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Influence of Selected Conditions on the Efficiency of Carbonate Precipitation of Cu(II), Ni(II), Pb(II) and Zn(II) Ions

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Optimal process conditions for carbonate precipitation of selected heavy metal ions were tested in laboratory conditions using Na_2CO_3. To the prepared synthetic monocomponent and binary multicomponent solutions of heavy metals with initial concentrations of 500 mg/L, Na_2CO_3 was added in certain doses at selected mixing speeds (0, 100, 300 and 800 rpm) and mixing time (0, 15, and 30 minutes). The results show the removal efficiency at optimal mixing speeds for monocomponent metal solutions were: Cu(II) 96.394% (300 rpm), Ni(II) 94.594% (0 rpm and 800 rpm), Pb(II) 75.968% (0 rpm), Zn (II) 99.311% (0 rpm). In binary multicomponent mixtures Cu(II)-Ni(II) and Pb(II)-Zn(II) the removal efficiency results at optimal mixing speeds were: Cu(II) 96.394% (100 rpm), Ni(II) 95.528% (800 rpm), Pb(II) 99.536% (300 rpm), Zn(II) 98.945% (100 rpm). Also, the results of the efficiency of heavy metal removal due to the influence of the contact time of the precipitant and heavy metal ions in monocomponent solutions show the following values: Cu(II) 99.940% (0 min), Ni(II) 94.612 % (0 min), Pb(II) 77.925 % (15 min), Zn(II) 99.324% (30 min), while in binary multicomponent mixtures Cu(II)-Ni(II) and Pb(II)-Zn(II) they were for Cu(II) 96.247% (30 min), Ni(II) 95.521% (0 min), Pb(II) 99.350% (30 min) and Zn(II) 98.944% (0 min). Examination of the influence of the mixing speed of monocomponent solutions showed that the efficiency of removing heavy metal ions was in most cases the best without mixing. Effect of metal-precipitant contact time on the efficiency of heavy metal ion removal showed that in half of the examined metals, the optimal values were chosen as the best (0 and 30 min). It can be concluded that this method based on chemical precipitation using Na_2CO_3 with optimal parameters such as contact time and mixing speed, can be used in the treatment of industrial wastewater.

Keywords: Carbonate precipitation; heavy metals; Na2CO3; removal efficiency; mixing speed; time.

