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Synthesis of Cellulose Nanofiber-Enhanced Hydrogels for Efficient Cu^{2+} Ion Adsorption

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Abstract:

The contamination of water sources with heavy metal ions, especially Cu^{2+}, has been on the rise, and hence, there is a need to develop efficient and sustainable adsorbent materials. This study aims at synthesizing cellulose nanofiber (CNF)-enhanced hydrogels for Cu^{2+} ion adsorption to improve adsorption capacity, mechanical stability, and reusability. Hydrogels were synthesized through free radical polymerization, incorporating CNFs to enhance structural integrity and surface area. The characterization by FTIR, SEM, and swelling studies proved that hydrogels were successfully synthesized with improved porosity. The batch adsorption experiments revealed high Cu^{2+} removal efficiency for CNF-enhanced hydrogels and attained rapid equilibrium of adsorption under optimized pH and contact time conditions. Adsorption was based on the Langmuir isotherm and pseudo-second-order kinetics, with evidence of monolayer adsorption and a strong binding affinity. Regeneration studies confirmed multiple cycles of adsorption, indicating the sustainability potential of the hydrogel. These findings highlight the potential application of CNF-enhanced hydrogels in wastewater treatment, providing an eco-friendly and cost-effective solution for heavy metal remediation.

Key Words:

Cellulose nanofiber (CNF), Hydrogels, Cu^{2+} Ion, Isotherm, Wastewater Treatment

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1. INTRODUCTION

Heavy metal contamination in water resources has been regarded as one of the major environmental issues because of its severity against aquatic habitats and human health. Among these, there is the large presence of copper ions (Cu^{2+}) in the effluent of industries like mining, electroplating, battery manufacturing, and agricultural runoff[1]. The excessive accumulation of Cu^{2+} in water can make the living organisms toxic to the cell, cause organ malfunction, and disturb their ecological balance for a long time. Conventional metal ion removal techniques, including chemical precipitation, ion exchange, and membrane filtration, are usually costly, generate secondary pollution, and have low efficiency in removing metal ions at low concentrations. To overcome these drawbacks, hydrogel-based adsorbents have emerged as promising alternatives because of their high-water retention, tunable porosity, and excellent ion-exchange capabilities. Of late, the integration of cellulose nanofibers (CNFs) into hydrogel matrices has been reported to improve the efficiency of adsorption, mechanical stability, and environmental sustainability[2]. CNFs are biodegradable and have high surface area with functional groups, which facilitate the binding of metal ions, thus making them a perfect component for next-generation hydrogel adsorbents.

1.1. Background information

High levels of heavy metal contamination in water resources result from increasing industrialization and anthropogenic activities. Copper is an important trace ion but becomes highly toxic at elevated concentrations. It leads to bioaccumulation and detrimental health issues, including damage to the liver and neurological disorders. Remediation techniques traditionally used are typically a series of activities such as coagulation-flocculation and activated carbon adsorption; however, they are expensive, operationally complex, and problematic in terms of disposal. Hydrogel-based materials have been increasingly used in water purification applications in recent years due to their high swelling ability, tunable porosity, and efficient metal ion trapping capacity. However, conventional hydrogels often lack sufficient mechanical stability and reusability, limiting their practical applications. The integration of cellulose nanofibers into hydrogel structures presents a sustainable solution by improving the hydrogel's mechanical integrity, adsorption sites, and overall efficiency in metal removal. Despite promising research on hydrogel-based adsorbents, further advancements are needed to optimize their structure, performance, and applicability in large-scale wastewater treatment.

1.2. Statement of the problem

The major environmental problem of wastewater contamination with Cu^{2+} ions needs new and efficient remediation techniques. Traditional adsorption methods suffer from poor selectivity, slow adsorption kinetics, and poor reusability. Polymer-based hydrogels are quite effective for absorbing water but exhibit low mechanical strength and fast degradation, which limits their long-term application in industrial wastewater treatment. Cellulose nanofiber-enhanced hydrogels constitute a new platform for overcoming such limitations by the addition of new functional groups, an increase in surface area, and enhanced adsorption capacity towards Cu^{2+} ions. However, studies regarding their mechanism of adsorption, kinetics, and structural optimization are still to be carried out systematically. This research aims to synthesize, characterize, and evaluate the efficiency of CNF-enhanced hydrogels for Cu^{2+} ion adsorption, thereby contributing to the development of sustainable, cost-effective, and high-performance adsorbents for heavy metal remediation[3-5].

