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Enhanced Heavy Metal Removal from Wastewater Produced by Chemical Analysis Laboratory Using Calcium Oxide Precipitation: pH Improvement and Characterization of Precipitated Phases

Intissar Loughlaimi[1]*, Zineelabidine Bakher[1], Abdeljalil Zouhri[1]

[1]HASSAN 1st University, Faculty of Sciences and Techniques, Department of Applied Chemistry and Environment, Settat, Casablanca road, km 3.5, P.C 539, Morocco.

Abstract: This article presents research results on the precipitation of heavy metals: Aluminum (Al), arsenic (As), cadmium (Cd), zinc (Zn), iron (Fe), chromium (Cr), copper (Cu), nickel (Ni), vanadium (V), and molybdenum (Mo) from wastewater generated in mining chemical analysis laboratory. Calcium oxide was used as the precipitating agent. The efficiency of heavy metal removal was achieved by increasing the dosage of precipitating reagent (8-28 g/L). Efficiencies greater than 90% are achieved. The efficiency of chemical precipitation depends on the pH of the process. Over a wide pH range from 6-11, the removal efficiency of zinc, iron, cadmium, and arsenic were approximately 99.9%. The optimum pH range for the removal of most elements was found to be between 8 and 11, where the removal efficiency of heavy metal ions reached up to 99%. Furthermore, X-ray diffraction results indicated that the metals in the wastewater precipitated in various forms as mentioned in Table 7, and not just as hydroxides, due to the presence of different ions in the solution.

Keywords: Precipitation, Heavy metals, Wastewater, Calcium oxide, Chemical analysis laboratory.

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***Corresponding author's E-mail:** i.loughlaimi@uhp.ac.ma

1. INTRODUCTION

A reliable supply of high-quality water is essential for human well-being, social and economic progress, and the sustainability of the global ecosystem (1). As water resources become scarcer, the need to provide water to various parts of the world, including Morocco, is a pressing issue and may become even more of a concern in the future (2).

Due to the fundamental importance of water for the sustenance of all living beings, the phenomenon of rapid urbanization and development has resulted in a substantial increase in the demand for this vital resource. Moreover, water plays a crucial role in a diverse range of residential and industrial processes, including but not limited to petroleum refineries, agricultural practices, medicinal and pharmaceutical procedures. These activities introduce many toxic pollutants and waste substances into water (1). These contaminants fall into three main groups: organic, inorganic, and biological particles. Industrial and urban activities that produce toxic heavy metals such as cadmium, copper, zinc, nickel, arsenic, chromium, and mercury are classified as hazardous and non-biodegradable (3-4). In this study we focus on inorganic groups containing heavy metal ions. Additionally, heavy metals are a common health issue. As they are teratogenic, carcinogenic and causes detrimental health problems (5). Even trace levels of heavy metals contamination can lead to bioaccumulation through the food chain. High levels of heavy metals are produced in the body when a person drinks water or eats food contaminated with heavy metals. For example, lead affects central nervous system disorders by altering the characteristics of early human physiological symptoms such as allergies, renal disorders, hepatic disorders, infertility, dermatitis, abdominal pain, gingivitis, migraines, and insomnia (6-7).

A number of special processes have been developed to remove metals from wastewater (8). The various treatment techniques include: chemical precipitation (9-10), coagulation/flocculation (11), adsorption (12), ion exchange (13), solvent extraction (14), electrochemical operation (15), biological operations (16-17), ultrafiltration (18) and membrane processes (19). Each technique has its own related advantages and drawbacks.

Among the various methods available, one viable option is to employ simple precipitation of metals in the form of insoluble hydroxides or carbonates. Chemical precipitation, extensively utilized in industrial applications, is particularly favored due to its inherent process control simplicity (3) and lower concentrations of all metals. The success of this method depends on the solubility of the precipitated metal compounds. If a metal has the ability to form an insoluble compound, it is possible to remove that compound through a clarification and filtration process. Although the number of precipitation methods available is limited, hydroxide and sulfide are predominantly used, with hydroxide precipitation being the most commonly used. The process involves a simple step: raising the pH of the effluent by adding lime (CaO) as a precipitant. This converts heavy metals to their corresponding hydroxides and immobilizes them (1).

