Universidad Continental

FACULTAD DE INGENIERÍA

Escuela Académico Profesional de Ingeniería de Minas

Tesis

Toxic Metals Reduction in Acid Mine Drainage through the Use of Calcium Carbonate: A Case Study in Canchayllo,Peru

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Para optar el Título Profesional de
Ingeniero de Minas

Huancayo, 2025

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Toxic Metals Reduction in Acid Mine Drainage through the Use of Calcium Carbonate: A Case Study in Canchayllo,Peru

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- https://trebuchet.public.springernature.app/get_content/5cf742b6-1b16-4e94-a0c0-ddc83723239d?format=pdf&from=springerlink

- DOI del libro Book: 10.1007/978-3-031-54684-6

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Toxic Metals Reduction in Acid Mine Drainage through the Use of Calcium Carbonate: A Case Study in Canchayllo, Peru"

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Abstract. Environmental liabilities comprise facilities, effluents, contaminated sites, and residues or waste deposits that have a real, potential, or permanent impact on people's health and environmental quality. This study focuses on the evaluation of calcium carbonate extracted from limestone as a neutralizing agent due to its abundance, low cost, and low toxicity for the treatment of acid mine drainage in the "La Calzada" mining environmental liability in Canchayllo, Peru. Wastewater analyses were conducted at Continental University, where metal levels and pH were determined. The results revealed that "La Calzada" mining liability has high levels of iron (3,266-9,287 mg/L), arsenic (0.24-0.35 mg/L), copper (1,560-2,313 mg/L), and an acidic pH of 3.14, exceeding environmental quality standards for water. To address this issue, calcium carbonate with 87% purity, obtained from crushed limestone, was used as a neutralizing agent. The application of this compound in the samples resulted in an increase in pH from 3.14 to 7.50, as well as a reduction in concentrations, yielding final concentrations of arsenic (0.039 mg/L), copper (0.107 mg/L), and iron (0.056 mg/L). These findings demonstrate the effectiveness of calcium carbonate as a flocculant in the treatment of acid mine drainage in the "La Calzada" mining liability, suggesting its potential application in other areas affected by similar mining environmental liabilities.

Keywords: Wastewater, acid mine drainage, limestone rock.

1 Introduction

Peru is renowned for its long history and mining tradition, particularly in the Andean region. Throughout the 20th century, there has been a significant increase in exploration efforts in this region, establishing mining as one of the country's leading extractive industries. Peru has stood out as the world's second-largest producer of copper, silver, and zinc, thanks to its vast mineral potential [1].

However, this mining development has had a considerable and inadequately managed environmental impact, resulting in the degradation of soils and critical water bodies essential for food production, ecosystem preservation, and industrial purposes [2]. The resulting contamination has rendered water from these sources unusable due to high levels of salts, microorganisms, pathogens, and heavy metals.

Mining Environmental Liabilities, which include effluents, emissions, abandoned structures, and waste generated by mining [3], have become a critical problem. Despite their documented presence in the inventory of the Ministry of Energy and Mines, which, as of June 30, 2021, numbered 7,668, with 921 of them classified as highly hazardous [4], the lack of government oversight and limited application of environmental regulations have hindered impact mitigation [5]. The main challenge is identifying responsible parties, often resulting in the state assuming the responsibility for remediation, increasing the burden on public resources [6].

This study addresses the mining environmental liability "La Calzada" in Canchayllo, Peru. This mining liability raises concerns due to its high levels of contaminants such as iron, arsenic, and copper in acid mine drainage [7]. These toxic elements are worrisome for their ability to accumulate in the food chain [8], posing a threat to wildlife and the natural environment. Additionally, these contaminants affect water quality [9], which, in turn, raises concerns for human health since there is a downstream pond with the same name used for industrial fish farming that is constantly contaminated by the acid mine drainage.

In pursuit of an effective and sustainable solution, this article focuses on the evaluation of calcium carbonate extracted from limestone as a neutralizing agent. The choice of this compound is based on its abundance in the region, its low cost, and low toxicity, making it a promising option to address the issue of acid mine drainage at "La Calzada." This process involves the neutralization of metals through chemical precipitation, a method that induces the formation of solid particles through chemical reactions where contaminants are converted into insoluble compounds [10]. The chemical reactions involved in this process are described in detail in the article.

