



## Evaluation and Comparative Analysis of Heavy Metal Leaching Efficiency by Nitric Acid, Perchloric Acid and Sulfuric Acid from Moroccan Phosphate Solid Waste

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**Abstract:** This article presents a comprehensive study into the leaching of heavy metals such as Al, As, Cd, Cu, Zn, Ni, Fe and Cr from Moroccan phosphate solid waste. Sulfuric acid, perchloric acid and nitric acid were used as leaching agents at various concentrations (0.5 M, 1 M, 2 M, 3 M and 4 M) under ambient temperature conditions, maintaining a particle size of 160  $\mu\text{m}$  and a stirring time of 1 hour. To optimize the efficiency of metal extraction, research focused on the subtle interplay between acid selection and concentration. Nitric acid appears as the front runner, consistently showing excellent leaching results for all metals, especially at higher concentrations. Although perchloric acid does not exceed the efficiency of nitric acid, its performance is competitive and position it as a viable alternative, with encouraging results, especially at moderate concentrations. Sulfuric acid shows different trends in metal leaching efficiency, highlighting the need for separate and customized approaches. At lower concentrations it shows moderate effectiveness, with leaching efficiency varying depending on the metal. As acid concentration increases, extraction efficiency changes, requiring careful consideration of specific metal properties. In the hierarchy of leaching agents, the position studied is nitric acid > perchloric acid > sulfuric acid, indicating the superior performance of nitric acid, followed by perchloric acid, compared to sulfuric acid during metal leaching.

**Keywords:** Leaching process, heavy metal, nitric acid, perchloric acid, sulfuric acid, Moroccan phosphate solid waste, comparative analysis.

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### 1. INTRODUCTION

Heavy metal pollution is one of the major environmental problems today. Most heavy metal ions are toxic to living organisms. Heavy metals are present in nature and industrial waste. The main source of heavy metal pollution is metal plating, mining, smelting, battery manufacturing, tanneries, petroleum refining, pigment manufacturing, etc. (Sulaymon et al., 2014; Yuntao Chen et al., 2024).

Heavy metals are defined as metals with a specific density of greater than 5  $\text{g/cm}^3$ , this definition includes the alkali metals, alkaline earth metals, lanthanides and actinides, which are not considered heavy elements in a chemical sense, while excluding some other elements such as arsenic, due to its chemical and ecological effects, it is often considered a heavy metal. One classification that can combine these properties and explain the similarities in mechanisms of heavy metal toxicity in different organisms is based on equilibrium constants describing the formation of metal ion ligand complexes.

Therefore, there are three classes of metals with different bonding preferences. Elements with an affinity for oxygen containing ligands belong to class A, elements with an affinity for nitrogen- or sulfur-containing ligands belong to class B, and elements with intermediate characteristics have an affinity for O<sup>-</sup>, S<sup>-</sup> or N<sup>-</sup> containing ligands belonging to borderline (Stanković et al., 2018).

Based on coordination chemistry and the effects on the environment, heavy metals are all in the class B and the borderline group (Stanković et al., 2018; Kumar Sharma et al., 2004).

The principal elements classified as heavy metals include Cr, Mn, Co, Cu, Zn, Mo, Hg, Ni, Sn, Pb, Cd, Sb, etc. (Sulaymon et al., 2014).

Cd with an atomic number of 48 and an atomic weight of 112.41, is classified within group XII of the periodic table of chemical elements. It naturally occurs at trace concentrations, associated with sulfide ores of zinc, lead and copper. Human activities including the combustion of fossil fuels and the release of leachate from landfill sites, agricultural areas and mining waste, particularly those linked to zinc and lead mining operations, contribute significantly to the contamination of the environment with cadmium (Genchi et al., 2020).

Cr is the seventh most abundant element in the earth. With an atomic number of 24 and a mass number of 52, it occupies a place in group 6 and period 4 of the periodic table. It exhibits various oxidation states, ranging from 0 to +6, with the most stable states being 0, +3 and +6. The primary source of chromium is chromite ore, where it predominantly exists in the +3 oxidation state. However, industrial processes produce hexavalent chromium and the elemental form of the metal (Mukherjee et al., 2013).

Cu is a transition metal, found in nature in two oxidation states: Cu (I) and Cu (II). Cu (I) is a soft and durable metal while Cu (II) is borderline according to the hard and soft acids and bases classification (Dupont et al., 2011). The mainly source of environmental contamination due to copper including mining, printed circuits, metallurgical, fiber production, pipe corrosion and metal plating industries. Additionally, other significant sectors releasing copper into their effluents including the paper and pulp, petroleum refining and wood preserving industries (Sulaymon et al., 2014).

Ni is the 24<sup>th</sup> most abundant element in the earth. With an atomic number of 28 and an atomic weight of 58.71. It is typically found in igneous rocks, either in its elemental form or in conjunction with iron (Amari et al., 2017). Nickel is naturally released into the environment through processes such as windblown, weathering of rocks and wildfires. Human made activities, including the

operations of foundries and battery manufacturing plants, also contribute to the environmental release of nickel. Additionally, nickel can be discharged into the environment through sources like tobacco use and the use of stainless-steel kitchenware (Kiran et al., 2021).

As is classified as a metalloid, is naturally present on earth, being the 20<sup>th</sup> most abundant element. It is a constituent of over 245 different minerals. The inorganic form, mainly consisting of arsenic and arsenate compounds, poses a health hazard to humans (Guha Mazumder, 2008). Arsenic is a common occurrence in nonferrous ores such as copper, lead, zinc, gold and uranium. As is a predominant component in specific ores, such as the copper mineral enargite, while it exists as a trace impurity in other ore types. Unregulated human activities such as smelting metal ores, using arsenic based pesticides, and using wood preservatives, can also lead to direct releases of arsenic into the environment (Choong et al., 2007).

As base metal prices have increased significantly in recent years, the extraction of these metals from various waste sources, including solid phosphate waste, has become increasingly important (Huang et al., 2011).

