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Desorption Studies on Pb^{2+} and Cr^{3+} ions Contaminant and Re-generation of the Exhausted Zeolite-A Nanocomposite Adsorbent from Petroleum Refinery Wastewater, Kaduna-Nigeria

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Abstract

This research work is aimed at studying the desorption and regenerative characteristics of previously adsorbed lead (II) (Pb^{2+}) and Chromium(III) (Cr^{3+}) ions on petroleum refinery wastewater and were tested by various desorption eluents such as tap water, de-ionized water, NaOH, HNO_3 and CH_3COOH. Among them, CH_3COOH was chosen as the best desorbing agents for Pb^{2+} and Cr^{3+} ions, due to its high desorption efficiency. The desorption efficiency of CH_3COOH for Pb^{2+} and Cr^{3+} was about 82.7% and 91.4%, respectively, at a concentration of 0.2 M for the elution agents. The exhausted nanocomposite adsorbent was regenerated up to five cycles, and the removal efficiency of Pb^{2+} and Cr^{3+} on the recycled zeolite-A nanocomposite adsorbent could be maintained at 91.9% and 97.1%, respectively, in the third cycle. The successive regeneration cycles resulted in the reduction of the desorption efficiency by 14.6 % and 3.3%, for lead (II) and Chromium(III) ions, respectively, after the third cycle. The results show that exhausted zeolite-A nanocomposite adsorbent could be recycled when used to remove Pb(II) and Chromium(III) ions from Pb(II) and Chromium(III)-polluted water and wastewaters.

Keywords: **Adsorbent, Biosorption, Desorption, Eluent, Regeneration , Heavy metals**

Introduction

Environmental agencies worldwide are increasingly alarmed by the severe health risks posed by heavy metals in water bodies, highlighting an urgent need for effective removal strategies. Industrial expansion has substantially elevated levels of heavy metals in water systems, particularly chromium (III) and lead (II). Nigeria is not an exception because of their toxicity and potential health risks posed to biological organisms when consumed beyond the acceptable limits[2].

It is a fact that agriculture, mining and refining industries remain the major key contributors to chemical pollution in Nigeria. Vegetable and crop farming in Nigeria agriculture is prone to either misuse or overuse of fertilizers, pesticides and fungicides. Diseases and pests pose big challenges and problems in crop and vegetable production that usually compelled farmers to use

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chemical pesticides even if they have no technical know-how in application procedures.

Furthermore, the rapid population growth along water sources in Nigeria has necessitated proper conservation and efficient utilization of freshwater bodies due to heavy metals and other toxic chemicals. This is because there has been accelerated deterioration of water quality within water bodies due to increased domestic, municipal, industrial, mining and agricultural activities [10]. The sudden increase in indiscriminate petroleum refinery wastewater alone river banks in the local communities, especially in Nigeria, has led to concerns about the level of environmental pollution coming from these refining activities [15]. The emission of heavy metals into the environment from refining operations [13] will pollute the surface and underground water sources as well as agricultural soil.

When agricultural soils are polluted, these metals are taken up by plants consequently accumulate in their tissues [19]. Humans and animals that eat and graze on such contaminated plants and drink from polluted waters, as well as aquatic organisms that breed in heavy metal polluted waters, accumulate such toxic metals in their tissues and milk if lactating [11]. These accumulated heavy metals tend to cause various diseases and disorders in living organisms [20]. Chromium (III) and lead (II) are being classified by the United States Environmental Protection Agency (USEPA) as potentially hazardous to most living species [15]. Chromium (III) is well known due to its healthrelated issues in humans including carcinogenic and mutagenic risks [6 ; 9] while neurological disorders in humans, particularly children and in a fetus, are due to the chronic consequences of lead (II) [5;18].

Therefore, it is necessary to remove metal-contaminated water and wastewaters before its discharge to the environment. However, the disposal of the used adsorbent is again an environmental problem and constitutes a disposal problem. Hence, there is also the need to desorbed toxic ions on the surface of the used adsorbent to regenerate it for further use or to ensure safe disposal. Recently, the interest in desorption and re-utilization of adsorbents in metal recovery processes from water and wastewaters has deepened [17].

