POTENTIAL OF USING $NaHCO_3$ AS A PRECIPITATION AGENT FOR THE REMOVAL OF CD(II) IONS

ORIGINAL SCIENTIFIC ARTICLE

Halid Junuzović✉*, Sabina Begić, Asija Cena, Mirsad Salkić, Amra Selimović, Indira Šestan, Melisa Ahmetović, Hana Alihodžić*

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Faculty of Technology, University of Tuzla

✉ halid.junuzovic@untz.ba

ABSTRACT:
Cadmium is recognized as one of the most hazardous heavy metals, ranking among the top ten in terms of toxicity. With the growing industrial reliance on cadmium for various manufacturing processes, concerns have risen within the scientific community regarding its presence in wastewater and the challenges associated with its removal. This research investigates the potential for removing Cd(II) ions from a synthetic aqueous solution by utilizing $NaHCO_3$ as a precipitating agent. The removal efficiency exceeded 99%, with the most effective conditions identified as: pH 8, a stirring speed of 300 rpm, a stirring duration of 5 minutes, and a precipitation agent of 90 mg. Additionally, removal efficiencies of 96.256% and 91.234% were achieved at cadmium concentrations of 150 mg/L and 300 mg/L, respectively. The removal of Cd(II) ions was found to be more efficient in a mixture of metals, with an efficiency above 98%, compared to when individual metals were considered in isolation.

KEYWORDS: Cd(II) ions, most effective conditions, removal efficiency

INTRODUCTION

Untreated or poorly treated wastewater, when discharged into natural water bodies, significantly contributes to the deterioration of water quality [1]. Wastewater contaminated with toxic heavy metals originates from several major industrial sectors, including textile production, mining operations, fertilizer manufacturing, leather tanning, and metal plating processes [2]. Heavy metals are used in various processes that include catalysis, then as parts of electrodes or some other industrial products, but after production they lose their value and become waste [3]. Toxic heavy metals present in wastewater is a great threat on human health, aquatic animals and plants [4]. The most commonly present toxic ions in wastewater are: Cd, Cr, Cu, Hg, Ni, Pb and Zn [5]. Accumulation of these elements in the human body may result in a range of harmful effects, posing significant risks to the nervous, hematological, digestive, urinary, and reproductive systems [6]. According to the Agency for Toxic Substances and Disease Registry, cadmium is on the list of the 10 most toxic metals [7]. High doses of Cd can cause problems with the proximal tubules in the kidneys and interfere with Ca metabolism in animals, which can lead to osteoporosis [8]. In children, it is most commonly absorbed in the gastrointestinal tract, due to more frequent hand-to-mouth behavior [9]. Adequate removal of toxic heavy metals from the environment is of great importance to the scientific community in order to ensure drinking water, protect the ecosystem and ensure sustainable food production [10]. Today, there are various methods of removing toxic heavy metals from water systems, which include: chemical precipitation [11], [12], [13] [14], adsorption [15], [16], [17], [18] ion exchange [19], coagulation-flocculation [20], flotation [21], reverse osmosis [22], membrane separation [23], [24], application of a magnetic field [25] and etc. Chemical precipitation method is a simple, inexpensive and with the minimal operational management for removing heavy metals from wastewater. It is typically non-selective for heavy metals, and is less sensitive to initial wastewater conditions [26]. The precipitation mechanism is reflected in the formation of insoluble metal precipitates by the reaction between free metal ions and the precipitation agent. The resulting precipitates are easily separated by sedimentation and filtration [27]. Many available relevant studies were conducted using hydroxide reagents such as NaOH, $Ca(OH)_2$ primarily due to their efficiency in wastewater treatment processes, however the use of these reagents may have certain limitations such as: high cost, use of larger amounts of reagents to achieve the appropriate pH considering that metals precipitate as hydroxy at higher pH values, huge amounts of gelatinous precipitate that is difficult to filter.

The use of sulfide reagents such as Na_2S for metal removal and extraction faces challenges due to the formation of a large number of submicron particles due to a high degree of supersaturation, which makes it difficult to control the precipitation process. Although metal sulfides have low solubility and theoretically high efficiency, practical application often results in significantly lower efficiency due to problems in separating the solid and liquid phases [28].