Ongoing global efforts include in depth research aimed at identifying efficient and cost-effective ways to process solid waste and extract valuable metals from it (Ajiboye et al., 2019).

In practice, innovative hydrometallurgical methods for metal extraction have emerged, including hydrothermal techniques (Zhang et al., 2006), subcritical water treatment (Zhang et al., 2006), and fungal bioleaching (Ishigaki et al., 2005). Although those methods have promising extraction capabilities, they often increase operating costs or extend leaching times. Therefore, it is recommended to give priority to performing the leaching process at ambient temperature, emphasizing the importance of careful selection of suitable leaching agents to achieve satisfactory treatment efficiency throughout the process (Huang et al., 2011).

Leaching is a technique for dissolving soluble substances from a mixture of insoluble solids. The solvent used in the leaching process is called a leaching agent (Yuliusman et al., 2018), and various leaching agents, such as inorganic acids such as sulfuric acid, hydrochloric acid and nitric acid (Hussaini et al., 2021), and organic acids (Fatima et al., 2024) such as citric acid (Wu et al., 2006), oxalic acid (Nugteren et al., 2001; Fengyuan et al., 2024), acetic acid (Fuoco et al., 2005), tartaric acid (Smichowski et al., 1998), and chelating agents such as nitrilotriacetic acid (NTA) (Wu et al., 2022), ethylenediaminetetraacetic acid (EDTA) (Opi et al., 1999) and diethylenetriamine-pentaacetic acid (DTPA) (Singh et al., 2013), and in some cases alkaline solutions such as ammonium

hydroxide and sodium hydroxide (Ntumba Malenga et al., 2015), can be used to extract heavy metals. Of the above-mentioned leaching agents, sulfuric acid and hydrochloric acid generally appear to be the most suitable (Huang et al., 2011; Shokrullah et al., 2024).

The experimental approach was to perform a leaching process of solid phosphate waste using different acid treatments, each with a specific concentration. Sulfuric acid, nitric acid and perchloric acid were selected as the main agents for metal extraction. The entire process was performed at ambient temperature. The purpose of this study was to determine the different effects on the efficiency of extraction of Fe, Al, Cd, Ni, Zn, Cr, As and Cu from waste materials by varying the concentration of these acids. Comparative analysis of these acid treatments provides valuable insights into optimal conditions for metal recovery from

Moroccan phosphate solid waste under ambient temperature conditions.

## 2. EXPERIMENTAL STUDIES

### 2.1. Materials And Reagents

The samples used for the leaching process in this study obtained from the Moroccan phosphate solid waste, we utilized a series of chemical reagents, including sulfuric acid, nitric acid and perchloric acid with different concentration (0.5 M to 4 M).

In our study of Moroccan phosphate solid waste, we used a variety of materials and techniques to analyze and characterize the samples. The GFL 3040 jar test from Gesellschaft Für Labortec Overhead Shakers; was used to stir the samples; to ensure efficient mixing and dispersion during the leaching process. This step is critical to achieve optimal leaching efficiency.

**Table 1:** Product utilization descriptions.

Product	Chemical formula	CAS number	Analysis method	Purity (conductivity and pH of water)
Sulfuric acid	H <sub>2</sub> SO <sub>4</sub>	7664-93-9	VT <sup>1</sup>	95%
Perchloric acid	HClO <sub>4</sub>	7601-90-3		70%
Nitric acid	HNO <sub>3</sub>	7697-37-2		69%
Distilled water	H <sub>2</sub> O	7732-18-5	CA <sup>2</sup>	Conductivity: 21.4 μS/cm pH: 7.61

1: Volumetric titration, 2: Conductimetric analysis

To measure the ion concentration after the leaching process, we used inductively coupled plasma (ICP) analysis. ICP allows to accurately quantify the concentrations of different ions in solution, and gain important insights into the leaching efficiency and distribution of elements in solid waste samples. Moreover, ICP was also instrumental in the analysis of metal ion concentrations within the solid waste, further enhancing our understanding of the waste's elemental composition.

Furthermore, for detailed analysis of a solid waste samples' crystalline structure and composition, we used X-ray diffraction (XRD). This technique uses diffraction patterns of X-ray to identify the crystal structure and composition of the samples providing valuable information about the chemical compounds present in the solid waste. X-ray diffraction analysis was conducted on all solid samples to assess the composition of solid phases within diverse saturated solutions. This analytical process was carried out using a BRUKER D2 phase shifter instrument equipped with a LYNXEYE detector, utilizing a Cu-Kα wavelength of 1.541874 Å. Each stable solid underwent analysis within a temperature range spanning from 10 to 80 °C for a duration of 20 minutes, allowing for a comprehensive characterization of the solid phases present.

### 2.2. Experimental Methodology

In a 250 mL flask, a precisely measured 10 grams of the solid waste sample were added. Subsequently, different concentrations of the previously prepared acid solutions were introduced into the flask, with concentrations ranging from 0.5 M to 4 M. These acids served as the leaching agents and were carefully selected to optimize the leaching process.

The entire setup was maintained at a constant temperature of 24°C throughout the experiments to ensure uniform conditions during the leaching process. The mixture is stirred continuously for one hour to ensure effective contact between the solid waste and the leaching agent.

After the stirring period, the samples were allowed to stand to facilitate the separation of solid and liquid phases. The decanted liquid was then filtered through a 0.45 μm filter to remove any remaining solid residue. This filtration process ensured that only the liquid phase without solid particles remained for later analysis.

The leachate obtained was analyzed using ICP for a comprehensive evaluation of the chemical composition, especially the concentration of various ions and metals. This analytical technique enabled accurate quantification of dissolved ions and provided valuable insight into leaching efficiency and liquid phase composition resulting

from the interaction of solid waste with different concentrations of leaching acid.

