

Comparative Evaluation of Lead Removal from Contaminated Water by Chemical Precipitation Using Different Reagents under Varying pH and Dosage Conditions

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ABSTRACT

Lead (Pb) contamination in water is a major environmental problem and necessitates efficient and economical treatment techniques. This study was designed to investigate the removal of Pb by chemical precipitation at different pH and precipitant dosage using sodium carbonate (Na_2CO_3), sodium sulphate (Na_2SO_4) and sodium hydroxide (NaOH). Experiments were conducted at pH 5, 7, and 9, with dosages of 0.10, 0.25, and 0.50 g/50 mL, using an initial Pb concentration of 53.58 mg L^{-1} . The removal efficiency was found to be quite different for various precipitants and operating conditions. The overall performance of Na_2CO_3 was the best, with a mean of 92.77% removal of lead, followed by Na_2SO_4 with 88.75% and NaOH with 43.55% removal of lead. The best Pb removal was 99.58% (residual concentration of 0.226 mg L^{-1}) for Na_2CO_3 at pH 7 and 0.10 g/50 mL. For Na_2SO_4 , the highest observed removal was 98.97% at pH 9 and 0.25 g/50 mL, while NaOH showed comparatively lower removal under the tested conditions. Chemical precipitation was found to be effective in removing lead from contaminated water, and Na_2CO_3 showed the highest observed removal among the tested conditions.

Keywords: Lead, cost-effective treatment, chemical precipitation, pH and dosage

1. INTRODUCTION

Water pollution caused by rapid industrialisation, urbanisation, and population growth has emerged as one of the most serious environmental problems in the world today. Heavy metals are particularly problematic pollutants because they are non-biodegradable, persistent and can bioaccumulate in aquatic systems and food chains, resulting in significant ecological and human health impacts. Heavy metals cannot be naturally degraded and are present in the environment for long periods of time, so effective treatment must be done before wastewater is discharged or reused [1, 2, 3].

The use of lead (Pb) is widespread in industry, and it is highly toxic, making it one of the most dangerous

heavy metals. It is widely used in the production of batteries and battery recycling, mining and smelting, electroplating, pigment production, cable production and metal finishing industries that generate lead wastewater. Lead is chemically stable and non-biodegradable and can easily be deposited in soils, sediments, and aquatic systems, where it can persist and pose a potential for long-term environmental contamination and human exposure via the food chain and drinking water [2, 3]. Low levels of lead have been linked to neurological damage, decreased mental function in children, kidney damage, heart and blood vessel disease, problems with reproduction, and lowered immunity in adults. Thus, international organisations like the World Health Organization (WHO) have established strict limits for lead in drinking water, which makes the use of effective and efficient treatment technologies important [3, 4].

Various physical, chemical, and biological technologies have been explored for the removal of lead from contaminated water, such as adsorption, membrane filtration, ion exchange, electrochemical treatment, coagulation–flocculation, phytoremediation, and chemical precipitation [1, 5, 6, 7]. Activated carbon, biochar, agricultural residues, nanomaterials, and metal oxides have been shown to have very high adsorption efficiencies but are often not used on a large scale due to the high cost of the materials, regeneration requirements, and the disposal of spent materials [8, 9, 10]. Similarly, membrane processes like reverse osmosis and nanofiltration have high separation efficiencies but demand significant capital investment, high energy consumption and frequent membrane maintenance; ion exchange and electrochemical methods are often limited by the high cost of resins and operating costs [6, 7]. This has kept the interest alive in simple, economical and scalable treatment methods applicable to industrial wastewater.

Chemical precipitation is one of the most widely used conventional technologies, as it is easy to operate, has fast reaction rates, is inexpensive in terms of chemicals used, and is compatible with existing wastewater treatment plants [6, 7, 3]. It involves the precipitation of dissolved metal ions to form sparingly soluble compounds which can be settled and filtered out. Precipitation is still one of the most promising methods for treating heavy-metal-contaminated effluents due to its robustness and capacity to effectively treat relatively high concentrations of contaminants [10, 11, 3].