Carbonate precipitation occurs at less pH values and sludges are repoted to have good filtration characteristics [29]. Therefore, refined $NaHCO_3$, food grade from Sisecam Soda, Lukavac, Bosnia and Herzegovina was used in this study because it is considered a more economical and environmentally friendly alternative compared to NaOH, $Ca(OH)_2$ and Na_2S, due to its lower price, less toxicity and easier handling. The efficiency of removing toxic metals from aqueous solutions is over 99% using the chemical precipitation process [30]. However, the efficiency of removing toxic heavy metals depends on precipitation parameters such as: pH, initial concentration of metals, the nature of the precipitation agent, which actually directly affects the efficiency of the process [31]. Hence, this study investigates the parameters of chemical precipitation using the batch method, in order to achieve more efficient removal of toxic cadmium ions from synthetic aqueous solutions, using sodium bicarbonate ($NaHCO_3$) as an environmentally acceptable precipitation agent. The examined parameters include the pH value, the speed and duratation of stirring the solution, the dose of added bicarbonate, the initial concentration of cadmium ions, as well as the influence of the presence of coexisting metal ions and their mixtures.

MATERIALS AND METHODS

Materials

- Refined sodium bicarbonate $NaHCO_3$, food grade; Sisecam Soda, Lukavac, Bosnia and Herzegovina
- Cadmium nitrate tetrahydrate ($Cd(NO_3)_2{\cdot}4H_2O$), p.a. >99%, Centrohem, Stara Pazova, Serbia;
- Potassium hydrogen pthalate ($C_8H_5KO_4$), p.a. >99,5%, Fluka Chemica, Switezrland;
- Potassium dihydrogen phosphate (KH_2PO_4), p.a. >99,5%, Semikem, Sarajevo, Bosnia and Herzegovina;
- Di-sodium tetraborate 10-hydrate ($Na_2B_4O_7$ $_{10}H_2O$), p.a. >99,5%, Gram-mol, Zagreb, Croatia
- Sodium hydroxide (NaOH), p.a. >98%, Centrohem, Stara Pazova, Serbia;
- Hydrochloric acid (HCl), p.a. >35%, Lach:ner, Neratovice, Czehch Republic;
- Nitric acidd (HNO_3), p.a. >65%, Kefo, Ljubljana, Slovenia;
- Barium nitrate, ($Ba(NO_3)_2$), p.a. >99%, Kemika, Zagreb, Croatia;
- Cupper nitrate, ($Ca(NO_3)_2$), p.a. >99%, Kemika, Zagreb, Croatia;
- Lead nitrate, ($Pb(NO_3)_2$, p.a.>99%, Centrohem, Stara Pazova, Serbia;
- Demineralized water, <1 μS/cm, Euroaquaing, Tuzla, Bosnia and Herzegovina.

Instruments

- pH/mV/ion/Temp meter, BT-675, BOECO, Germany;
- Magnetic strirrer, IKA RCT basic;
- Optical Emission Spectrometer, OPTIMA 2100 DV, Perkin Elmer preciesly.

Buffer preparation

Buffer solutions of the desired pH values were prepared in 100 mL volumetric flasks as follows:

- pH 3: 0.1021 g of $C_8H_5KO_4$ and 22.3 mL of 0,1 M HCl;
- pH 4: 0.1021 g of $C_8H_5KO_4$ and 0.1 mL of 0,1 M HCl;
- pH 5: 0.1021 g of $C_8H_5KO_4$ and 22.6 mL of 0,1 M NaOH;
- pH 6: 0.681 g of KH_2PO_4 and 5.6 mL of 0,1 M NaOH;
- pH 7: 0.681 g of KH_2PO_4 and 29.1 mL of 0,1 M NaOH;
- pH 8: 0.681 g of KH_2PO_4 and 46.7 mL of 0,1 M NaOH;
- pH 9: 0.477 g of $Na_2B_4O_7{\cdot}10H_2O$ and 4.6 mL of 0,1 M HCl;
- pH 10: 0.477 g of $Na_2B_4O_7{\cdot}10H_2O$ and 18.3 mL of 0,1 M NaOH;