Metal precipitation is the process of converting metal ions dissolved in water into solid metal compounds. This precipitation is influenced by two important factors: Concentration of metals and pH of water. Heavy metals in wastewater are typically present in relatively low concentrations ranging from 1 to 100 mg/L. These concentrations are considered diluted. Also, the pH of water containing these heavy metals is usually neutral or slightly acidic (pH<7.0). When metals are introduced into the treatment procedure, they exist in a dissolved state within the water, maintaining stability and being unable to solidify. The objective of employing hydroxide precipitation for metals' treatment is to manipulate the water's pH in such a way that the metals can undergo a transformation, forming precipitates that are insoluble. The concentration of hydroxide ions in the water is directly proportional to the pH level, therefore, a higher pH signifies elevated hydroxide concentrations. However, when lime is added to water containing dissolved metals, the metals react with hydroxide ions to form metal hydroxide solids. Once the metals have precipitated and formed solids, they can be easily removed, and the water, now depleted of metals, can be drained or reused (20).

In this article, calcium oxide was used to precipitate heavy metals present in wastewater: aluminum (Al), arsenic (As), cadmium (Cd), zinc (Zn), iron (Fe), chromium (Cr), copper (Cu), nickel (Ni), vanadium (V) and molybdenum (Mo). The objective of this study was to attend the maximum removal efficiency of all heavy metal ions from solution. In addition, the agglomeration of the precipitates during the treatment was investigated and characterized by inductively coupled plasma ICP to identify the different phases of precipitate.

2. EXPERIMENTAL STUDIES

2.1. Materials and Reagents

The wastewater of mining chemical analysis laboratory, which contains heavy metals, was used to be treated with chemical precipitation. The precipitating agent used in this treatment is calcium oxide (CaO) with different masses (8 g/L up to 28 g/L).

Table 1: Descriptive of products utilized.

Product	Descriptive	Provider
CaO	Purity: 99.9%	Sigma Aldrich[a]
Distilled water	Conductivity: 21.4 µS/cm pH: 7.610	

[a]: Analyzed by XRD

2.2. Measuring Instrumentations and Equipment

In the context of wastewater treatment, determining the optimal dosage of a precipitating agent can be a challenging task that necessitates the use of various analytical techniques. In this study, the GFL 3040 jar test from Gesells chaft Fuer Labortec Overhead Shakers was utilized to assess the effectiveness of different doses of a precipitating agent in treating wastewater.

To evaluate the changes in the pH of the solution during the treatment process, a Mettler Toledo pH meter was employed. This tool helped in understanding the impact of the dosage on the pH levels, as the addition of a precipitator can alter the pH of the solution.

The concentration of metal ions in the wastewater was analyzed using Inductively coupled plasma (ICP), a technique that uses plasma to ionize samples for analysis. This method provides highly accurate and precise measurements of trace elements, such as metals, in complex samples like wastewater.

In addition to Inductively coupled plasma ICP analysis, X-ray diffraction (XRD) examination was utilized to identify the important chemical compounds present in the sludge samples. This technique utilizes the diffraction pattern of X-ray to identify the crystal structure and composition of the sample, providing valuable information on the chemical compounds present in the wastewater. X-ray diffraction analysis was performed on all solids to characterize the solid phases present in various saturated solutions. This analysis was performed on the BRUKER D2 phase shifter instrument with a LYNXEYE detector using a Cu-Ka wavelength of 1.541874 Å. Each stable solid was analyzed over a temperature range of 10 to 80 °C for 20 minutes.

