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Column Study and Desorption Performance of Coffee Biomass-Calcium-Alginate Beads to Remove Pb^{2+} Ions from Aqueous Solutions

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Abstract

Heavy metal pollution, particularly by lead (Pb^{2+}), poses a serious environmental threat and can significantly impact human health. This study aims to evaluate the performance of an adsorbent composed of a mixture of coffee waste and calcium alginate (Coffee-Calcium-Alginate/CCA beads) in removing Pb^{2+} ions through column experiments, as well as to assess the desorption efficiency and reusability of the adsorbent. Column studies were conducted at 1 ppm Pb^{2+} ions initial concentration, a flow rate of 2.2 mL/min, using a column with a length of 10 cm and a diameter of 7 mm to determine the breakthrough time of the adsorbate, while desorption was performed using HNO_3 to evaluate the efficiency of Pb^{2+} ions release and the potential for adsorbent reuse. The results demonstrated that the CCA adsorbent was able to retain high efficiency over two adsorption–desorption cycles, with desorption efficiencies of 96.35% and 86.64%. The desorption mechanism involves protonation of carboxylate groups and competition between H^+ and Pb^{2+} ions at the active sites. Overall, CCA beads exhibit promising potential as an effective and reusable adsorbent for treating water contaminated with heavy metals. Future research could explore the effects of varying initial Pb^{2+} ions concentrations and column height to gain a more comprehensive understanding of desorption dynamics within the column system.

Keywords: *adsorption, coffee biomass, calcium alginate, Pb^{2+}, leads*

Introduction

Water Water serves as a fundamental resource for sustaining life. However, anthropogenic activities, particularly the discharge of domestic and industrial waste, have significantly compromised both the quality and availability of water. According to WHO, significant vector for the spread of infectious diseases such cholera, diarrhoea, dysentery, hepatitis, typhoid, and polio is contaminated water, with more than 251.4 million individuals require medical intervention for schistosomiasis, a chronic disease resulting from exposure to polluted water sources. According to the UNESCO (2021) World Water Development Report, around 1,000,000 people die each year as a result of using unsafe drinking water and lacking sufficient sanitation. This figure includes an estimated 395,000 children under the age of five. Therefore, comprehensive strategies are needed to mitigate the negative impacts of water pollution on plant and animal life as well as human health.

Water pollution is primarily caused by industrial, agricultural, and waste processing activities. Major contributing industries include those involved in paper production, food processing, metal manufacturing, and nuclear operations (Lin *et al*., 2022). These industries generate various hazardous chemical by-products, including organic, inorganic, toxic, and volatile organic compounds. Without proper management, these substances can lead to significant water contamination (Chowdhary *et al*., 2020). Common pollutants found in contaminated water include heavy metals such as cadmium, arsenic, lead, and chromium. The presence of these hazardous pollutants is largely caused by the industrial sector (Chen *et al*., 2019).

Heavy metals are widely recognized for their hazardous nature and their detrimental effects on human health. These elements, particularly when bioaccumulated, impair physiological functions, notably those of the kidneys and the brain, by interfering its regular function. Lead (Pb) is considered as one of the most hazardous of these, having serious effects on the environment and living organisms. Lead exerts its toxicity across multiple organ systems, with the nervous system being the most significantly affected. Due to its persistent nature, lead accumulates in the human body over time, contributing to neurological, gastrointestinal, and respiratory dysfunction (Wani *et al*., 2015). The presence of lead in aquatic environments is particularly concerning, as it often originates from both natural and anthropogenic sources. Natural sources include geological processes such as volcanic activity, while anthropogenic contributions stem predominantly from industrial and transportation-related activities (Zhang, 2015). Lead emissions from vehicles, primarily due to the combustion of leaded gasoline, as well as effluents from the metal-processing industry, have been shown to negatively affect soil and plant quality. Additional sources include battery manufacturing, coal combustion, and the production of household appliances. Once released, lead contaminants may become airborne, deposit onto terrestrial surfaces, and eventually leach into aquatic systems. According to Government Regulation of the Republic of Indonesia No. 22 of 2021, the maximum concentration of lead in water is 0.03 mg/L. Data reveal that in Semarang, Demak, and Kendal, Pb concentrations in sediments Semarang bay waters have been measured at 0.0522, 0.0557, and 0.0603 mg/L, respectively, surpassing the standards set by the Indonesian River Quality Standard, as assessed using the SNI 6989.46:2009 test method (Yusfaddillah *et al*., 2023).