Method

An aqueous synthetic solutions of Cd(II) ions was prepared by accurately weighing a known mass of $Cd(NO_3)_2{\cdot}4H_2O$ into a 100 mL volumetric flask and diluting to volume with a buffer solution of the appropriate pH. The pH of the resulting solution was then measured, and the final pH was adjusted to the desired value by adding 1 M NaOH or HCl, using a pH meter for control. Precipitation was carried out at room temperature (±23 °C) in a 250 mL laboratory beaker by adding an appropriate amount of the precipitating agent $NaHCO_3$, with continuous stirring on a magnetic stirrer at a specified speed and duration. Afterward, the suspension was filtered using black and blue filter paper, and the residual concentration of the metal ion (mg/L) was determined using an ICP–OES instrument. The removal efficiency of Cd(II) ions from synthetic aqueous solutions using $NaHCO_3$ was calculated according to the following formula:

$$R = Ci - Cf/Ci$$

Where: R – removal efficiency (%), Ci – initial concentration of Cd(II) ions (mg/L), Cf – residual concentration of Cd(II) ions (mg/L).

RESULTS AND DISCUSSION

The precipitation of heavy metal ions from aqueous solutions primarily depends on two parameters: the initial concentration of metal ions and the pH value of the solution [32].

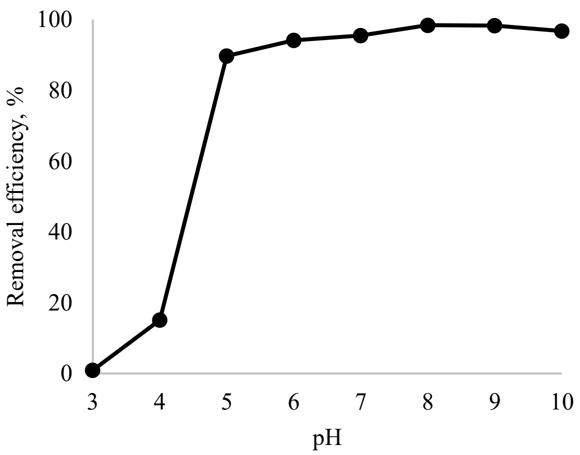

Figure 1. Influence of the pH value of the solution on the efficiency of Cd(II) ion removal using $NaHCO_3$

Bicarbonate precipitation conditions: initial concentration of Cd(II) ions (100 mg/L), solution stirring speed (300 rpm), dose of added precipitation agent (80 mg) and precipitation time (6 minutes)

Figure 1 shows the influence of the pH value of the solution on the precipitation of Cd(II) ions from aqueous synthetic solutions. Loughlaimi et. al. 2024 found that increasing the pH from 2 to 11 increases the efficiency of Cd(II) ion removal using CaO, in such a way that increasing the pH also increases the concentration of OH^- ions, creating an insoluble ion, which reduces the concentration of heavy metals in the solution [33]. Influence of pH value was examined in the interval of 3-10, since heavy metal ions in aqueous solutions show different behavior depending on the pH of the environment, because pH itself affects the solubility of metals [34]. At lower examined pH values, a lower removal efficiency of Cd(II) ions from aqueous synthetic solutions was observed (R<20%). However, by increasing the pH value, a significant increase in the removal efficiency of Cd(II) ions was observed, reaching a maximum of 98.406% at pH 8. The optimum pH that was found for using lime to remove Cd(II) ions by chemical precipitation was above 9.5, suggesting that a higher amount of precipitation agent is required to reach this value, given that metal ions in wastewater are typically present in an acidic pH range. [35]. By further increasing the pH value, a lower removal efficiency of Cd(II) ions was observed, due to the possible re-dissolution of the metal carbonate into the aqueous solution. Based on the obtained experimental data, adjusting the pH of the solution to a value of 8 was used for all other experimental examinations.

