



Enhancing Nitrate Ion Removal from Water using Fixed Bed Columns with Composite Chitosan-based Beads

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Abstract

Water contamination is a pressing global concern, especially regarding the presence of nitrate ions. This research focuses on addressing this issue by developing an effective adsorbent for removing nitrate ions from aqueous solutions. two adsorbents Chitosan-Zeolite-Zirconium (Cs-Ze-Zr composite beads and Chitosan-Bentonite-Zirconium Cs-Bn-Zr composite beads were prepared. The study involved continuous experimentation using a fixed bed column with varying bed heights (1.5 and 3 cm) and inlet flow rates (1 and 3 ml/min). The results showed that the breakthrough time increased with higher bed heights for both Cs-Ze-Zr and Cs-Bn-Zr composite beads. Conversely, an increase in flow rate led to a decrease in breakthrough time. Notably, Cs-Ze-Zr and Cs-Bn-Zr demonstrated impressive removal efficiencies, reaching 87.23% and 92.02%, respectively. The optimal conditions for peak performance were found to be an inlet flow rate of 1 ml/min, a bed height of 3 cm, and initial concentrations of 400 mg/L and 600 mg/L for Cs-Ze-Zr and Cs-Bn-Zr, respectively.

Keywords: Adsorption, Chitosan, Nitrate, Fixed bed column, Composite beads, Zeolite, Bentonite.

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1- Introduction

Nowadays, the swift pace industrialization, of urbanization, and intensified agricultural activities collectively contribute to the pollution of water sources [1]. Water quality issues have been brought to the forefront due to the presence of diverse contaminants, such as inorganic anions, synthetic organic compounds, and metal ions. Several drinking water sources have been demonstrated to contain potentially harmful levels of certain inorganic anions, with nitrate emerging as a prominent global issue [2]. Since nitrate is highly soluble in water, its removal from aquifers poses a challenge[3]. The accumulation of nitrate in the human body can lead to serious health risks, including cancer, blue-baby syndrome, and kidney failure [1]. Recognizing these dangers, the World Health Organization has set a nitrate concentration limit of 50 mg/L in drinking water to protect people from its detrimental effects [4].

To address this challenge, various established techniques aim to remove nitrate from water samples. Examples of such techniques include ion exchange [5], reverse osmosis[6, 7], and electrodialysis [8]. These methods play crucial roles in mitigating the health hazards associated with elevated nitrate levels and contribute to ensuring the safety and quality of drinking water.

Adsorption stands out as a practical and common method for efficiently removing contaminants. It offers various benefits, including ease of use, affordability, and a significant industrial processing capacity. Deliberate functionalization enhances adsorption's selectivity and efficiency, making it a preferred choice for water purification due to its simplicity, low cost, and minimal energy requirements [9, 10].

The crucial role of the adsorbent type in the adsorption process has been widely acknowledged. Numerous studies have recommended a variety of adsorbents, including organic materials, inorganic substances, and agricultural by-products such as activated carbon [11], silica gel [12], and date pits [13]. Among these, Chitosan has demonstrated successful application in removing toxic ions [14, 15], offering practical advantages such as forming flexibility, biodegradability, and biocompatibility[16, 17]. Moreover, Chitosan's significant content of amino and hydroxyl groups facilitates protonation in acidic media [1].

Despite the acknowledged advantages of Chitosan, certain drawbacks, including poor selectivity, limited adsorption capacity, and low mechanical strength, have been identified [18]. Consequently, recent approaches focus on overcoming these challenges by proposing



different modification methods to enhance the physicochemical properties of Chitosan. These methods encompass both physical and chemical modifications, such as composite blend forms [19], metal loading [20], crosslinking [21], and other innovative approaches.

Chitosan is commonly subjected to crosslinking to enhance its mechanical strength, so it can be used in water treatment applications [22]. Crosslinking involves the interconnection of Chitosan's macromolecular chains [23], a process facilitated by various chemicals such as glutaraldehyde, epichlorohydrin, and ethylene glycol diglycidyl ether [24, 25].