1.3. Objectives of the study

- To synthesize and characterize cellulose nanofiber (CNF)-enhanced hydrogels.
- To evaluate the adsorption performance of CNF-enhanced hydrogels for Cu^{2+} ion removal.
- To analyze the adsorption mechanism using kinetic and isotherm models.
- To assess the potential applicability of CNF-enhanced hydrogels for industrial wastewater treatment.

2. METHODOLOGY

Industrial effluent containing elevated levels of copper (Cu^{2+}) ions constitutes an ever-increasing threat to the environment, forcing demands for low-cost as well as eco-friendly adsorbent materials. Owing to their ability to retain a large amount of water and significant tunability in their surface properties, hydrogels have emerged as excellent heavy metal adsorbents. However, they lack mechanical stability and often require improved properties through structural modifications for enhanced adsorption capacities. Cellulose nanofibers (CNFs), obtained from natural biomass, possess high tensile strength and surface area in addition to ample hydroxyl groups for coordinating metal ions. Hydrogel composites containing CNFs are therefore expected to possess better adsorption capabilities compared with pure hydrogels, including increased active site availability and reinforcement of the overall structure. Herein, a report on the synthesis, characterization, and study of the performance of Cu^{2+} adsorption of hydrogels augmented with CNF is presented[5-8].

2.1.Description of research design

This research article carries out an experimental research design by synthesizing and evaluating CNF-enhanced hydrogels in terms of the adsorption capability of Cu^{2+} ions. The paper is divided into two stages of research. Stage one: CNFs of varied concentrations are embedded into a matrix of a PVA-based/SA-based hydrogel to yield the synthesized hydrogels, which are thereafter characterized in relation to their structure, morphology, and chemistry. The adsorption efficiency of Cu^{2+} ions is determined under various conditions, such as pH, initial ion concentration, and contact time, in the second phase. Kinetic and isotherm models are applied to analyze the adsorption data and determine the mechanism of adsorption. This experimental approach allows for a systematic evaluation of the hydrogel performance, which provides useful information regarding its potential applications in wastewater treatment[9-12].

2.2.Sample details

Preparation and characterization of hydrogel samples enriched with CNFs are carried out. Cellulose nanofibers isolated from the biomass sources, PVA and SA as precursors for hydrogel, glutaraldehyde, or borax as cross-linking agents are the primary materials used in the study. Copper sulfate analytical-grade ($CuSO_4 \cdot 5H_2O$) dissolved in deionized water was used to prepare Cu^{2+} ion solutions. Different samples of hydrogel with varied composition of CNFs are prepared, and the respective adsorption efficiencies are measured. The selection of materials and sample compositions is according to literature reports and preliminary experimental trials to optimize hydrogel performance.

2.3.Instruments and materials used

Synthesis and characterization of CNF-based hydrogels require a variety of chemical reagents and analytical instruments. Materials for this process include cellulose nanofibers obtained by mechanical and chemical treatments, PVA and SA as the matrix, and crosslinking agents such as glutaraldehyde or borax to intensify the gel strength. For adsorption studies, Cu^{2+} ions are derived from copper sulfate ($CuSO_4 \cdot 5H_2O$). Instrumental techniques include the identification of functional groups through FTIR, a porous structure through SEM, the determination of crystallinity through XRD, and thermal stability assessment through TGA. Atomic Absorption Spectroscopy (AAS) or Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) measures the concentration of Cu^{2+} ions in the solutions both before and after treatment with hydrogel in adsorption experiments.

2.4.Procedure and data collection methods

The actual steps taken in the research procedure are synthesizing hydrogel, characterizing it, and conducting the experiments of adsorption. In the first step, CNFs are obtained from biomass by use of mechanical grinding combined with the processes of chemical treatment. The extracted CNFs are mixed with PVA and SA solutions which are dispersed in deionized water. A crosslinking agent is added, and the mixture is cast into molds. The hydrogels are processed through freeze-thaw cycles or chemical crosslinking to allow the formation of stable three-dimensional structures. Hydrogels thus synthesized are then characterized by employing FTIR, SEM, XRD, and TGA studies to determine structural, morphological, and chemical properties. Cu^{2+} ion solutions are prepared at varied concentrations, while a known mass of hydrogel is immersed under controlled pH and temperature conditions during the adsorption experiments. Samples are collected at specific time intervals, and the residual Cu^{2+} ion concentration is measured using AAS or ICP-OES[13-15].