The limestone extraction area is located at the convergence of various geological formations in the districts of Canchayllo, Llocllapampa, and Yanacancha, where limestone outcrops with varying contents of calcium oxide (CaO) and magnesium oxide (MgO) can be found, resulting in variations in limestone purity. In mine drainage treatment, there are generally two approaches: active treatment, which requires constant human intervention and external energy [11], and passive treatment, which relies on natural processes and the use of natural construction materials [12]. Both approaches can be applied in situ, reducing costs and environmental risks associated with excavating and transporting contaminated materials [13]. However, in this case, a passive treatment is proposed as the optimal solution.

This research aims not only to address the challenges of contamination in the "La Calzada" mining environmental liability but also to contribute to the understanding of effective and sustainable solutions for managing mining environmental liabilities in similar contexts. The choice of passive treatment is based on the nature of the geological formations present in the limestone extraction area, which provides an opportunity to leverage natural processes. This not only enhances the efficiency and sustainability of the solution but also reduces constant human intervention and dependence on external energy, thereby lowering costs and associated risks.

2 Methods and Materials

2.1 STUDY AREA

The research study was conducted at the Former La Calzada Mining Unit, a Mining Environmental Liability, located in the town of Canchayllo, in the province of Jauja, within the department of Junín, Peru. It is situated at an altitude of 4,340 meters above sea level and has UTM coordinates 10247.373E 8677594.408N. It is registered in the catalog of the Ministry of Energy and Mines as number 200-2021 with ID 1338. The coordinates for this location are East 408642, North 8678957, Zone 18, Datum WGS84, and it is classified under Mining Law Code 620006715;01049700 6;620005310;010417 895;010100799 [4].

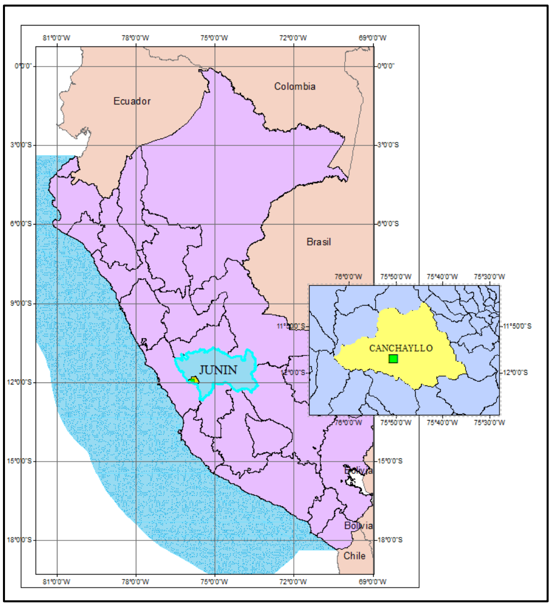

Fig. 1. Geographical Location of La Calzada Mining Environmental Liability

Fig. 2. Location of the Environmental Liability and La Calzada Lagoon, obtained from the official GEOCATMIN *website.*

This environmental liability is responsible for the ongoing contamination occurring in the lagoon with the same name located in the Department of Junín, specifically in the Province of Jauja, District of Canchayllo, with Latitude: -11.9576, Longitude: -75.8331. The lagoon has an approximate perimeter of 4.84 kilometers and a distance between its ends of 2.24 kilometers.

2.2 Maximum Permissible Limit

Table 1 shows the parameters of the Maximum Permissible Limit (MPL) established by Supreme Decree No. 004-2017-MINAM, Category 4 - Conservation of the aquatic environment in lakes and lagoons. However, in this category, the MPL for Iron in this environment is not specified. Therefore, as a reference, Category 1-A1 is used, which recommends a maximum permissible limit of 0.3 mg/L for consumption. This figure will be used because the water found in this lagoon is used several kilometers downstream for domestic consumption without prior advanced purification treatment [14].

Table 1. Maximum Permissible Limits - ECA Category 4-E1 & Category 1-A1: Conservation of the Aquatic Environment - Lakes and Lagoons, Category 1 - Population.