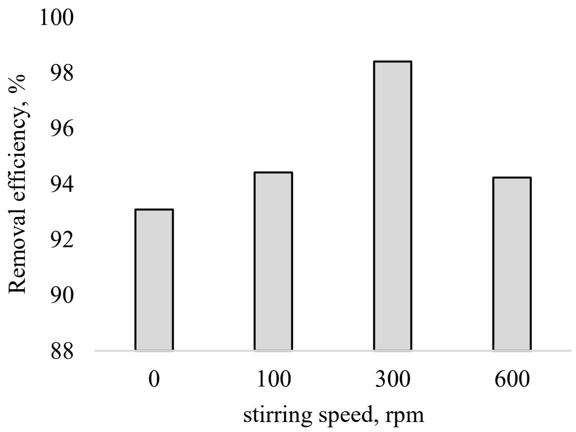

Figure 2. Influence of the stirring speed of the solution on the efficiency of Cd(II) ion removal using $NaHCO_3$

Bicarbonate precipitation conditions: pH value (8), initial concentration of Cd(II) ions (100 mg/L), dose of added precipitation agent (80 mg) and precipitation time (6 minutes)

The study of the influence of solution stirring speed on the removal of Cd(II) ions from aqueous synestetic solutions using $NaHCO_3$ as a potential precipitation agent is shown in Figure 2. The influence of stirring speed was investigated on a magnetic stirrer with the possibility of adjusting the stirring speed in the interval from 100 to 600 rpm. The removal efficiency of Cd(II) ion was also investigated in this study. Experimental data showed that the values of Cd(II) ion removal for stirring speeds of 0, 100 and

600 rpm were in a narrow range from 93% to 94%. However, the highest efficiency of Cd(II) ion removal was observed at 300 rpm (R>98%) and therefore this speed was chosen for the following experimental examinations. Junuzović et al. 2025 examined the removal efficiency of Pb(II) ions using hydrated lime as a precipitation agent at different speeds in the interval of 100-600 rpm and found that the highest removal efficiency was achieved, as in this study, at 300 rpm, while further increasing the stirring speed decreased the removal efficiency and explained that at lower stirring speeds, better contact between the metal ion and the precipitation agent was ensured [36].

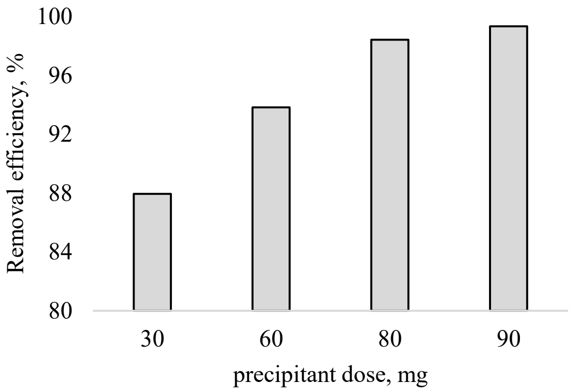

Figure 3. Influence of dose of added precipitant on the efficiency of Cd(II) ion removal using $NaHCO_3$

Bicarbonate precipitation conditions: pH value (8), stirring speed (300 rpm), initial concentration of Cd(II) ions (100 mg/L) and precipitation time (6 minutes)

Examination the influence of the dose of added precipitation agent, $NaHCO_3$, was performed at a predefined pH and stirring speed, and was examined in the interval of 30-90 mg as shown in Figure 3. The lowest Cd(II) ion removal efficiency was observed after adding 30 mg of precipitation agent and was 87.940%. By further increasing the dose of the added precipitation agent, a linear increase in the removal efficiency of Cd(II) ions was observed, and thus already at the addition of 60 mg, the removal efficiency was 93.820%. The efficiency of removing heavy metal ions is improved by increasing the dose of the precipitation agent [37]. The highest removal efficiency was seen at the highest dose of added precipitation agent (90 mg) and was 99.32%, which means that only 0.68 mg/L of unremoved Cd(II) ion remained in the solution. Based on the obtained experimental data, it can be concluded that with further increase of the precipitation agent dose, it would be possible to completely remove Cd(II) ions from the synthetic aqueous solution using $NaHCO_3$. Therefore, a dose of 90 mg was chosen, in addition to pH 8 and a stirring speed of 300 rpm in further studies for the removal of Cd(II) ions using $NaHCO_3$.