1. INTRODUCTION

The definition of heavy metals most often refers to metals whose specific density is greater than 5 g/cm^3 (Jaishankar et al., 2014). Heavy metals are found in nature in very low concentrations, however, in larger quantities they are very dangerous (Das et al., 2011). Heavy metals are major environmental pollutants and are mainly of anthropogenic origin, most often commercial and industrial, but they are also naturally found in the biosphere. The growth of the world's population and industrialization are creating large amounts of wastewater that contain large amounts of heavy metals and are therefore a threat to the environment (Mitra et al., 2022). The reason why researchers around the world are assessing the concentrations of heavy metals in the air, water and soil is that millions of people are affected by this problem (Balali-Mood et al., 2021). A global problem today is the pollution of natural waters with heavy metals such as Cu(II), Ni(II), Pb(II) and Zn(II) which are persistent in the environment, bioaccumulate and biomagnificate in the food chain and are toxic (MathuMitha et al., 2021). Exposure to heavy metals can cause consequences such as ingestion, inhalation, and dermal absorption (Abd Elnabi et al., 2023). Therefore, there is great importance for the removal of heavy metals from the environment, and according to a review of the literature, there are various methods for removing these toxic substances. Methods for removing heavy metals from natural waters include adsorption, flotation, ion exchange, membrane-based filtration, coagulation, flocculation, phytoremediation, electrochemical methods and chemical precipitation (Gahrouei et al., 2024, Türkmen et al., 2022, Fei and Hu, 2023, Dhokpande et al., 2024). These techniques, apart from being economically expensive, have disadvantages like incomplete metal removal, high amount of reagents or energy requirements, and generation of toxic sludge or other waste products that require disposal (Abbar et al., 2017) Of these methods, chemical precipitation is most widely practiced in industry, mainly for the simplicity of process control, effective over a wide range of temperature and low cost of operation (Chen et al., 2018, Stec et al., 2020). Chemical precipitation refers to the method by which heavy metals are removed by converting into solid particles under the influence of an appropriate agent by adjusting the pH (Mazur et al., 2018). Moreover, chemical precipitation enhances the overall quality of treated wastewater by purging it of harmful contaminants through precipitate formation, rendering it safer for discharge or reuse (Saengchut et al., 2024). The thermodynamic driving force causing precipitation is called supersaturation (Lewis, 2017). However, the method is very pH sensitive and with small changes in the pH of the solution, it is therefore very important to maintain the pH at the specified value (Rodriguez-Freire et al., 2020). The main advantage of carbonate precipitation compared to hydroxide precipitation is actually the fact that better removal efficiency of heavy metal ions is achieved at lower pH values, usually in the range of pH 7 to 9, which is the reason for choosing pH 8 in this research (Selimović et al., 2020). It could be achieved using sodium carbonate or calcium carbonate. It could have less sludge volume, but it could release CO_2 bubbles and needs higher reagents for efficient precipitation (Qasem et al., 2021). Apart from pH as the main factor on the efficiency of removing heavy metal ions, a very important parameter is the initial concentration of heavy metal ions (Maulin et al., 2021). Elzahabi and Yong, 2001 state in their research that the most frequently found metals in leachate solution are lead, copper, zinc, cadmium, chromium and nickel. The concentration of these heavy metals varies from 0 to 100 ppm in municipal solid waste leachate to 100–10,000 mg/L in sewage sludge, mining wastes and various industrial wastes (Elzahabi and Yong, 2001). Therefore, lead, copper, zinc and nickel ions were selected for this research and their initial concentration was 500 mg/L in order to simulate wastewater from sewage sludge. The most commonly used precipitant for the removal of heavy metals is lime, however, a good alternative that also respects the principles of green chemistry and provides good removal efficiency is actually carbonate precipitation using $CaCO_3$ and Na_2CO_3 (Li et al., 2020). Therefore, based on the previous experience of the authors of this paper on the topic of carbonate precipitation of metal ions using Na_2CO_3, this paper investigates the influence of very important process conditions on the precipitation of metal ions in monocomponent and binary multicomponent solutions, namely the mixing speed (rpm) and the mixing time of the solution (min.). The novelty of this research work is its aim to investigate the influence of mixing speed and metal and precipitant contact time on the efficiency of removing heavy metal ions Cu(II), Ni(II), Pb(II) and Zn(II) in monocomponent and binary multicomponent solutions using Na_2CO_3 as an alternative precipitant. Also, the importance of this research is reflected in the lack of available scientific papers on the topic of the influence of mixing speed and contact time on the efficiency of metal removal using carbonate precipitation.

2. MATERIALS AND METHODS

2.1 Instrumentation

Instruments used for the experimental part of this work were: Atomic absorption spectrometer, Perkin Elmer Aanalyst 200, pH meter GLP Crison, analytical balance Kern ADDB, (0,001 g)

2.2 Chemicals and Reagents

During the experimental work, the following chemicals were used: standard solution of copper 1000 mg/L Cu(II) in 0.5 M nitric acid (from $Cu(NO_3)_2$), standard solution of nickel 1000 mg/L Ni(II) in 0.5 M nitric acid (from $Ni(NO_3)_2$), standard solution of lead 1000 mg/L Pb(II) in 0.5 M nitric acid (from $Pb(NO_3)_2$) and standard solution of zinc 1000 mg/L Zn(II) in 0.5 M nitric acid (from $Zn(NO_3)_2$) from Merck, Germany. Standard solutions of the mentioned heavy metal ions by dilution were used to prepare a series of standard solutions of exactly known concentrations, in order to determine the concentration of heavy metal ions by the FAAS method after the carbonate precipitation process. As precipitant in this work was used Na_2CO_3, min. 99.30% from Sisecam Soda Lukavac, Bosnia and Herzegovina. Nitrate salts of heavy metals were used to prepare monocomponent and binary multicomponent metal solutions with an initial concentration of 500 mg/L to simulate wastewater: $Cu(NO_3)_2 \cdot 3H_2O$, Pliva Zagreb, $Ni(NO_3)_2 \cdot 6H_2O$, Semikem, Sarajevo, $Pb(NO_3)_2$, Alkaloid, Skopje, $Zn(NO_3)_2 \cdot 6H_2O$, Kemika, Zagreb. These salts were pure analytical grade (p.a > 99%), blue and black ribbon circle, Fioroni, France.