The toxicity and environmental persistence of lead ions necessitate methods for their removal from groundwater and drinking water. To remove lead ions from water sources, a number of methods have been investigated, including adsorption, chemical precipitation, ions exchange, liquid membrane separation, and electrochemical treatment (Abdullah *et a*l., 2019; Azimi *et al.*, 2017). Among these, adsorption is the most widely used method due to its simplicity, cost-effectiveness, and high removal efficiency. Common commercial adsorbents include zeolite, alumina microsphere, biomass, and synthetic polymers (Shi *et al.,* 2017; Moreira & Genova, 2024; Sawicka *et al.,* 2024; Rekha *et al.,* 2024). In recent years, alginate—a naturally derived polysaccharide extracted from brown seaweed cell walls—has gained popularity as a promising sorbent for heavy metal ions. Alginate consists of 1,4-glycosidic linkages between guluronic acid (G) and mannuronic acid (M), forming a biopolymer structure that enables efficient binding with metal ions.Extensive research has been conducted on the removal of Pb^{2+} ions using alginate-based materials.

Alginate is frequently combined with other substances to enhance its adsorption capacity and mechanical stability. The incorporation of a secondary component into the primary polymer matrix not only introduces additional functional groups but also significantly improves the mechanical strength of the material—an essential property for ensuring the reusability and operational durability of adsorbents. Notable examples include the removal of Pb2+ and Ni(II) ions using an alginate–magadiite composite, achieving removal efficiencies of 88–96% for Pb2+ ions and 93–99% for Ni(II) (Attar et al., 2019); the use of calcium alginate beads doped with Caryota urens seed carbon for Pb2+ ions removal, with an adsorption capacity of 86.9 mg/g and 96% removal efficiency (Ravulapalli and Kunta, 2018); and the application of Chlorella vulgaris alginate (CVA) beads for Pb2+ ions adsorption, yielding a capacity of 133.8 mg/g and 99.4% removal (Goher et al., 2016). Additionally, immobilized coffee-alginate composites demonstrated even higher adsorption capacities, reaching 176.99 mg/g, respectively (Hanifah et al., 2021). These findings underscore the significant potential of alginate and its composites as efficient and sustainable adsorbents for the removal of Pb^{2+} ions contaminated aqueous systems.

Numerous studies have investigated the adsorption of heavy metal ions, many of which have applied the batch method (Al-Tameemi *et al.*, 2012; Yan *et al.*, 2022; Vancea *et al.*, 2023; Chauhan & Dikshit, 2023). In batch studies, the adsorbent is placed into a container containing the analyte, allowing contact between the two under controlled conditions. Key parameters such as pH, contact time, and adsorbent dosage are systematically varied to evaluate the adsorption capacity. This process occurs within a closed system, with no inflow or outflow of solution. In contrast, this focus on column approach for the adsorption of Pb^{2+} ions using coffee-biomass-alginate (CCA). This dynamic system is designed to simulate real-world conditions in wastewater treatment, thereby providing a more representative model for practical applications. Furthermore, the long-term sustainability of an adsorbent depends on its reusability. Accordingly, desorption studies were conducted to assess the regeneration potential of the adsorbents in this study. Overall, this study aims to conduct a column study to determine the time required for complete adsorption of Pb^{2+} ions and to evaluate the effectiveness of the adsorption-desorption process in a column system using CCA, which was previously reported to have a higher adsorption capacity than Ca-Alginate (Hanifah *et al.,* 2021).

Methods

Synthesis of CCA

The synthesis of coffee-calcium alginate (CCA) followed the procedure described by Hanifah *et al.* (2021), in which a 2% alginate solution was dropped into a 0.1 M $CaCl_2$ solution, with the addition of 0.25 grams of coffee biomass to the 2% alginate solution. The resulting CCA beads were soaked in $CaCl_2$ for 2 hours, then washed with distilled water to remove excess $CaCl_2$ until the pH reached 7. Subsequently, the CCA beads were dried for 48 hours.

