

## CO<sub>2</sub>-Mineralization Insights from Non-Ferrous Metallurgy Slags Copper and Lead Slags' Behavior during CO<sub>2</sub> (G)-Induced Dissolution

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### Abstract

Since dissolution is typically regarded as a key rate-limiting step during CO<sub>2</sub>-mineralization, the prospect of using non-ferrous slags for CO<sub>2</sub>-mineralization is investigated in this study by examining their dissolving behaviours in CO<sub>2</sub>-environments. At combinations of two temperatures (30 and 60 °C) and two CO<sub>2</sub>-pressures (1 and 10 barg), dissolution of two copper slags and a lead slag is studied at the liquid to solid ratio (w/w) of 1000 over time (30, 60, 120, and 240 min). The lead slag exhibits Fe-dissolution of up to 10% and the copper slags up to 5–6% within four hours among the systems in which the slags are dissolved in CO<sub>2</sub>-environments[1-15]. Nearly majority of the observations had a solution pH between 4 and 5. The slags dissolve at rates that are following the dissolution that occurs during the first 30 to 60 minutes, systems with higher temperatures and higher CO<sub>2</sub> pressures also exhibit greater (or comparable and solution-pH values. Additionally, the systems with higher temperatures and CO<sub>2</sub> pressure continue to show faster dissolving rates throughout the trial even if they initially showed higher solution pH after 30 to 60 minutes of dissolution. They are suggested as prospective sources for CO<sub>2</sub>-mineralization since residues like non-ferrous copper and lead slags are easily accessible in contrast to equivalent natural minerals (such olivines), which often need to be pre-processed before carbonation.

### Introduction

One of the main methods for achieving the long-term CO<sub>2</sub> concentration targets is thought to be CO<sub>2</sub>-mineralization. As a result, alkaline sources that can be utilised to mineralize CO<sub>2</sub> include both natural minerals and industrial waste. Although frequently mentioned in tandem, it is typically stated that industrial wastes have substantially larger carbonation reactivities than their natural analogues. However, it should be noted that the majority of studies that have led to these conclusions have concentrated on the direct aqueous carbonation of relatively reactive alkaline sources like ferrous metallurgy slags, byproducts of cement production, and concrete that has reached the end of its useful life. Items, leftover ashes, etc. Such generalizations, while possibly true for a particular set of sources, do not apply to all industrial leftovers. Copper and lead slags in particular have not attracted much attention as CO<sub>2</sub>-mineralization sources. Their low reactivities, which were also shown in the outcomes of attempts to use them as supplemental cementation materials can be attributable to this lack of interest. Therefore, three non-ferrous metallurgical slags (NFMS)—two from copper production and one from lead production—are explored for CO<sub>2</sub>-induced dissolving in this work as a first step toward expanding the field of mineral carbonation to NFMS. In systems with dissolved CO<sub>2</sub> (without additions) and parent minerals, CO<sub>2</sub>-sequestration by direct aqueous carbonation typically entails two basic steps: dissolving parent minerals to release cations (Ca/Mg/Fe) and precipitating these cations as low-solubility carbonates. Dissolution (Reaction 1) during the carbonation of the majority of natural minerals is typically regarded as the rate-limiting process. To this purpose, it is thought that the acidic species produced by CO<sub>2</sub>-dissolution (H<sup>+</sup>/H<sub>2</sub>CO<sub>3</sub>/HCO<sub>3</sub><sup>-</sup>) are crucial for Reaction 1. Regarding the direct aqueous carbonation of industrial wastes, the function of these acidic species hasn't gotten much consideration. It appears that one of the primary causes of this lack of interest is that even the hydrolytic disintegration of the major species in such pH ranges (Bjerrum plot) when it comes to the direct aqueous carbonation of alkaline residues, Reaction 1 has received little attention; however, because NFMS are thought to have low reactivities, Reaction 1's effectiveness has to be thoroughly examined. This is the primary goal of the effort.

### Subjective Heading

Numerous research have examined how NFMS dissolves as a function of pH, however not from the standpoint of rapid carbonation. Although the majority of these research have concentrated on the behaviour of Si dissolving from parent minerals, it has been amply demonstrated that slag dissolution generally increases in a slightly acidic environment. These pH ranges are anticipated for the CO<sub>2</sub>-H<sub>2</sub>O solutions used in this study to study NFMS dissolution. From a different angle, the increase in solution pH due to mineral dissolution via Reaction 1 is given major attention in the studies focusing on accelerated weathering and ocean alkalinity enhancement employing natural minerals. Clear from such research that, in a closed system, the pH of the solution may rise with an increase in the degree of mineral dissolution, and that the resulting change in solution composition may also affect the reaction processes. Therefore, the impact of such an evolution of the solution composition with mineral-dissolution must be taken into account when researching the carbonation of industrial leftovers. Since the systems with the largest CO<sub>2</sub>-solubilities are anticipated to have the highest dissolution rates and thus maximum degrees of the evolution of the solutions, if this is overlooked it may result in contradicting observations. As a result, this study also covers how the composition of the solution changes as the dissolution progresses.