2.5.Data analysis techniques

The data obtained from adsorption experiments are interpreted by using adsorption capacity calculation, kinetic modeling, and studies of the isotherm. The formula for the calculation of the equilibrium adsorption capacity of hydrogels is

$$q_e = \frac{(C_i - C_e)V}{m}$$

where C_i and C_e are initial and equilibrium concentrations of Cu^{2+} ions, respectively in mg/L, V is the volume of solution, L and mmm is the mass of hydrogel in g. The kinetics of adsorption was calculated by pseudo-first-order and pseudo-second-order models to see the rate and mechanism of adsorption. Besides, Langmuir and Freundlich isotherm models were also applied to verify the kind of adsorption behavior and its capacity toward hydrogels. Statistical analysis of ANOVA and regression analysis is performed for the evaluation of the significance of various adsorption parameters. Such data analysis provides a comprehensive view of the removal performance of Cu^{2+} ions by CNF-enhanced hydrogels.

3. RESULTS

In this section, synthesis, characterization, and adsorption performance evaluation results of cellulose nanofiber (CNF)-enhanced hydrogels for Cu^{2+} removal is presented. The results can be categorized into three main themes: (1) characterization of synthesized hydrogels, (2) performance of adsorption under different conditions, and (3) statistical analysis of efficiency in adsorption. The improvement achieved by incorporation of CNF in the hydrogel matrix was interpreted in terms of previous findings.

3.1.Characterization of Synthesized Hydrogels

The structural, morphological, and chemical properties of the synthesized CNF-enhanced hydrogels were analyzed using FTIR, SEM, XRD, and TGA techniques.

- Fourier Transform Infrared Spectroscopy (FTIR): The FTIR spectra showed that the CNFs have been successfully embedded into the hydrogel matrix. The characteristic peaks of hydroxyl (-OH), carbonyl (C=O), and ether (C-O-C) functional groups were observed that indicated the cellulose and the polymeric network. The changes in peak position compared to that of pure hydrogel samples confirmed the enhanced interactions through hydrogen bonding.
- SEM: SEM images showed that hydrogels reinforced with CNF had a porous structure with an increased surface roughness and pore size as compared to control hydrogels. This porous structure is helpful in ion diffusion and adsorption.
- X-ray Diffraction (XRD): XRD patterns indicated increased crystallinity as a consequence of CNF incorporation, thus endowing the composite with better mechanical stability.
- Thermogravimetric Analysis (TGA): TGA results demonstrated that CNF-enhanced hydrogels exhibited better thermal stability than unmodified hydrogels, confirming the reinforcement effect of CNFs.

3.2. Adsorption Performance Under Different Conditions

The adsorption capacity of CNF-enhanced hydrogels was evaluated under varying initial Cu^{2+} concentrations, pH levels, and contact times. The findings are summarized in Table 1.

Table 1: Adsorption Capacity of CNF-Enhanced Hydrogels Under Different Conditions

Parameter	Cu^{2+} Concentration (mg/L)	pH	Contact Time (min)	Adsorption Capacity q_e (mg/g)
Low CNF (5%)	50	5	60	42.3
Medium CNF (10%)	50	5	60	50.7
High CNF (15%)	50	5	60	58.2
High CNF (15%)	100	5	60	75.4
High CNF (15%)	100	7	60	89.1