### 2.3. Characterization Of Phosphate Solid Waste

#### 2.3.1. Preparation of samples

Before further analysis, the solid waste samples underwent a careful pretreatment process to ensure suitability for subsequent experiments. The first step was to dry the samples in an oven set at 135°C for 30 min to 1 h. This controlled drying helped remove interparticle moisture, an important step to prevent clogging that may occur during the subsequent grinding process.

The samples were milled using a rotating disc mill consisting of a fixed steel disc and a movable counterpart. This grinding process reduces the sample to a fine consistency, typically reaching a

fineness of around 160  $\mu\text{m}$ . The purpose of this grinding process was to completely homogenize the sample.

After the grinding process, the obtained material was sieved through 160  $\mu\text{m}$  sieve. The material that did not pass the sieve was manually ground using a mortar and pestle and mixed with the material that passed the sieve.

To make certain the entire elimination of any ultimate moisture, the homogenized pattern underwent a very last drying step. It turned located in an oven set at a decrease temperature, commonly 105°C, for a length of hours, taking into account the full removal of residual humidity. This complete pattern guidance ensured that the solid waste was in an optimal state for subsequent analytical procedures and minimized the capacity for moisture associated interference.

#### 2.3.2. ICP characterization

**Table 2:** Analysis of the utilized phosphate solid waste in this study.

Elements	Units	Values for
Mg	%	14.2
Fe	%	8.2
Al	%	15.2
Cd	Ppm	418
Ni	%	50.96
Zn	%	426.4
Cr	%	229.8
As	%	10.96
Cu	%	50.59

**Table 3:** Operation conditions for ICP.

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

#### 2.3.3. Solid waste characterization

X-ray diffraction (XRD) analysis of our solid phosphate waste samples (Figure 1) revealed the presence of several significant crystalline compounds. The most prominent diffraction peaks in the spectrum correspond to several different minerals such as  $\text{Mg}_3\text{AlSi}_3\text{O}_{12}$ ,  $\text{As}_8\text{Cu}_{12}\text{S}_{18}$ ,  $\text{Mg}_4\text{FeCa}_4\text{Si}_4\text{O}_{16}$ ,  $\text{Zr}_4\text{Cr}_4\text{NiCuO}_{14}$  and  $\text{CdMg}_5\text{C}_6\text{O}_{18}$ . Each peak in the XRD spectrum is closely related to a specific interplanar distance and corresponding  $2\theta$  diffraction angle, forming a unique signature for each compound. The relative

intensities of these peaks provide valuable insight into the abundance of each mineral in the sample.

The presence of this compound is confirmed by the distinct peaks observed in the diffraction pattern. This XRD analysis provides a comprehensive understanding of the mineral composition of the samples which is essential for further studies. Our results provide a solid foundation for a detailed study of the chemical leaching processes involved in the removal of elements such as Mg, Cd, Fe, Al, Ni, Zn, Cr and As from samples.

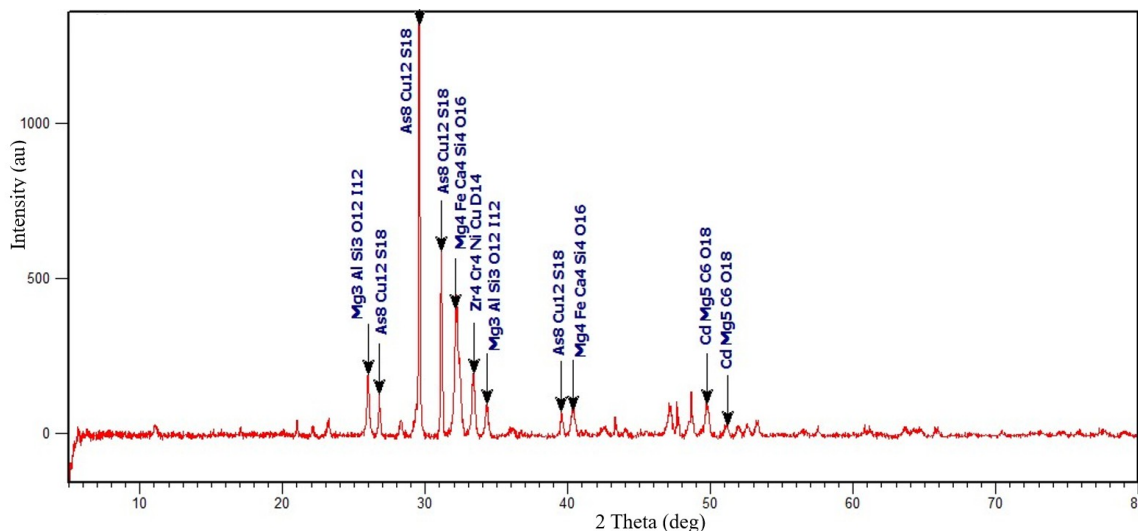


Figure 1: X-ray diffraction pattern of phosphate solid waste sample.

Table 4: Elemental analysis references, content of pattern from X-pert Highscore.

Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
96-150-9454	20	Ag (Mg <sub>0.5</sub> Zn <sub>0.5</sub> )	0.054	Mg <sub>0.50</sub> Zn <sub>0.50</sub> Ag <sub>1.00</sub>
96-901-0226	12	Magnesite	0.178	Cd <sub>0.60</sub> Mg <sub>5.40</sub> C <sub>6.00</sub> O <sub>18.00</sub>
96-900-0637	11	Monticellite	0.134	Mg <sub>3.72</sub> Fe <sub>0.28</sub> Ca <sub>4.00</sub> Si <sub>4.00</sub> O <sub>16.00</sub>
96-900-2230	0	Clinochlore	11.732	Mg <sub>2.97</sub> Al <sub>0.99</sub> Si <sub>3.04</sub> O <sub>12.00</sub> I <sub>12.00</sub>
96-900-0465	14	Sinnerite	0.348	As <sub>8.00</sub> Cu <sub>12.00</sub> S <sub>18.00</sub>
96-100-8717	12	Zirconium chromium copper nickel deuteride	0.061	Zr <sub>4.00</sub> Cr <sub>4.80</sub> Ni <sub>1.60</sub> Cu <sub>1.60</sub> D <sub>14.04</sub>

