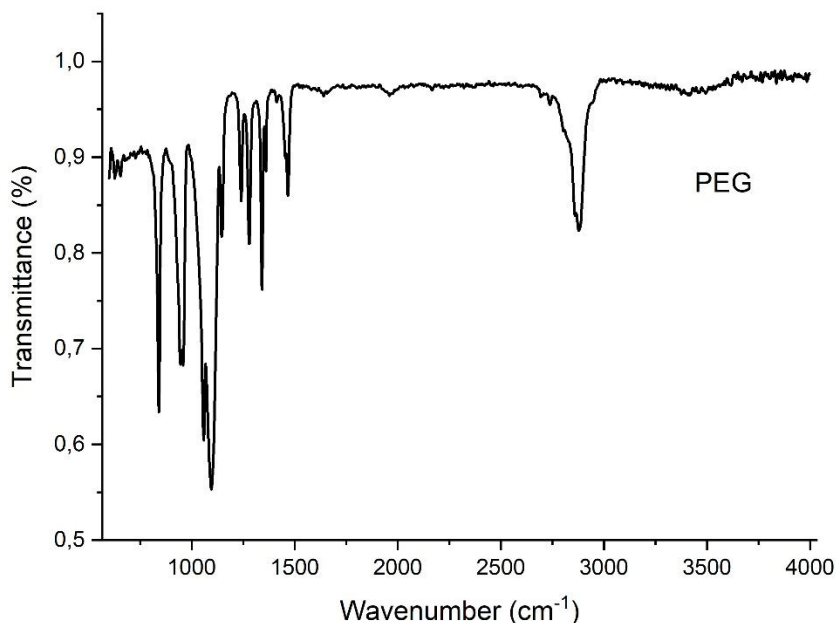


Figure 6. FTIR spectrum of polyethylene glycol (PEG) used as the pore-forming agent for composite membrane fabrication.

The FTIR spectrum exhibited a characteristic absorption band at 2877.80 cm^{-1} , corresponding to the stretching vibration of aliphatic C–H bonds from the methylene ($-\text{CH}_2-$) groups that constitute the polymer backbone. Additional absorption bands were observed at 1466.37 cm^{-1} and 1340.66 cm^{-1} , which are attributed to the bending and deformation vibrations of methylene groups, respectively. These absorption bands are characteristic of the polyethylene glycol molecular structure and confirm the integrity of its polymeric backbone.

The spectrum also exhibited characteristic vibrations associated with the ether linkage (C–O–C), which represents the fundamental repeating unit of PEG. The presence of this functional group confirms the polyether structure of the material and is closely related to its hydrophilic properties. Because of its high affinity for water, PEG can be readily removed from the polymer matrix during membrane washing, leaving behind interconnected voids that subsequently develop into membrane pores. This mechanism is responsible for improving membrane porosity, permeability, and the accessibility of adsorption sites within the composite membrane [57].

Furthermore, absorption bands observed at 958.06 cm^{-1} and 840.40 cm^{-1} correspond to skeletal vibrations of the PEG polymer chain, further confirming the characteristic molecular structure of polyethylene glycol. No unexpected absorption bands were detected, indicating that the PEG maintained its chemical stability before membrane fabrication.

Fourier Transform Infrared (FTIR) Analysis of the Composite Membrane

The FTIR spectrum of the fabricated composite film is presented in Figure 7. FTIR characterization was used to assess the retention of characteristic functional groups of chitosan, polyethylene glycol (PEG), and the carbon-containing component after film fabrication and to evaluate whether the characteristic functional groups of each component were preserved after membrane fabrication. Retention of these functional groups is essential because they govern the chemical interactions between the membrane surface and dissolved Fe ions during adsorption [58].

