



Valorization of the saline slags generated during secondary aluminium melting processes as adsorbents for the removal of heavy metal ions from aqueous solutions



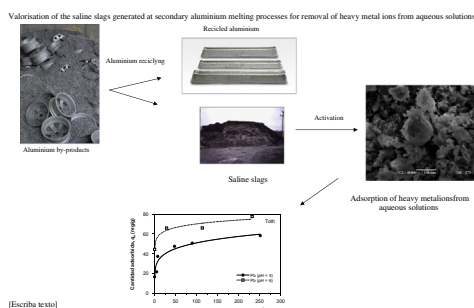
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HIGHLIGHTS

- Valorisation of aluminium saline slags as adsorbents.
- Chemical activation of saline slags.
- Activated saline slags as adsorbents of heavy metal ions from aqueous solutions.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 17 February 2014
Received in revised form 11 April 2014
Accepted 12 April 2014
Available online 24 April 2014

Keywords:

Saline slags
Secondary aluminium
Industrial wastes
Heavy metal cations
Adsorption

ABSTRACT

This work describes the surface properties of saline slags generated during secondary aluminium melting processes and the application of these materials as adsorbents for the removal of Cd(II), Cu(II), Pb(II) and Zn(II) from aqueous solutions. Saline slags were chemically treated with aqueous solutions of HNO₃, H₂SO₄ and NaOH of varying concentrations (0, 0.5, 1, 1.5 and 2 mol/dm³) for 0.5, 4 and 24 h. Nitrogen adsorption at −196 °C was used to study the textural properties of the solids. The specific surface area and pore volume decrease as the chemical reagent concentrations and duration of the treatments increase. Solids with a surface area of up to 109 m²/g are obtained. The adsorption of heavy metal ions has been studied in terms of pseudo-first-order and pseudo-second-order kinetics, and several isotherm models have also been applied to the equilibrium adsorption data at various pH values. The adsorption capacity of the saline slags was maintained after several cycles of reuse.

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1. Introduction

Aluminium recycling is an essential part of the aluminium industry. The aluminium destined for recycling can be divided into two categories: preconsumer by-products from primary aluminium production, and scrap, which is associated with postconsumer aluminium. The by-products have their origin in the manufacturing process for aluminium-based materials. As such, their quality

and composition are known and they can therefore be melted without prior treatment. In contrast, scrap is aluminium-containing material obtained from articles used and rejected at the end of their life cycle. This type of scrap therefore requires treatment and prior separation in order to recover the highest possible quantity of aluminium. The by-products and scrap are transformed into various other forms by melting for subsequent commercialization.

From an environmental point of view, the recycling process has a lower environmental impact than aluminium production from the reduction of aluminium oxide, known as the Hall-Héroult

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process [1,2]. As regards waste generation, one of the waste by-products of the alkaline extraction of aluminium oxide from bauxite in the primary process is red mud. The quantity of this waste, as well as its physicochemical properties, depends on the type of bauxite employed. Red mud mainly consists of oxides of iron, aluminium, silicon and titanium, amongst others and it is usually managed by means of controlled landfill disposal. Recently, several applications for this type of waste have been reported in the literature [3–10]. Various types of wastes, including powders from treatment of the melting furnace gases and grinding processes, foams, skimmings, and saline slags, may also be generated during the secondary aluminium smelting process. The most important of these, in terms of the amount generated, is saline slag.

The saline slags from secondary aluminium smelting processes are cataloged as hazardous wastes with EWL (European Waste List) code 100308 [11]. They are generated when various salts are used to cover the molten material in order to prevent its oxidation, to increase the performance of the process, to enhance its thermal efficiency and to mechanically scatter the oxides and metallic or non-metallic substances present in the smelting furnace. The amount of fondants used, and saline slags generated, depends on the impurities present in the aluminium scrap and byproducts, the type of furnace used, and the foundry conditions. The average composition of saline slag can be summarized as metallic aluminium, several oxides (this fraction being known as non-metallic products), flux brines (salts, normally NaCl and KCl in a molar ratio of 70/30) and other components in smaller proportions (NaI, Al₄C₃, Al₂S₃, Si₃P₄, Na₂SO₄, Na₂S, criolite, C, etc.) [2]. Due to its composition, aluminium saline slag is an important byproduct of considerable economic value that is worth recovering provided the process can be made economically viable.