In order to study the effects of precipitant dosages and initial pH on the removal of heavy metals, a series of polyethylene graduated flasks (500 mL) containing wastewater solution were prepared. Different masses of calcium oxide (CaO) (8 to 28 g/L) were added into each flask at a temperature of 24 °C. The flasks were then subjected to stirring at a speed of 200 rpm for 15 minutes to allow for decantation, after which the mixture was filtered through a 0.45 µm membrane to obtain two phases; liquid and solid.

Subsequently, the final pH of the liquid was measured, and the remaining metal in the liquid phase was measured using ICP. In addition, the solid phase was analyzed using XRD. Through this process, a comprehensive understanding of the impact of precipitant dosages and initial pH on heavy metal removal can be gained (see Figure 1).

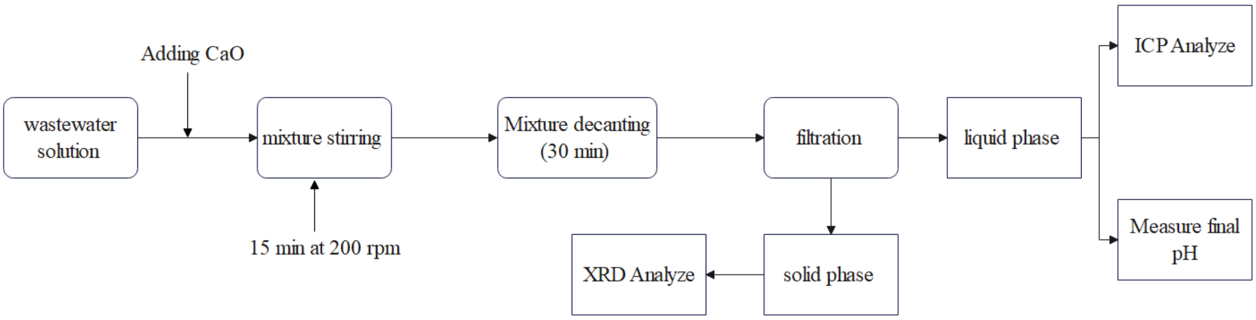

Figure 1: Representative schema of the process.

To calculate metal removal efficiency, we used the following equation:

$$R(\%) = \frac{C_i - C_f}{C_i} \times 100$$

R: Metal removal efficiency
C_i: Initial concentration of the metal
C_f: Final concentration of the metal

Table 2: Characterization of wastewater used in this study.

Parameters	Unites	Values for wastewater
pH	-	0.313
Conductivity	mS/cm	164.8
Al	%	0.992
Cu	ppm	50.59
Cd	ppm	23.76
Cr	ppm	229.8
As	ppm	10.96
Fe	%	0.484
Mo	ppm	8.666
Ni	ppm	50.96
V	ppm	203.2
Zn	ppm	426.4

Table 3: Operating conditions for ICP.

ICP	
Rf power (w)	1300.0
Plasma Ar flow (L/min)	12.0
Auxiliary Ar flow (L/min)	0.2
Nebuliser Ar flow (L/min)	0.7
Delay time (s)	40.0
Measurement mode:	
Axial	All
Radial	Iron, Aluminum, CaO

3. RESULTS AND DISCUSSION

The pH is considered an important operational factor that governs the interactions of a surface-active substance and metal ions in flotation processes because the pH affects the stability and precipitation behavior of heavy-metal ions in the solution.

Tables 4 and 5 present the outcomes of using various dosages (8, 12, 16, 20, 24, and 28 g/L) of calcium oxide to assess the most effective dosage for wastewater purification. The parameters analyzed include the final pH, residual metal concentration, and metal removal efficiency. Increased amounts of calcium oxide ranging from 8 to 28 g/L were examined in order to determine the optimal dosage for achieving effective wastewater purification.

As shown in Tables 3 and 4, the removal efficiency of zinc, iron, cadmium, and arsenic was approximately 100% over a wide pH range from 6-11, and for a dose lower than 12 g/L of lime. These results are similar to those stated in the references (15-21).