Desorption of ions can be performed to restore the adsorbent for further use [9, 6]. After desorption, the metals can be recovered by extraction from the liquid phase so that the naturally low-cost adsorbents could be preserved. Biosorbent regeneration is the supreme fundamental method in assessing its commercial applicability, especially, in both batch and continuous processes, when expensive and selective sorbents are used [10]. During desorption procedures, ions accumulated on the biomaterial can be removed easily under appropriate operating procedures, and the adsorbent recycled several times to decrease material cost [22]. Desorption can be

performed by proton exchange using acids, chelating agents or exchange with other ions [16].

The choice of desorbing agents depends on the kind of adsorbent used and the metals adsorbed. The removal of Cr(III) and Pb(II) ions from wastewater has been widely studied using nanoparticles and these studies have already shown that Cr(III) and Pb(II) ions could be adsorbed by zeolite-A nanocomposite, which has economic advantage compared to other adsorbents. Unfortunately, however, there is little study on the recovery of the Cr(III) and Pb(II) ions-laden adsorbents and regeneration of these adsorbents. The present study seeks to provide a greater understanding of the dynamic characteristics for desorption and regeneration of Cr(III) and Pb(II) ions adsorbed onto zeolite-A nanocomposite using different desorbing agents.

Recent research supports the growing interest in natural and modified biosorbents for Cr(III) removal [13,8], and underscores advancements in adsorption-desorption and regeneration techniques [8 ,22]. Zeolite-A nanocomposite adsorbent a readily available, low-cost biosorbent offers a promising solution, yet studies exploring its regeneration after desorption remain sparse. This study aims to elucidate the desorption behavior and regeneration potential of zeolite-A nanocomposite after adsorption of Pb(II) and Cr(III), using various eluents (tap water, deionized water, NaOH, HNO_3, CH_3COOH), focusing particularly on recovery efficiency and adsorbent reusability[5]. [9] investigated sorption and desorption studies of chromium (III) from the nonviable cyanobacterium Nostoc muscorum biomass.

Sorption–desorption of chromium into inorganic solutions and distilled water were observed and the study indicated that the biosorbent could be regenerated using 0.1 M HNO_3 and EDTA with up to 80 % recovery. The biosorbent was reused in five biosorption–desorption cycles without a significant loss in the biosorption capacity. [15] studied the regeneration of natural zeolite polluted by lead (II) and zinc (II) in wastewater treatment systems. Several desorbing solutions such as HNO_3, NaCl, KCl and NH_4Cl were examined for the removal of Pb(II) and Zn(II) from zeolite, and the highest desorption efficiency was obtained for 3 M KCl and 1 M KCl, respectively. KCl was the most effective solution for the desorption process exhibiting very high desorption efficiencies (> 98.5 %) for both metals. [17] conducted a study on the desorption and regeneration characteristics for previously adsorbed indium ions to phosphorylated sawdust. Various chemical reagents such as HCl, HNO_3, NaCl, ethylenediamine tetraacetic acid and nitrilotriacetic acid were tested on the desorption characteristics of indium ions adsorbed on phosphorylated sawdust. Among them, HNO_3 was chosen as the best desorbing agent with a desorption efficiency of 98% at a concentration of 0.5 M. A study was conducted by [12] on the adsorption and desorption of copper (II) ions onto garden grass. In their study, the desorption of

adsorbed Cu(II) from exhausted garden grass was explored using eight types of desorption eluents including tap water, Milli-Q water, distilled water, 0.1 M H_2SO_4, 0.1 M HCl, 0.1 M HNO_3, 0.1 M NaOH and 0.1 M CH_3COOH. It was found that 0.1 M H_2SO_4 was a suitable eluent, which could be used up to five cycles of adsorption-desorption.

