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The effect of microstructure on the leaching behaviour of electric arc furnace (EAF) carbon steel slag

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ABSTRACT

Electric arc furnace (EAF) slag could be exploited in several fields of application, such as land filling, road constructions and concrete production, since their physical properties are similar, or even better, than natural materials like gravel. Environmental concerns related to pollutants leaching (Ba, V, Cr, chlorides, fluorides, cyanides, etc.), are the primary hindrance limiting the effective reuse of such material. Thus, chemical and structural stability are fundamental requirements to be fulfilled, especially when the slag comes in contact with water. The slag microstructure has a non-negligible influence on metals concentration in leachate, even if the phases that react with water have not yet been clearly identified. In this study, different classes of carbon steel EAF slag were investigated in order to correlate their leaching behaviour with microstructural and crystallographic features. Fine particles were chosen to enhance the dissolution rate of the phases involved in the leaching process and to quantify the effects of the fine fraction on leachate concentration with respect to the bulk material. Qualitative tests at different liquid-to-solid (L/S) ratios were also performed to detect dissolution of the phases, which would otherwise be non-appreciable in standard conditions. The analyses also allowed for the identification of the phases responsible for toxic metal leaching (Ba, Cr, V), as well as those that were not involved in dissolution phenomena, thereby consolidating the results proposed elsewhere on slag stabilization.

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1. Introduction

The growing interest in the use of steel slag as an alternative to traditional stone materials has prompted researchers to study in detail the chemical, physical and mechanical properties of these industrial by-products. Environmental concerns related to toxic metal leaching, are the primary hindrance limiting the effective reuse of such material, especially for slag from the electric steelmaking route. For this reason, several studies (Lind et al., 2001; Chaurand et al., 2007; van Zomeren et al., 2011; Engelsen et al., 2010, 2012) have investigated the leaching behaviour of slag aggregates, highlighting that such

aggregates could potentially release dangerous chemical species (Ba, V, Cr, chlorides, fluorides, cyanides, etc.). Recently, strong correlations between leaching behaviour and the chemical composition of different classes of EAF slag have been highlighted and important considerations relating to the improvement of pollutant retaining substances were stated, concerning the standard test required by the EN 12457-2 regulation (Mombelli et al., 2014, 2016). Chemical composition plays a fundamental role and provides important indications on the leaching behaviour of a slag, since it determines, with cooling way, the slag microstructure (Tossavainen et al., 2007; Albertsson et al., 2013, 2014). Thus, microstructure has a non-negligible influence on metals

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concentration in leachate. As a consequence, one of the most important issues to solve is the identification of the mineralogical phases responsible for the release of toxic metals. In fact, not all the slag particle surface reacts with water, but only specific phases are involved in hydration and dissolution processes (Engström et al., 2013; Mombelli et al., 2016; Strandkvist et al., 2015). Several studies (Barella et al., 2012; Mombelli et al., 2012; Gelfi et al., 2010; De Windt et al., 2011; Faellman, 2000; Engström et al., 2013; Strandkvist et al., 2015) indicate that calcium-rich crystalline phases (in particular larnite, calcium aluminate and calcium ferrite) are the most probable structural constituents involved in releasing phenomena, but which phases exactly react with water is not clearly identified. Moreover, the conditions under which the leachates are produced vary in relation to different physical parameters like granulometry, temperature or flow conditions, influencing the resulting concentrations of metals in the leachates (Faellman and Hartlén, 1996). Even if the different standard tests are carried out on bulk samples, a certain amount of fine particles is admitted, and these fine fractions negatively influence the test results, i.e. enhancing the leaching (Mizutani et al., 2006; Vítková et al., 2011; Moser and Römbke, 2009). In fact, fine particles can lead to a variation of the pH of the eluate and consequently to a different leachability of pH sensitive constituents. For this reason, the European Standards usually specify that the material should not be finely ground and plan to limit particle size reduction in order to maintain the physical state of the waste. However, no indication about the minimum admitted size and amount of fine particles is indicated in the standards. In addition, since fine particles are usually employed as filler material in cementitious components, the leaching behaviour of such manufactures could be significantly changed by the filler weight fraction. Nevertheless, since the interaction between slag and water was experimentally demonstrated to be only cortical, interesting only the particle surface, the analysis of fine particles could allow to better identify the hydration and dissolution mechanisms that rule the pollutants leaching (Mombelli et al., 2014). Effectively, the increase in the surface-to-volume (S/V) ratio of fine particles, coupled with a high liquid-to-solid (L/S) ratios, favours the identification of dissolved phases. This is because it maintains the pH far from basic values, thus working in conditions a long way from the saturation limit, enhancing the dissolution rate as reported by Nicoleau et al. (2013).