Lakes and lagoons	Parameters	Unit of measurement
Copper	0.1	mg/l
Zinc	0.12	mg/l
Arsenic	0.15	mg/l
Lead	0.0025	mg/l
Iron	0.3	mg/l

Table 2. Maximum Permissible Limits for the Discharge of Liquid Effluents from Mining and Metallurgical Activities

Parameters	Unit of measurement	Limit at any time	Limit for the annual average
Total Copper	mg/l	0.5	0.4
Total Arsenic	mg/l	0.1	0.08
Total Lead	mg/l	0.2	0.16
Iron (dissolved)	mg/l	2	1.6
pH		6-7	6-9

Note: Table 2 displays the maximum allowable limits for the discharge of liquid effluents from mining and metallurgical activities, as stipulated by Supreme Decree No. 10-2010-MINAM-Annex 01. Exceeding these limits would pose a threat to health, human well-being, and the environment [15].

2.3 Sampling Method

The recommended materials were used in accordance with the guidelines of the National Protocol for Monitoring the Quality of Surface Water Resources, along with established procedures approved by Judicial Resolution No. 010-2016-ANA [16]. Following the chain of custody procedure for environmental monitoring, relevant points in the area of the La Calzada Mining Environmental Liability were selected for the analysis of metal content and pH levels in the wastewater.

Samples were collected using sterilized 1000 ml containers, to which 20 drops of nitric acid were added for metal extraction. Analytical research was conducted in a laboratory certified by INACAL (National Institute of Quality). The testing methodology used by the laboratory to determine metal content and pH is detailed in Table 3. The results of the analysis of two collected samples are presented in Tables 5 and 6.

Table 3. Standards Applied in the Analysis of Heavy Metals and pH

Practice	Reference Method	Description
Copper	1SMEWW-APHA-AWWA-WEF Parte 3500-CU B, 23ª ed.2017	Cu: Atomic Absorption Spectrometry Method.
Zinc	SMEWW-APHA-AWWA-WEF Parte 3500-Zn B, 23ª ed.2017	Zn: Atomic Absorption Spectrometry Method.
Iron	SMEWW-APHA-AWWA-WEF Parte 3500-FE B, 23ª ed.2017	Fe: Atomic Absorption Spectrometry Method.
Arsenic	SMEWW-APHA-AWWA-WEF Parte 3500-As B, 23ª ed.2017	As, Atomic Absorption Spectrometry Method.
Lead	SSMEWW-APHA-AWWA-WEF Part5210-PB B, 23ª ed.2017	Pb: Atomic Absorption Spectrometry Method.
pH	SMEWW-APHA-AWWA-WEF Part4500 - H+ B, 23ª ed.2017	Valor de pH. Potentiometric Method.
% Calcium carbonate $\mathbf{CaCO_3}$	NOM-021-RECNAT-2000-AS-29	Determination of Calcium Carbonate Equivalent, Acid Neutralization Method

2.4 . Limestone rock preparation

Table 4. Limestone rock preparation

Standards	Procedure	
Specific research method	- Several samples of limestone were extracted near PAM La Calzada, and their reaction to hydrochloric acid was verified. - The samples were cleaned to remove any organic residues. - A 4" x 8" laboratory stone crusher was used to reduce the particle size. - The Ro Tap sieving machine was used to separate different particle sizes. Subsequently, only the results from the 10 mm sieve and the 0 mm sieve (limestone sand) were used. - The CaCO3 purity of the limestone rock was analyzed in a laboratory accredited by INACAL, "Laboratorio Ambiental S.A.C." which determined a purity level of 84%. - Finally, the Jar test machine was used to perform multiple experiments at various concentrations.	Calcium carbonate equivalent determination, acid neutralization method

Note: Table 4 describes the process carried out in the laboratory of Continental University to obtain crushed limestone and limestone sand.

It is worth emphasizing that the use of calcium carbonate as a flocculant provides notable advantages compared to quicklime. This flocculant is more cost-effective as it doesn't require a prior calcination process, and it is easier to handle [17]. Its use will help increase the water's pH, which is beneficial in the neutralization process.

3 Results

3.1 Water Quality of the Inflow

The results from the laboratory tests, as evidenced in Table 5 and Table 6, reveal that the water from the La Calzada mining environmental liability has a high presence of iron, with concentrations ranging from 3.266 mg/L to 9.287 mg/L. Furthermore, arsenic content ranging between 0.24 mg/L and 0.35 mg/L is detected, along with copper content between 1.56 mg/L and 2.313 mg/L. Additionally, a pH level of 3.14 is observed, indicating acidity.

