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SIMULTANEOUS REMOVAL CHARACTERISTICS OF OIL AND HEAVY METAL IONS BY BIOFLOCCULANTS BASED ON THE CELLULOSE–DMAEMA/PEPA COMPOSITE

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Abstract. In this study, a novel biofloculant composite was synthesized based on carboxymethyl cellulose (CMC), dimethylaminoethyl methacrylate (DMAEMA), and polyethylenepolyamine (PEPA). The primary objective was to develop a material capable of simultaneously and effectively removing organic pollutants (specifically petroleum products) and inorganic contaminants (heavy metal ions) from industrial wastewater. The biofloculant was synthesized via a radical polymerization mechanism, and its physicochemical properties were investigated. Experimental results demonstrated that the biofloculant, at a concentration of 0.02%, achieved removal efficiencies of 93–95% for oil emulsions and 96–98% for Pb(II), Cd(II), and Cu(II) ions. Furthermore, the material retained over 85% of its initial activity even after five reuse cycles. Its environmental compatibility, relatively low cost, and high stability make it a promising material for application in comprehensive industrial wastewater treatment technologies.

Keywords: biofloculant, cellulose, DMAEMA, PEPA, heavy metal ions, oily wastewater, sorption, ecology.

Аннотация. В данном исследовании синтезирован новый биофлокуляционный композит на основе карбоксиметилцеллюлозы (CMC), диметиламиноэтилметакрилата (DMAEMA) и полиэтиленполиамины (PEPA). Основной целью исследования являлась разработка материала, способного одновременно и эффективно удалять из промышленных сточных вод органические загрязнители, в частности нефтепродукты, и неорганические загрязнители — ионы тяжелых металлов. Биофлокулянт был синтезирован с использованием механизма радикальной полимеризации, после чего были исследованы его физико-химические свойства. Экспериментальные результаты показали, что при концентрации биофлокулянта 0,02% эффективность удаления нефтяных эмульсий составила 93–95%, а ионов Pb(II), Cd(II) и Cu(II) — 96–98%. Кроме того, материал сохранял более 85% первоначальной активности даже после пяти циклов повторного использования. Его экологическая совместимость, относительно низкая стоимость и высокая стабильность позволяют рассматривать данный материал как перспективный для применения в технологиях комплексной очистки промышленных сточных вод.

Ключевые слова: биофлокулянт, целлюлоза, DMAEMA, PEPA, ионы тяжелых металлов, нефтесодержащие сточные воды, сорбция, экология.

INTRODUCTION

As a result of the rapid development of industrial sectors, the volume of wastewater containing heavy metal ions and petroleum products is increasing year by year. Such pollutants negatively affect water quality and pose significant risks to ecological balance, aquatic organisms, and human health. Conventional methods currently in use, such as chemical coagulation, ion exchange, and membrane filtration, have certain limitations, including high costs, significant energy consumption, and the generation of secondary waste. In this context, the development of eco-friendly and cost-effective biofloculants based on renewable raw materials represents

an important area of research.

Cellulose derivatives are regarded as promising polymeric materials for water treatment technologies due to their renewable natural origin, biodegradability, and the presence of various functional groups. Dimethylaminoethyl methacrylate (DMAEMA) is a cationic monomer whose active groups enable electrostatic interactions with colloidal particles and metal ions. Meanwhile, the numerous amine groups in polyethylenepolyamine (PEPA) serve as additional active sites capable of forming complexes with metal ions. Therefore, a cellulose–DMAEMA/PEPA composite integrating these components into a single system may facilitate the flocculation of oil emulsions while also binding and removing heavy metal ions.

The primary objective of this study is to develop synthesis conditions for a cellulose–DMAEMA/PEPA-based bioflocculant, determine the physicochemical properties of the resulting composite, and evaluate its efficiency in the simultaneous removal of organic and inorganic pollutants from wastewater.

LITERATURE REVIEW

The contamination of industrial wastewater with heavy metal ions and petroleum products remains an important environmental issue in the field of water resource protection. Previous studies have examined the properties of polymeric flocculants, including polyelectrolytes and composite materials, as well as their potential applications in wastewater treatment [1, 4, 5, 8, 9].