Many alternative approaches have been adopted to improve the properties of adsorbents, including removal efficiency and adsorption capacity. Loading Chitosan with other substances, particularly multivalent metal ions, has emerged as an effective solution. For instance, ions with higher positive charges, such as calcium, magnesium, aluminum, zirconium, and lanthanum ions, are widely used for loading [26]. Zirconium exhibits notable advantages due to its high positive charge, biological and chemical inertness, and resistance to oxidation, reductants, alkalis, and acids [27].

Zirconium, among various multivalent metal ions, enhances the selectivity of the adsorbent by binding to appropriate functional groups (e.g., carboxyl, hydroxyl, and amino groups) and forming stable complexes with anions like nitrate and phosphate [28]. Additional materials, such as activated carbon, cellulose, and alginate, have been incorporated to enhance the properties of Chitosan, including surface area, chemical stability, mechanical strength, and adsorption performance. In this study, Chitosan was blended with Zeolite and Bentonite to achieve these objectives.

The current research introduces two types of composite adsorbents, Cs-Ze-Zr and Cs-Bn-Zr, consisting of Chitosan blended with Zeolite and Bentonite, respectively. These composites were crosslinked with glutaraldehyde and subsequently loaded with Zirconium ions. The study investigates the feasibility of these proposed composite adsorbents for nitrate removal from water, exploring breakthrough curves through continuous mode operations with variations in bead height and flow rate.

2- Experimental Method

The preparation, system setup, procedure, and characterization for the composite bead (Cs-Bn-Zr) are described in detail in our previous work [29]. A similar procedure and system setup was used for the (Cs-Ze-Zr).

3- The Continuous Adsorption Experiments

A continuous fixed-bed adsorption column was designed to investigate the adsorbent's dynamic behavior during the nitrate removal process.

Fig. 1 shows a photograph and schematic diagram of the continuous system utilized to conduct the experiments. A QVF glass tube with a diameter of 1.8 cm and a length of 15 cm was used as a column to conduct the continuous adsorption process. The column was filled with the prepared adsorbents to achieve the desired bed height, and a fine sieve gauze plate was placed at the base to capture any adsorbents, preventing their loss in the effluent.

To ensure efficient flow distribution, glass beads of the same size as the adsorbent were placed on the upper layer of the bead. Nitrate solution, with a specified initial concentration, was pumped into the column at varying flow rates using a peristaltic pump. The study systematically investigated the impacts of key parameters on the adsorption process performance, such as bed height (1.5 and 3.0 cm) and feed flow rate (1 and 3 ml/min) for each adsorbent.

To assess the NO_3^- content, spectrophotometer readings of adsorption column effluent samples were taken at different intervals, providing valuable insights into the adsorption process performance.

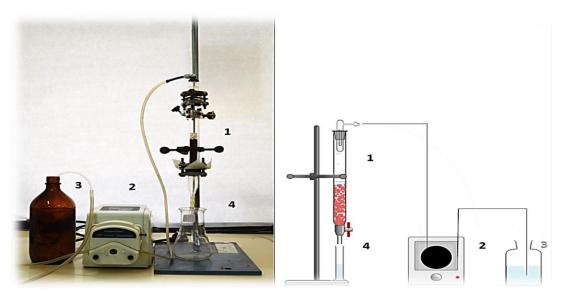


Fig. 1. Photograph and Schematic Diagram of the Continuous System. 1- Adsorption Column, 2- peristaltic pump, 3-Feed source, 4- Collection Reservoir

4- Results and Discussion

4.1. Effect of Flowrate

The effect of the inlet flow rate on the breakthrough curves of nitrate ion adsorption from water by Cs-Ze-Zr was examined. Two different inlet flow rates, namely 1 ml/min and 3 ml/min, were considered in this study. The bed height was 3 cm, and the nitrate concentration was 400 mg/L. The breakthrough curves for Cs-Ze-Zr beads were analyzed by plotting the ratio of the effluent concentration to the initial concentration (C_t/C_o) , as a function of time Fig. 2.

Fig. 2 illustrates a noticeable trend when comparing the breakthrough curves at different flow rates. By increasing the flow rate from 1 ml/min to 3 ml/min, the breakthrough curve shifts to the left in comparison to the stander behavior. This shift is indicative of a shortened breakthrough time, which represents the time for the ultimate limit concentration for nitrate in water 50 mg/L. At a flow rate of 1 ml/min, the breakthrough time was approximately 120 min. However, when the flow rate increased to 3 ml/min, the value of C_t/C_o exceeded the limited concentration of nitrate at the initial time of the experiment.