Saline slag is readily managed by separation of its components for possible recovery or by storage in controlled landfills [12]. The recovery process consists of an initial separation of the metallic aluminium present in saline slag by means of electromagnetic and mechanical procedures [13]. This process is viable if the metallic aluminium content is higher than 4–6 wt.%. The remaining waste consists of nonmetallic products and fluxes, which can be valorized using the HANSE process [2]. In this process the saline fraction is separated from the oxides and is then initially concentrated by means of electrodialysis, for example, for subsequent separation of the salts by evaporation [14]. Similarly, the collection of salt-free oxide fractions is also of interest. It is noteworthy that the non-metallic products composition can be highly variable and depends on the nature of the material recycled. As such, it is difficult to propose generic recovery methods. For certain specific applications, additional problems arise due to the need for this fraction to be free from salts [13]. It is considered to be free from salts when the salt content is lower than 2 wt.%. Finally, it should be noted that recovery of the saline fraction and non-metallic products is economically viable only if a concentrated flow of salt and a waste that is free from non-metallic products can be obtained. The main drawback, aside from possible subsequent applications, is that these two determining factors cannot be ensured simultaneously, thus meaning that the process is not economically viable. As such, the best solution is currently to maximize aluminium recovery and to store the waste in controlled landfills [15–17].

Studies using saline slag residues generated during secondary aluminium melting processes as non-conventional adsorbents for water and wastewater treatment purposes are motivated by the fact that these materials are a mixture of oxides, with high surface reactivity, that are capable of removing several contaminants. To the best of our knowledge, such materials have not been reported as adsorbents of heavy metals or organic pollutants from water. Herein we present the surface characteristics of non-treated and

chemically activated salt cakes and their use as adsorbents for the removal of heavy metals from water.

2. Experimental procedure

2.1. Starting materials and chemical activation procedure

The material used in the present study was obtained from *Iberica de Aleaciones Ligeras, Ltd., IDALSA*, Spain. The typical chemical composition of the saline slags from secondary aluminium melting processes is summarized in Table 1. The fractions with a particle size of less than 1 mm were used for chemical activation, which was carried out using aqueous solutions of HNO₃ (65%, Panreac), H₂SO₄ (98%, Panreac) and NaOH (Panreac). Chemical activation was performed using five aqueous solutions of each chemical reagent at concentrations of between 0 and 2 mol/dm³. The reaction time for each of these solutions (0.5–24 h) was also studied as a parameter. In each activation, 10 g of saline slag was added to 100 cm³ of aqueous reagent solution and the resulting suspension stirred at a speed of 500 r.p.m. After the reaction time, the slurries were centrifuged at 4500 r.p.m. for 20 min at room temperature using a Rotonta 460 S to separate the saline slag from the activation solution. This process was repeated several times, replacing the solution with deionized water each time in order to free the saline slag from chemical reagent. The solids obtained were dried in an oven at 60 °C and atmospheric pressure for 16 h. The nomenclature used for every sample is X–Y–t, where X is the concentration of the aqueous solution used, Y is the reagent used in the chemical activation and t the contact time between the saline slag and the aqueous solution in hours. Thus, for example, sample 0–N–4 is a saline slag treated with an aqueous solution of 0 mol/dm³ HNO₃ (N) (i.e. water only) for 4 h.

2.2. Characterization techniques

Nitrogen (Air Liquide, 99.999%) adsorption was carried out at –196 °C using a static volumetric apparatus (Micromeritics ASAP 2010 adsorption analyzer). Prior to the adsorption measurements, the samples were outgassed at 120 °C for 24 h under a vacuum better than 0.1 Pa.

Scanning electron microscopy (SEM) was performed using a JEOL microscope (model JSM5610LV). A drop of powder suspension was deposited on a copper grid and the selected area recorded.

Metal cation concentrations were determined by inductively coupled plasma-optical emission spectroscopy (ICP-OES) using a Varian Vista-MPX instrument.

2.3. Adsorption procedure

Stock solutions of Cd(II), Cu(II), Pb(II) and Zn(II) ions were prepared by dissolving Cd(NO₃)₂·4H₂O, Cu(NO₃)₂·3H₂O, Pb(NO₃)₂ and Zn(NO₃)₂·6H₂O separately in 1 dm³ of distilled water. Each

Table 1
Typical chemical composition of the saline slags from the secondary aluminium melting processes.

Compound	(wt.%)
Al ₂ O ₃	29.60
NaCl	19.50
KCl	10.40
SiO ₂	2.75
Na ₂ O	2.35
MgO	1.90
CaO	1.43
Fe ₂ O ₃	1.37
N	0.40
SO ₃	<0.10

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