2.3 Dose of Added Precipitant (Na_2CO_3)

In this research, the precipitant Na_2CO_3 was added with a concentration of 2 g/L as an alternative to a more toxic and expensive precipitant such as NaOH. Table 1. shows the doses of precipitant used in this work in order to achieve the appropriate pH 8 at which the carbonate precipitation of the mentioned ions from monocomponent and binary multicomponent systems was performed.

2.4 General Procedure

Carbonate precipitation was carried out in such a way that in 100 mL solutions of monocomponent metals Cu(II), Ni(II), Pb(II) and Zn(II) initial concentrations of 500 mg/L and binary multicomponent solutions of metals Cu(II)-Ni(II) and Pb(II)-Zn(II) initial concentrations of 500 mg/L added a certain dose of precipitant Na_2CO_3. Precipitant was also added to adjust pH 8 for all metals in monocomponent and binary multicomponent solutions. After the specified time of mixing the solutions, the solutions were filtered first through the black and then through the blue ribbon circle. On the same day, metal samples were measured on FAAS, and then the efficiency of removal of metal ions from the solution was calculated (E, %):

$$E = Ci – Cf /Ci \cdot 100$$

where: E, % removal efficiency, Ci – initial metal concentration (mg/L), Cf – final metal concentration (mg/L). Data are given as the mean of three replicates.

Table 1. Dose of precipitant Na_2CO_3

Metals	Dose of Na_2CO_3, mL/100 mL
Cu(II)	75
Ni(II)	2
Pb(II)	7
Zn(II)	40
Cu-Ni(II)	3
Pb(II)-Zn(II)	10

3. RESULTS AND DISCUSSION

3.1 Influenence of solution mixing speed on the removal efficiency of Cu(II), Ni(II), Pb(II), Zn(II), Cu(II)-Ni(II) and Pb(II)-Zn(II) solutions

Fig. 1. – Fig. 4. show the effect of mixing the solution (rpm) on the efficiency of removal of Cu(II), Ni(II), Pb(II), Zn(II) ions at different speeds from monocomponent solutions. The experimental results show that the mixing speed for each individual heavy metal ion had a different effect on the removal efficiency. Thus, for Cu(II) ions the highest removal efficiency was 99.952% at 300 rpm, for Ni(II) ions the highest removal efficiency was 94.594% at speeds of 800 rpm and without mixing, for Pb(II) ions the highest removal efficiency was 76.67% at 100 rpm and the highest removal efficiency of Zn(II) ions was 99.311% without mixing. It can be seen from the results that the efficiency of removing heavy metal ions is affected by the mixing speed individually, which means that the type of metal is still a key factor in determining the optimal mixing speed. Hummadi et al. 2023 investigated the removal of Cu(II), Ni(II) and Zn(II) ions and found that increasing the mixing speed of the solution resulted in a decrease in the removal efficiency (Hummadi et al., 2023).

In Fig. 5 and Fig. 6 show the influence of mixing on the efficiency of ion removal in binary multicomponent solutions Cu(II)-Ni(II) and Pb(II)-Zn(II). The highest removal efficiency of Cu(II) ions was 96.3394% at a speed of 100 rpm, Ni(II) 95.528% at a speed of 800 rpm, Pb(II) 99.536% at a speed of 300 rpm and Zn(II) 98.954% at a speed of 100 rpm respectively. Abu-Zurayk et al. 2017 investigated the influence of mixing speed on the efficiency of removing heavy metals Pb(II) and Cr(III) in binary multicomponent solution in the interval of 200 – 1000 rpm and found that at 800 and 1000 rpm the percentage of heavy metal ions removal was the same and concluded that higher efficiency is definitely related to the formation of strong turbulence, which consequently will decrease in the external mass transfer resistance thickness around the metal particles. At higher mixing speed, the decrease in efficiency may be due to improper contact time between the metal ions and the binding sites (Rund et al., 2017).