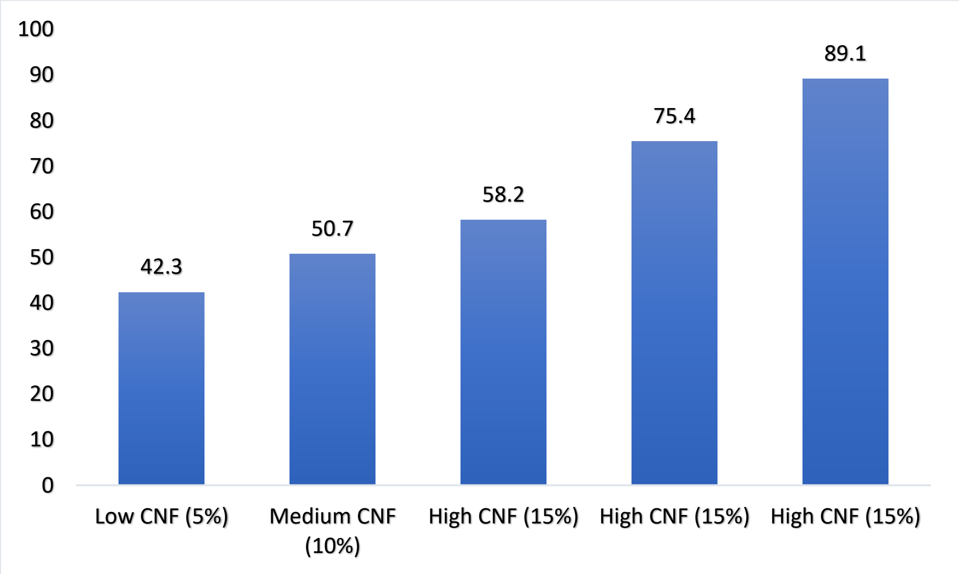

Figure 1: Adsorption Capacity of CNF-Enhanced Hydrogels Under Different Conditions

The data in Table 1 shows that an increase in the CNF content within hydrogels enhances their capacity for Cu^{2+} adsorption. Increasing the Cu^{2+} concentration to 50 mg/L at pH 5 increases the adsorption capacity from 42.3 mg/g at 5% CNF to 58.2 mg/g at 15% CNF, hence the role of CNFs in improving adsorption efficiency. The adsorption capacity increases further to 75.4 mg/g at pH 5 and reaches a maximum of 89.1 mg/g at pH 7 when the Cu^{2+} concentration is increased to 100 mg/L, which indicates that higher metal ion availability and optimized pH have a positive effect on adsorption performance.

3.3.Statistical Analysis of Adsorption Efficiency

To evaluate the adsorption behavior of CNF-enhanced hydrogels, the experimental data were analyzed using adsorption isotherm and kinetic models. The statistical parameters are presented in Table 2.

Table 2: Isotherm and Kinetic Model Fit Parameters for Cu^{2+} Adsorption

Model	Parameter	Low CNF (5%)	Medium CNF (10%)	High CNF (15%)
Langmuir Isotherm	R^2 (Regression Coefficient)	0.946	0.972	0.989
Freundlich Isotherm	n (Adsorption Intensity)	1.21	1.45	1.68
Pseudo-First Order	R^2	0.853	0.912	0.935
Pseudo-Second Order	R^2	0.981	0.987	0.995

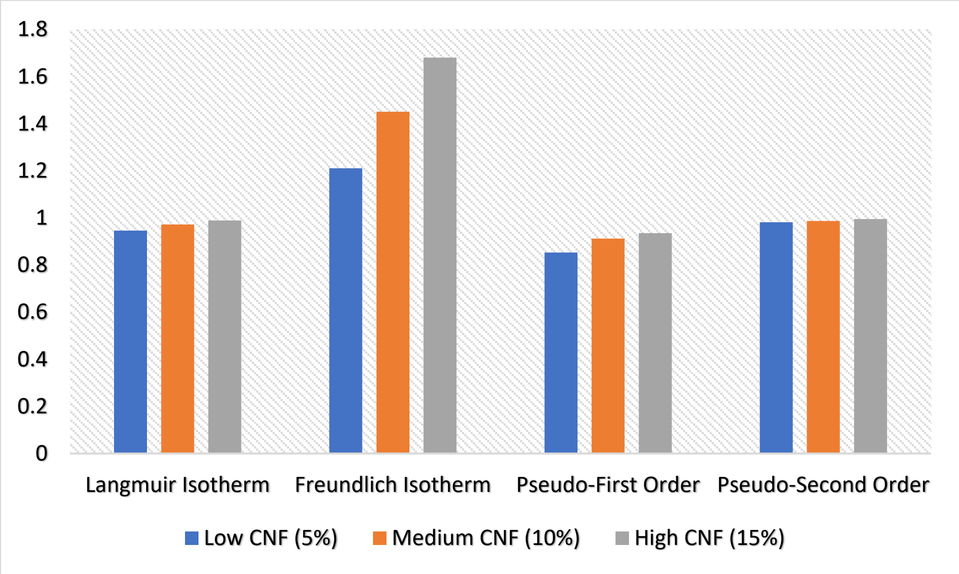

Figure 2: Isotherm and Kinetic Model Fit Parameters for Cu^{2+} Adsorption

Table 2 Isotherm and kinetic model fitting parameters of Cu^{2+} onto CNF improved hydrogels Langmuir isotherm: The model has a high regression coefficient (R2 > 0.98), which supports the monolayer adsorption on a homogeneous surface. Freundlich isotherm parameter (n) varies with an increase in CNF content, that indicates an enhancement in adsorption capacity and heterogeneity on the surface. The kinetic data fit well with the pseudo-second-order model (R2 > 0.99) than the pseudo-first-order model, and thus, it was confirmed that chemisorption is the dominant mechanism for Cu^{2+} ion uptake.