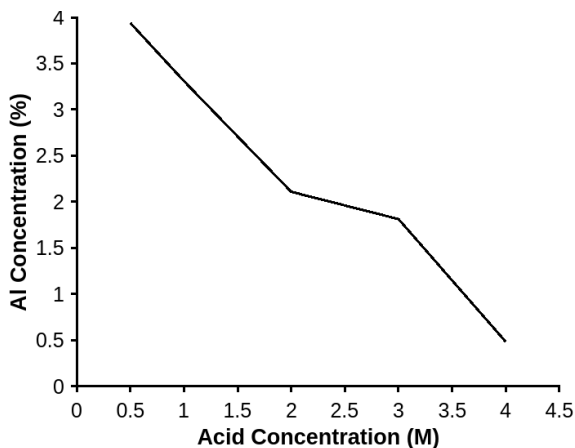
### 3. RESULTS AND DISCUSSION

#### 3.1. Leaching Metals With Sulfuric Acid

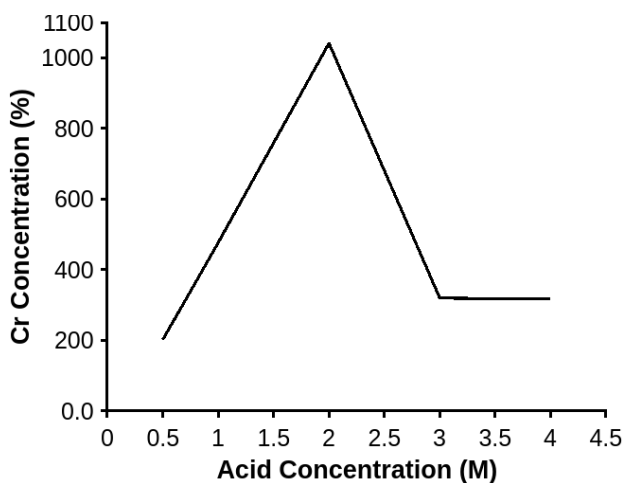
In our experimental study, we examined the interaction between sulfuric acid concentration H<sub>2</sub>SO<sub>4</sub> (M) and aluminum concentration dissolved (%). As shown in Figure 2 if we gradually increase the H<sub>2</sub>SO<sub>4</sub> concentration, we observed a steady decrease in the aluminum content in the solution. At an initial H<sub>2</sub>SO<sub>4</sub> concentration of 0,5M, the Al extraction rate was relatively high. However, as the acid concentration increases, the Al content shows a significant decrease. When the acid reaches 4 M, the Al concentration has dropped significantly to only 0.484%. This downward indicates that sulfuric acid is effective in extracting aluminum, as evidenced by the inverse relationship between acid concentration and Al concentration. Our results are consistent with previous studies in this area, which have consistently shown that increasing the molar concentration of sulfuric acid results in a decrease in the aluminum content in the solution. This phenomenon is well documented in studies of the leaching of aluminum and other metals from various matrices. For example, Wang P et al (Wang P et al., 2021) in their study of extraction of aluminum from Coal Fly Ash observed further increase in H<sub>2</sub>SO<sub>4</sub> concentration did not result in significant or substantial increases in Al extraction.

As shown in the Figure 3, at an initial sulfuric acid concentration of 0.5 M, the chromium concentration was 201.9%. However, when the sulfuric acid concentration was increased to 1 M, the value increased significantly to 476.4%. This increase was not consistent, as at 2 M of sulfuric acid, the chromium concentration reached 1041%, indicating a significant increase. However, when we switched to 3 M the chromium concentration dropped to 319.8%. This decrease continued with 4 M.

These results highlight the sensitivity of the chromium leaching process to sulfuric acid concentration. They highlighted a critical optimization point where a specific concentration of sulfuric acid promotes the maximum chromium extraction. Beyond this point, higher acid concentrations do not necessarily result in more efficient chromium extraction. Previous studies in this field have revealed a complex relationship between acid concentration and chromium leaching efficiency. Our results are consistent with this established knowledge in several aspects. Notably, increasing the sulfuric acid concentration did not improve the chromium concentration in solution, a trend that mirrors observations in previous studies (Silva J et al., 2005).



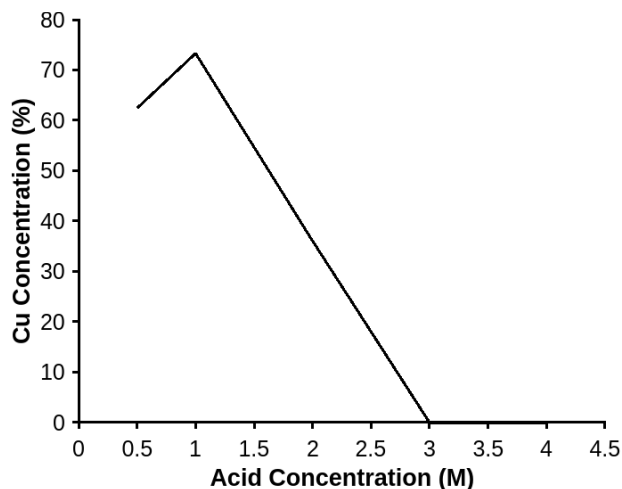
**Figure 2:** Effect of sulfuric acid concentration on the extraction of Al from phosphate solid waste.