3.2 Influenence of Time on the Removal Efficiency of Cu(II), Ni(II), Pb(II), Zn(II), Cu(II)-Ni(II) and Pb(II)-Zn(II) Solutions

In the case of the influence of time on the removal efficiency of heavy metals, Cu(II) and Ni(II), it was shown that the highest removal efficiency was without mixing and these percentages were 99.940% and 96.612%, respectively. Unlike Cu(II) and Ni(II), Pb(II) ions had the highest removal efficiency at 15 minutes (77.925%), while Zn(II) ions needed the longest time to achieve the highest efficiency removal (99.324%). Fathy et. al 2024 examined the effect of time on the removal efficiency of metal ions Pb(II), Cu(II) and Cd(II), and the results of the study showed that the mentioned metals under the influence of different contact times had different removal efficiency values, which is in accordance with this study (Fathy et al., 2025).

When it comes to mixtures of heavy metal ions, the influence of time on the removal efficiency of heavy metal ions was such that Cu(II) ions in a mixture with Ni(II) needed a time of 30 min to achieve the best removal efficiency, namely 96,247 %, while Ni(II) ions needed the shortest time (0 min) to achieve the best removal efficiency, 95.521%. Junuzović et al. 2019, investigated the removal of Cu(II) and Ni(II) from their binary multicomponent aqueous solutions in which the initial concentrations of both metals were 500 mg/L, both metals showed the same precipitation kinetics, with the efficiency of Cu(II) removed being slightly higher than Ni(II) at conditions of 300 rpm and a contact time of 5 minutes (Junuzović et al., 2019). Pb(II) and Zn(II) ions in the binary multicomponent mixture behaved similarly to Cu(II) and Ni(II) ions in the mixture, so the highest removal efficiency of Pb(II) ions was achieved at the longest contact time between the precipitant and the metal (30 min) and was 95.350%, while for Zn(II) ions the removal efficiency was 98.944% at the shortest mixing time (0 min.).

3.3 Statistical Analyses

Since in each statistical factor the agreement between the measured values is good, it is less than the critical factor.

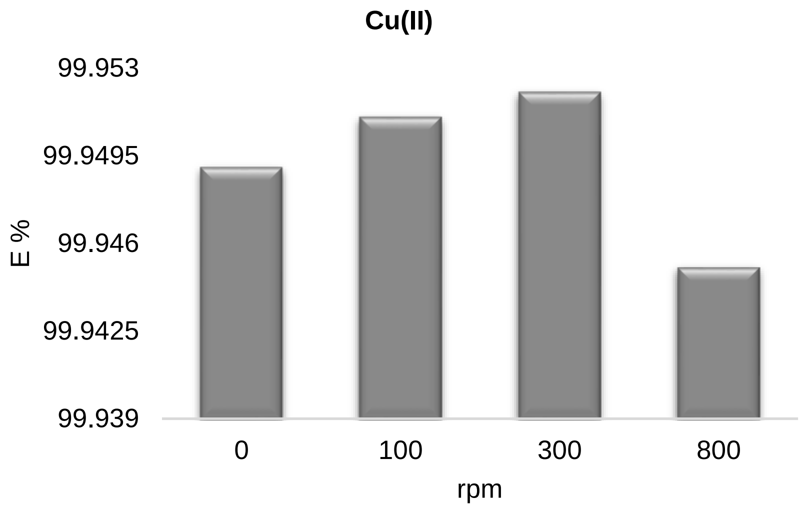

Fig. 1. Influence of solution mixing speed (rpm) on the removal efficiency of Cu(II) ions using sodium carbonate