Materials and methods

Adsorbent material and characterization

Doped zeolite-A nanocomposite powder was the adsorbent of interest in this study. The physicochemical composition of the zeolite-A nanocomposite was determined in our previous studies using Fourier transform infrared spectrometer (FT-IR), N_2 - adsorption for determining the specific surface areas (BET), X-ray Difraction(XRD), UV and scanning electron microscopy (SEM) [16].

Adsorbate solutions

The raw wastewater used in this study was collected from Kaduna refinery and petrochemical company (KRPC) Kaduna State, Nigeria. The wastewater was analyzed for physico chemical parameters based on the recommendation of standard methods of water and crude oil wastewater analysis at Federal Ministry of Agriculture Goning Gora Kaduna State, Nigeria. The heavy metals concentration of Pb (II) and Cr (III) ions were analyzed using Perkin Elmer A Analyst 3100 Atomic Absorption Spectrophotometer (AAS) using an air acetylene flame. The hollow cathode lamp was operated at 15 mA for Lead and at 25 mA for Chromium.

Desorption and Regeneration Experiment

The desorption, regeneration and reusability experiment was carried out. Reusability of the adsorbents is an important issue in the economic development for adsorption process [1].

A 2 g portion of the desorbed adsorbent was introduced into 100 cm^3 Erlenmeyer flasks containing 25 cm^3 of water. After equilibration for 60 min under room temperature, the adsorbent was recovered and the adsorption capacity determined.

Residual contaminant on the used adsorbent surface were detached through washing for three consecutive times using distilled water and then dried in an oven at 120 °C to constant mass. Five different desorption agents were used, tap water, de-ionized water, 0.2 M NaOH, 0.2 M HNO_3 and 0.2 M CH_3COOH, were used to desorbed the contaminant from the adsorbent. Fifty cm^3 of the desorption agents was kept in 100 cm^3 Erlenmeyer flasks with the recovered adsorbent and agitated for 60 min with continuous agitation at a 300 rpm at a temperature of 40 °C. The adsorbate adsorbent mixtures were filtered after equilibration using Whatman No.1 filter paper. The concentration of the contaminants in the filtrate was determined spectrophotometrically (AAS (A London, model 5000)) [3,6] methods, respectively.

The adsorbent used was zeolite-A nanocomposite, with surface area of

650.162(m^2/g), stock solutions of Cr(III) and Pb(II) (1000 mg/L) were prepared from petroleum refinery wastewater, then diluted to 25 mg/L working solutions [5]. Adsorption trials were conducted by mixing 2 g/L of zeolite-A nanocomposite with metal ion solutions and allowing equilibration for 60 minutes at ambient temperature.

After separation, the spent zeolite-A nanocomposite was washed, dried, and then subjected to desorption using 40 mL of different eluents (tap water, deionized water, 0.2 M NaOH, 0.2 M HNO_3, 0.2M CH_3COOH), agitated for 60 minutes at 120 rpm and 30 °C.

Desorption efficiency was calculated using standard formulas, and the dried zeolite-A nanocomposite was reused in up to five adsorption-desorption cycles to assess regeneration performance[5].

Percentage desorption was calculated using the equation below [6]:

$$\text{Desorption efficiency} = \frac{C_{des}}{C_{ads}} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

or

$$\text{Desorption (\%)} = \frac{\text{Released target pollutants}}{\text{Initially sorbed target pollutants}} \times 100 \ldots\ldots\ldots\ldots (2)$$

where C_{ads} denotes the amount of metal ions adsorbed and C_{des} is the concentration of metal ions desorbed. The recovered adsorbent was dried at 120 °C to constant mass, and so the regenerated adsorbent was used in adsorption–desorption cycles to determine the reusability of zeolite-A nanocomposite.

The reusability of the zeolite-A nanocomposite was conducted in five successive adsorption–desorption cycles. The amount of each contaminants desorbed from the zeolite-A nanocomposite into the solution per unit mass of adsorbent, Q_d (mg/g) at equilibrium was calculated by [6]:

$$Q_d = \frac{C_{des}}{m} \times V \ldots\ldots\ldots\ldots (3)$$

where C_{des} (mg/) is the liquid phase physicochemical and metal concentration detected in the desorbing solution at equilibrium conditions, m is mass of adsorbent and V (L) is the volume of the desorbing solution.