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## Discussion

With combined availabilities of > 80 Mt/y, copper and lead-slags can be voluminous sources for CO<sub>2</sub> mineralization. Presently, if at all utilized, they are used for low-value applications such as aggregates for concrete production or civil works, for grit blasting, and for ceramic production. While the utilization pattern of lead slag is hard to find, according to an estimate of the EU utilization pattern of copper slags provided by the Copper Institute (personal communication, 2020) 58% is used as aggregate, roads, and filling, ~29% for abrasive blasting, 4% as aggregate for concrete, and 8% as raw material and SCM in cement. Only 15-20 percent of the copper slags are used, and the remainder is dumped, according to another rough estimate in the FISSAC H2020 project. Additionally, numerous attempts have been undertaken to extract multiple target metals present in these slags using various pyrometallurgical, hydrometallurgical, and beneficiation. The bulk byproducts of such processes can still be used for high-volume applications like CO<sub>2</sub>-mineralization and construction materials, and it should be highlighted that such target metals do not make up more than 10% of the overall slag. Most recent attempts to make use of them functionally have centred on chemical or mechanical activation to make use of their silicate component in the creation of alkali-activated binder. Additionally, their potential use in Ordinary Portland Cement (OPC) as Supplementary Cementitious Materials (SCM) is being examined. Recent attempts to examine the dissolving of copper slags were concentrated on Si-dissolution at a very alkaline pH suitable to such systems since such applications depend on the Si-dissolution from slags in alkaline environments to generate Si-based binders. But when minerals dissolve in an acidic environment, incongruent dissolution causes the elements Ca, Mg, and Fe to dissolve preferentially, leaving behind a Si-rich residue. Recently, the adoption of such a technique has also been suggested. When the carbonated slags are utilised as SCM, CO<sub>2</sub> acts as an activator for steel/stainless steel slags so that both the freshly created calcite and the leached Si-rich layers can take part in the cement hydration reaction. The essential knowledge gained from this work will also be useful in the study of CO<sub>2</sub>-induced activation of fayalitic copper and lead slags such that both the newly created leached Si-rich layer and Fe-rich carbonates can take part in cementation reaction.

The Ca-rich carbonates that precipitate during the rapid carbonation of slags can be advantageously employed as a carbonate cement in the case of Ca-rich residues (for example, steel slags). Recently, rapid carbonation of Fe(O) has also been used to create Fe-rich carbonate binders. In a similar vein, CO<sub>2</sub>-mineralization of the Fe and Ca found in NFMS may provide a practical path to its application in Fe, Ca-rich carbonate. The long-term goal of this study is to support the use of NFMS in this way. Because of their poor hydrolytic solubilities, the inability of dissolved CO<sub>2</sub> to induce their solubility, and the need to comprehend the cementing properties of Fe-carbonates, should they occur in the future, there is currently little interest in this area.

This study aims to gain a better knowledge of the CO<sub>2</sub>-induced dissolution of NFMS as a first step towards their usage for CO<sub>2</sub>-mineralization. The dissolution of NFMS is typically thought to be the rate-limiting phase in this. In order to do this, it must be remembered that recent research has demonstrated both the capacity of dissolved CO<sub>2</sub> to dissolve an insoluble source such as Fe as well as the ability of Fe-carbonates to cement materials. Additionally, it is important to note that there have been a few investigations on the dissolution behaviour of naturally occurring fayalite in CO<sub>2</sub>-environments because fayalite (the crystalline form of Fe<sub>2</sub>SiO<sub>4</sub>) is thought to be a significant component of NFMS. Consequently, it is anticipated that the carbonation.

In this work, slags from three distinct sources are examined. Two slags from two distinct copper production techniques (NFMS-2 and NFMS-3) as well as one slag from secondary lead production (NFMS-1) are examined. A thorough pre-processing procedure was used to ensure the homogeneity of observations across various platforms. A Retsch planetary ball mill with Tungsten carbide jars and balls was used to grind all of the as-received slags until the entire powder sample passed after being wet-sieved with isopropanol, passing through a 63 μm sieve. The sieved powder was further ultrasonically treated in ethanol for five minutes to eliminate any ultrafine particles that were present as agglomerates. The ultrasonicated solution was then given a 2-minute rest period before being decanted. The supernatant was repeatedly decanted and subjected to ultra-sonication until it appeared clear. The bottom residue was vacuum filtered using a Whatman 589/3 filter and a Buchner flask after the final decantation and rinsed with distilled water. When the residue reached a steady weight, it was dried in an oven at 40 °C. was reached. The dried residue was used for the dissolution experiments.