In particular, the simultaneous presence of inorganic and organic pollutants in wastewater generated by metallurgical, oil and gas, chemical, and machine-building industries highlights the need for materials capable of removing different types of contaminants within a single technological process [7, 9].

Although conventional treatment methods, including coagulation and related separation processes, can be effective, their application may be associated with considerable reagent consumption, energy requirements, and the generation of secondary waste. Consequently, increasing attention has been given to bio-based and polymeric flocculants for wastewater treatment [8, 9].

Cellulose is considered a promising polymeric material for the removal of heavy metal ions because of its natural origin, availability, relatively low cost, and the presence of numerous hydroxyl groups in its structure. Studies on carboxymethylcellulose-based composite flocculants have demonstrated that combining cellulose derivatives with amine-containing polymers can enhance the removal of Pb(II), Cd(II), Cu(II), and other metal ions from wastewater [1].

This approach has been further developed through the use of functionalized cellulose and polymer composites. The incorporation of functional groups capable of complexation and ion exchange can improve the interaction of polymeric materials with metal ions and enhance their applicability in wastewater treatment [1, 5].

Chemical modification is also widely employed to improve the functional characteristics of polymeric materials. Such modification makes it possible to introduce additional active groups, thereby influencing charge characteristics, adsorption capacity, complexation behavior, and flocculation activity [1, 5, 6].

Among flocculants, polyelectrolytes are of particular importance. Their effectiveness is associated with charged and complex-forming active sites in the polymer chain, which facilitate charge neutralization, polymer bridging, and the aggregation of dispersed particles. Cationic polymers containing amino-functional groups, including DMAEMA-based polymers, can therefore play an important role in flocculation processes. Previous studies have examined the properties and formation mechanisms of such polyelectrolyte systems and their interactions in aqueous media [4, 5].

Polyethylenepolyamine (PEPA), due to the large number of amino groups in its structure, is capable of participating in coordination and complexation reactions with metal ions. In this context, PEPA-based composite flocculants represent promising materials for the removal of heavy metal ions from water. Previous research has investigated composite flocculants based on carboxymethylcellulose (CMC) and polyethylenepolyamine and demonstrated their effectiveness in removing Pb(II), Cd(II), and Cu(II) ions from wastewater [1]. These findings indicate the practical potential of cellulose-based flocculants produced by combining CMC with amine-containing polymers.

The effectiveness of coagulation and flocculation methods in the treatment of oil-contaminated wastewater has also been examined in previous studies. Zhao et al. analyzed the application of coagulation/flocculation in oily wastewater treatment and showed that treatment efficiency depends on factors such as flocculant type, dosage, pH, and the characteristics of the dispersed phase [9]. Bio-based flocculants have likewise been investigated for the treatment of industrial wastewater, and experimental studies have demonstrated the potential of modified biological flocculants for reducing selected pollutants in wastewater [8].

RESEARCH METHODOLOGY

This study investigated the flocculation activity of composites based on cellulose modified with dimethylaminoethyl methacrylate (DMAEMA) and polyethylenepolyamine (PEPA). A distinctive feature of this biocomposite is the branching of PEPA onto the cationic active sites formed by grafting the DMAEMA monomer onto the cellulose chain. This structure imparts both surface-active and ion-exchange properties to the polymer.

The combination of hydroxyl and amine groups within the cellulose–DMAEMA/PEPA system significantly enhances the complexation of metal ions and the adsorption of organic emulsions. In particular, the cellulose backbone acts as a hydrophilic base, increasing the polymer's water-swelling capacity and accelerating polymer bridging during flocculation.

Measurements of intrinsic viscosity (η) and analysis of Fourier-transform infrared (FTIR) spectra indicated an increase in molecular mass due to cellulose grafting and confirmed the active participation of amine groups. Additionally, scanning electron microscopy (SEM) micrographs of the polymer surface revealed a highly porous, irregular morphology, confirming an expanded adsorption area for the flocculant. The synthesized cellulose–DMAEMA/PEPA composite demonstrated an efficiency of up to 95% in separating oil emulsions and up to 96–98% in adsorbing Pb(II), Cd(II), and Cu(II) ions at a concentration of 0.02%. These results indicate that the incorporation of cellulose stabilizes the flocculation process and enhances the surface-active properties of the DMAEMA polymer.