Results and Discussion

Desorption of Cr(III) and Pb(II) ions loaded on the zeolite-Ananocomposites.

Studies on desorption was conducted to choose the optimum desorbing agents (solution) to be employed in successive regeneration cycles.

The desorption experiments shown in Figure 1 prove that the 0.2 M CH_3COOH and 0.2M HNO_3 recovered about 76.1% and 56.0% of Cr(III) adsorbed on the adsorbent surface, respectively. Conversely, in Figure 2, 0.2 M CH_3COOH and 0.2M HNO_3 were able to recover 82.1% and 74.2% of the Pb(II) adsorbed on the adsorbent surface, respectively.

Desorption Performance

Acidic eluents outperformed others in recovering metal ions from the zeolite-A nanocomposite . Specifically, 0.2 M CH_3COOH achieved 76.1% desorption of Cr(III), while 0.2M HNO_3 recovered 82.1% of Pb(II). Other eluents water variants and NaOH were considerably less efficient, typically recovering less than 35% of ions [5]. The driving mechanism is likely ion exchange facilitated by functional groups (hydroxyl, carboxyl) on zeolite-A nanocomposite surfaces, rather than mere chemical sorption [6, 9]. The field has seen growing exploration of biosorbents such as activated palm coconut husks and groundnut husks achieving Cr(III) removal efficiencies ranging from 85% to over 91%, validating groundnut-based approaches [6,8].

Regeneration and Reusability

Desorption cycles showed diminishing returns: for Cr(III), removal efficiencies dropped from 73.4% (cycle 1) to 53.5% (cycle 3); Pb(II) declined similarly, from 81.3% to 54.6%. By the third cycle, overall desorption efficiency had decreased by approximately 20% (Cr) and 26.7% (Pb) [5]. This decline suggests irreversible sorption likely chemisorptions begins to dominate with repeated use. While the initial three cycles show promising reuse potential, efficacy deteriorates thereafter, limiting long-term economic viability. Comparatively, recent studies highlight engineered biochars and nanomaterials achieving near-total Cr(III) removal, often with superior regeneration robustness [2,10,4].

The desorption, regeneration and reusability experiment was carried out. Reusability of the adsorbents is an important issue in the economic development for adsorption process [1].