The oxide and elemental compositions of the slags were determined by ED-XRF (Energy dispersive X-ray fluorescence)<sup>1</sup> using Spectro XEPOS HE model XEP03. The samples were prepared as melts using Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> as flux. The oxide compositions and the corresponding elemental compositions of the respective slags are shown. In the second from last column of the 'average ± standard-deviation' values of the oxides reported based on the review of more than 160 publications on copper slags is provided for comparison with the literature. Furthermore, in the last column of the table, the ranges of the elemental compositions of the secondary lead slags based on another review are provided. The table shows that the copper slags examined in the literature are fairly well represented by NFMS-2 and NFMS-3. The oxide compositions of NFMS-1 are also within the typical ranges noted in the literature, despite the fact that the composition of secondary lead slags can be highly varied. From the table, it is possible to see NFMS-1's unique characteristics in comparison to the other slags: it has a 10% lower Fe-oxide content, a 10% higher Ca-oxide content, and a 6% lower Si-oxide content. XRD was used to determine the mineral compositions of the powdered slags (63 μm) (PANalytical Empyrean diffractometer, Cobalt anode). To do this, the powdered slags were put into the instrument and back-loaded into the sample holders. The data were then gathered throughout a 5-120° 2θ angular range with a total measurement period of 30 min. To enhance particle statistics, the samples were rotated around the vertical goniometer axis while being measured. X'Pert Highscore Plus v 4.7a software was used to analyse the collected data using Rietveld and phase identification (Analytical). The amorphous contents were tested and quantified independently using a rutile external standard. Utilizing the oxide compositions that were examined by XRF for analysis mass absorption coefficients were computed to determine the amount of amorphous content. The supplemental data allows for observation of the slags' XRD patterns (S2). A laser particle size analyzer (Horiba LA-350) was used to measure the particle size distribution in an isopropanol solution. 200–400 mg of the powder sample were dissolved in 20 mL of isopropanol and fed into the device after being ultrasonically mixed for two minutes. The gadget takes three readings and then computes the average. The cumulative volume distribution as a function of particle size as seen in the mass distributions in NFMS-1 and NFMS-3 are primarily bimodal, in contrast to NFMS-2, which displays a normal distribution of mass. This should be taken into consideration in particular when normalising the slags' dissolving rates depending on their unique surface. The liquid samples were taken during the experiment at 30, 60, 120, and 240 minutes. A tap on the instrument enables sampling without pausing

the experiment. The samples of liquid were obtained by allowing the solution to flow through the sampling tap into a 20 mL plastic syringe that was fitted with a disposable syringe filter (Chromafil AO-45/25, Macherey-Nagel, pore diameter of 0.45 μm), which was then used to filter the solution. For each sampling time, approximately 14 mL of the solution was filtered in this manner. The remaining 4 mL of the filtered solution was used to measure the pH of the solutions, and about 10 mL of the solution was preserved for elemental analysis using ICP-AES (Inductively coupled plasma atomic emission spectroscopy).

The samples were supplemented with 0.5 mL of HNO<sub>3</sub> (Fisher Scientific, optima grade, 67-69 percent assay to preserve them for ICP-AES. A sample that had been diluted 100 times, ten times, and not at all were measured for the analysis. Utilizing PerkinElmer Avio 500 equipment, ICP-AES was carried out. After the liquid sample was extracted from the reactor and filtered, the pH of the filtered liquid samples was assessed using a Mettler Toledo Seven multi dual metre. Temperature correction was applied to the measured pH values. Every day, the equipment was calibrated using pH 4.01, 7.00, and 9.21 commercial buffer solutions. As an additional check, the pH of a buffer solution with a pH of 11 was measured.

## Conclusion

From the standpoint of rapid carbonation, the dissolving behaviour of NFMS-1 is very intriguing. The primary components of NFMS-1 are Fe (24.6%), Ca (10.1%), and Si (11.2%), according to its elemental makeup. As a result, this section goes into great length about the dissolution behaviours of these elements. The concentrations of Fe, Ca, and Si in the solutions are depicted in as functions of temperatures, CO<sub>2</sub> pressures, and durations the figures show that Ca, Fe, and Si hardly ever dissolve in N<sub>2</sub>-H<sub>2</sub>O environments, whereas the addition of dissolved CO<sub>2</sub> is shown to have significantly increased their dissolution rates. This increase can be mostly ascribed to the pH of water dropping when CO<sub>2</sub> was introduced. The pH of each CO<sub>2</sub>-H<sub>2</sub>O system is as follows, according to the modeling of the CO<sub>2</sub>-H<sub>2</sub>O systems under the reaction circumstances examined in this work in the next subsections, the effects of temperature, CO<sub>2</sub>-pressure, and duration are each covered in detail.

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## Conflict of Interest

The authors declare that they are no conflict of interest

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