Conditions: Cu(II) 500 mg/L, pH 8, t = 5 minutes

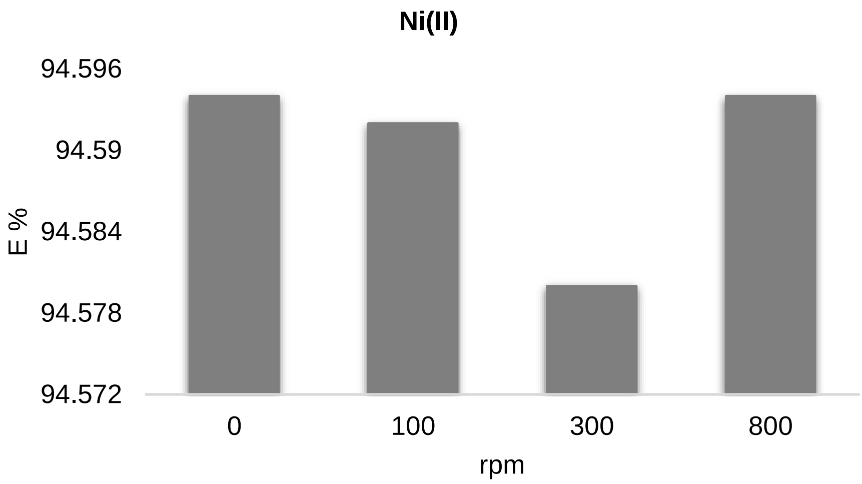

Fig. 2. Influence of solution mixing speed (rpm) on the removal efficiency of Ni(II) ions using sodium carbonate

Conditions: Ni(II) 500 mg/L, pH 8, t = 5 minutes

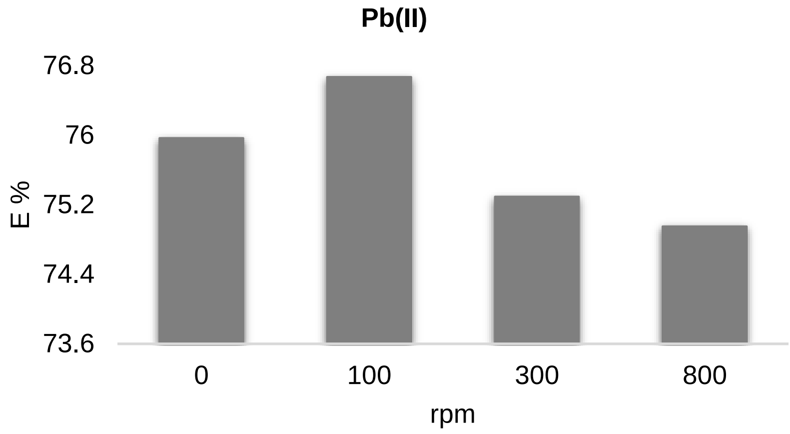

Fig. 3. Influence of solution mixing speed (rpm) on the removal efficiency of Pb(II) ions using sodium carbonate

Conditions: Pb(II) 500 mg/L, pH 8, t = 5 minutes

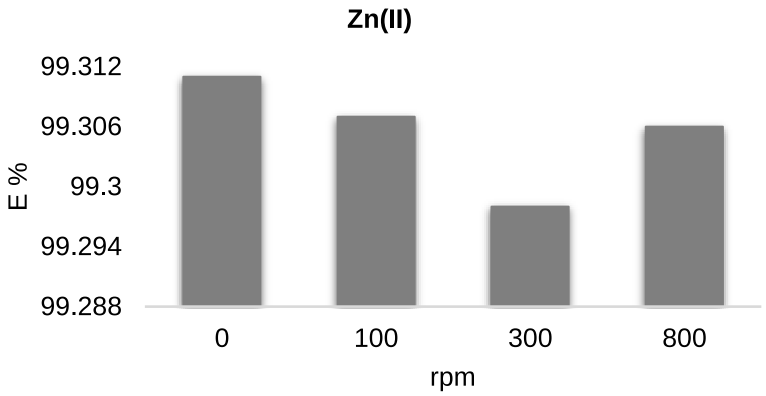

Fig. 4. Influence of solution mixing speed (rpm) on the removal efficiency of Zn(II) ions using sodium carbonate