Column Study

The adsorption of Pb^{2+} ions in the column study was conducted at a flow rate of 2.2 mL/min, using a column with a length of 10 cm and a diameter of 7 mm. A total of 0.5 g of CA was soaked in 20 mL of distilled water for 3 hours before being placed into the column. Deionized water at pH 4 was then flowed through the column. The Pb^{2+} ions feed solution was delivered via a hose connected to a peristaltic pump, allowing the solution to pass through and saturate the CA within the column. A second hose was used to recirculate the effluent back into the Pb^{2+} ions feed solution. To determine the duration required for completing adsorption of Pb^{2+} ions by the adsorbent, 5 mL samples of the feed solution were collected every 30 minutes. The Pb^{2+} ions concentration in each fraction was then analyzed using atomic absorption spectroscopy (AAS). An illustration of the column setup is shown in Figure 1. The result of column study is plotted as a C/Co ratio, where C is the Pb^{2+} ions concentration after adsorption, and Co is the initial Pb^{2+} ions concentration.

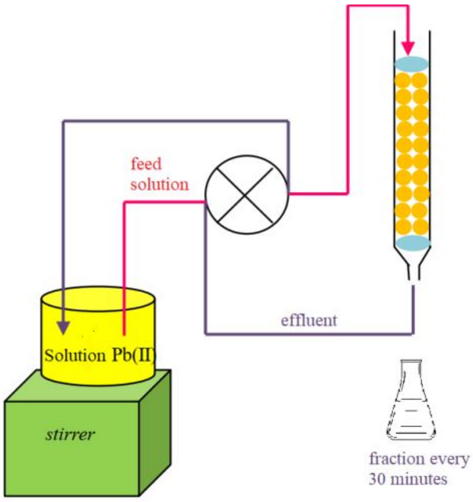

Figure 1. Column Study Set Up on Pb^{2+} Adsorpsion

Desorption of Pb^{2+}

Desorption of Pb^{2+} ions from the CCA adsorbent in the column was performed using 20 mL of 1 M HNO_3 at a flow rate of 2.2 mL/min. The HNO_3 solution was passed through the adsorbent, and effluent fractions were collected in every 5 mL volume. Each fraction was analyzed using atomic absorption spectroscopy (AAS) to determine the Pb^{2+} concentration. The percentage of desorption (% desorption) per fraction was calculated using the following equation, where Cf is the concentration of Pb^{2+} ions desorbed in each fraction (mg/L), $VHNO_3$ is the volume of HNO_3 per fraction (L), and mg Pb^{2+} is the total mass of Pb^{2+} adsorbed (mg).

$$\%\,Desorption = \frac{C_f\ V\ HNO_3}{mg\ Pb^{2+}}\ x\ 100\% \quad (1)$$

Adsorbent Reusability

Reusability refers to the ability of an adsorbent to repeatedly adsorb Pb^{2+} ions. To evaluate the reusability, the adsorbent was subjected to two adsorption–desorption cycles, and its performance was observed throughout each cycle.

% Adsorption

The percentage of removal (% adsorption) of Pb^{2+} ions by the adsorbent was determined by the following formula. The symbol % adsorption is the percentage of metal removal, Ci is the initial concentration of Pb^{2+} ions (mg/L), and Ce is the concentration of Pb^{2+} ions at equilibrium (mg/L). The %desorption formula, follow the % desorption equation above.

$$\%\,adsorption = \frac{Ci - Ce}{Ci}\ x\ 100\% \quad (2)$$

Result and Discussion

Column Study

The column adsorption method was applied to determine the minimum contact time required for CCA to adsorb all Pb^{2+} ions from the feed solution. Feed solution of 1 ppm Pb^{2+} ions solution was passed through the CCA packed column, and the resulting effluent was recirculated into the feed reservoir to maintain continuous contact with the adsorbent. This process was repeated until complete removal of Pb^{2+} ions by the CCA adsorbent was achieved. The minimum contact time required for total adsorption is presented in Figure 2.