This observed phenomenon can be attributed to the shortened breakthrough time with a higher flow rate, meaning that nitrate ions spend less time in contact with the adsorbent [30]. Consequently, the nitrate removal efficiency decreases from 87.23% at the flow rate of 1ml/min to 52.13% at 3 ml/min.

The breakthrough curve exhibits a slight steepening at a reduced volumetric flow rate of 1 ml/min, allowing nitrate ions more time for diffusion and adsorption onto the active sites. As a result, the removal efficiency is enhanced at a lower flow rate of 1 ml/min. These results agree with those obtained by Tabrizi and Yavari [31].

The effect of the inlet flow rate on the breakthrough curves of adsorptive nitrate ions from water, using Cs-Bn-Zr beads, was investigated under the same conditions above, with the inlet nitrate ions concentration maintained at 600 mg/L Fig. 3.

Fig. 3 shows that as the inlet flow rate rises, the breakthrough curves shift to the left from the origin compared to the ideal behavior. Specifically, the breakthrough time was 60 min at a flow rate of 1 ml/min., while the value of C_t/C_o exceeded the limited concentration of nitrate at the initial time of the experiment at 3 ml/min. In addition, the nitrate removal efficiency decreased from 92.02% at a flow rate of 1 ml/min. Additionally, there was a subtle steepening of the breakthrough curve when the volumetric flow rate was reduced to 1 ml/min, attributed to the reasons outlined above.

4.2. Effect of Bed Height

The effect of bed height on nitrate ion removal was investigated at two different bed heights, namely 1.5 cm and 3 cm, for Cs-Ze-Zr. The experiments were conducted with a constant flow rate of 1 ml/min and an initial nitrate concentration of 400 mg/L. The experimental breakthrough curves C_t/C_o as a function of time are plotted in Fig. 4.

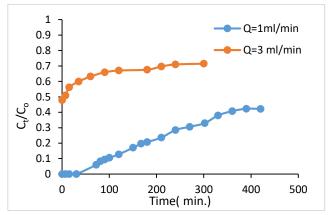


Fig. 2. Experimental Breakthrough Curves for Adsorption of Nitrate onto Cs-Ze-Zr Composite Beads at Different Flow Rates

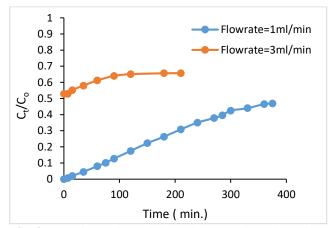


Fig. 3. Experimental Breakthrough Curves for Adsorption of Nitrate onto Cs-Bn-Zr Composite Beads at Different Flow Rates

As shown in Fig. 4, an increase in bed height from 1.5 cm to 3 cm results in a reduction in the slopes of the breakthrough curves, accompanied by a rightward shift. The breakthrough times increased from 72 min at a bed height of 1.5 cm to approximately 120 min. for bed heights of 3 cm. This observed trend can be attributed to the increased number of active sites and the prolonged residence times for nitrate ions to interact with the adsorbent.

The impact of bed height on Cs-Bn-Zr bead performance was studied under similar conditions, with the initial nitrate concentration set at 600 mg/L. The experimental breakthrough curves are shown in Fig. 5.

From Fig. 5, it can be noticed that at a bed height of 1.5 cm, the curve did not initiate from the origin and shifted to the left. This indicates that the bed height is insufficient to achieve an ideal adoption process. Consequently, this suggests that the mass transfer zone (MTZ), representing

(the zone through which the material transfer occurs) is greater than the height of the bed.

The curve started from the zero point at a bed height of 3 cm and increased gradually. This behavior indicates that the MTZ is either equal to or smaller than the bed height. In this scenario, the pollutant does not appear in the initial effluent drops from the column.