Each metal precipitates over a narrow pH range, above which the metal redissolves, as is the case for molybdenum. For pH 9.394 a concentration of 6.049 can achieved. For aluminum Al the concentration decreased to 0.023% at a pH of 6.93 which is close to the reported value obtained by Wei X Viadero R Buzby K (21), and for chromium the removal was 100% at a pH between 6.93 and 9.394. These results are in line with Dr C, Ramakrishnaiah P (22). It can also be seen that the removal of copper Co is 94.57% for pH= 6.93. The remaining for nickel Ni and vanadium V decreased from 50.96 ppm and 203.2 ppm to 8.647 ppm (pH= 8.614) and 2.979 ppm (pH= 11.345) respectively.

Table 4: Remaining concentrations of metals after treatment by chemical precipitation.

Dose of lime (g/L)		0.000	8.000	12.00	16.00	20.00	24.00	28.00
Final pH		0.637	6.930	7.974	8.614	9.394	11.34	11.75
Remaining concentration	Al (%)	0.992	0.023	0.024	0.024	0.023	0.021	0.023
	As	10.96	0.084	0.000	0.000	0.000	0.000	0.000
	Cd	23.767	0.316	0.000	0.000	0.011	0.000	0.000
	Cr	229.8	0.000	0.000	2.387	0.000	7.284	4.516
	Cu	50.59	2.749	4.269	6.426	8.562	4.877	3.251
	Fe	0.484	0.000	0.000	0.000	0.000	0.000	0.000
	Mo	8.666	29.85	22.32	28.01	6.049	9.041	172.3
	Ni	50.96	67.96	24.2	8.647	9.400	10.70	11.32
	V	203.2	63.20	59.45	14.4	5.745	2.979	6.148
	Zn	426.4	0.000	0.000	0.000	0.000	0.000	0.000

Table 5: Removal efficiency of metals.

Element	**Dose of lime**	**Final pH**	**Removal (%)**
Al	8.000	6.930	97.68
	12.00	7.974	97.58
	16.00	8.614	97.58
	20.00	9.394	97.68
	24.00	11.34	97.88
	28.00	11.75	97.58
As	8.000	6.930	99.23
	12.00	7.974	100.0
	16.00	8.614	100.0
	20.00	9.394	100.0
	24.00	11.34	100.0
	28.00	11.75	100.0
Cd	8.000	6.93	98.67
	12.00	7.974	100.0
	16.00	8.614	100.0
	20.00	9.394	99.95
	24.00	11.34	100.0
	28.00	11.75	100.0
Cu	8.000	6.930	94.57
	12.00	7.974	91.56
	16.00	8.614	87.30
	20.00	9.394	83.07
	24.00	11.34	90.30
	28.00	11.75	93.57
Fe	8.000	6.930	100.0
	12.00	7.974	100.0
	16.00	8.614	100.0
	20.00	9.394	100.0
	24.00	11.34	100.0
	28.00	11.75	100.0
Mo	8.000	6.930	NC
	12.00	7.974	NC
	16.00	8.614	NC
	20.00	9.394	30.20
	24.00	11.34	NC
	28.00	11.75	NC
Ni	8.000	6.930	NC
	12.00	7.974	52.51
	16.00	8.614	83.03
	20.00	9.394	81.55
	24.00	11.34	79.00
	28.00	11.75	77.79
V	8.000	6.930	68.90
	12.00	7.974	70.74
	16.00	8.614	92.91
	20.00	9.394	97.17
	24.00	11.34	98.53
	28.00	11.75	96.97
Zn	8.000	6.930	100.0
	12.00	7.974	100.0
	16.00	8.614	100.0
	20.00	9.394	100.0
	24.00	11.34	100.0
	28.00	11.75	100.0

(NC: Not calculated)

3.1. Effect of pH

As shown in Figures 2 and 3 the effect of pH value on the removal of heavy metals, when the pH is increased from 2 to 11, the remaining concentration of metal ions decreases and the removal efficiency increases. The pH of the solution is an important factor that has an effect on precipitation. When the pH is low, the hydroxide metals will not form due to the large amount of H^+, which decreases the capacity of precipitation. As the pH increases, the $[OH^-]$ concentration increases to form an insoluble ion, which reduces the remaining concentration of heavy metals in solution.