**Figure 3:** Effect of sulfuric acid concentration on the extraction of Cr from phosphate solid waste.

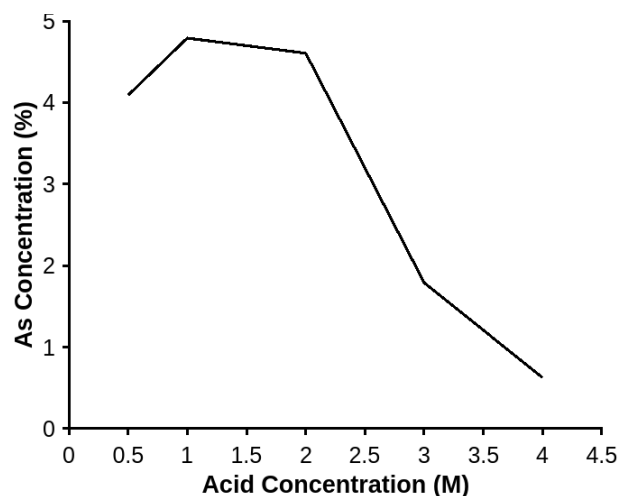
Figure 4 shows the impact of varying sulfuric acid concentration on the leaching efficiency of Cu from our solid phosphate waste sample. The initial increase in acid concentration significantly increased the amount of copper extracted. However, at higher acid concentrations, a decrease in copper leaching efficiency was observed, and leaching finally stopped at 3 M and 4 M sulfuric acid. This observation highlights the dynamic relationship between sulfuric acid concentration and copper leaching efficiency. Our results initially support the assumption that low to moderate acid concentrations promote copper extraction, but also highlight an important threshold. At some point (shown here at 2 M  $H_2SO_4$ ), increasing the acid concentration does not proportionally improve copper leaching. The subsequent complete cessation of leaching at 3 M and 4 M highlights the importance of precise control of acid concentration to optimize copper recovery while minimizing waste. Although our results support previous studies such as those by Zhang Y et al (2019), who emphasized the improvement of copper extraction with increasing acid concentration, they also highlighted the

importance of the optimal range of acid concentration. Beyond this range, further increases provide no additional benefit.



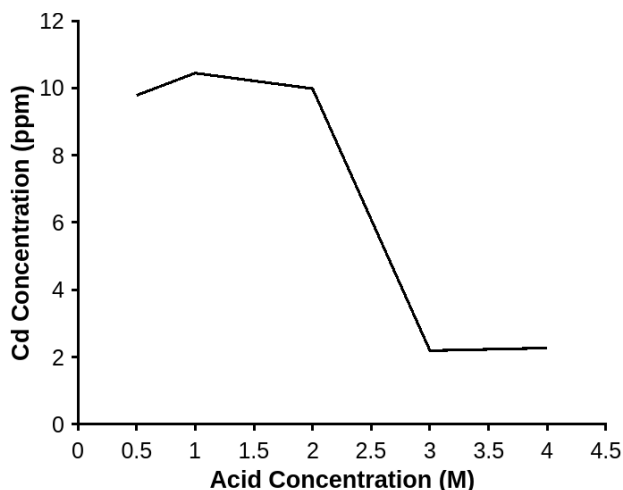
**Figure 4:** Effect of sulfuric acid concentration on the extraction of Cu from phosphate solid waste.

Building on our investigation of the effect of sulfuric acid on metal leaching efficiency, we shifted our focus to leaching of As, another important element presents in solid phosphate waste samples. As shown in Figure 5 at an initial  $H_2SO_4$  concentration of 0.5 M, the arsenic concentration is determined to be 4.089%. As the acid concentration was gradually increased, the amount of arsenic leached continued to increase. This increase persisted at 2 M. However, a significant change was observed at 3 M as the arsenic concentration decreased significantly to 1.791%. This reduction in leached arsenic continues at 4M. The results indicate a critical point, around 3 M of  $H_2SO_4$ , where continued increases in acid concentration do not result in a proportional increase in leached arsenic concentration.



**Figure 5:** Effect of sulfuric acid concentration on the extraction of As from phosphate solid waste.

As can be seen from Figure 6, there is a significant trend in cadmium leaching efficiency as the sulfuric acid concentration changes. The initial increase in acid concentration increased the amount of cadmium extracted. However, as the acid concentration continued to increase, a decrease in yield was observed, highlighting the existence of an optimal acid concentration range for efficient cadmium recovery. These results confirm the findings obtained by Safarzadeh et al (Safarzadeh et al., 2009).



**Figure 6:** Effect of sulfuric acid concentration on the extraction of Cd from phosphate solid waste.

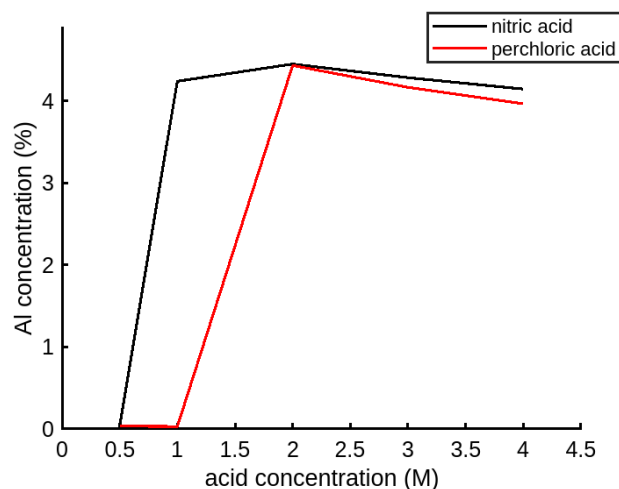
In conclusion, based on all these results, we studied the leaching process of several elements, namely Al, Cr, As, Cu and Cd, using different concentrations of sulfuric acid  $H_2SO_4$ . Our results show significant trends for each element. For aluminum, we observed a gradual decrease in concentration with increasing  $H_2SO_4$  concentration, highlighting the importance of acid optimization. The concentration of chromium Cr increases up to a certain point, and then decreases again, indicating that there is an optimal range. As exhibits a similar reaction to chromium, with significantly lower concentrations at higher acid levels. In contrast, the Cu concentration increased with increasing  $H_2SO_4$  concentration, which is consistent with the trend observed in other studies. Finally, the concentration of Cd initially increases and then gradually decreases at higher acid levels. These results highlight the importance of precise control of sulfuric acid concentration during the leaching process of these elements, especially for optimizing their recovery and waste reduction.