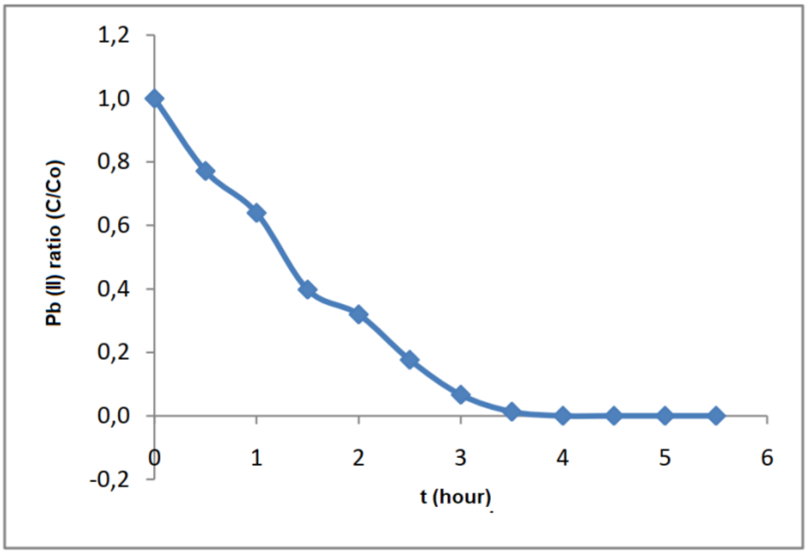

Figure 2. Ratio Pb^{2+} (C/Co) over time

Figure Figure 2 illustrates the variation in the residual Pb^{2+} ions concentration ratio (C/C_0) over time, where C represents the concentration of Pb^{2+} ions remaining in the solution and C_0 denotes the initial concentration. The observed decreasing trend signifies progressive adsorption of Pb^{2+} ions onto the CCA adsorbent, with complete adsorption achieved after approximately 3.5 hours. Initially, the adsorption rate is rapid due to the abundance of available active sites on the adsorbent surface. However, as these sites become progressively occupied, the adsorption rate decreases. This deceleration is attributed to increased competition among Pb^{2+} ions for the remaining limited active sites, thereby lowering the chance of further adsorption. Moreover, since adsorption is a dynamic equilibrium process, the rate of adsorption eventually approaches the rate of desorption, resulting in a plateau in the C/C_0 curve. This plateau, observed between 3.0 and 3.5 hours. This condition indicates that equilibrium has been reached and the maximum adsorption capacity adsorbent has been attained (Ponnuchamy *et al.*, 2021). This behavior highlights the finite nature of available adsorption sites on the adsorbent surface.

The performance of the CCA beads in this study can also be compared with other dynamic column adsorption studies. Nguyen et al. (2021) synthesized glue-free NaA-zeolite-coated metakaolin granules and applied them in fixed-bed columns to remove Pb^{2+} ions. Their findings indicated that at analyte concentration of 100 mg/L and a flow rate of 2.5 mL/min, with breakthrough occurring between 66 and 120 hours depending on column height and flow rate. These values required longer saturation times. In contrast, the CCA beads used in this study achieved complete Pb^{2+} ions removal within 3.5 hours at a lower concentration (1 ppm), highlighting their efficiency under lower contaminant loads and shorter operational timeframes.

In addition to the availability of active sites, factors such as flow rate, bed height, initial Pb^{2+} ions concentration, and adsorbent particle size significantly influence the adsorption kinetics and overall performance of the column (Nguyen *et al.*, 2010). Future research could explore the optimization of these parameters or the use of surface-modified adsorbents to enhance adsorption efficiency and delay saturation.