Table 5. Results of influent 1 - Test report No. AL/IE-2022-025

Customer code	Lab code	Practice	Result	Unit
PM-1-CANCHAYLLO	M-22063	Copper	2.313	mg/l
		Zinc	0.138	mg/l
		Iron	3.266	mg/l
		Arsenic	0.353	mg/l
		Lead	<0,001	mg/l

Table 6. Results of Influent 2 - Test Report No. AL/IE-2022-030-Sample 3 - Water inlet to the lagoon

Customer code	Lab code	Practice	Result	Unit
PM-1-CANCHAYLLO	M-22063	Copper	1.560	mg/l
		Iron	9.287	mg/l
		Arsenic	0.24	mg/l
		pH	3.14	mg/l
		% calcium carbonate $CaCO_3$	87	%

3.2 Importance of pH in Neutralization

pH is a crucial factor for achieving proper precipitation. The following figure shows that as the pH increases, ferric iron precipitates at values close to 4.

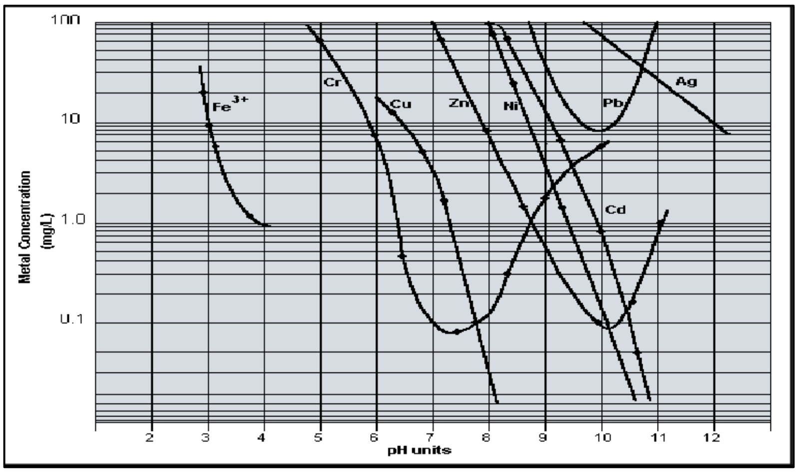

Fig. 3. Solubility of metal hydroxides as a function of pH

This process carries arsenates, which result from the oxidation of arsenic, as arsenates (i.e., As(V)) can be removed from an aqueous solution by precipitation with ferric iron (i.e., Fe (III)) [18], and the amounts adsorbed can be significant even when the concentrations of arsenic in the solution are low [19]. Meanwhile, copper settles at values above 6.2.

The following reactions describe that, as the pH value increases, the anions become available.

$$H_2O \rightarrow H^+_{(ac)} + OH^-_{(ac)} \quad (1)$$

$$H_2CO_{3\,(ac)} \leftrightarrow H^+_{(ac)} + HCO^-_{3\,(ac)} \quad (2)$$

$$H_2CO^-_3 \leftrightarrow H^+_{(ac)} + CO^{-2}_{3\,(ac)} \quad (3)$$

$$H_2S_{3\,(ac)} \leftrightarrow H^+_{(ac)} + HS^-_{3\,(ac)} \quad (4)$$

$$H_2S^-_{(ac)} \leftrightarrow H^+_{(ac)} + O^{-2}_{(ac)} \quad (5)$$

Limestone and dolomitic material react to neutralize the acidity as follows:

$$CaCO_3 + H^+ \rightarrow Ca^{+2} + HCO_{-3} \quad (6)$$

Precipitation of Metals

$$Cu^{2+} + 2oH^- \rightarrow Cu(OH)_2 \quad (7)$$

$$Fe^{2+} + 2oH^- \rightarrow Fe(OH)_2 \quad (8)$$

$$Ar^{3+} + 2oH^- \rightarrow Cu(OH)_2 \quad (9)$$

3.3 . NEUTRALIZATION OF PH

A total of 6 tests were conducted using water from the acid mine drainage of tributary 2 (Table 6). Volumes of 700 ml and 1000 ml of acid mine drainage water were used along with different concentrations of limestone to determine the minimum amount of limestone required to achieve viable results in contaminant removal. It is important to note that higher concentrations of iron were found in this sample, and limestone has been observed to be highly effective in removing this contaminant due to reaction kinetics that involve various factors such as the nature of the substance, particle size, concentration, temperature, and the presence of catalysts. Additionally, the precipitation of iron in the form of hydroxides and carbonates can also contribute to the removal of other contaminants. It has been demonstrated that certain ionic species, such as arsenic and molybdenum, co-precipitate or adsorb ferric hydroxides. Furthermore, there is evidence that some of these reactions can be facilitated by microorganisms [20], opening an intriguing window to a deeper and promising field of research. Subsequently, an evaluation was carried out to determine if the particle size of the limestone had any impact on the process.