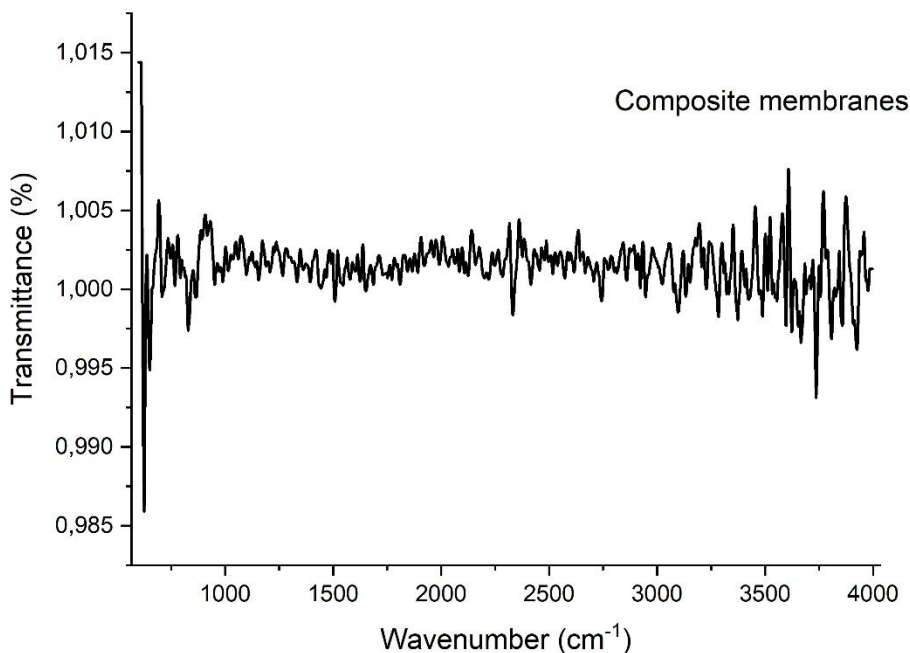


Figure 7. FTIR spectrum of the fabricated chitosan/activated carbon composite membrane.

The composite membrane exhibited a broad absorption band within the 3424.37–3284.05 cm^{-1} region, corresponding to the overlapping stretching vibrations of hydroxyl ($-\text{OH}$) and amino ($-\text{NH}$) groups. Compared with pristine chitosan, this band became broader and slightly shifted, indicating the formation of intermolecular hydrogen bonding among chitosan, PEG, and activated carbon within the composite matrix. Such spectral changes suggest that the membrane components interact primarily through physical interactions rather than the formation of new covalent bonds, thereby preserving the chemical integrity of the constituent materials.

Characteristic C–H stretching vibrations were observed at 2946.55 cm^{-1} and 2922.13 cm^{-1} , confirming the presence of the polymeric backbone after membrane fabrication. Meanwhile, absorption bands at 1652.41 cm^{-1} and 1545.92 cm^{-1} correspond to the amide and amino groups characteristic of chitosan. The preservation of these functional groups is particularly important because amino and hydroxyl groups constitute the primary adsorption sites responsible for binding Fe ions through electrostatic attraction and surface complexation mechanisms.

Additional absorption bands at 1443.98 cm^{-1} and 1333.00 cm^{-1} were assigned to CH_2 bending and C–N stretching vibrations, respectively, while the bands at 1154.77 cm^{-1} and 950.94 cm^{-1} correspond to C–O–C and C–O vibrations associated with the polysaccharide backbone of chitosan and the ether structure of PEG. The persistence of these characteristic peaks demonstrates that the membrane fabrication process did not alter the fundamental chemical structures of the polymer components, indicating good chemical compatibility among the membrane constituents.

A weak absorption band was also observed at 828.67 cm^{-1} , which may be associated with carbonaceous structures originating from activated carbon. However, because activated carbon generally exhibits broad and less distinctive FTIR absorption due to its predominantly amorphous structure, this peak alone cannot be considered conclusive evidence of activated carbon incorporation. Instead, the FTIR results are consistent with incorporation of the carbon-containing component, but the present data are not sufficient to establish this conclusively; direct complementary characterization would be required.

It is further acknowledged that the FTIR spectra of the pristine chitosan and PEG (Figure 5 and Figure 6) display a degree of baseline drift, and the composite spectrum (Figure 7) exhibits a comparatively higher noise level; these features are attributed to the transmittance-mode measurement and limited control over sample film thickness. Consequently, while the retained peak positions support preservation of the constituent functional groups, the FTIR evidence alone is not fully conclusive regarding successful chitosan/PEG modification of the membrane matrix, and future work employing ATR-FTIR with baseline correction is recommended for clearer spectral resolution.

Statistical Analysis

The relationship between the experimental and predicted Fe concentrations is illustrated in the Actual versus Predicted Plot (Figure 8). Most of the experimental data points are distributed close to the diagonal line, indicating good agreement between the predicted and observed responses. This distribution suggests that the regression model adequately captures the overall trend of the experimental data, with only limited deviations between predicted and measured values.