Chemical precipitation is highly dependent on the precipitating agent used. The most industrial method is hydroxide precipitation, which is the formation of insoluble metal hydroxide by the addition of alkaline reagents like sodium hydroxide or lime. But its performance can be limited due to amphoteric metal behaviour, incomplete precipitation and significant sludge formation [7, 10]. Other reagents like carbonate and sulphate salts yield highly insoluble lead compounds like lead carbonate (PbCO_3) and lead sulphate (PbSO_4) with different precipitation mechanisms and solubilities. Comparative investigations have shown that carbonate precipitation can result in lower residual lead levels than conventional hydroxide precipitation when conditions are optimised, and sulphate precipitation can yield better selectivity, depending on the wastewater chemistry [11, 12, 13, 14]. Moreover, it has been recently reported that optimising precipitation chemistry can lead to a significant increase in removal efficiency with a decrease in the amount of chemicals used and sludge produced [10, 3].

The efficiency of precipitation is strongly dependent on operational parameters, such as the pH of the solution and the amount of precipitant added. The speciation of lead, the stability of precipitates and the solubility of the compounds are all controlled by solution pH, and the dosage of chemicals affects the completeness of precipitation and the amount of sludge produced [10, 12, 14]. While many studies have examined these parameters individually, few have systematically examined the effect of hydroxide-, carbonate-, and sulfate-based precipitation under the same experimental conditions and optimized both pH and dosage. This makes it difficult to compare precipitation strategies directly and to determine which is the best treatment method for lead-contaminated wastewater [12, 13, 14], [10], [11].

The objective of the present study is to systematically compare the effectiveness of hydroxide-, carbonate-, and sulphate-based chemical precipitation for removing lead from contaminated water under different pH and dosages to optimise the removal process. This study aims at optimising the parameters of precipitating agent, solution pH and chemical dose for the removal of lead from the solution to maximise the removal efficiency with minimum chemical consumption and sludge formation. The results will help to develop a simple, low-cost and scalable solution to treat industrial wastewater and manage water resources sustainably.

2. MATERIALS AND METHODS

2.1 Chemicals and Reagents

Analytical-grade lead nitrate ($\text{Pb}(\text{NO}_3)_2$) was used for preparing synthetic lead-contaminated water. Chemical precipitating agents were sodium carbonate (Na_2CO_3), sodium sulphate (Na_2SO_4) and sodium hydroxide (NaOH). All solutions were prepared with deionised water in the course of the study. To make the solutions safe for experimentation at home, the pH was adjusted with food-grade vinegar to make the solution acidic and with freshly prepared 5% (w/v) sodium hydroxide solution to make the solution alkaline.

2.2 Preparation of Lead Stock Solution

A working lead solution was prepared by dissolving 0.12 g of lead nitrate in about 500 mL of deionised water, giving a final concentration of 50 mg L^{-1} . The solution was transferred to a volumetric flask and diluted to 1.5 L with deionised water to make a homogeneous working solution. In all precipitation experiments the prepared solution was well mixed before use to obtain a constant initial concentration of lead.

2.3 Experimental Design

A full-factorial experimental design was used to evaluate the removal of lead with precipitating agent, solution pH and chemical dosage. Three precipitants (Na_2CO_3 , Na_2SO_4 and NaOH) were used at pH 5, 7 and 9. There were 27 experimental treatments, each precipitating agent being tested at three doses, 0.10, 0.25 and 0.50 g/50 mL contaminated water. Additionally, control experiments were conducted without the use of chemical precipitants to determine the removal of lead in the absence of precipitants at each pH level.

2.4 Chemical Precipitation Procedure

The prepared lead solution was pipetted into a clean reaction vessel for each treatment (50 mL). Universal pH strips were used to monitor the pH of the solution as vinegar or 5% sodium hydroxide solution was added dropwise to give the desired pH (5, 7 or 9). When the desired pH was reached, the required precipitating agent (0.10, 0.25 or 0.50 g) was added to the solution. The suspension was stirred for about 30 min to ensure complete dissolution and to promote precipitation reactions, and it was allowed to settle for 20 min to allow the formation and sedimentation of the insoluble lead precipitates. The treated water was then filtered using laboratory-grade filter paper to remove the solids that precipitated out of the treated water. The reaction time and the amount of solution were the same in all experiments, and the mixing conditions were the same throughout the study, and all experiments were carried out at ambient temperature in the laboratory (approx. $25 \pm 2^\circ\text{C}$).