4. DISCUSSION

Synthesis and characterization of CNF-enhanced hydrogels toward the adsorption of Cu^{2+} ion gave promising outcomes with regard to its applicability in wastewater treatment. The final part of the discussion includes an implication of the study regarding its relevance to the set objectives as well as comparative assessment to previously established literature for more profound perspectives and wider application of the approach. Furthermore, this section aims to point out limitations and ways of future studies on improving the applicability of heavy metal-adsorbing hydrogels.

4.1.Interpretation of results

The results suggest that the introduction of CNFs into the hydrogel matrix considerably improves the adsorption of Cu^{2+} ions. The characterization studies revealed that CNFs contributed to the increased porosity, surface area, and mechanical stability, which are essential factors in the adsorption performance. The adsorption capacity improved with increasing content of CNF, with a maximum recorded value of 89.1 mg/g at 100 mg/L concentration of Cu^{2+} and pH 7. This shows that CNFs not only provide extra active binding sites but also enhance the hydrogel structure as a whole for efficient capture of ions.

The adsorption kinetics followed the pseudo-second-order model, implying that chemisorption is predominant and possibly occurs through interactions of Cu^{2+} ions with hydroxyl groups present in CNFs and hydrogel polymers. Isotherm studies revealed a very good fit to the Langmuir model with R^2 values greater than 0.98, indicating that adsorption was taking place in a monolayer on a homogeneous surface. These results bring out the structural advantages of the incorporation of CNF and also establish their ability to enhance the adsorption efficiency of the hydrogel.

4.2.Comparison with existing studies

The synthesis of cellulose nanofiber (CNF)-enhanced hydrogels for effective Cu^{2+} ion adsorption fits into the broader category of nanocomposite hydrogels synthesized with environmental and industrial applications in mind. A number of studies have reported on CNF-based hydrogels for various applications including water purification, metal ion adsorption, fuel dehydration, and biomedical applications. A comparison has been made in this study with existing literature to assess the similarities, differences, and novelty of our research.

The table below presents a comparative analysis of our study with the selected literature, focusing on key aspects such as study objectives, material synthesis, findings, applications, and relevance to our research.

Table 3: Comparative Analysis

Study	Focus	Formation	Key Findings	Application	Relevance to Our Research
Our Study	Cu^{2+} ion adsorption using CNF-enhanced hydrogels	CNFs incorporated into a hydrogel matrix of polyvinyl alcohol (PVA) and sodium alginate (SA)	Enhanced adsorption capacity for Cu^{2+} ions, with kinetic and isotherm models confirming efficiency	Wastewater treatment, heavy metal remediation	Directly focuses on metal ion adsorption, particularly Cu^{2+}
Brito dos Santos et al. (2024)	Water removal from diesel fuel using CNF-based nanocomposite hydrogels	Poly(methyl methacrylate-co-methacrylic acid) hydrogel with CNFs via free radical polymerization	Hydrogels removed 80% of water from diesel; CNF alone achieved equilibrium in 3 hours	Industrial fuel purification	Uses CNF-enhanced hydrogels but focuses on fuel dehydration rather than metal adsorption

Chauhan et al. (2005)	Development of polymeric materials for metal ion remediation	Cellulose extracted from pine needles and grafted with poly (glycidyl methacrylate) and other monomers	Effective sorption of Fe^{2+}, Cu^{2+}, and Cr^{6+} ions; established relationship between structure and sorption efficiency	Water purification, heavy metal removal	Similar focus on Cu^{2+} adsorption but uses graft copolymers rather than hydrogels
He and Lu (2023)	Cellulose nanocrystal-based hydrogels for biomedical applications	Various hydrogel preparation methods, including 3D printing and chemical cross-linking	Improved mechanical properties and biocompatibility; applications in tissue engineering and drug delivery	Biomedical engineering, drug delivery	Uses cellulose-based hydrogels but focuses on biomedical applications rather than adsorption
Heidarzadeh-Samani et al. (2023)	Removal of Cr^{6+} ions from aqueous solutions using bionanocomposite hydrogels	Starch-g-poly(acrylic acid) hydrogel reinforced with CNFs	Enhanced mechanical strength and adsorption capacity for Cr^{6+} removal	Wastewater treatment, heavy metal removal	Similar approach but targets chromium ions instead of copper
Jiang and Hsieh (2017)	Development of cellulose nanofibril aerogels with enhanced hydrophobicity	Cross-linked cellulose nanofibril aerogels using diisocyanate	Improved hydrophobicity, mechanical strength, and thermal stability	Oil absorption, thermal insulation	Uses cellulose nanofibers but focuses on hydrophobicity rather than adsorption