Desorption

The desorption process is an important step because, during this process, a number of Pb^{2+} ions previously adsorbed onto the CCA adsorbent are released. Desorption technology is practically applied to retrieve the maximum amount of adsorbed molecules from used adsorbents and to regenerate them for repeated use (Bayuo *et al.,* 2020). After the Pb^{2+} ions are removed, the CCA adsorbent can be reused for subsequent adsorption cycles, allowing the determination of its reusability through analysis of adsorption–desorption percentages. Desorption of Pb^{2+} ions was carried out using 1 M HNO_3, selected due to nitric acid's effectiveness in eluting metal ions from various adsorbents. The high concentration of H^+ ions facilitate ion exchange, thereby displacing Pb^{2+} ions from the adsorbent surface. Desorption was conducted via the column method by passing HNO_3 through the CCA packed column. The eluate was collected in 5 mL fractions and the amount of desorbed Pb^{2+} ions was quantified using atomic absorption spectrophotometry (AAS).

The mechanism of Pb^{2+} ions desorption from the CCA adsorbent involves two main pathways. The first mechanism is pH-driven: the carboxylate groups, which are abundant in alginate and coffee biomass (Choi *et al.,* 2021; Ningrum & Prayitno, 2024)—and are critical for Pb^{2+} ions binding—undergo protonation at low pH (pKa 3.38 and 3.65), leading to the release of Pb^{2+} ions (Hong *et al.,* 2016; Wu *et al.,* 2010). The second mechanism involves competitive binding, in which H^+ ions from HNO_3 displace Pb^{2+} ions from the active sites on the adsorbent. Understanding these mechanisms is essential for optimizing desorption conditions and enhancing the operational stability of the adsorbent in repeated use cycles. The average desorption results of Pb^{2+} ions are shown in Figure 3.

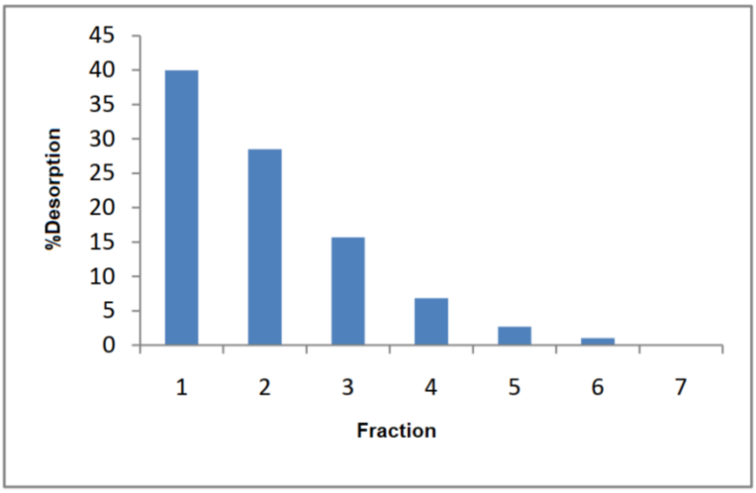

Figure 3. Percentage of Pb^{2+} Desorption per Fraction (v = 5 mL)

The first 5 mL fraction yielded the highest desorption percentage, followed by a gradual decline in subsequent fractions. By the seventh fraction, no detectable Pb^{2+} ions was eluted, indicating that most Pb^{2+} ions had already been desorbed. These findings confirm the effectiveness of 1 M HNO_3 in achieving rapid and complete desorption. Moreover, the desorption profile suggests that a relatively small volume of acid is sufficient, which could be advantageous in minimizing chemical usage and waste in practical applications.

Reusability

Reusability, or the ability of the adsorbent to be reused, was evaluated through two successive adsorption–desorption cycles using consistent experimental parameters. As shown in Figure 3, the desorption efficiency of Pb^{2+} ions decreased in the second and third cycles. This decline may be attributed to the strong interactions—such as electrostatic attraction or complex formation—between Pb^{2+} ions and the active sites on the adsorbent. The selection of acid type and its concentration can therefore serve as a reference for the development of a column-based desorption system. Despite the gradual decrease, the adsorbent demonstrated good reusability, with desorption efficiencies of 96.35%, and 86.64% as shown in figure 4. The desorption efficiency of CCA showed excellent performance compared to several other alginate and biomass-based adsorbents that have been reported. Attar *et al.* (2021) reported Pb^{2+} ions desorption efficiency of 93–98% in the first cycle using alginate–magadiite composite beads with 1 M HNO_3, while Ravulapalli & Kunta (2018) obtained a desorption efficiency of around 90% in the first cycle using calcium alginate beads doped with *Caryota urens* seed powder. Goher *et al.* (2016) also reported a desorption efficiency of 95% using Chlorella vulgaris alginate beads. This shows that CCA beads not only have good adsorption capacity, but also advantages in terms of regeneration and reuse, so they have the potential to be applied sustainably in the treatment of heavy metal-contaminated water.