2.5 Determination of Lead Removal

The filtrates obtained after chemical precipitation were analysed for residual lead concentration using Atomic Absorption Spectroscopy (AAS) at a certified analytical laboratory. Standards were prepared in the laboratory before analysis, following the laboratory protocol. The amount of residual lead in each experimental sample was measured using only one AAS measurement. The concentration obtained was used in calculating the removal efficiency of the lead (C_f). The efficiency of lead removal was determined by comparing the initial and final concentration of lead as shown in Equation (1):

$$\text{Removal Efficiency (\%)} = \{(C_i - C_f)/C_i\} \times 100$$

where C_i represents the initial lead concentration (mg L^{-1}) and C_f represents the residual lead concentration after treatment (mg L^{-1}).

2.6 Experimental Controls

Three control samples were prepared with the lead solution at pH 5, 7 and 9 without adding any precipitating agent to the solution to account for any change in lead concentration due to pH adjustment alone. Blank samples with deionised water containing no lead were also made to check for background contamination during the experimental procedure.

2.7 Data Analysis

The percentage removal of lead was determined for each treatment, and the effect of precipitating agent, solution pH and chemical dosage was compared to find the optimum precipitation conditions. The effectiveness of the treatment was determined by comparing the amount of lead remaining in the water and the percentage of lead removed from all 27 experimental treatments. The ease of precipitation and ease of filtration were also noted, and observations were made on the formation and settling characteristics of precipitates, which were also recorded to assess the practical applicability of each precipitation method.

3. RESULTS

The nominal concentration of the Pb stock solution was 50 mg L⁻¹, but analytical measurement gave an initial concentration of 53.58 mg L⁻¹. This measured concentration was taken as the initial concentration for all subsequent calculations used for the removal of lead and is reported in the results.

3.1. Comparative performance of the precipitating agents

Lead removal varied among the three precipitants tested (Table 1). In all the nine conditions tested, the maximum mean removal of Pb was found to be by Na₂CO₃ (92.77%), followed by Na₂SO₄ (88.75%) and NaOH (43.55%) (Table 2). Therefore, the removal of Pb by carbonate- and sulphate-based precipitation was higher than the removal of Pb by hydroxide precipitation under the experimental conditions studied.

Precipitant	pH	Dosage (g/50 mL)	Initial Pb (mg L ⁻¹)	Residual Pb (mg L ⁻¹)	Removal (%)
Na ₂ CO ₃	5	0.10	53.58	10.371	80.64
Na ₂ CO ₃	5	0.25	53.58	8.309	84.49
Na ₂ CO ₃	5	0.50	53.58	4.678	91.27
Na ₂ CO ₃	7	0.10	53.58	0.226	99.58
Na ₂ CO ₃	7	0.25	53.58	1.719	96.79
Na ₂ CO ₃	7	0.50	53.58	3.421	93.62
Na ₂ CO ₃	9	0.10	53.58	0.517	99.04
Na ₂ CO ₃	9	0.25	53.58	1.205	97.75
Na ₂ CO ₃	9	0.50	53.58	4.404	91.78
Na ₂ SO ₄	5	0.10	53.58	23.953	55.29
Na ₂ SO ₄	5	0.25	53.58	12.305	77.03
Na ₂ SO ₄	5	0.50	53.58	12.646	76.40
Na ₂ SO ₄	7	0.10	53.58	0.785	98.53
Na ₂ SO ₄	7	0.25	53.58	1.071	98.00

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Na ₂ SO ₄	7	0.50	53.58	1.564	97.08
Na ₂ SO ₄	9	0.10	53.58	0.569	98.94
Na ₂ SO ₄	9	0.25	53.58	0.553	98.97
Na ₂ SO ₄	9	0.50	53.58	0.818	98.47
NaOH	5	0.10	53.58	21.520	59.84
NaOH	5	0.25	53.58	31.690	40.85
NaOH	5	0.50	53.58	51.109	4.61
NaOH	7	0.10	53.58	29.831	44.32
NaOH	7	0.25	53.58	28.980	45.91
NaOH	7	0.50	53.58	32.084	40.12
NaOH	9	0.10	53.58	26.813	49.96
NaOH	9	0.25	53.58	23.569	56.01
NaOH	9	0.50	53.58	26.638	50.28
Control 1	5	-	53.58	53.58	0.00
Control 2	7	-	53.58	53.58	0.00
Control 3	9	-	53.58	53.58	0.00

Table 1. Experimental conditions and Pb-removal performance.

Precipitant	Mean removal (%)
Na ₂ CO ₃	92.77
Na ₂ SO ₄	88.75
NaOH	43.55

Table 2. Overall Pb-removal performance of the tested precipitants.

