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Purification of slag-derived leachate and selective carbonation for high-quality precipitated calcium carbonate synthesis

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ABSTRACT

Much research has been performed to synthesize precipitated calcium carbonate (PCC) from calcium extracted from blast furnace (BF) slag. A significant challenge has been obtaining a chemically pure sample with a homogeneous crystal structure and narrow particle size distribution, which can have profitable marketability (e.g. in the papermaking industry). The aim of this study was to investigate the influence of alternative and combinatory processing strategies on the chemical, morphological and mineralogical properties of BF slag-derived PCC. In a first phase, the physicochemical removal of impurities from the leachate, prior to the carbonation, was investigated, by means of temperature reduction (20 → 1 °C) and pH elevation (4.4 → 8.4); this led to selective precipitation of Al, Mg and Si. In a second phase, the operational conditions of the carbonation step were optimized, by lessening the severity of the operational conditions (90 → 30 °C and 6 → 2 bar, CO₂) and by reducing the amount of acid-neutralizing base (1.0 → 0.85 M, Na⁺/M, CH₃COO⁻) added during the carbonation process; this led to more selective calcium carbonation. Using these strategies, it was possible to synthesize chemically pure PCC (>98% Ca) with uniform morphology (scaleno-hedral), homogeneous mineralogy (>88% calcite) and narrow (1.09 uniformity), small ($D_{50} = 1.1 \mu\text{m}$) particle size distribution.

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1. Introduction

Increased carbon dioxide emissions give rise to elevated concentrations in the atmosphere, which has a possible long-term negative effect on the global climate. The five industrial sectors with the highest carbon dioxide emissions are: (1) the power industry; (2) cement production; (3) refineries; (4) iron and steel industry and (5) petrochemical industry (IPCC, 2005). Currently, the potential for reuse of the emitted carbon dioxide is low. Therefore, more focus has to be placed on long-term

capture and storage of emitted carbon dioxide, namely carbon capture and storage (CCS). A possible CCS route is mineral carbonation: chemically reacting carbon dioxide with calcium or magnesium containing materials, producing calcium- or magnesium carbonates (Seifritz, 1990).

In recent years, research has focused on employing waste residues from the iron and steel industry, rather than natural minerals, as source materials for the mineral carbonation, since these waste residues are inexpensive (or even have a negative price), are readily available, and are often produced

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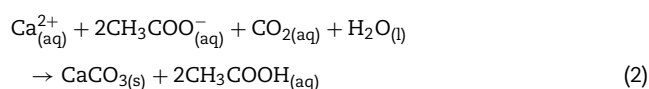
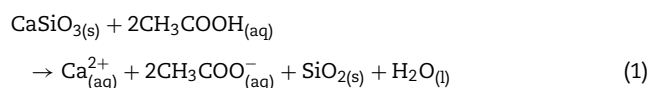
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near large CO₂ sources (Eloneva et al., 2008). Two main types of mineral carbonation exist: direct and indirect carbonation. In the former, the source material is directly carbonated, while in the latter, the calcium or magnesium is first extracted (e.g. using an organic lixiviant such as acetic acid or ammonium acetate) and subsequently carbonated. This results in post-carbonation precipitates with a higher purity and thus increasing the economic value, compared to direct carbonation (Eloneva et al., 2008). Moreover, special attention has been given to waste materials with a high calcium content, mainly blast furnace (BF) slag and basic oxygen furnace (BOF) slag (Eloneva et al., 2008; Teir et al., 2007; Bao and Li, 2012; Said et al., 2013; Chiang et al., 2014). The choice for calcium-rich materials stems from the fact that precipitated calcium carbonate (PCC) has a larger market demand than magnesium carbonates (Teir et al., 2007).

Kakizawa et al. (2001) proposed a reaction scheme (see Eqs. (1) and (2)) to indirectly synthesize PCC from calcium present in wollastonite (CaSiO₃). Teir et al. (2007) stated that, in this reaction scheme, wollastonite may be replaced by BF slag, which consists of mainly calcium silicate minerals. Subsequently, Eloneva et al. (2008) experimentally synthesized non-commercial grade PCC starting from BF slag, based on the proposed reaction scheme. In a first step, the calcium was leached out from the BF slag with an acetic acid solution. Next, the leachate was separated from the post-extraction BF slag and subsequently carbonated.