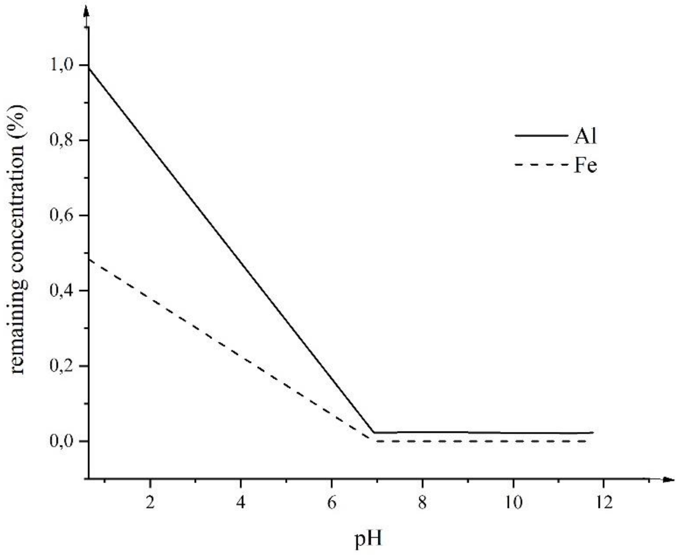

Figure 2: Effect of pH on removal of Al and Fe.

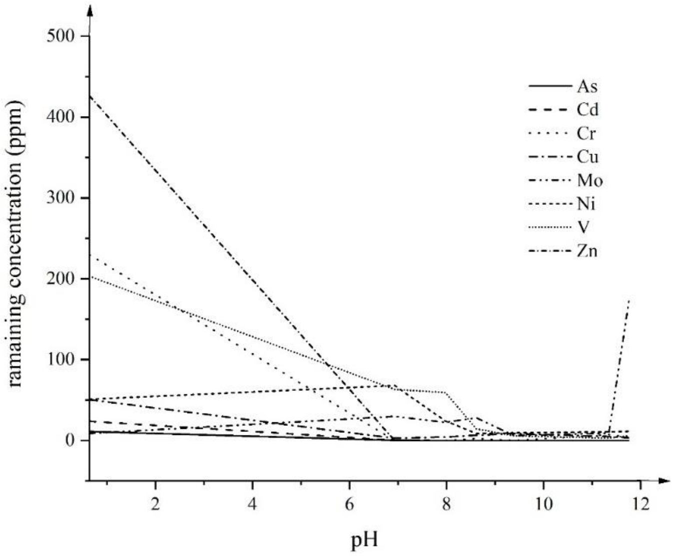

Figure 3: Effect of pH on removal of other metals.

3.2. Effect of Lime Dosage on pH

As shown in Figure 4, the pH value of solution can be increased by adding more calcium oxide to it. This increase in pH value results from the release of hydroxide ions (OH^-) in solution, which combines with the metal ions present in the solution to form insoluble hydroxides. The formation of these insoluble hydroxides precipitates metal ions, making them easier to remove from the solution.

It is important to note that the addition of CaO to the solution must be carefully controlled to avoid excessively increasing the pH value. An excessively high pH value can lead to the formation of unwanted precipitates, making it more difficult to remove the targeted metal ions from the solution.

In conclusion, the addition of CaO to the solution can increase the pH value, which is an important factor in the chemical precipitation method for removing heavy metal ions.