### 3.2. Leaching Metals with Perchloric Acid and Nitric Acid

Experimental results (Figure 7) on leaching aluminum varying concentrations of nitric acid and perchloric acid provided valuable insights into the leaching efficiency of these two acids. Remarkably, the results show a marked contrast in their performance. Nitric acid shows significant

advantages in the extracting aluminum especially as the acid concentration increases. At a low concentration of 0.5 M, the aluminum concentration extracted with nitric acid was only 0.039%, indicating limited effectiveness under mild conditions. However, when the nitric acid concentration increased to 1 M, the aluminum extraction efficiency increased significantly to 4.241%, indicating a significant improvement in leaching performance. This trend continues as the concentration continues to increase. In contrast, the efficiency of perchloric acid in extracting aluminum was relatively low, and at most concentrations, the extraction rate is significantly lower than that of nitric acid. Even at 2 M concentration, where nitric acid shows significant efficiency, perchloric acid can only achieve a concentration of 4.434%. As the concentration increases, the difference between the two acids becomes more pronounced, with perchloric acid concentration 4 M reaching 3.966%.

The trend of higher aluminum extraction efficiency with increasing nitric acid concentration is consistent with the literature. The study by Pepper R et al (2016) showed that higher concentrations of nitric acid led to an increased aluminum dissolution, which is consistent with our experimental results. Compared to nitric acid, perchloric acid is used less frequently in aluminum leaching, and its efficiency tends to be less.

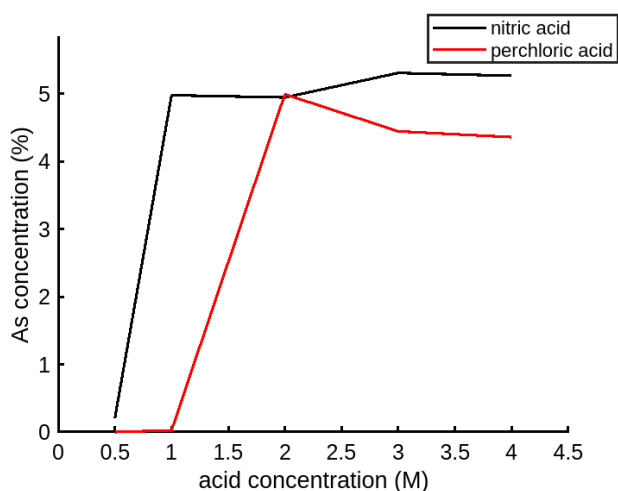


**Figure 7:** Effect of nitric acid and perchloric acid concentration in the extraction of Aluminum from phosphate solid waste.

As shown in the Figure (8), the results of As leaching experiments using varying concentrations of nitric acid and perchloric acid provide valuable insights into the efficiency of these acids in arsenic extraction. Nitric acid shows clear advantages in arsenic extraction, especially as the acid concentration increases. At a low concentration of 0.5 M only a small amount of arsenic was extracted with nitric acid. However, as the nitric acid concentration increases to 1 M, the extraction rate of arsenic increases significantly, reaching a

concentration of 4.978%. This increase continues as the concentration continues to rise. In contrast, perchloric acid showed limited arsenic leaching efficiency at lower concentrations, with negligible arsenic extraction at 0.5 M. At a concentration of 1 M, a small amount of arsenic was extracted. However, at higher concentrations, the arsenic extraction yield by perchloric acid improved. Despite this improvement, the efficiency of perchloric acid has always lagged behind that of nitric acid.

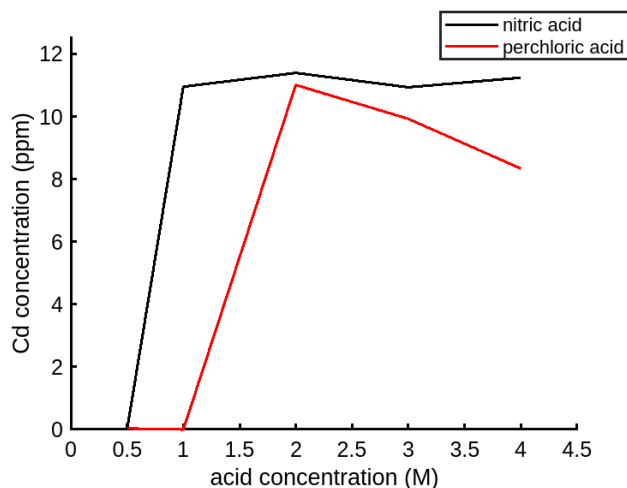
Compared with previous studies by Ling H et al (2022), our study showed a similar trend in increasing arsenic concentration with increasing nitric acid concentration. Notably, Ling H et al achieved these results while conducting experiments at a higher temperature of 60°C, which requires the expenditure of energy. In contrast, our study has a clear advantage because it achieves equivalent arsenic extraction efficiency without increasing the temperature. This represents a potentially more energy efficient and practical method for arsenic leaching. Our approach is not only consistent with existing literature, but also introduces an innovative aspect that reduces reliance on high temperatures, thereby providing potential cost and sustainability benefits.



**Figure 8:** Effect of nitric acid and perchloric acid concentration in the extraction of As from phosphate solid waste.