During the carbonation step, an acid neutralizing agent was added to induce precipitation of calcium carbonate crystals; sodium hydroxide was selected. The PCC synthesized by Eloneva et al. (2008), however, was not commercial grade; the calcium content of the post-carbonation precipitate was only between 63 and 89 wt%, as small amounts of aluminum, magnesium and silicon present in the initial BF slag also ended up in the post-carbonation precipitate. Furthermore, calcite, aragonite and calcite-magnesian phases were detected in the PCC sample, making for a mineralogically and morphologically heterogeneous product.

Chiang et al. (2014) attempted to optimize the extraction step by decreasing the acetic acid concentration, which led to a more selective leaching of calcium from the BF slag. However, concurrently, the overall calcium extraction extent became smaller. To balance the extent and selectivity of the calcium extraction, Chiang et al. (2014) proposed a two-step leaching process. This by-passed the negative effect of high acidity on the selectivity while still maintaining a sufficient calcium extraction yield. Still, after carbonation of the leachates, the calcium concentration of the post-carbonation precipitates remained in the range of 69–90 wt%, and different mineralogical phases were also present. Based on these past studies, it appears that improvement of PCC purity may only be reached by removing the main impurities prior to the carbonation step, for example by selective precipitation (Bao et al., 2014).

The focus of the present work is on the indirect carbonation of calcium originating from BF slag, with the goal to synthesize marketable PCC intended for use as filler in paper products. For this application, the carbonate has to be chemically pure (~98 wt% calcium), should have a homogeneous mineralogical structure, a small average particle size and a narrow size distribution (Gill, 2005). The approach was to investigate the influence of alternative and combinatory processing strategies on the chemical, morphological and mineralogical properties of BF slag-derived PCC, in view of generating a high-quality product. The research focus was divided in three stages:

- the separation of the leachate from the post-extraction BF slag, targeting the removal of nearly all suspended particles (mainly silica) and residual BF slag prior to the carbonation of the leachate;
- investigation towards the physico-chemical removal of the most prevalent impurities in the leachate, i.e. silicon, aluminum and magnesium, prior to its carbonation; and
- optimization of the carbonation conditions to precipitate PCC with a sufficiently high calcium content, homogeneous crystal structure, small average particle size and narrow size distribution.

To finalize the present study, an optimized process set-up is suggested and experimentally tested.

2. Materials and methods

2.1. Materials

Ground granulated BF slag was obtained from a Belgian industrial steelworks. For the different experiments, the BF slag was manually milled with mortar and pestle. The resulting material had a D_{50} value of 2.1 mm and a uniformity coefficient (U) of 12 ($U = D_{60}/D_{10}$); these values were determined via classic sieving classification. Wavelength dispersive X-ray fluorescence (WDXRF, Panalytical PW2400) was used to determine the chemical composition. The slag was composed of the following oxides: CaO (41.2 wt%), SiO₂ (35.3 wt%), Al₂O₃ (10.1 wt%), MgO (7.0 wt%); metallic components were Ti (0.8 wt%), Fe (0.4 wt%) and Mn (0.3 wt%). X-ray Diffraction (XRD, Philips PW1830) showed the slag to be largely amorphous; amorphicity is caused by the granulation process whereby the slag is rapidly cooled, preventing crystallization.

BF slag was chosen over BOF slag because of the following advantages: (i) higher production output (0.25–0.30 t BF slag per tonne crude iron); (ii) a more consistent mineralogical and chemical composition; and (iii) a higher aluminum content (10–14 wt% Al₂O₃) (Chiang et al., 2014). The last characteristic is important with respect to the feasibility of the two-way valorization of ironmaking slag, as proposed by Chiang et al. (2014). In the proposed process, in addition to producing PCC, the post-extraction ironmaking slag is upgraded to a marketable zeolitic material in order to help off-set the costs of CCS. To enable zeolite synthesis, however, the calcium extraction from the slag must be very extensive; therefore it was also a focus of the present work to maximize calcium extraction from BF slag.

The lixiviant used was analytical grade acetic acid (CH₃COOH), the pH adjusting base used was analytical grade sodium hydroxide (NaOH), the carbon dioxide used

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