In this study, different classes of carbon steel EAF slag powder originating from different steel productions (reinforced bar steel, high alloyed steel and quality steel) were investigated, in order to correlate their leaching behaviour with microstructural and crystallographic features. Fine particles were chosen to enhance the dissolution rate of the phases involved in the leaching process and to quantify the effects of the fine fraction on leachate concentration with respect to the bulk material. Moreover, qualitative tests at different L/S ratios were also performed to detect the dissolution of the phases, otherwise non-appreciable in standard conditions. The main goal of the present work is to identify which crystalline phases are responsible for the toxic metal leaching (Ba, Cr, V). The analyses have also allowed for the identification of the phases not involved in dissolution phenomena, thereby consolidating the results proposed elsewhere on slag stabilization by Mombelli et al. (2014; 2016). The results discussed in the present paper show quantitatively the relationship between leaching and the fraction of some mineral phases considered as key factors controlling the leaching behaviour of EAF slag.

2. Experimental procedure

2.1. Material

EAF slag were provided by different Italian and European electric steelworks and associated to different steel productions: reinforcing bar steel (group A, 7 samples), high-alloyed steel (group B, 4 samples) and quality steels (group C, 4 samples).

The investigated samples represent a selected subset from a larger sampling, previously analyzed by the same authors (Mombelli et al., 2016).

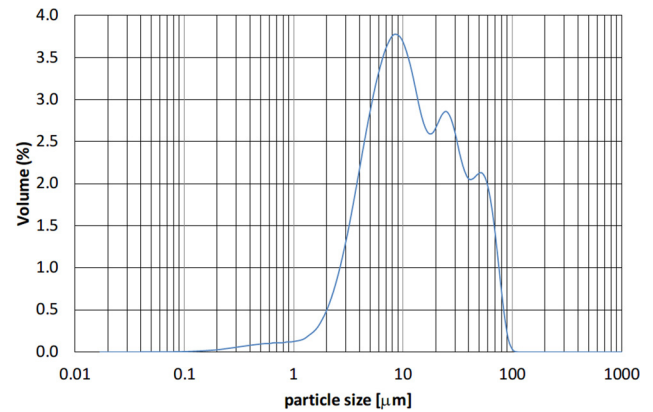


Fig. 1 – Particle size distribution of investigated slag powder.

The slag chemical composition was provided by ED-XRF analysis by means of Ametek Spectro Xepos spectrometer in He atmosphere on 5 g of powdered slag (Table 1).

The samples, in the form of powder obtained by ring mill grinding (particle size distribution in Fig. 1), were morphological and microstructural characterized by means of XRD and SEM analysis, and leaching tests were carried out in deionized water at pH 7, varying the liquid-to-solid ratio. To determine which phases were responsible for metals release, leaching tests on the polished section were also performed. Leaching tests results were correlated to chemical composition and crystalline phase fraction.

2.2. Crystallographic and microstructural characterizations

X-ray diffraction (XRD) data was collected using a Bruker D8 Advance diffractometer in a θ - θ configuration employing the Cu K α radiation ($\lambda=1.54 \text{ \AA}$) with a fixed divergence slit size 0.5° and a rotating sample stage. The samples were scanned between 10° and 80° (step size of 0.007°) with the Vantec detector. The qualitative analysis was performed with EVA software whereas a semi-quantitative analysis was performed by Crystal Match! Software, exploiting the Reference Intensity Ratio method (RiR-method) (de Wolff and Visser, 1988), calibrating the results on the base of a experimentally determined larnite fraction. Larnite fraction was determined through a selective dissolution in methanol-salicylic acid solution (Klemm and Skalny, 1977). 2.5 g of slag powder was mixed in 150 ml of methanol acidified with 10 g of salicylic acid. The solution was stirred for 2 h at 100 rpm and decanted for 15 min before filtering in a $0.45 \mu\text{m}$ vacuum system. The residual slag powder was dried at 105°C for 1 h and weighed again (balance accuracy: 0.0001 g). The weight difference is a good estimation of larnite fraction.

Morphological and microstructural characterization was performed by Zeiss EVO50 Scanning Electron Microscopy (SEM) equipped with an Oxford Inca EDS probe. Slags were moulded in araldite-based resin, ground and polished.

2.3. Leaching test

Slag powder leaching behaviour was investigated by varying the L/S ratio (10, 100, 1000 l/kg). Weight loss, water pH and conductivity were measured after each test and the dried powders were investigated by XRD. The test consisted in immersing the slag powder in deionized water for 1 day,

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