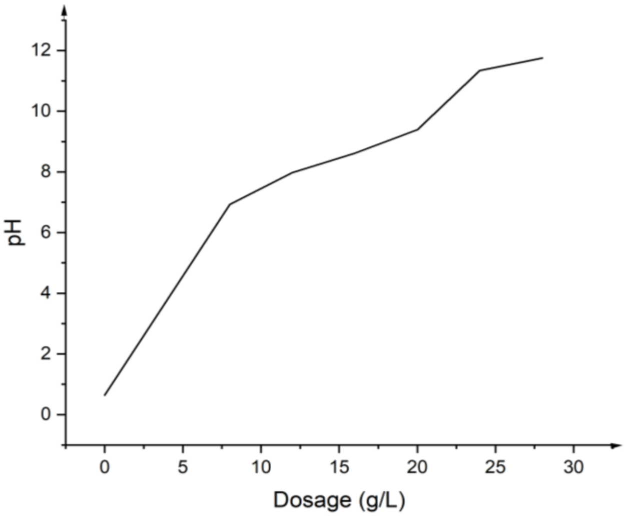

Figure 4: Effect of dosage calcium oxide on pH solution.

3.3. Process Description

Precipitation, as a chemical unit process, is used to remove unwanted soluble metal ions and specific anions from water or wastewater by converting them into insoluble forms. It is a widely used processing technology to remove heavy metals, phosphorus, and hardness. This processing method involves altering the ion balance to form precipitates that are insoluble and can be easily separated. The chemical precipitation process is typically followed by a solid's removal step, which may include coagulation and filtration to remove the formed precipitates. In certain cases, a chemical reduction step may precede the precipitation process to modify the properties of the metal ions, enabling their precipitation (23).

The main chemical reactions during the precipitation process and the conversion of the compound in the precipitate are as follows (24):

- The process begins with the hydrolysis of lime, followed by its partial dissolution, resulting in a pH increment due to the formation of hydroxyl ions (reactions 1 and 2).
- Then, the hydroxyl ions follow a series of interactions with divalent and trivalent cations present (reactions 3 and 4).
- If any free sulfate exists, may involve simultaneous precipitation of gypsum (reaction 5), as shown in Table 6.

Table 6: Reactions of process.

Number of reaction	Reaction	Free Energy	Reference
1	$Ca0_{(s)} + H_2O \rightarrow Ca(OH)_{2\,(s)}$	$\Delta G°_{25°C} = -13.815\,Kcal$	-
2	$Ca(OH)_{2(s)} \rightarrow Ca^{2+}_{(aq)} + 2\,OH^{-}_{(aq)}$	$\Delta G° = -6.438\,Kcal$	(24)
3	$Me^{2+}_{(aq)} + 2OH^{-}_{(aq)} \rightarrow Me(OH)_{2(s)}$		
4	$Me^{3+}_{(aq)} + 3OH^{-}_{(aq)} \rightarrow Me(OH)_{3(s)}$		
5	$Ca^{2+}_{(aq)} + SO^{\acute{}2-}_{4(aq)} \rightarrow CaSO_{4(s)}$	$Ksp_{25°C} = 7.97 \times 10^4$	(24)
		$\Delta G°_{25°C} = -10.463\,Kcal$	-

(-) calculated in this study

3.4. Sludge Analyzing

The results presented in Figure 5 highlight the presence of additional phases in the system. This observation suggests that the precipitation mechanism of hydroxide metal has not been fully identified. The lack of identification of the hydroxide metal precipitation mechanism may be attributed to the presence of different metals in the solution. These metals may have an influence on the precipitation mechanism by altering the chemical and physical properties of the system. Moreover, the presence of other phases in the system may also suggest that there are complex chemical reactions occurring in the solution. The mechanisms of these reactions may be influenced by several factors, including the concentration of metal ions and the pH of the solution.

In summary, the results presented in Figure 5 suggest that the precipitation mechanism of heavy metal ions in the industrial wastewater may be influenced by the presence of multiple metals. This finding underscores the importance of considering the presence of multiple metals when investigating the precipitation of heavy metal ions in industrial wastewater. The presence of multiple metals can have a significant impact on the precipitation mechanism and can potentially result in the formation of additional phases.

Table 7: Elemental analysis references, content of patterns from X-pert highscore.