For Cd, Figure 9 shows that nitric acid is an efficient leaching agent, especially as the acid concentration increases. This trend is consistent with previous research conducted by Saleh M et al (2021) (32). At a low concentration of 0.5 M, no cadmium was extracted. However, as the nitric acid concentration increased to 1 M, the cadmium extraction yield increased significantly to 10.95 ppm, highlighting the improvement in leaching performance. This trend continues to increase. On the other hand, perchloric acid showed relatively low cadmium leaching efficiency at low concentrations and extraction results were

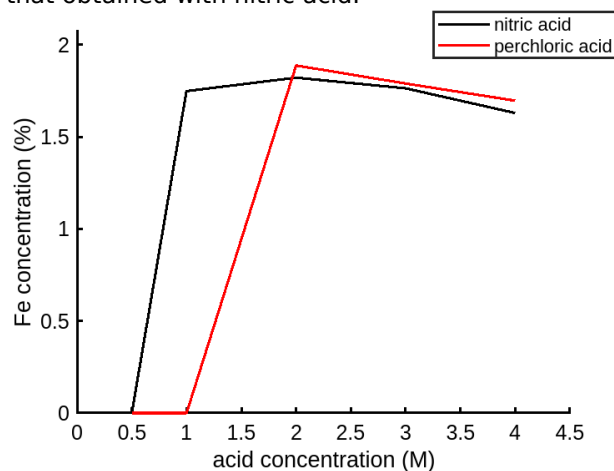
moderate. However, as the perchloric acid concentration increased, an improvement in cadmium extraction was observed. It is important to note that the effectiveness of perchloric acid still lower than that obtained with nitric acid at higher concentrations.



**Figure 9:** Effect of nitric acid and perchloric acid concentration in the extraction of Cadmium from phosphate solid waste.

As we can see in Figure 10, for Fe leaching, nitric acid exhibited a limited efficiency in extraction, as evidenced by the low extraction yield at different concentrations. The lack of significant iron extraction at a concentration of 0.5 M is consistent with the difficult nature of iron leaching, especially in the presence of nitric acid. The small amount of iron extracted, even at higher concentrations, suggests that nitric acid may not be the ideal choice for iron leaching in this context.

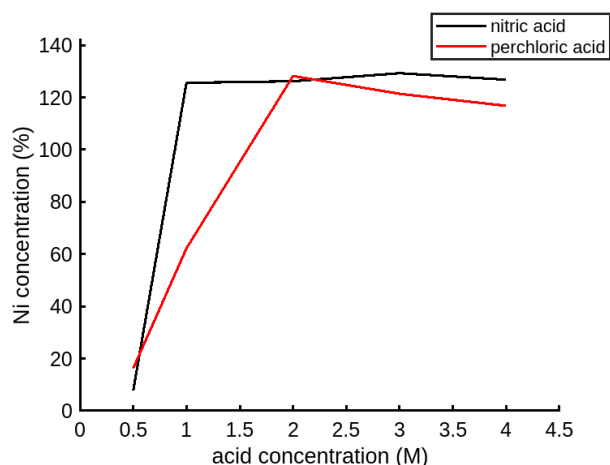
In contrast, perchloric acid exhibited slightly improved iron separation efficiency, especially at higher concentrations. The extraction efficiency with perchloric acid was significantly better than that obtained with nitric acid.



**Figure 10:** Effect of nitric acid and perchloric acid concentration in the extraction of iron from phosphate solid waste.



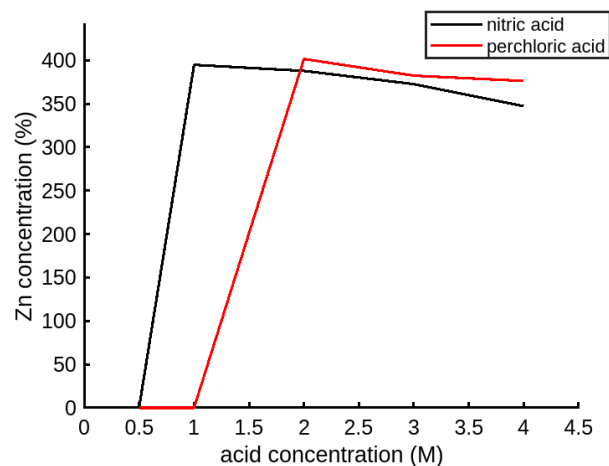
For Ni leaching results (Figure 11) show that both nitric acid and perchloric acid are effective agents for nickel extraction, each with distinct effectiveness trends. Nitric acid has significant ability to dissolve nickel, and this efficiency increases with increasing acid concentration. At a concentration of 1 M, the nickel extraction yield increased significantly to 125.6%, highlighting the effectiveness of nitric acid leaching nickel from samples. The trend continues to improve and reaches a pick at a concentration of 3 M. These results highlight the suitability of nitric acid for nickel leaching, especially at higher acid concentration, without the need for additional factors such as high temperatures. On the other hand, perchloric acid also proved to be an effective nickel leaching agent, with high leaching efficiency observed at concentrations 2 M and 3 M. Significant extraction yields of 128.2% and 121.4% were obtained at these concentrations, respectively, highlighting the effectiveness of nickel perchlorate leaching in this specific experimental setting. These results indicate that perchloric acid, especially at the concentrations mentioned above, can be a valuable alternative for nickel extraction and provide flexibility in the leaching process.



**Figure 11:** Effect of nitric acid and perchloric acid concentration in the extraction of Ni from phosphate solid waste.