Conditions: Zn(II) 500 mg/L, pH 8, t = 5 minutes

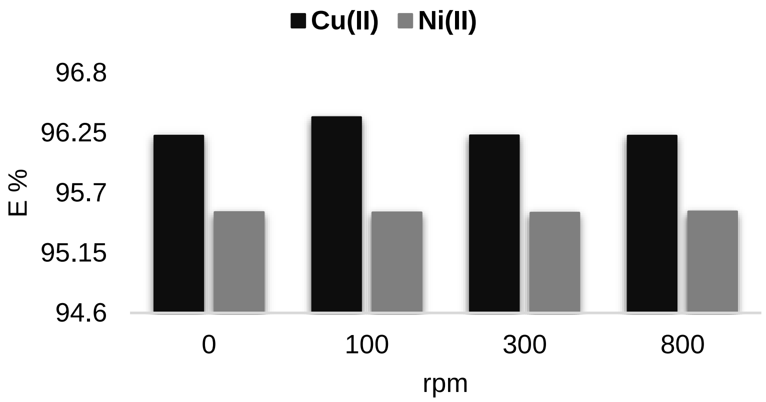

Fig. 5. Influence of solution mixing speed (rpm) on the removal efficiency of Cu(II) and Ni(II) ions in a binary multicomponent system using sodium carbonate

Conditions: Cu(II) and Ni(II) 500 mg/L, pH 8, t = 5 minutes

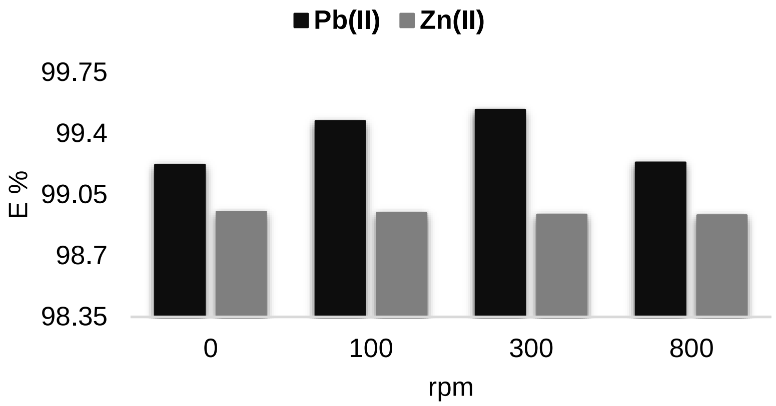

Fig. 6. Influence of binary solution mixing speed (rpm) on the removal eficiency of Pb(II) and Zn(II) ions in a binary multicomponent system using sodium carbonate
Conditions: Pb(II) and Zn(II) 500 mg/L, pH 8, t = 5 minutes

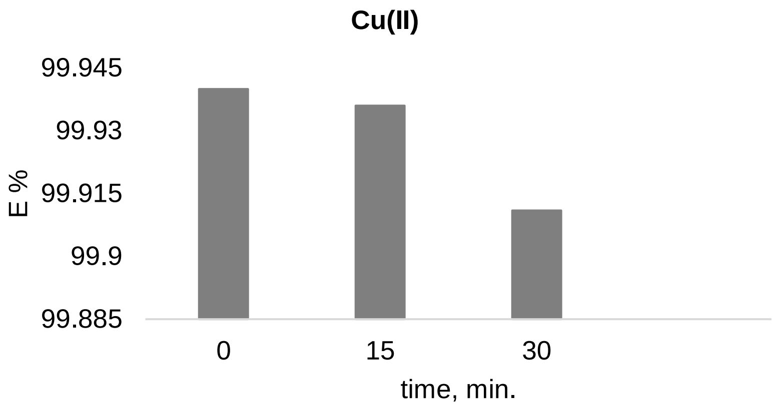

Fig. 7. Influence of time (minutes) on the removal efficiency of Cu(II) ions using sodium carbonate
Conditions: Cu(II) 500 mg/L, pH 8, 300 rpm