Visible	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
*	00-019-0636	55	Iron Titanium	0.713	FeTi
*	01-085-0849	47	Calcium Carbonate	0.195	$CaCO_3$
*	01-073-0257	20	Aluminum Lithium Silicate	0.029	$LiAlSiO_4$
*	01-081-0119	33	Strontium Vanadium Oxide	0.057	$SrVO_{2.93}$
*	01-072-1214	58	Calcium Carbonate	0.484	$CaCO_3$
*	01-085-1989	18	Lithium Chromium Manganese Oxide	0.089	$Li(CrMnO_4)$
*	00-046-1217	61	Copper Nickel Zinc Oxide	0.544	$Cu_{1.02}ZnNi_{3.27}O_{5.29}$
*	01-075-0272	53	Nickel Zinc Oxide	0.471	Ni_7Zn_3O
*	00-043-1159	25	Aluminum Titanium Vanadium	0.103	$Al_3V_{0.33}Ti_{0.666}$
*	00-036-0811	21	Beryllium Arsenide	0.073	$BeAs_2$
*	00-029-0913	13	Molybdenum Boron Carbide	0.149	Mo_2BC
*	00-033-1442	13	Vanadium Oxide Phosphate	0.088	$V(PO_3)_3$
*	01-075-0272	58	Nickel Zinc Oxide	0.651	Ni_7Zn_3O
*	00-031-0871	25	Molybdenum Carbide	0.105	Mo_2C
*	00-036-0811	19	Beryllium Arsenide	0.123	$BeAs_2$
*	00-029-1380	10	Vanadium Oxide Hydroxide	0.185	$V_2O_2(OH)_3$
*	01-072-0132	27	Cadmium Thorium	0.065	Cd_2Th
*	01-077-2308	31	Lithium Zinc Chromium Oxide	0.578	$LiZn_2CrO_4$
*	00-034-1269	20	Potassium Arsenate	0.364	$K_4As_2O_7$
*	00-030-0203	8	Cadmium Arsenide	0.179	Cd As
*	00-036-0811	20	Beryllium Arsenide	0.064	$Be As_2$
*	01-075-0272	59	Nickel Zinc Oxide	0.474	Ni_7Zn_3O
*	00-048-1710	0	Aluminum Arsenide	0.218	AlAs
*	00-041-0406	13	Cadmium Bismuth Germanium Oxide	0.254	$CdBi_2GeO_6$

4. CONCLUSION

The treatment of industrial wastewater to remove heavy metal ions is a complex process that involves several factors. One of the most critical factors that influence the efficiency of this process is the pH value. Chemical precipitation method is widely used to remove heavy metal ions from industrial wastewater. In this method, a chemical reagent is added to the wastewater to form a precipitate that can be separated from the liquid phase.

In this study, the effect of pH value on the removal of heavy metal ions from industrial wastewater was investigated. The results of this study showed that the removal efficiency of heavy metal ions increases with an increase in pH value. Moreover, the optimum pH range for the removal of most elements was found to be between 8 and 11, where the removal efficiency of heavy metal ions reached up to 99% to 100%.

The pH value can be controlled by adjusting the dosage of the chemical reagent added to the wastewater. Calcium oxide (CaO) is a commonly used chemical reagent that can increase the pH value of wastewater. The results of this study showed that the optimal mass of calcium oxide required for the removal of heavy metal ions was approximately 16 to 24 g/L, which corresponds to a pH value range of 8.614 to 11.345.

It is important to note that the presence of other elements in the wastewater can also affect the pH value of the precipitation for each metal. X-ray diffraction (XRD) was used in this study to determine the form of the precipitate. The results of XRD analysis showed that the presence of other elements in the wastewater can influence the form of the precipitate, which can affect the efficiency of the removal process.

In conclusion, the pH value is an important factor that should be carefully controlled in the chemical precipitation method for the removal of heavy metal ions from industrial wastewater. The optimal pH range for the removal of most elements is between 8 and 11, and the dosage of calcium oxide should be carefully controlled to achieve the optimal pH value. The presence of other elements in the wastewater should also be considered when designing the treatment process.