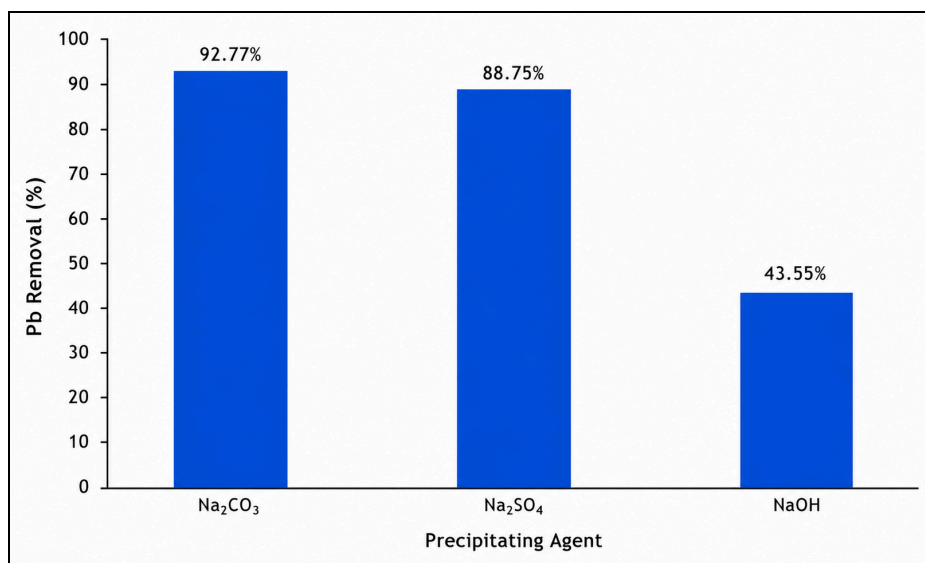


Figure 1. Mean Pb removal efficiency of the tested precipitating agents across the experimental conditions.

3.2. Effect of pH on Pb removal

The effect of pH differed among the precipitants (Table 3; Figure 2). For Na₂CO₃, mean Pb removal increased from 85.47% at pH 5 to 96.67% at pH 7, followed by a slight decrease to 96.19% at pH 9. Na₂SO₄ showed a progressive increase in removal from 69.58% at pH 5 to 97.87% at pH 7 and 98.79% at pH 9. NaOH exhibited comparatively low removal, ranging from 35.10% at pH 5 to 43.45% at pH 7 and 52.08% at pH 9.

Overall, the highest mean removal for Na₂CO₃ was at pH 7, while Na₂SO₄ and NaOH had the highest mean removal at pH 9.

Parameter	Level	Na ₂ CO ₃ (%)	Na ₂ SO ₄ (%)	NaOH (%)
pH	5	85.47	69.58	35.10
	7	96.67	97.87	43.45
	9	96.19	98.79	52.08
Dosage (g/50 mL)	0.10	93.09	84.26	51.37
	0.25	93.01	91.33	47.59

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Parameter	Level	Na ₂ CO ₃ (%)	Na ₂ SO ₄ (%)	NaOH (%)
pH	5	85.47	69.58	35.10
	7	96.67	97.87	43.45
	0.50	92.22	90.65	31.67

Table 3. Mean Pb removal according to pH and precipitant dosage.

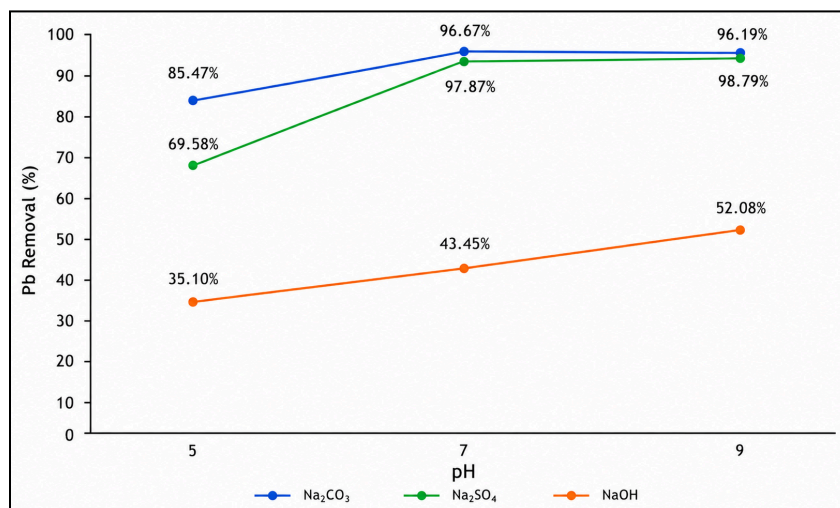


Figure 2. Effect of pH on Pb removal efficiency for the tested precipitating agents.