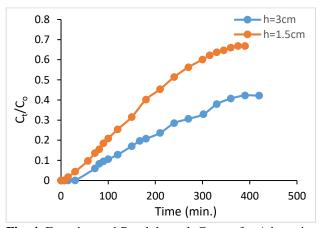


Fig. 4. Experimental Breakthrough Curves for Adsorption of Nitrate onto Cs-Ze-Zr Composite Beads at Different Bed Heights

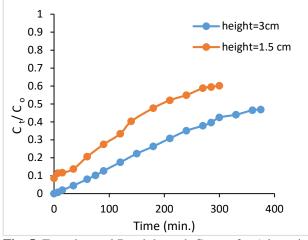


Fig. 5. Experimental Breakthrough Curves for Adsorption of Nitrate onto Cs-Bn-Zr Composite Beads at Different Bed Heights

5- Conclusions

In this research, the properties of Chitosan were improved by blending it with Zeolite and Bentonite to create composite beads. These beads were subsequently crosslinked with Glutaraldehyde solution and loaded with Zirconium for the removal of nitrate from water. The research further investigated the effects of key parameters, such as liquid flow rate and bed height, on the performance of fixed bead systems in the adsorption process. The results demonstrated that the optimal flow rate was 1 ml/min for both bead types. A significant reduction in breakthrough time was observed when the flow rate was increased to 3 ml/min. At the breakthrough point, the removal efficiency was 87.23% for Cs-Ze-Zr and 92% for Cs-Bn-Zr at a flow rate of 1 ml/min. Moreover, an increase in the bed height resulted in an extended breakthrough time for both prepared beads.

Nomenclature

Symbols	Description	Units
Co	Inlet nitrate concentration	mg/L
Ct	Outlet nitrate concentration	mg/L

Abbreviation

Symbol	Description
Cs-Ze-Zr	Chitosan-Zeolite-Zirconium composite
	beads
Cs-Bn-Zr	Chitosan-Bentonite-Zirconium composite beads

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تعزيز ازالة ايونات النترات من الماء بأستخدام أعمدة الحشوة الثابتة مع حبيبات مركبة العزيز ازالة ايونات النترات من الماسها الجيتوسان

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الخلاصة

يعد تلوث المياه مصدر قلق عالمي ملح، خاصة فيما يتعلق بوجود أيونات النترات. يركز البحث الحالي على تطوير مادة ممتزة فعالة لإزالة أيونات النترات من المحاليل المائية. تم تحضير مادتين مازتين حبيبات مركب الجيتوسان-زيولايت-زركونيوم-Bn-Zr Cs وحبيبات مركب الجيتوسان-بنتونايت-زركونيوم-Bn-Zr Cs. تم الجيتوسان-زيولايت-زركونيوم) باستخدام عمود حشوة ثابت بارتفاع (٥,١ و ٣ سم) اجراء البحث في الدراسة المستمرة (continuous study) باستخدام عمود حشوة ثابت بارتفاع (٥,١ و ٣ سم) يزداد ومعدل التدفق الداخل (١ و ٣ مل / دقيقة). أظهرت النتائج أن زمن الاختراق (breakthrough time) يزداد مع زيادة ارتفاع الحشوة لاحمد مع زيادة المتوا المركبة علي المركبة الحمد حموة تابت بارتفاع (٥,٢ و ٣ مم) مع زيادة ارتفاع الدائسة المركبة الحموة النتائج أن زمن الاختراق (breakthrough time).

وعلى العكس من ذلك، أدت الزيادة في معدل التدفق إلى انخفاض في زمن breakthrough. والجدير بالذكر أن الممتزات CS-Ze-Zr و CS-Bn-Zr أظهرا كفاءات إزالة مثيرة للإعجاب، حيث وصلت إلى مر ٨٧,٢٣ و ٩٢,٠٢%، على التوالي. وقد وجد أن الظروف المثالية لأداء الذروة كان عند معدل تدفق قدره ١ مل / دقيقة، وارتفاع الحشوة ٣ سم، والتركيزات الأولية ٤٠٠ ملغم / لتر و ٦٠٠ ملغم / لتر لـ CS-Ze-Zr وCS-Bn-Zr، على التوالي.

الكلمات الدالة: الامتزاز، الجيتوسان، النترات، عمود الحشوة الثابت، حبيبات مركبة، الزيولايت، البنتونايت.