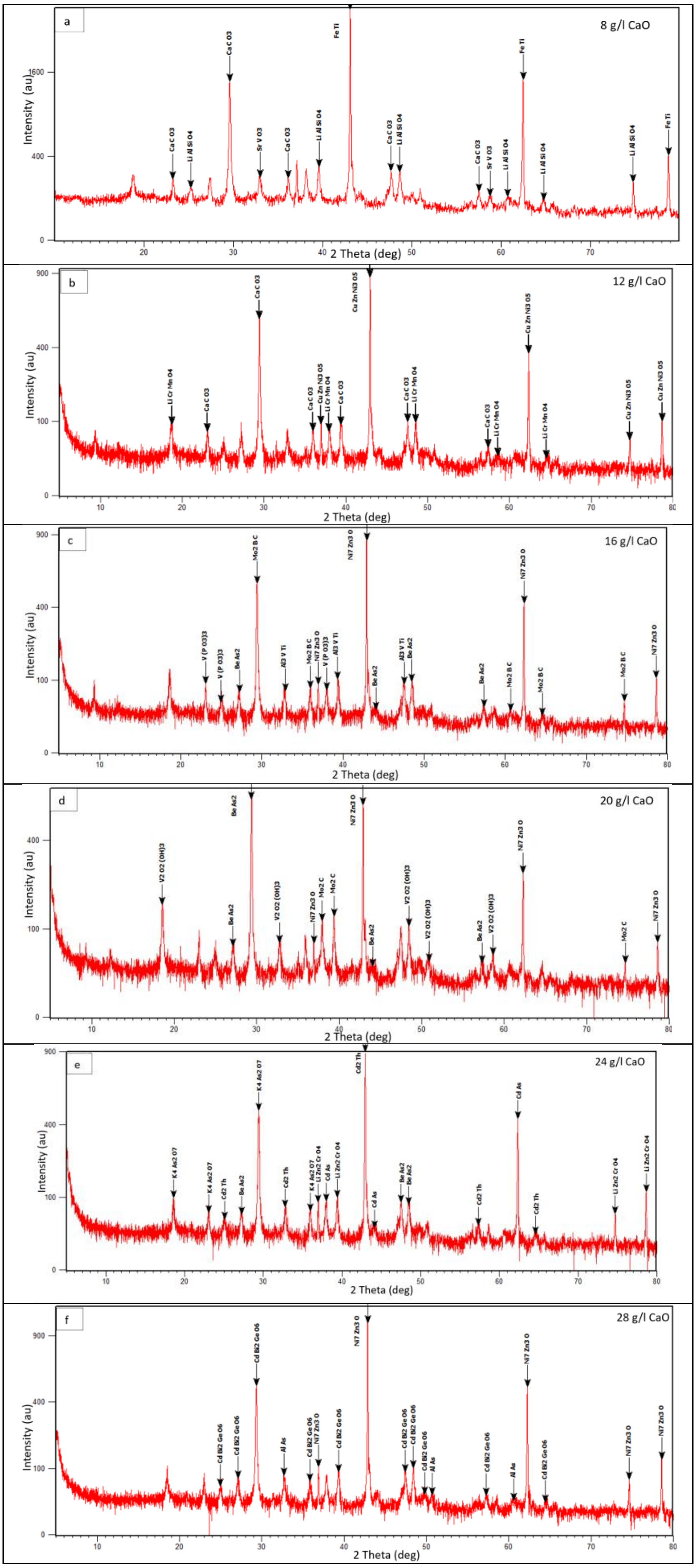

Figure 5: Diffractograms for sludges produced using different dosages of CaO by XRD: a) 8 g/L CaO, b) 12 g/L CaO, c) 16 g/L CaO, d) 20 g/L CaO, e) 24 g/L CaO, f) 28 g/L CaO.

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