Desorption of Cr(III), Pb(II), loaded on zeolite-A nanocomposites

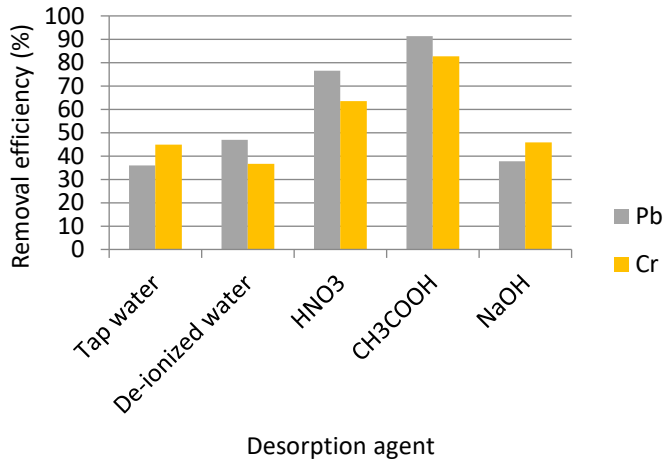


Figure 1: Desorption of Pb(II) and Cr(III) ions on to zeolite-A nanocomposites

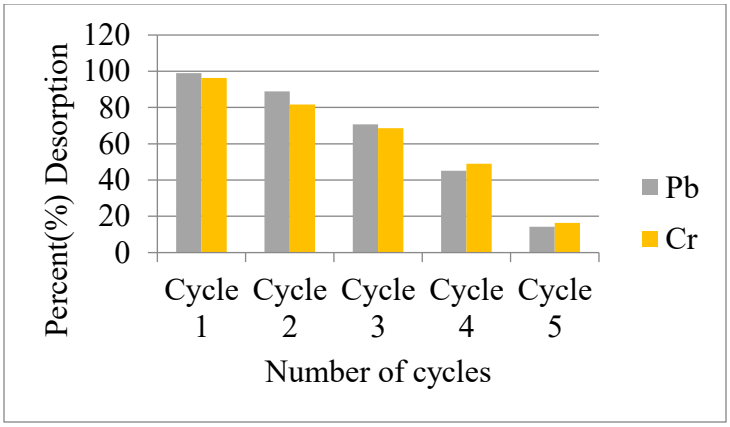


Figure 2: Adsorption–desorption of Pb(II) and Cr(III) ions and reusability of adsorbent Using 0.2M CH_3COOH as desorbing agent

Studies on desorption of Cr(III) and Pb(II) ions Loaded on to zeolite-A nanocomposites

Studies on desorption were conducted to choose the optimum desorbing solution to be used in successive regeneration cycles. The desorption experiments shown in Figure 1, 0.2 M CH_3COOH was able to recover 91.4 % and 82.7 % Pb(II) and Cr(III)ion adsorbed on the adsorbent surface, respectively using zeolite-A nanocomposites. Tap water, distilled water, HNO_3 and NaOH also showed low desorption percentages of 37.7 %, 37.1 %, 29.4 %, 28.9%, 41.7 % ; 46.6 %, 46.3 %, and 41.7 % for Pb(II) and Cr(III), ions, respectively using zeolite-A nanocomposite.

The study indicated that Cr(III), Pb(II) ions, accumulated onto the zeolite-A nanocomposite could be desorbed and maximum percentage recovery for zeolite-A nanocomposite was 91.4 %, 82.7 %, for Pb and Cr with 0.2 M CH_3COOH acid respectively. Therefore, 0.2 M CH_3COOH solutions was chosen, as desorption reagents for removing Cr(III) and Pb(II) ions from the zeolite-A nanocomposite surfaces.

The resultant desorption phenomenon observed in Figures 1 for Cr(III) and Pb(II) ions using CH_3COOH as desorption agents, might be attributed to ion exchange type interaction rather than chemical sorption [19]. Desorption is useful forth recovery and separation of contaminants in addition to the regeneration of the exhausted bisorbent [14]. The hydroxyl and carboxylic groups in the zeolite-A nanocomposite made it so to easy desorption and regeneration with the acid solutions [5,14,19,21]. The results of this study contradict the finding of [7] who studied on regeneration of chitosan after Cr(III) sorption. In their study, 0.1M HCl was the best desorption agent and maximum desorption efficiency of Cr(III) was found to be 88 % under the following optimum conditions: agitation speed of 300 rpm, a contact time of 60 min and temperature of 40 °C.

Furthermore, [7] investigated the desorption of Pb(II) and Cu(II) ions from chitosan-coated sand in a batch system using tap water and diluted HCl solutions. The results signified that more metal ions could be recovered by the HCl with desorption efficiency of 97.91 % and 99.22 % for Pb(II) and Cr(III), respectively.

Regeneration and reusability of the zeolite-A nanocomposites.

To ascertain adsorption-desorption several cycles were conducted for Cr(III) and Pb(II) ions, to determine the adsorption-desorption potential of the zeolite-A nanocomposite, once it had been regenerated several times. The reusability of the used adsorbent was investigated by performing five successive adsorption–desorption cycles of Cr (III) and Pb(II) ions on the adsorbent using 0.2 M CH_3COOH as desorption eluents. There was a gradual decrease in Cr (III) and Pb(II) ions, removal efficiencies with an increasing number of cycles as shown in figures 2. It was observed that cycles 1, 2 and 3 showed good retrieval of both contaminants accumulated on the adsorbent surface. For Cr(III) and Pb(II) ions for cycles 1, 2 and 3 were found to be 91.9 %, 97.1 %, 99.0 %, 96.4 % and 69.6 %, 75.5 %, as presented in