The developed factorial model exhibited a coefficient of determination (R^2) of 0.92, indicating that approximately 92% of the variation in the response variable can be explained by the model. Such a high R^2 value generally reflects a strong goodness-of-fit and suggests that the selected process variables collectively account for most of the observed

exhibited the highest p-value ($p = 0.9423$). These results indicate that the observed variations in Fe adsorption could not be attributed to any individual factor with sufficient statistical confidence under the current experimental design.

Nevertheless, the ranking of LogWorth values provides useful information regarding the relative influence of the investigated variables. The comparable contributions of PEG concentration and activated carbon loading suggest that membrane adsorption performance is primarily governed by membrane composition rather than by processing time. Furthermore, the relatively higher contribution of the PEG $\times$ activated carbon interaction compared with the remaining interaction terms indicates that these two variables may act synergistically. This observation is consistent with the experimental adsorption results, in which improved Fe removal was achieved only when higher activated carbon loading was accompanied by sufficient PEG content to generate an interconnected pore structure.

The absence of statistically significant effects may be attributed to the limited number of experimental runs and the resulting low statistical power of the factorial model. Under these conditions, relatively large experimental variability can mask the individual contributions of the investigated factors, even when meaningful trends are observed experimentally. Increasing the number of replicates or expanding the design space would reduce the experimental error and improve the ability of the model to distinguish significant factor effects.

The Residual versus Predicted Plot is presented in Figure 9 and was used to evaluate the adequacy of the factorial regression model. Residual analysis is an essential diagnostic tool because it assesses whether the assumptions underlying the regression model are satisfied and whether the model adequately represents the experimental data. Ideally, residuals should be randomly distributed around zero without any systematic pattern, indicating that the model errors are independent and exhibit constant variance (homoscedasticity) (Quezada et al., 2021).

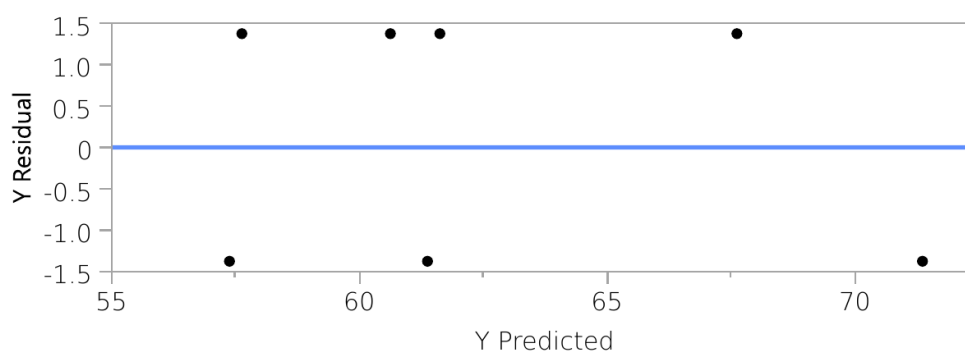


Figure 9. Residual versus predicted plot of the factorial regression model for Fe adsorption by the fabricated composite membrane.

As shown in Figure 9, the residuals are randomly scattered around the zero reference line, with no apparent increasing, decreasing, or curved trend. This random distribution suggests that the prediction errors are independent of the fitted values and that the model does not exhibit systematic bias within the investigated experimental region. The absence of a discernible pattern also indicates that the assumption of constant error variance is reasonably satisfied, suggesting that the regression model provides an adequate description of the observed experimental responses.

Furthermore, no extreme residuals were observed, indicating the absence of obvious outliers that could disproportionately influence the regression coefficients or compromise model stability. The relatively balanced distribution of positive and negative residuals suggests that the model does not consistently overestimate or underestimate the response, providing additional evidence that the prediction errors are randomly distributed.

Although the residual analysis supports the adequacy of the regression assumptions, these results should be interpreted together with the overall statistical performance of the model. As discussed in the previous section, the regression model was not statistically significant at the 95% confidence level ($p = 0.4915$). Therefore, while the residual plot indicates that the model is free from obvious diagnostic problems such as heteroscedasticity or systematic prediction bias, it does not imply that the investigated factors exert statistically significant effects on the response. Rather, the residual analysis confirms that the regression model is appropriate for describing the available experimental data, whereas its inferential and predictive capabilities remain limited by the statistical significance of the fitted model.