. **Table 7.** Experimentation for pH Neutralization with Rock Resulting from Sieve Size 10

Sample Sieve 10	Relationship	Mine acid drainage	Limestone $CaCO_3$	Initial pH	10 mins.	20 mins.	30 mins.	60 mins.
M1	10/1	700 ml.	70g.	4.51	5.85	6.58	6.75	6.68
M2	3/1	700 ml.	230g.	4.34	5.62	6.67	6.76	7.21
M3	2/1	1000 ml.	500g.	3.14	6.2	7.0	7.4	7.45

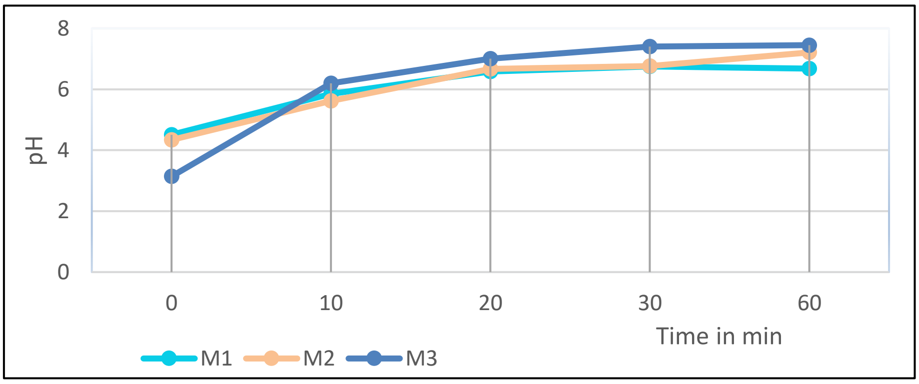

Fig. 4. Experimentation for pH neutralization using rock resulting from sieve 10.

Table 8. Experimentation for pH Neutralization with Rock Resulting from Sieve Size 0 (Limestone Sand)

Sample Sieve 0	Relationship	Mine acid drainage	Limestone $CaCO_3$	Initial pH	10 mins.	20 mins.	30 mins.	60 mins.
M4	1.5/1	700 ml	450g.	3.86	5.55	7.01	7.12	7.15
M5	2/1	700 ml	350 g.	3.79	5.40	6.7	6.8	7.1
M6	3/1	700 ml	350 g.	4.04	5.60	6.7	6.74	7

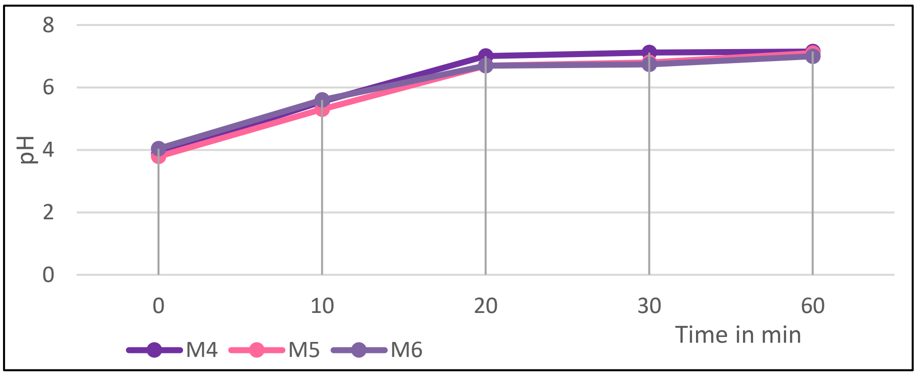

Fig. 5. Experimentation for pH neutralization using rock resulting from sieve 0 (limestone sand)

3.4 . LIMESTONE AS A FLOCCULANT FOR Cu, Fe, Ar

Three tests were conducted using acid mine drainage water from tributary 1 (Table 5). 1000 ml of water with 1000 mg of crushed limestone sieved at mesh 10 were used. The tests were monitored and separated from the flocculant after 10 minutes, 30 minutes, and 60 minutes, respectively. For this purpose, filter paper was used to separate the water from the sediment. After labeling each 500 ml sample container, they were delivered to the "Ambiental Laboratorios" laboratory for atomic absorption testing, following the chain of custody protocol.