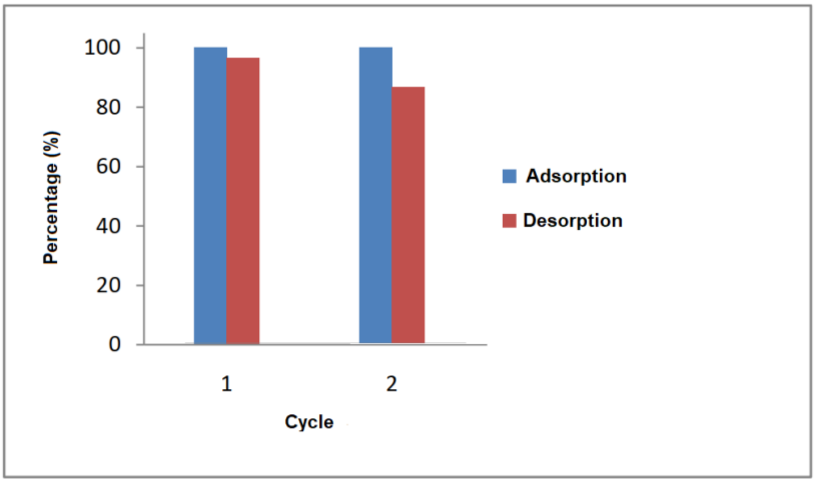

Figure 4. Adsorption and Desorpion (%) of Pb2+ in two cycles

The decrease in desorption efficiency in each cycle generally occurs due to several factors that affect the adsorbent's ability to optimally release heavy metal ions after repeated use. One of the main causes is the alteration or degradation of the adsorbent's physical and chemical structure, resulting from exposure to strongly acidic desorbing agents such as HNO_3. Repeated desorption processes may lead to the degradation, loss, or blockage of active functional groups (e.g., carboxylate groups in alginate or coffee biomass), thereby reducing the number of available active sites for binding and releasing Pb^{2+} ions in subsequent cycles (Attar *et al.*, 2021; Azimi *et al.*, 2017). Moreover, the occurrence of irreversible adsorption—where Pb^{2+} ions become strongly trapped within the adsorbent matrix and cannot be completely removed by the desorbing agent—also contributes to the reduction in desorption efficiency (Ravulapalli & Kunta, 2018). Another contributing factor is the morphological changes on the adsorbent surface, such as cracking or the breakdown of bead structures caused by repeated drying and rehydration cycles. These changes reduce the effective surface area and limit access to active sites. Additionally, residual metal ions or desorbing agents remaining on the adsorbent surface may block active sites, further hindering the desorption process in subsequent cycles (Goher *et al.*, 2016).

Conclusion

This study highlights the potential of coffee-calcium-alginate (CCA) beads as an effective and reusable adsorbent for the removal of Pb^{2+} ions from aqueous solutions. The CCA beads demonstrated significant lead adsorption capacity due to the presence of carboxyl functional groups derived from both alginate and coffee biomass. The column study informed that CCA beads have been effectively remove all the Pb^{2+} ions from the solution by 3,5 hours. The desorption mechanism was found to be influenced primarily by pH-driven protonation and competitive ion exchange with H^+ ions, enabling effective release of Pb^{2+} ions. By the seventh fraction, no detectable Pb^{2+} ions was eluted, indicating that most Pb^{2+} ions had already been desorbed from the CCA beads. Through two adsorption–desorption cycles, the CCA beads retained considerable reusability, with desorption efficiencies of 96.35%, and 86.64%. Further optimization of acid types and desorption conditions is recommended for enhancing column-based regeneration systems.

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