3.3. Effect of precipitant dosage

The dosage effect varied with the precipitant and was not always linear with the amount of precipitant added (Table 3; Figure 3). In the case of Na₂CO₃, the mean Pb removal showed little variation with increasing dosage, ranging from 93.09% at 0.10 g/50 mL to 92.22% at 0.50 g/50 mL. Na₂SO₄ showed an increase from 84.26% at 0.10 g/50 mL to 91.33% at 0.25 g/50 mL, followed by a slight decrease to 90.65% at 0.50 g/50 mL. In contrast, NaOH removal decreased from 51.37% at 0.10 g/50 mL to 31.67% at 0.50 g/50 mL.

These findings suggest that there was not a consistent increase in the removal of Pb with an increase in precipitant dosage within the range of dosages tested.

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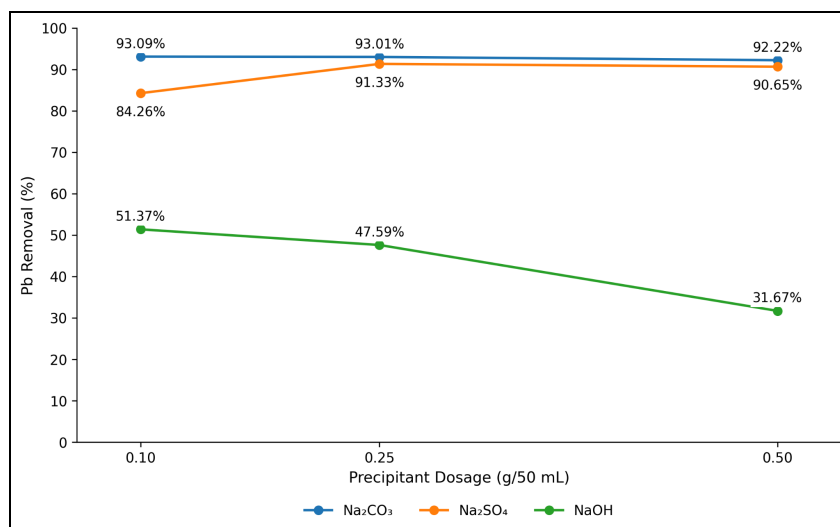


Figure 3. Effect of precipitant dosage on Pb removal efficiency for the tested precipitating agents.

3.4. Identification of the best-performing treatment condition

The maximum removal of Pb was 99.58% with Na₂CO₃ at pH 7 and 0.10 g/50 mL, which decreased the Pb concentration from 53.58 to 0.226 mg L⁻¹ (Table 4). Na₂CO₃ at pH 9 and 0.10 g/50 mL produced a comparable removal of 99.04%. For Na₂SO₄, the highest observed removal was 98.97% at pH 9 and 0.25 g/50 mL, while NaOH showed an observed removal of 59.84% at pH 5 and 0.10 g/50 mL.

Precipitant	Best tested pH	Best tested dosage (g/50 mL)	Removal (%)
Na ₂ CO ₃	7	0.10	99.58
Na ₂ SO ₄	9	0.25	98.97
NaOH	5	0.10	59.84

Table 4. Observed Pb removal under the tested pH and precipitant dosage conditions.

Based on the results, the best-performing tested condition was found to be the pH 7 of Na₂CO₃ and 0.10 g/50 mL. This is the the best-performing tested condition was observed and should not be considered as a statistically verified optimum.

4. DISCUSSION

The results show that the type of precipitant, pH and dosage of precipitant affected the removal of Pb. Na_2CO_3 exhibited the highest overall mean removal (92.77%), while Na_2SO_4 exhibited a mean removal of 88.75% and NaOH exhibited a mean removal of 43.55% under the conditions investigated. This suggests that the chemical nature of the precipitating reagent had an effect on precipitation performance. Heavy-metal precipitation systems also have been found to be sensitive to precipitant chemistry and operating conditions [3], [14], [15].