Table 9. Laboratory Results from "Ambiental Laboratorios"

Sample Sieve 10	Lab code	Mine acid drai-nage	Limes-tone $CaCO_3$	Initial pH	Cu mg/L	Fe mg/L	Ar mg/L
Start of test	M-22084	1000 ml.	1000g.	3.14	2.313	3.266	0.353
15 minutes	M-22085	1000 ml.	1000g.	7.5	0.135	0.090	0.060
30 minutes	M-22086	1000 ml.	1000g.	7.51	0.112	0.060	0.044
60 minutes	M-22087	1000 ml.	1000g.	7.5	0.103	0.056	0.039

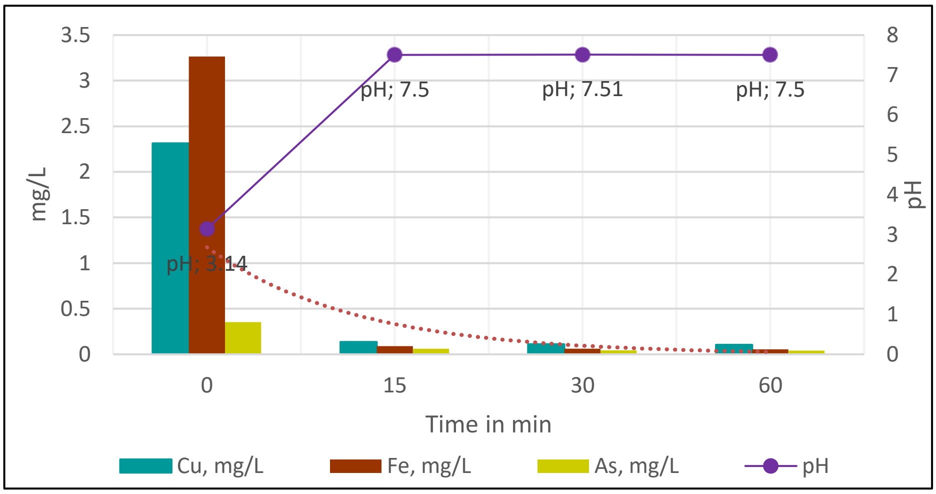

Fig. 6. Variation in Heavy Metal Concentration and pH Value.

The results of atomic absorption obtained from the laboratory analysis indicate that the calcium carbonate present in the limestone reduced the concentrations of Cu by 96%, Fe by 98%, and As by 89%.

4 Discussion

In this study, the effectiveness of using calcium carbonate (CaCO3) for the removal of heavy metals in acid mine drainage was examined. The results obtained indicate that calcium carbonate is highly effective in removing metals such as copper, iron, and, under certain circumstances, arsenic.

For this research, limestone with a particle size of 10 mm and limestone sand were used for flocculation. It was observed that calcium carbonate has the ability to precipitate these heavy metals, forming insoluble compounds that can be easily separated from the water. Evaluations of the samples before and after treatment revealed a significant decrease in the concentrations of heavy metals, reaching levels below those allowed by environmental regulations. The tests, where an increase in pH was documented, were conducted at a 1:1 ratio (1000 ml per 1000g), yielding similar results even at ratios of 10:1 (700 ml per 70g). Due to the minimal difference between both particle sizes used, no significant variability was found between the tests with a 10 mm particle size and limestone sand. However, it is worth noting that according to the International Network for Acid Prevention, the most effective systems for Acidic Drainage (ALD) treatments have used limestone ranging from 5 to 20 cm [21] in size. Ziemkiewicz suggests that an open limestone channel, as indicated by its name, has free-flowing water with a lining of coarse limestone, meaning grains larger than 10 cm [22]. The use of rocks of this size allows for proper permeability and water flow through the system. Larger rocks can obstruct water flow, while smaller rocks may not provide a sufficient reaction surface for acid neutralization and metal precipitation [13].