Figures 2. Hence, after the sequence of three cycles, the Cr(III) and Pb(II) ions using zeolite-A nanocomposite uptake capacity on the adsorbent was reduced from 91.5 to 14.6 % for zeolite-A nanocomposite. The regeneration experiments presented in Figures 2 showed that the zeolite-A nanocomposite could be reused repeatedly in the adsorption and recovery of Cr (III) and Pb (II) ions without significantly losing it's adsorption properties.

Generally, regeneration is a key factor in improving the economy of the adsorption process and accessing the potential of the adsorbent for commercial applications. From the regeneration studies presented in Figures2,Cr (III)and Pb(II) ions uptake capacity on zeolite-A nanocomposite was reduced after a sequence of three cycles. After this, no significant desorption was observed which indicated that a considerable part of the contaminants adsorbed was due to irreversible sorption.

Hence, chemisorption has an important influence on contaminants removal [7]. The loss in the uptake capacity of the zeolite-A nanocomposite for Cr(III) and Pb(II) ions uptake during the first three cycles was found to be 54.2 %. This could be attributed to the ignorable adsorbent load lost for the period of the adsorption–desorption cycles. The zeolite-A nanocomposite could be reutilized repeatedly in Cr(III) and Pb(II) ions adsorption processes since the Cr(III) and Pb(II) ions could be desorbed and adsorbent regenerated up to three cycles by 0.2 M CH_3COOH. This means that the adsorbent offered an economic benefit to be recycled repeatedly in Cr (III) and Pb (II) ions adsorption process. It has been found that if the used adsorbent could be regenerated and used again at the end of the first cycle, then the adsorbent is said to be economical and could be used commercially [23]. The findings of this study showed that 0.2 M CH_3COOH was the best desorbing agent and could be compared to the findings of other studies on desorption and regeneration of exhausted biosorbents.

In most studies, it is found that the desorption of contaminants in acidic media appeared to be rapid and higher than in basic and neutral media [22].

Conclusions

This research work was successfully conducted using a multiple doped zeolite-A nano adsorbent for petroleum refinery wastewater treatment with high adsorption capacity and high regeneration capacity. The synthesized adsorbents were characterized using XRD, XRF, SEM, UV-vis, FT-IR, EDX and BET surface analyzer. The XRD results revealed well crystalline adsorbents and the surface area of the zeolite-A nanocomposite was 650.162 m^2/g.

It can be concluded from various analysis conducted, that the developed adsorbents was suitable for refinery wastewater treatments (zeolite-A nanocomposite).

Batch adsorption–desorption studies were performed. It was observed that cycles 1, 2 and 3

showed good retrieval of both contaminants accumulated on the adsorbent surface. For Cr(III) and Pb(II) ions the removal efficiencies of cycles 1, 2 and 3 were found to be 91.9 %, 97.1 %, 99.0 %, 96.4 %, and 69.6 %, 75.5 %, as presented in Figures 2. Hence, after the sequence of three cycles, the Cr(III) and Pb(II) ions, using uptake capacity on the adsorbent was reduced from 91.5 to 14.6 % zeolite-A nanocomposite. The regeneration experiments presented in Figures 2 showed that the zeolite-A nanocomposite could be reused repeatedly in the adsorption and recovery of Cr(III) and Pb(II) ions without significantly losing its adsorption properties. After treating the spent adsorbent by varying pH of 4-12, the highest regeneration efficiency was achieved at a pH 7 by 0.2 M CH_3COOH after five cycles for zeolite-A nanocomposite.

Author contribution statement Kantoma Dogara: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper. Fatima Abubakar: Contributed reagents, materials, analytical tools or data, analyzed and interpreted the data. James Yusuf: Analyzed and interpreted the data.

Data availability statement Data will be made available on request.

Declaration of competing interest The authors declare no competing interests.

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