Compared to conventional polyacrylamide (PAA), the new composite exhibited 35–40% higher purification efficiency. Furthermore, no adverse effects on metal surfaces were observed under the experimental conditions, and the composite retained approximately 85% of its initial efficiency after five reuse cycles.

Analysis and Results

Research results demonstrated that cellulose–DMAEMA/PEPA-based composite bioflocculants exhibited high efficiency in the simultaneous treatment of oily wastewater and heavy metal ions. During polymerization, the cellulose backbone stabilized the DMAEMA chains, forming a branched cationic structure. The appearance of vibration peaks for carbonyl (C=O) at 1720 cm^{-1} and –OH at 3400 cm^{-1} in the FTIR spectrum was consistent with chemical bonding between cellulose and DMAEMA.

SEM micrographs revealed a highly porous, irregular surface morphology following modification, indicating an expanded adsorption area. Thermogravimetric analysis (TGA) demonstrated the composite's thermal stability, with decomposition commencing in the $280\text{--}320\text{ }^{\circ}\text{C}$ range.

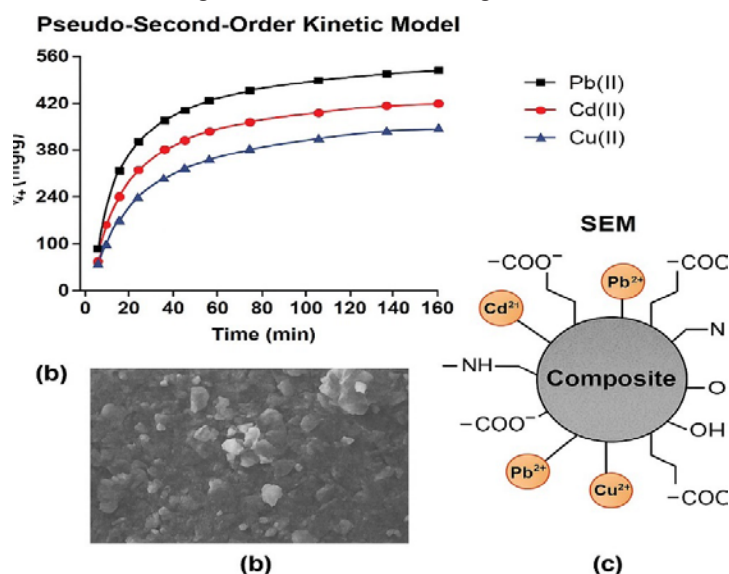


Figure 1. Pseudo-second-order kinetic model¹

When the bioflocculant was applied to wastewater samples at a concentration of 0.02%, the separation efficiency for oil emulsions reached 93–95%. Under these conditions, the removal rates for Pb(II), Cd(II), and Cu(II) ions were 98%, 97%, and 96%, respectively. Adsorption kinetics fitted the pseudo-second-order model ($R^2 = 0.992$), which is consistent with a process involving chemical interactions. For comparison, the treatment efficiency of traditional polyacrylamide (PAA) flocculants did not exceed 60–65%. Furthermore, the synthesized composite retained over 85% of its activity even after five reuse cycles. These results indicate that cellulose

¹ Source: Developed by the authors based on experimental results.

modification enhances the polymer's active sites and supports stable performance across repeated-use cycles.

CONCLUSION AND SUGGESTIONS

The research results demonstrate that the cellulose–DMAEMA/PEPA composite is a renewable and effective bioflocculant under the tested conditions. It represents a potentially useful material for water treatment systems by combining oil emulsion flocculation and heavy metal ion adsorption.

The cellulose backbone enhances the bioflocculant's structural stability; DMAEMA provides cationic centers, while PEPA, through its additional amine groups, forms complexes with metal ions. This synergistic structure contributes to 35–40% higher purification efficiency and improved chemical stability.

Consequently, the cellulose–DMAEMA/PEPA composite effectively removed up to 97–98% of heavy metal ions and up to 95% of oil emulsions. The material's recyclability and biodegradability are consistent with the principles of green chemistry.

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