It is important to note that the pH of acid mine drainage water plays a crucial role in the effectiveness of calcium carbonate as a metal removal agent. It was found that neutralizing the acidic pH by adding calcium carbonate promotes the precipitation of heavy metals and enhances the efficiency of the removal process [23]. However, Faulkner and Skousen reported both successes and failures among 11 limestone anoxic drains (ALDs) treating mine water in West Virginia in 1994. In all instances, the water's pH level increased after ALD treatment, and the acidity of the water in these drainage systems decreased by 50% to 80%. Yet, three of the sites had pH values below 5.0, indicating that the ALD did not work completely, possibly due to limestone coating or obstruction since when it functions correctly, the water's pH values in the ALDs should be at least 6.0. They suggest that some limestone coating or obstruction likely occurred [24]. As a result, this method requires constant monitoring, which is why an Open Limestone Drain (OLD) is proposed as an effective method for the current case. In a study by Ziemkiewicz et al. in 2003 on the removal efficiency of acid by 83 different types of passive water treatment technologies located in the United States, including 10 OLDs, it was concluded that while the removal of heavy metals is lower compared to other passive treatment systems, this is the method with the lowest average construction cost and the second-lowest cost in acidity removal rate [25]. Furthermore, if the OLD is designed and built correctly, it should require no maintenance and treat acidic mine waters for decades.

On the other hand, while calcium carbonate demonstrated high efficiency in removing heavy metals in this study, it is important to consider the associated limitations and challenges. Implementing this at a full-scale may require designing treatment systems tailored to the specific conditions of each mining site and considering economic and operational factors [26].

In general, the results of this study support the feasibility of using calcium carbonate as a promising option for the removal of heavy metals in acid mine drainage. However, further research is needed to optimize treatment conditions, assess the removal of other metals, and consider the sizing or tonnage of limestone required for a viable environmental remediation, as these require a comprehensive analysis and were not the subject of investigation in this study.

5 In-Situ Treatment Proposal

The use of the in-situ passive treatment method known as the Open Limestone Drain (OLD) is recommended due to its simplicity and efficiency in treating acid mine drainage. In the study by Ziemkiewicz et al. [27], it was suggested that OLD systems would be useful in abandoned mine reclamation projects, where installation costs are one-time, and regular maintenance is not feasible, as in the present case.

There are two approaches to implement open limestone channels. The first involves constructing a drainage trench using limestone, responsible for collecting water contaminated with acid mine drainage. The second approach involves placing limestone fragments directly in a contaminated stream or pit. For this case, the second option is considered more viable because there are already moderate-flow streams and pits with a slope greater than 20%, which would hinder the limestone coating process but favor its dissolution.

It's important to consider that the coating of limestone by precipitates of Fe (CO3)3 and Fe (OH)3 generated through neutralization can decrease alkalinity generation. Therefore, a considerable amount of limestone is required to ensure long-term success.

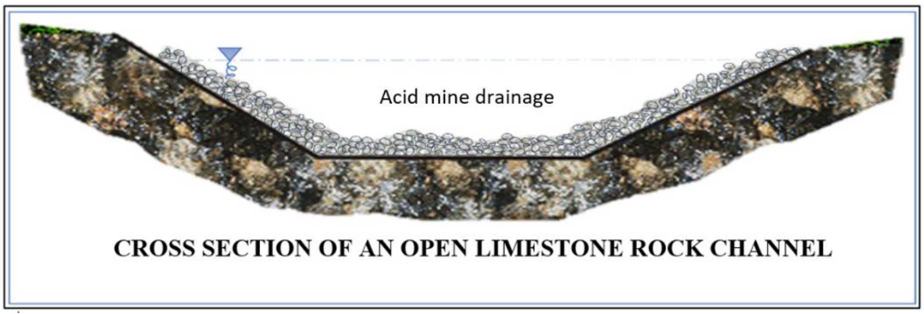

Fig. 7. Proposal for Treatment with Open Limestone Drainage

6 Conclusions

In summary, this study provides evidence of the effectiveness of calcium carbonate obtained from crushed limestone as an efficient option for the removal of heavy metals in the acid mine drainage of the environmentally responsible "La Calzada" mine. The addition of calcium carbonate raises the pH from 3.14 to 7.5, facilitating the precipitation of metals such as copper, iron, and arsenic, significantly reducing their concentrations in treated water by 95.55%, 98.29%, and 88.96%, respectively.

The neutralization of acidic pH and the formation of insoluble compounds are key mechanisms involved in the precipitation removal process of Cu, Fe, and As. However, it is essential to consider the specific conditions of each mining site, including the presence of other ions and the characteristics of the drainage water, to optimize treatment efficiency.

While the results are promising, as they provide a solution to contamination without the need for a prior calcination process, further research is recommended to address aspects such as the removal of other heavy metals and the optimization of treatment conditions.

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