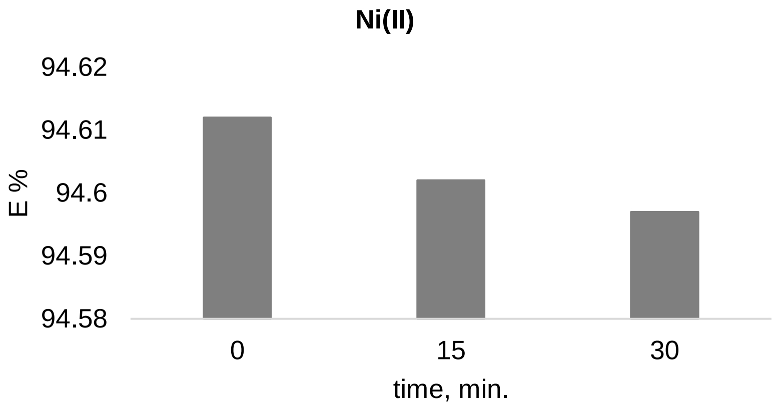

Fig. 8. Influence of time (minutes) on the removal efficiency of Ni(II) ions using sodium carbonate
Conditions: Ni(II) 500 mg/L, pH 8, 300 rpm

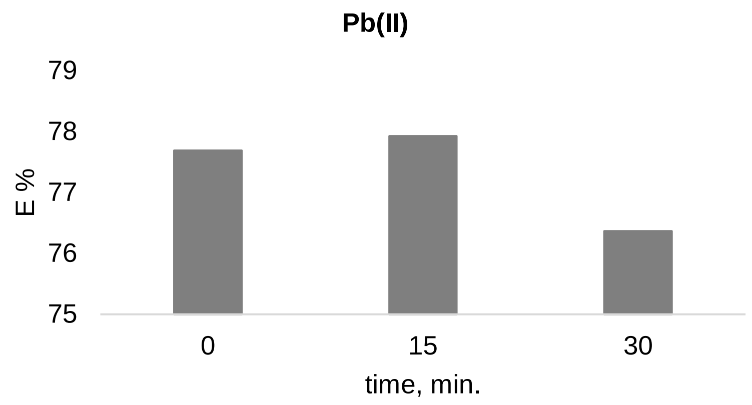

Fig. 9. Influence of time (minutes) on the removal efficiency of Pb(II) ions using sodium carbonate

Conditions: Pb(II) 500 mg/L, pH 8, 300 rpm

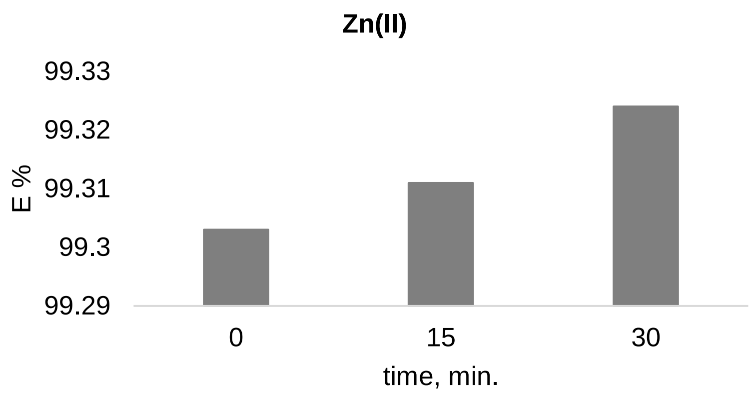

Fig. 10. Influence of time (minutes) on the removal efficiency of Zn(II) ions using sodium carbonate

Conditions: Zn(II) 500 mg/L, pH 8, 300 rpm

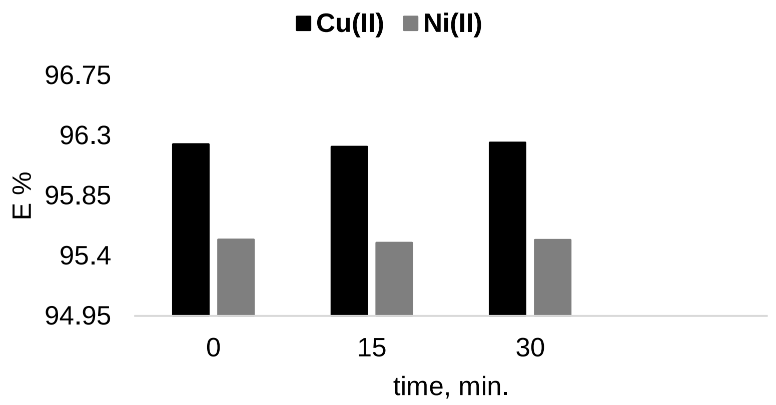

Fig. 11. Influence of time (minutes) on the removal efficiency of Cu(II) and Ni(II) ions in a binary multicomponent system using sodium carbonate

