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Metal-Sorption Properties of Modified Silk Fibroin

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Abstract

Chemically modified silk fibroin has gained attention as an important biomaterial due to its biodegradability, abundance of functional groups, and unique mechanical properties. Nitrated silk fibroin contains a large number of functional groups, which enable the binding of metal ions through electrostatic interactions and chelation processes. Based on this binding capacity, silk proteins can be used to develop eco-friendly and effective sorbents for removing heavy metals from aqueous environments. This article presents preliminary research findings regarding the potential of nitrated silk fibroin to sorb (absorb/adsorb) specific metal ions.

Keywords: Modification, silk, nitronated silk fibroin, heavy metals, sorption.

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1. Introduction

Bombyx mori silk fiber has been used as a textile material for many years [1,2]. Pure silk fiber consists of a core of silk protein—silk fibroin (SF)—and an outer layer of silk protein known as sericin. Specific procedures are carried out to remove sericin from the silk [3], as silk fibroin coated with sericin exhibits poor sorption properties. Since silk fibroin is a multifunctional biopolymer, its sorption capacity can be enhanced through the introduction of additional functional groups. This study focuses on investigating the heavy metal sorption properties of nitrated silk fiber. According to literature, approximately 12% of the chemical dyes used are discharged into wastewater, and 20% of this amount eventually enters the natural environment from treated wastewater [4, 5].

Bombyx mori L. silk is composed of silk fibroin (SF) and sericin; it has an elliptical cross-section and a diameter of approximately 30 μm. The SF component consists of two individual fibers, each with a triangular or semi-elliptical cross-section and a diameter of approximately 10 μm. Silk is composed of 70–80% SF and approximately 20–

30% sericin [6–8].

SF is a macromolecular, fibrous protein composed of three elementary units: the fibroin heavy chain (H-chain), the fibroin light chain (L-chain), and a glycoprotein (P25) [9, 10]. The H-chain consists of glycine (45.9%), alanine (30.3%), serine (5.3%), tyrosine (4%), valine (1.8%), and 15 other amino acids. It comprises four distinct domains: a crystalline domain, an amorphous domain, an N-terminal, and a C-terminal [11-12]. The crystalline domain is primarily composed of several repeating polypeptide segments—notably two hexapeptides (Gly-Ala-Gly-Ala-Gly-Ser(GAGAGS) and Gly-Ala-Gly-Ala-Gly-Tyr (GAGAGY)) which account for approximately 70% of its structure, thereby simplifying the amino acid sequence of the crystalline domain.

The amino acid composition of silk fibroin protein serves as the basis for its interaction with metal ions. Specifically, the oxygen atoms within the carboxyl groups and the nitrogen atoms of the amino acids contain lone pairs of electrons. Furthermore, terminal carboxyl groups, terminal amino groups, and certain groups within the side chains of amino acid residues (such as the hydroxyl group of serine [13, 14]) can also contribute to the sorption of metal ions through coordination. Silk proteins possess amphoteric properties. When the pH of the solution exceeds their isoelectric point [15, 16], the proteins release protons and become negatively charged; consequently, they can bind to metal ions via electrostatic interactions.

The interaction of silk proteins with metal ions involves electrostatic interactions and chelation. Electrostatic interaction is typically an ionic interaction between the negatively charged functional groups of silk proteins and positively charged metal ions. Silk proteins contain a large number of negatively charged polar functional groups, including -COOH, -OH, -CO-NH-, C=O, and -S-S- [17, 18]. These negatively charged functional groups provide binding sites for metal ions. Furthermore, these polar functional groups contain atoms such as oxygen (O) and nitrogen (N) that possess lone pairs of electrons [19–21]. These atoms tend to chelate with electron-deficient metal ions, forming coordination bonds. Both electrostatic interactions and chelation result in a strong affinity of silk proteins for metal ions.

The interaction between silk proteins and metal ions is influenced by several factors, such as the pH of the environment [22], temperature [23], and the specific properties of the metal ions [24, 25]. The binding energy of silk proteins to various metal ions depends primarily on the properties of the metal ions, including their charge-accepting capacity, ionic radius, and valence state. Electrostatic interactions between negatively charged ligands in silk proteins and metal ions as well as charge-dipole interactions between metal ions and uncharged ligands become stronger when the metal ions possess a higher net charge.

2. Required Materials and Equipment

Fibrous waste of silk (Cleaned of additives. Urganch Bahmal, LLC, Urgench, Uzbekistan), Sodium bicarbonate (purity 99,9%) was purchased from Chimreaktivinvest (Uzbekistan), Seeds.Bidistilled water is obtained from the “GFL 2104 Double distillation water still” device (Germany)., Microware radiation was performed on a ME81ARW (Samsung) device at a frequency of 2450 MHz. Crystallinity degree of the samples were measured by powder x-raydiffraction (XRD, Rigaku, The SmartLab SE, Japan (Khorezm Mamun Akademy lab)) using Cu Ka irradiation at 40 kV, 50 mA. ATR-IR Fourier spectroscopy analyses were performed on a “FTIR 4600 JASCO” (Japan) spectrophotometer.