The analysis of variance (ANOVA) for the factorial regression model is summarized in Table 7. ANOVA was performed to evaluate the overall statistical significance of the regression model by comparing the variability explained by the model with the unexplained residual error. The regression model produced an F-ratio of 2.0248 with a corresponding p-value (Prob F) of 0.4915. Because the p-value is substantially greater than the significance level of 0.05, the null hypothesis cannot be rejected, indicating that the regression model is not statistically significant at the 95% confidence level. Consequently, the investigated factors, including PEG concentration, activated carbon loading, and stirring time, cannot be considered to have statistically significant effects on Fe adsorption within the investigated experimental range.

Table 7. Analysis of variance (ANOVA) for the factorial regression model describing Fe adsorption by the fabricated composite membrane.

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	6	183.75000	30.6250	2.0248
Error	1	15.12500	15.1250	Prob > F
C. Total	7	198.87500		0.4915

The absence of statistical significance should be interpreted in the context of the experimental design. As shown in Table 7, the model was fitted using only one degree of freedom for the residual error (DF = 1). Such a limited residual degree of freedom substantially reduces the statistical power of the F-test and limits the precision of the estimated experimental error. Under these conditions, relatively large differences among treatment combinations may fail to reach statistical significance because the error variance cannot be estimated reliably.

This limitation is also consistent with the previous model diagnostics. Although the regression model exhibited good agreement between predicted and experimental responses and the residual analysis indicated no obvious violations of regression assumptions, the ANOVA results demonstrate that the available experimental evidence is insufficient to establish statistically significant relationships between the investigated variables and the response. Therefore, the model should be regarded as a descriptive representation of the observed experimental trends rather than a statistically validated predictive model.

The low residual degree of freedom further suggests that the current factorial design provides limited information for hypothesis testing. Increasing the number of experimental runs or incorporating replicate measurements would improve the estimation of experimental error, increase the statistical power of the ANOVA, and provide greater confidence in identifying significant main effects and interaction terms. Alternatively, a more comprehensive optimization approach, such as response surface methodology (RSM), could be employed to capture potential nonlinear relationships among the fabrication parameters.

The estimated regression coefficients for the factorial model are presented in Table 8. These coefficients describe the direction and magnitude of the effects of the investigated variables and their interactions on the predicted Fe adsorption response.

Before interpreting individual terms, it is important to state the main limitation of this analysis upfront: the experimental design used eight runs with no replicates or center points, leaving very few degrees of freedom to estimate error, which limits the statistical power to detect real effects even when they are present. With that caveat, the regression model produced a statistically significant intercept (Estimate = 61.875, $p = 0.0141$), representing the baseline response of the fitted model. In contrast, none of the investigated process variables or interaction terms were statistically significant at the 95% confidence level ($p > 0.05$), indicating that no individual factor could be identified as an independent predictor of the response within the investigated experimental region given the current design's limited power.

Table 8. Regression parameter estimates for the factorial model describing Fe adsorption by the fabricated composite membrane.

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	61.875	1.375	45.00	0.0141*
X1	2.625	1.375	1.91	0.3072
X2	2.625	1.375	1.91	0.3072
X3	0.125	1.375	0.09	0.9423
X1*X2	2.375	1.375	1.73	0.3341
X1*X3	-0.125	1.375	-0.09	0.9423
X2*X3	1.875	1.375	1.36	0.4028

Among the main effects, PEG concentration (X_1) and activated carbon loading (X_2) exhibited identical positive regression coefficients (Estimate = 2.625), suggesting that increasing either factor tended to improve the adsorption performance. Although these coefficients were not statistically significant ($p = 0.3072$), their positive values are consistent with the experimental observations, where higher PEG concentrations promoted pore formation and greater activated carbon loading increased the availability of adsorption sites. The similarity of both coefficients also indicates that these variables contributed comparably to the predicted response.

In contrast, stirring time (X_3) exhibited a very small positive coefficient (Estimate = 0.125) with the highest p-value (0.9423), indicating a negligible individual contribution to the response. This result agrees with the Effect Summary analysis, which identified stirring time as the least influential factor among the investigated variables. Within the selected experimental range, extending the stirring time produced only marginal improvements in adsorption performance, suggesting that membrane composition exerted a greater influence than the processing duration.