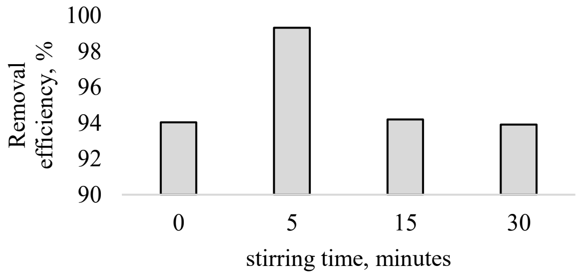

Figure 4. Influence of stirring time on the efficiency of Cd(II) ion removal using $NaHCO_3$

Bicarbonate precipitation conditions: pH value (8), stirring speed (300 rpm), dose of added precipitation agent (90 mg), initial concentration of Cd(II) ions (100 mg/L)

Figure 4 shows the influence of the stirring time of the synthetic aqueous solution of Cd(II) ions on the removal efficiency using $NaHCO_3$ as a potential ecofriendly precipitation agent. The influence of stirring times of 0, 5, 15 and 30 minutes was examined with previously defined precipitation parameters such as: pH value (8), stirring speed (300 rpm), dose of added precipitation agent (90 mg), while the initial concentration of Cd(II) was 100 mg/L. A high value of Cd(II) ion removal efficiency of 94.047% was achieved immediately after the contact of the precipitation agent and the metal ion (0 minutes). At a mixing time of 5 minutes, the highest removal efficiency of R>99% was recorded, while with a longer stirring time, the removal efficiency decreased, so that R=94.197% at 15 minutes and R=93.916% at 30 minutes. Therefore, a solution stirring time of 5 minutes was chosen for the following experimental examinations. Kartal et. al 2023 examined the influence of time on the efficiency of Cu(II) ion removal using Na_2S, and from their graphical results it is clearly visible that extending the precipitation time from 0 to 3500 seconds decreases the percentage of ion removal from 300 mg/L to 161.35 mg/L respectively [38].

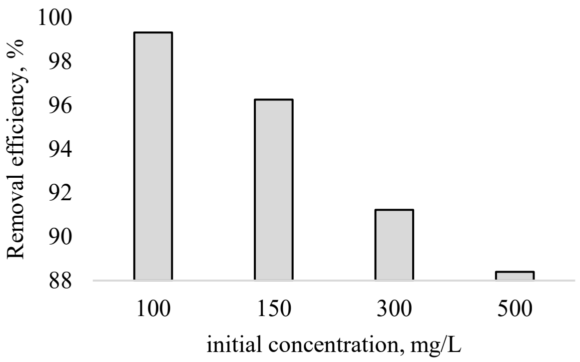

Figure 5. Influence of the initial concentration of Cd(II) ion on the efficiency of Cd(II) ion removal using $NaHCO_3$
Bicarbonate precipitation conditions: pH value (8), stirring speed (300 rpm), dose of added precipitation agent (90 mg) and precipitation time (5 minutes)

The examination of the influence of the initial concentration on the efficiency of Cd(II) ion removal using sodium bicarbonate is shown in Figure 5 with previously defined precipitation parameters such as pH value (8), stirring speed (300 rpm), dose of added precipitation agent (90 mg) and precipitation time (5 minutes). All tested concentrations of Cd(II) ions in this study were above the permitted values for the discharge of wastewater enriched with Cd(II) ions into surface water systems. The highest removal efficiency was achieved at the lowest initial metal ion concentration (100 mg/L) and was R>99%. However, increasing the metal ion concentration resulted in a decrease in the removal efficiency of metal ions, so already at a concentration of 150 mg/L, the removal efficiency was 96.256%. The removal efficiency of 91.234% was achieved at an initial concentration of 300 mg/L, while the lowest Cd(II) ion removal value was recorded at a concentration of 500 mg/L and was 88.4%. Although the mechanism of removal of some metal ions differs, the influence of initial concentration on the efficiency of metal ion removal is actually similar. In the study by Li et al. [39], the authors examined the removal efficiency of Cd(II), Cu(II), and Pb(II) ions by adsorption and graphically presented the dependence of removal efficiency on the initial metal ion concentration. The results clearly show that increasing the metal ion concentration leads to a decrease in removal efficiency, which can also be expected in the precipitation process. Also, the authors confirmed that the removal rate of heavy metal ions using lignite and MML as adsorbents decreases with the increase of initial concentration [40].