Conditions: Cu(II) and Ni(II) 500 mg/L, pH 8, t = 5 minutes

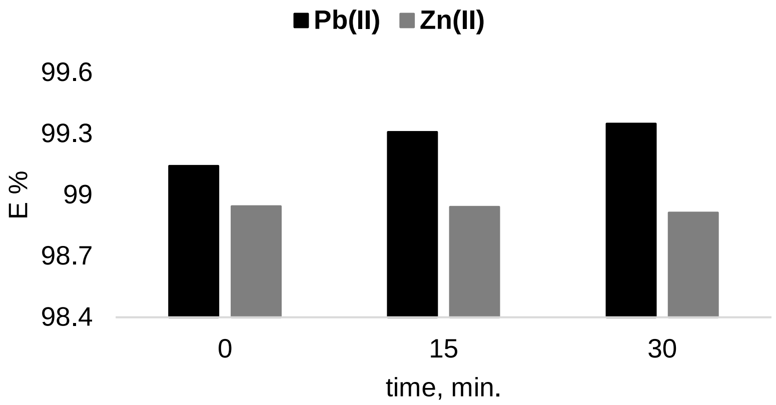

Fig. 12. Influence of time (minutes) on the removal efficiency of Pb(II) and Zn(II) ions ions in a binary multicomponent system using sodium carbonate
Conditions: Pb(II) and Zn(II) 500 mg/L, pH 8, t = 5 minutes

Table 2. ANOVA single factor for monocomponent metal solutions (mixing speed)

Groups	Count	Sum	Average	Variance
rpm	4	1200	300	126666,7
Cu(II)	4	399,797	99,94925	9,58E-06
Ni(II)	4	378,36	94,59	4,53E-05
Pb(II)	4	302,885	75,72125	0,578149
Zn(II)	4	397,222	99,3055	2,97E-05

Table 3. ANOVA single factor for binary multicomponent metal solutions (mixing speed)

Groups	Count	Sum	Average	Variance
rpm	4	1200	300	126666,7
Cu(II)	4	385,066	96,2665	0,007228
Ni(II)	4	382,087	95,52175	2,36E-05
Pb(II)	4	397,464	99,366	0,026266
Zn(II)	4	395,765	98,94125	8,09E-05

Table 4. ANOVA single factor for monocomponent metal solutions (time)

Groups	Count	Sum	Average	Variance
min	3	45	15	225
Cu(II)	3	299,787	99,929	0,000247
Ni(II)	3	283,811	94,60367	5,83E-05
Pb(II)	3	231,984	77,328	0,707539
Zn(II)	3	297,938	99,31267	0,000112

Table 5. ANOVA single factor for binary multicomponent solutions (time)

Groups	Count	Sum	Average	Variance
min	3	45	15	225
Cu(II)	3	288,697	96,23233	0,000261
Ni(II)	3	286,538	95,51267	0,000162
Pb(II)	3	297,8	99,26667	0,012097
Zn(II)	3	296,799	98,933	0,000301

4. CONCLUSION

The selected heavy metal ions precipitated differently in the form of insoluble carbonates both individually and in binary multicomponent mixtures. Thus, Ni(II) and Zn(II) ions in monocomponent solutions had the highest removal efficiency at 0 rpm, while Cu(II) and Pb(II) had it at speeds of 300 rpm and 100 rpm, respectively. In mixtures, Cu(II) and Pb(II) ions again required higher mixing speeds to be better removed from the solution, compared to Zn(II) which again showed that no mixing was required at all to achieve the best removal efficiency. The effect of time on the removal efficiency showed that Cu(II) and Ni(II) ions precipitated very quickly in the form of solid particles (0 min), while the remaining two heavy metal ions required a longer time to achieve a better result. Ni(II) and Zn(II) ions showed better removal efficiency at the shortest time of the precipitation process (0 min), while Cu(II) and Pb(II) ions needed a longer time to achieve better removal efficiency.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc.) and text-to-image generators have been used during the writing or editing of this manuscript.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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