The interaction terms also exhibited limited contributions to the regression model. The positive coefficient for the PEG $\times$ activated carbon interaction ($X_1 \times X_2$; Estimate = 2.375) suggests a potential synergistic effect between pore formation and adsorbent loading. This trend is consistent with the adsorption experiments, where improved Fe removal was observed only when increased activated carbon content was accompanied by sufficient PEG concentration. However, the interaction was not statistically significant ($p = 0.3341$), indicating that the observed synergy could not be confirmed statistically under the current experimental design.

The interaction between PEG concentration and stirring time ($X_1 \times X_3$) exhibited a small negative coefficient (Estimate = -0.125) with a non-significant p -value (0.9423), suggesting that simultaneous changes in these variables had virtually no measurable influence on the response. Similarly, the activated carbon $\times$ stirring time interaction ($X_2 \times X_3$) showed a positive coefficient (Estimate = 1.875), although this effect also remained statistically insignificant ($p = 0.4028$). These findings indicate that the interaction effects were relatively weak compared with the main compositional variables.

The significance of the main effects and two-factor interactions included in the factorial regression model was evaluated using the Effect Tests, as summarized in Table 9. This analysis determines whether each model term contributes significantly to the observed variation in Fe adsorption after accounting for the remaining variables in the regression model.

Table 9. Effect tests for the main factors and two-factor interactions in the factorial regression model describing Fe adsorption by the fabricated composite membrane.

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
X3	1	1	0.125000	0.0083	0.9423
X2*X3	1	1	28.125000	1.8595	0.4028
X2	1	1	55.125000	3.6446	0.3072
X1*X3	1	1	0.125000	0.0083	0.9423
X1*X2	1	1	45.125000	2.9835	0.3341
X1	1	1	55.125000	3.6446	0.3072

Among the investigated main effects, PEG concentration (X_1) and activated carbon loading (X_2) exhibited the largest F-ratios (3.6446), followed by the interaction between PEG concentration and activated carbon loading ($X_1 \times X_2$, $F = 2.9835$). In contrast, stirring time (X_3) and its interaction with PEG concentration ($X_1 \times X_3$) produced extremely small F-ratios (0.0083), indicating negligible contributions to the regression model. The interaction between activated carbon loading and stirring time ($X_2 \times X_3$) exhibited an intermediate contribution ($F = 1.8595$), although its effect remained relatively weak.

Despite these differences in F-ratio values, none of the investigated factors or interaction terms were statistically significant at the 95% confidence level. The lowest p -values were obtained for PEG concentration and activated carbon loading ($p = 0.3072$), whereas stirring time and the PEG $\times$ stirring time interaction exhibited the highest p -values ($p = 0.9423$). These results indicate that the observed variations in Fe adsorption cannot be attributed to any individual processing parameter with sufficient statistical confidence under the current experimental conditions.

The relative ranking of the F-ratios nevertheless agrees with the previous analyses. The larger F-ratios obtained for PEG concentration and activated carbon loading indicate that membrane composition exerted a greater influence on adsorption performance than stirring time. Likewise, the comparatively higher F-ratio of the PEG $\times$ activated carbon interaction suggests that these two variables may interact to influence membrane performance, supporting the experimental observation that improved Fe removal was achieved only when higher activated carbon loading was accompanied by sufficient PEG to generate an effective pore structure. However, because the interaction did not reach statistical significance, this apparent synergistic effect should be regarded as a qualitative trend rather than a statistically verified relationship.

The lack of significant factor effects is consistent with the results of the ANOVA, parameter estimates, and effect summary analyses, all of which indicated limited statistical evidence for the investigated variables. This outcome is likely associated with the limited number of experimental observations and the resulting low residual degrees of freedom, which reduce the statistical power of the regression analysis. Consequently, moderate differences among treatment combinations may not be detected as statistically significant despite observable experimental trends.

The Prediction Profiler generated by the factorial regression model is presented in Figure 10 and was used to evaluate the influence of each process variable on the predicted Fe adsorption response while simultaneously identifying the operating conditions that maximize the model response. This graphical tool illustrates the sensitivity of the predicted response to changes in individual factors while maintaining the remaining variables at their selected levels.