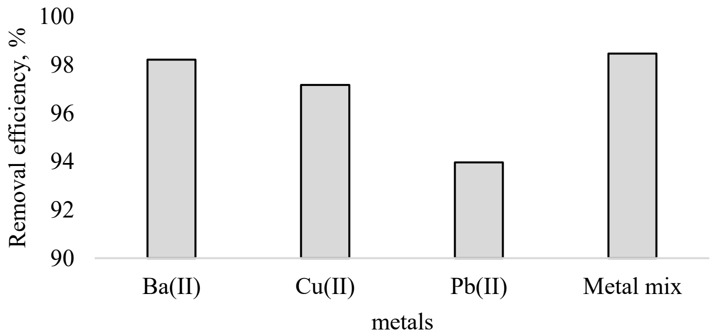

Figure 6. Influence of co-existing metal ions and metal ion mixture on the efficiency of Cd(II) ion removal using $NaHCO_3$
Bicarbonate precipitation conditions: pH value (8), stirring speed (300 rpm), dose of added precipitation agent (90 mg), stirring time (5 minutes) and initial concentration of Cd(II) ions (100 mg)

The influence of co-existing ions and a mixture of metal ions with an initial concentration of 100 mg/L on the precipitation of Cd(II) ions under predefined conditions of bicarbonate precipitation in Figures 1-5 was examined. The selected initial concentration of 100 mg/L of co-existing ions was chosen in order to demonstrate the selectivity of binding and the specificity of the interaction between metal ions, and in this way, the competitiveness of binding can be examined and to understand how different ions compete for the same place in the process of bicarbonate precipitation, which is of great importance for understanding at the molecular level. The efficiency of precipitation, in addition to pH, type and concentration of metal, used precipitation agent, and reaction time, depends on the presence of some other components that can inhibit the main reaction [41]. The lowest removal efficiency of Cd(II) ions was observed in the presence of Pb(II) ions (R = 93.969%). Better removal efficiency of Cd(II) ions was in the presence of Cu(II) ions and amounted to R=97.164%, while slightly better removal efficiency was in the presence of Ba(II) ions (R=98.213%). However, the best removal efficiency of Cd(II) ions was actually achieved when Ba(II), Cu(II) and Pb(II) ions co-existed in the solution together forming a mixture (R=98.466%). The highest removal efficiency of ions (Cu(II), Al(III), Fe(III), Mn(II) and Zn(II)) in the mixture using NaOH as a precipitant from acid mine drainage was at the same pH of 8.2 [42]. Metal ions in real systems are mostly in mixtures, they co-exist at the same time, and these results show that it is possible to effectively remove Cd(II) ions using $NaHCO_3$ from a mixture of metal ions, and the results are of great importance for understanding the specificity of interaction and selectivity of Cd(II) ion binding.

CONCLUSION

This study investigated bicarbonate precipitation of Cd(II) ions and was performed by the batch method at room temperature using $NaHCO_3$ as an environmentally acceptable precipitant. Refined food-grade $NaHCO_3$ was used due to its large-scale availability and lower cost compared to other precipitation agents such as NaOH, $Ca(OH)_2$, and Na_2S. The bicarbonate precipitate obtained was less gelatinous, which reduced the filtration time, and the handling of this precipitant in the precipitation process is less toxic compared to the others mentioned. Precipitation conditions were tested, including: pH, initial concentration, solution stirring speed, solution stirring time, dose of added precipitant, as well as the influence of coexisting metal ions in order to achieve the highest removal efficiency. By adjusting the buffered solution of Cd(II) ions with an initial concentration of 100 mg/L to pH 8 and maintaining the stirring speed and time at 300 rpm and 5 minutes with the addition of 90 mg of $NaHCO_3$, it is possible to achieve a removal efficiency of even 99.3196%. Precipitation of Cd(II) ions directly depended on the initial pH and metal ion concentration. Increasing pH and initial concentration above the most favorable conditions led to a decrease in removal efficiency, respectively. Also, high Cd(II) ion removal efficiency was achieved even at the highest metal ion concentration (500 mg/L) of 88.4%.

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