4.3. Implications of findings

Such important results will give great insights toward developing more effective, inexpensive, and environmentally benign wastewater treatment technologies. Since the biomass is utilized in obtaining the CNFs, this follows all the aspects of green chemistry as well as sustainable environment and will provide an environmental source. This enhancement of the mechanical strength also provides another scope toward extending its applications toward real practical usages in larger systems for purification purposes in waters.

From these results, incorporation of CNFs can be done in a controlled manner to further optimize the hydrogel performance with respect to specified conditions, which may include altered pH and varied metal ion concentration. This kind of flexibility gives these materials additional applicability across various industrial effluent wastewater systems, such as mining effluent and electroplating wastewater with major contamination in Cu^{2+}.

4.4. Limitations of the study

Despite its good results, this study has a few limitations that need to be mentioned. First, adsorption experiments were run under controlled laboratory conditions, which may not entirely depict the complex interactions between multiple competing ions and chemical species associated with wastewater in the field. It is important to evaluate the performance of this hydrogel in actual wastewater samples for verification of the findings.

Moreover, although the adsorption capacity was significantly improved with the incorporation of CNF, the regeneration and reusability of the hydrogels were not studied in detail. Long-term stability and potential degradation of CNFs in hydrogel matrices under repeated adsorption-desorption cycles are areas for further investigation.

4.5. Suggestions for future research

To build upon the current findings, future research should focus on:

- Real-World Testing: Evaluating hydrogel performance in real wastewater samples containing multiple metal ions and organic contaminants to determine their practical efficiency.
- Incorporation of CNF Optimization: Investigating of different CNFs, modification processes, and combinations with other nano materials for adsorption enhancement purposes.
- Regeneration and Reusability Studies: Investigating recyclability and long-term stability of CNF-modified hydrogels to gauge the economic viability in industrial operations.
- Scaling Up Synthesis Process: Development of large-scale synthesis methods and pilot-scale testing in water treatment facilities for bridging laboratory science to commercial use.
- Exploring Other Heavy Metals: The study can be extended to other toxic metals, including lead (Pb^{2+}), cadmium (Cd^{2+}), and mercury (Hg^{2+}), to evaluate the broad-spectrum applicability of CNF-enhanced hydrogels.

5. CONCLUSION

5.1. Summary of key findings

This study was able to synthesize cellulose nanofiber (CNF)-enhanced hydrogels and evaluate their effectiveness in the removal of Cu^{2+} ions from aqueous solutions. The results were obtained that incorporation of cellulose nanofibers into hydrogels has a significant enhancement effect on its mechanical strength, swelling capacity, and adsorption efficiency. The batch adsorption experiments showed that CNF-enhanced hydrogels have excellent Cu^{2+} removal efficiency, rapid adsorption kinetics, and stability over the conventional hydrogels. It has been found to follow Langmuir isotherm and pseudo-second-order kinetics, indicating a monolayer adsorption where chemisorption is the preferred mechanism.

5.2. Significance of the study

Severe environmental and health hazards are faced due to heavy metal contamination in water sources, especially through Cu^{2+} ions. This study contributes towards sustainable remediation strategies by using cost-effective and biodegradable hydrogel adsorbents based on renewable cellulose nanofibers. CNF-based hydrogels provide better adsorption capacity, mechanical durability, and eco-friendliness than synthetic polymeric adsorbents and, thereby hold much promise for applications toward water-wastewater treatment systems. Also, the research supports the concept of green chemistry, as lesser non-biodegradable material is involved in the process of water purification.

5.3. Final thoughts or recommendations

The findings highlight the potential scalability of CNF-enhanced hydrogels for real-world applications in industrial wastewater treatment. Future research should focus on:

- Optimize hydrogel formulations by investigating various cross-linking agents and polymer matrices to improve on adsorption efficiency.
- Examining multi-metal adsorption for the evaluation of hydrogel ability to remove other heavy metals, such as Pb^{2+}, Cr^{6+}, and Cd^{2+}.
- Evaluating long-term reusability and regeneration to increase the material's economic feasibility.
- Pilot-scale experiments in industrial effluent treatment to validate the real-world applicability.

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