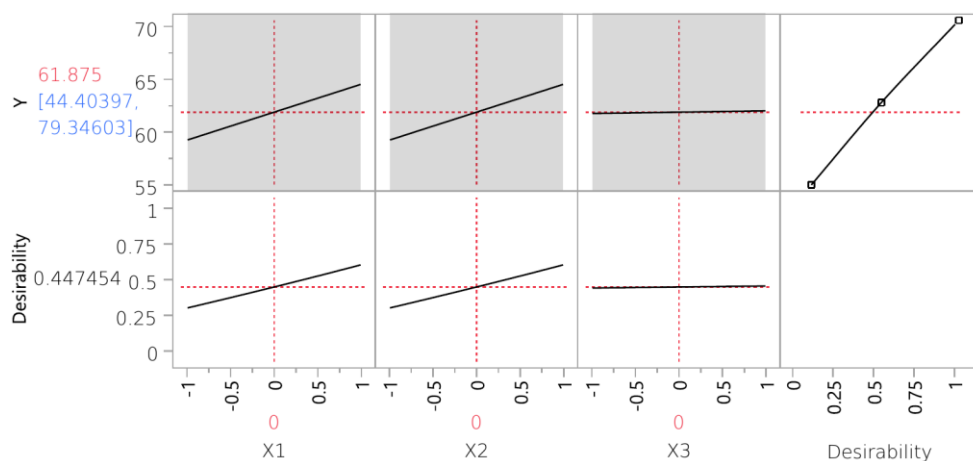


Figure 10. Prediction profiler of the factorial regression model showing the effects of PEG concentration (X_1), activated carbon loading (X_2), and stirring time (X_3) on the predicted Fe adsorption response.

As shown in Figure 10, the prediction profiles for PEG concentration (X_1) and activated carbon loading (X_2) exhibit positive slopes, indicating that increasing either variable results in an increase in the predicted response within the investigated experimental range. In contrast, the profile associated with stirring time (X_3) is nearly horizontal, suggesting that variations in stirring time have only a limited influence on the predicted adsorption performance. The relative slopes of the prediction profiles describe the model-estimated response within the tested design space; because the model was not statistically significant, these slopes should be interpreted as exploratory rather than as evidence of confirmed factor effects.

The profiler predicts an optimum response of 61.875 under the selected factor combination. This value represents the response estimated by the regression model rather than a directly measured experimental result. Consequently, the predicted optimum should be interpreted as the model's estimation of the most favorable operating condition within the investigated design space and should ideally be confirmed through additional validation experiments.

The optimization analysis also yielded an overall desirability value of 0.4475. In desirability-based optimization, values approaching 1.0 indicate that the selected factor combination closely satisfies the optimization objective, whereas lower values reflect a less satisfactory solution. The moderate desirability obtained in this study suggests that the identified operating conditions provide only partial optimization of the response and that additional improvement remains possible. This relatively low desirability is consistent with the statistical analyses presented previously, in which the regression model did not achieve overall significance. Consequently, the profiler provides an exploratory model-based condition within the current experimental domain, not a statistically validated optimum.

The prediction profiler is also consistent with the results of the Effect Summary, Effect Tests, and Parameter Estimates, all of which identified PEG concentration and activated carbon loading as the dominant contributors to the predicted response, while indicating only a minor contribution from stirring time. These outputs are consistent with the descriptive trends observed experimentally, but they do not establish statistically significant factor effects.

CONCLUSION

A chitosan-activated carbon composite adsorbent film was successfully fabricated and demonstrated measurable Fe^{2+} removal under the laboratory conditions investigated. FTIR spectra were consistent with retention of the characteristic functional groups of the constituent materials, but baseline drift and spectral noise limit the extent to which FTIR alone can demonstrate specific interactions or successful modification of the composite matrix. Among the eight experimental runs, the combination of 750 mg PEG, 40 wt% activated carbon, and 4 h stirring produced the lowest observed residual Fe concentration (7.750 mg L^{-1}). This condition is reported as the best-performing experimental run within the tested design space rather than as a statistically validated optimum. Although the factorial model gave $R^2 = 0.924$, the overall model and individual factor effects were not statistically significant at the 95% confidence level, and only one residual degree of freedom was available for error estimation. The present results therefore support Fe^{2+} removal by the fabricated composite film under the tested conditions but do not establish which formulation or processing factor controls adsorption or provide a direct characterization of the film's porosity and internal structure. Replicated or center-point experiments, together with direct porosity measurements and microscopy, are recommended to establish factor significance, structure-performance relationships, and the adsorption mechanism more rigorously.

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