Na_2CO_3 and Na_2SO_4 showed a pronounced response to changes in pH. The mean removal of Na_2CO_3 was the highest at pH 7, and that of Na_2SO_4 was the highest at pH 9. These differences are in line with the solution chemistry dependency of metal precipitation, such as solubility and precipitation equilibria [3, 14]. Importantly, the optimum pH range for the removal of Pb was not consistent for all precipitants used, suggesting that the optimum pH range is dependent on the precipitant used.

Dosage was also reagent dependent. The mean removal did not increase with increasing Na_2CO_3 dosage (0.10 to 0.50 g/50 mL), but it did peak at 0.25 g/50 mL Na_2SO_4 . The removal of NaOH decreased with increasing dosage. The mean removal did not increase consistently with increasing Na_2CO_3 dosage (0.10 to 0.50 g/50 mL), while Na_2SO_4 removal increased from 98.53% at 0.10 g/50 mL to 98.97% at 0.25 g/50 mL before decreasing slightly to 98.47% at 0.50 g/50 mL. The slight decrease may suggest an intermediate dose, at which point adding more Na_2SO_4 did not yield more precipitation benefit, and could also mean that the approach was towards a concentration of Na_2SO_4 that is beyond optimal for further gains in Pb removal. The removal of NaOH , on the other hand, demonstrated that it decreases significantly with the increase of its dosage. At pH 5, removal decreased from 59.84% at 0.10 g/50 mL to 40.85% and 4.61% at 0.25 and 0.50 g/50 mL, respectively. The amphoteric nature of the hydroxide precipitate, which is formed during the hydroxide precipitation process, may be the reason behind this behavior. An increase in the concentration of OH^- can encourage the precipitation of $\text{Pb}(\text{OH})_2$ but too much OH^- can encourage some redissolution to form soluble hydroxo complexes like $[\text{Pb}(\text{OH})_4]^{2-}$. Therefore, the amount of excess NaOH might have caused the net removal of Pb to be less under certain conditions. This is shown here as a mechanism but not as confirmed pathway due to the fact that dissolved form of Pb was not directly characterized. From these observations, it is concluded that the quantity of reagent is not necessarily the best way to improve the precipitation of the lead and that reagent-specific conditions should be taken into account when deciding on an operating dose.

The maximum percentage removal of Pb was achieved in the single experimental condition (99.58%) at the pH of 7 and dosage of 0.10 g/50 mL of Na_2CO_3 . Also, the study employed synthetic water with a high concentration of Pb and did not consider the effects of competing ions, precipitate composition, sludge generation, or precipitate stability. These factors can affect the performance of treatment in a real wastewater system.

Some uncertainty in maintaining the target pH values may also have been caused by using universal pH

strips for pH adjustment. The present results should be confirmed in future studies with independent replicates and calibrated pH measurements as well as with other experimental conditions and statistical analysis. Additionally, food grade vinegar and homemade NaOH were used for pH adjustment which could also have introduced variance in acetate and hydroxide concentrations and, potentially, have affected the observed removal of Pb.

Chemical precipitation removes Pb from the aqueous phase to a solid precipitate but does not remove the contaminant. Therefore, to reduce the potential for secondary contamination, it is essential to collect, stabilise, recover or dispose of solids containing lead in an appropriate manner [14]. In order to further understand the practical application of the process, it is necessary to investigate the characteristics of the precipitates, sludge generation, stability and sludge treatment performance of actual industrial wastewater.

5. CONCLUSION

The efficiency of Na_2CO_3 , Na_2SO_4 and NaOH for removal of Pb from contaminated water was comparatively evaluated under different pH and precipitant dosages. The overall mean Pb removal (92.77%) was obtained with Na_2CO_3 among the tested reagents. The maximum removal efficiency of 99.58% was obtained with Na_2CO_3 at pH 7 and a dose of 0.10 g/50 mL, which reduced the concentration of Pb from 53.58 to 0.226 mg L^{-1} . The results also showed that the amount of precipitant was not always in a direct correlation with the removal of Pb, which indicates that the operating conditions are reagent specific.

The results show that the precipitation of lead under the experimental conditions is possible by carbonate-based precipitation. These are all single-run pilot experiments, and are therefore not statistically validated optimum performance values. Further research with replicated experiments, careful control of pH, actual wastewater matrices, characterisation of precipitates, and evaluation of sludge containing Pb should be performed to determine the repeatability and feasibility of the treatment method.

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