As shown in Figure 12, Zn leaching experiment results showed that nitric acid could effectively leach zinc at a lower concentration, when the concentration is 1 M, the extraction rate is significantly reaching 394.9%. However, it is worth noting that the zinc extraction efficiency decreases with increasing nitric acid concentration, as shown by the decreasing concentrations of 2 M, 3 M and 4 M. This pattern suggests that moderate concentrations of nitric acid may be most effective

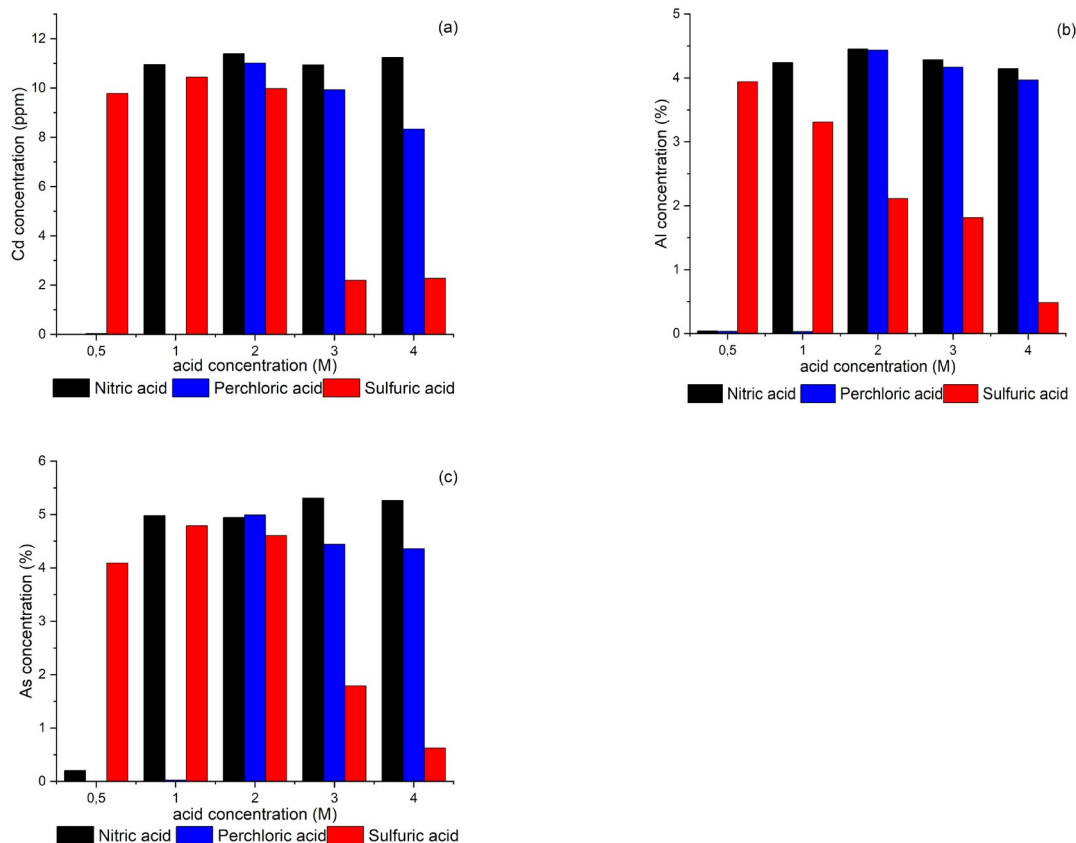
for zinc leaching. In contrast, perchloric acid showed relatively stable and higher efficiency in zinc extraction, especially at higher concentrations (2 M, 3 M and 4 M). The extraction yields remained very constant. This suggests that perchloric acid may be a suitable choice for zinc leaching, especially when higher concentrations are required.



**Figure 12:** Effect of nitric acid and perchloric acid concentration in the extraction of Zinc from phosphate solid waste.

### 3.3. Comparison Between Different Acids

A comprehensive comparison of the leaching of cadmium Cd, aluminum Al and arsenic As from solid phosphate waste using sulfuric acid, nitric acid and perchloric acid revealed complex patterns. For cadmium leaching (Figure 13 (a)), sulfuric acid consistently showed relatively low efficiency at all concentrations, while nitric acid showed a significant increase in efficiency, peaking at concentration 2, and perchloric acid showed competitive efficiency at the same concentration. In aluminum leaching (Figure 13 (b)), nitric acid has proven to be the most effective leaching agent, with the leaching efficiency increasing significantly as the concentration increases, while perchloric acid maintains a relatively stable leaching rate. However, as the concentration increases, the effectiveness of sulfuric acid gradually decreases. For the leaching of arsenic (Figure 13 (c)), sulfuric acid showed constant efficiency at concentration 1 M, while nitric acid showed a significant increase and reached a maximum at concentration 3 M, and perchloric acid showed efficient extraction, especially at concentration 2 M. The different trends between the three elements emphasize the different interactions of acid selection and concentration in optimizing the leaching process. Nitric acid is consistently an effective leaching agent, while perchloric acid is competitively efficient.



**Figure 13:** Comparison of the extraction efficiency of Cadmium (a), Aluminum (b) and Arsenic (c) with nitric acid, sulfuric acid and perchloric acid.

### 3. CONCLUSION

Recovering metals from solid phosphate waste presents multifaceted challenges, and the selection of a suitable leachate plays a vital role in effective recovery. In this study, the effectiveness of sulfuric acid, nitric acid and perchloric acid in dissolving Al, Cd, As, Cu, Cr, Fe, Ni and Zn in waste samples was examined. An important aspect of this study was to perform experiments at ambient temperature conditions, consciously varying acid concentrations (0.5 M, 1 M, 2 M, 3 M and 4 M). Additionally, the particle size of the samples was standardized to 160  $\mu\text{m}$  to ensure uniform leaching. The stirring time was maintained at one hour, which helped control the experimental conditions.

Nitric acid has consistently proven to be an effective leaching agent, with significantly improved efficiency for all elements, especially at higher concentrations. Although perchloric acid is not more efficient than nitric acid, its performance is competitive and positions it as a viable alternative. Sulfuric acid, on the other hand, showed a different trend, highlighting the importance of developing a customized approach based on the specific properties of the element.

In summary, this study provides valuable insights into the optimized use of sulfuric acid, nitric acid and perchloric acid for the extraction of metals from solid phosphate waste. The findings not only expand our understanding of leaching kinetics but also provide a solid foundation for the development of sustainable and efficient extraction methods. These findings are expected to impact waste management practices and provide potential solutions to the challenges posed by metal leaching from complex waste matrices.

### 4. CONFLICT OF INTEREST

The authors have no conflicts of interest to declare.

### 5. ACKNOWLEDGMENTS

Not applicable.

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