3. Experimental Part

Natural silk fibroin fibers were used in the study. A 10% nitric acid (HNO_3) solution was employed for the nitration of the fibers. During the nitration process, the fibers turned yellow, but their fibrous structure remained intact. The nitrated fibers were isolated, and any residual acid was neutralized using a saturated sodium bicarbonate ($NaHCO_3$) solution. Subsequently, the samples were washed with hot distilled water until all traces of $NaHCO_3$ and other ions were completely removed, after which they were dried at 70 °C.

The sorption properties of the nitrated silk fibroin regarding Fe^{3+}, Pb^{2+}, and CrO_4^{2-} ions were investigated. In studying the sorption process, solutions containing varying concentrations of metal ions were used, with a mass-to-volume (g:mL) ratio of 1:1000 between the sorbent and the solution. Sorption experiments were conducted at a temperature of 298 K.

4. Results and Discussion

To study the sorption of iron(III) ions onto nitrated silk

fibroin fibers, iron(III) chloride solutions with concentrations ranging from 10-4 to 10-5 mol/L were used. The sorption process was carried out at a temperature of 298 K for 24 hours. The maximum experimental sorption capacity (for iron(III) ions) observed in these solutions was 8.34 mg/g. The experimental results were used to investigate the mechanisms of sorption.

For the Dubinin-Radushkevich isotherm, the equation lnQe= -4×10-9 ε2+3.5152 was obtained. From this equation β=4×10−9 mol2/J2 and the theoretical maximum sorption capacity Qm=33.6 mg/g were determined. The mean sorption energy, calculated using the formula E=(2β)-0.5 was found to be 11.2 kJ/mol. This value indicates that the sorption mechanism primarily involves ion exchange and electrostatic interactions. For the D–R model, R2= 0.9475, which provided a better description of the experimental results.

Thus, it was determined that the sorption of Fe(III) ions onto nitrated silk fibroin fibers is a process occurring on a heterogeneous surface; it is a favorable process primarily associated with ion exchange and electrostatic attraction. The theoretical maximum capacity calculated based on the Dubinin–Radushkevich isotherm is 33.6 mg/g, a value significantly higher than the experimental result (8.34 mg/g). This discrepancy is explained by the fact that, at low initial concentrations, not all active sites on the sorbent are fully occupied.

The results indicate that nitrated silk fibroin fibers possess a high sorption potential, effectively adsorbing Fe(III) ions even at low concentrations. The sorption process occurs at a heterogeneous interface and is primarily characterized by ion exchange and electrostatic interactions.

Sorption experiments were conducted using Pb(II) ion solutions with concentrations of 0.0042, 0.024, 0.0487, and 0.097 mol/L. The ratio of sorbent to solution was 1:1000 (g:mL). The nitrated fibroin fibers were immersed in the solutions and maintained at a temperature of 298 K for 24 hours. Following the sorption process, the residual concentration of Pb(II) ions in the solutions was determined via conductometric titration using a 0.05M Na2SO4 solution. The experimental results demonstrate that the sorption of Pb(II) ions by the nitrated fibroin fibers increases as the solution concentration rises. The maximum sorption capacity at 298 K was found to be 1242 mg/g. This value is significantly higher than the sorption capacities for Pb(II) reported in the literature for many other natural and modified sorbents [26].

To study the sorption of chromate ions by nitrated silk fibroin, potassium chromate solutions with concentrations of 5.186×10^{-6}, 7.26×10^{-6}, 8.65×10^{-5}, 1.89×10^{-4}, and 8.11×10^{-6} mol/L were used. The sorption process was carried out at a temperature of 298 K for 24 hours. The maximum experimental sorption capacity (for chromate ions) observed in these solutions was 9.7 mg/g. The results indicate that nitrated silk fibroin fiber possesses a high sorption potential, effectively adsorbing CrO_4^{2-} ions even at low concentrations.

5. Conclusion

Nitrated silk fibroin fibers exhibit a high capacity for the sorption of Fe(III) ions from aqueous solutions, with a maximum sorption capacity of 8.34 mg/g determined at 298 K. They also demonstrate high efficiency in the sorption of Pb(II) ions, reaching a maximum sorption capacity of 1242 mg/g at 298 K. For $CrO4^{2-}$ ions, the maximum sorption capacity at 298 K was found to be 9.7 mg/g. This result represents a significant step toward the development of inexpensive, environmentally safe, and biodegradable sorbents based on modified natural polymers. Future research into the reusability of the sorbent, as well as its sorption kinetics and thermodynamic properties, will further expand the potential for the practical application of this material.

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