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Treatment of salt cakes by aqueous leaching and Bayer-type digestion

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Abstract

Salt cakes are toxic waste products which are produced when aluminium drosses are remelted under a salt cover to recover some of the metallic aluminium present. They are expensive to dispose of in waste dumps because they contain many toxic compounds and many water-soluble compounds. However, they also contain many relatively valuable compounds, some of which may be worth recovering. This paper shows that 90% of the Cl, 55% of the Na, and 45% of the K can be extracted by aqueous leaching of -2 mm salt cake for 1 h at 25 °C.

Treatment of the aqueous leach residue by Bayer-type digestion at 100 °C and 145 °C for 15 min was also investigated. It was found that elpasolite (K_2NaAlF_6) and metallic aluminium were fully extracted from fine ground material at both temperatures, that all aluminium nitrides were destroyed, and that all the resultant aluminium oxides and hydroxides were dissolved. Some other compounds were also dissolved. The total Al_2O_3 extraction was about 42%; this was comparable with that from typical bauxites.

Corundum (Al_2O_3) and diaoyudaoite $(NaAl_{11}O_{17})$ were insoluble in the solution and increased in proportion in the final residue. Sodalite $(Na_6[Al_6Si_6O_{24}] \cdot 2NaCl)$ had precipitated in all the residues. Crown Copyright © 2007 Published by Elsevier Ltd. All rights reserved.

Keywords: Salt cake; Aqueous leaching; Bayer-type digestion

1. Introduction

Salt cakes are complex toxic waste products which are formed when aluminium drosses are melted under a salt cover to recover some of the metallic aluminium present (Schlesinger, 2007). They are one of the ultimate waste products from the processes used to convert bauxite into metallic aluminium. Salt cakes are commonly disposed of in toxic waste dumps at considerable expense. However, they contain potentially valuable components such as metallic aluminium, salt (sodium and potassium chlorides), and many aluminium compounds. Beckman (1991) has described a process which is used in Germany to recover some of the constituents in salt cakes. In a previous paper (Bruckard and Woodcock, 2007) it was shown, after detailed characterisation of two Australian salt cakes, that some of the metallic aluminium could be recovered in a

high grade product by grinding and screening and that most of the water-soluble salts could be recovered in high grade products by aqueous leaching and solar evaporation. Although none of the alumina-containing phases present were recovered, it was suggested, after some preliminary experiments, that it should be possible to recover some of the alumina by Bayer-type digestions. Hence, a more detailed study was undertaken to determine whether freshly-produced salt cake, or salt cakes after aqueous treatments to recover the salt and aluminium, would be suitable feedstocks for the Bayer process.

Some of this work was conducted at CSIRO Minerals in Clayton, Melbourne, Victoria, and some at CSIRO Minerals in Waterford, Perth, Western Australia.

2. Experimental

Experimental work, following methods discussed by Bruckard and Woodcock (2004), included sample preparation, leaching with aqueous solutions at 25 °C and 60 °C,

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leaching with Bayer-type strongly-alkaline liquors at 100 °C and 145 °C, chemical analysis of solid and solution products by various techniques, and identification of the phases present in feed and products by X-ray diffraction (XRD) methods.

2.1. Sample preparation

The original salt cake sample weighed about 4 kg and consisted of rounded lumps up to about 150 mm in size with many small fragments. The sample was crushed to -2 mm using a jaw crusher in closed circuit with a 2 mm aperture screen. Particles of distorted or flattened metallic aluminium contaminated with non-metallic material were picked out when seen and put aside for separate weighing and analysis. The -2 mm product was riffled into various size sub-samples for analysis and/or treatment.

One 100 g sub-sample was pulverised to $-850 \, \mu m$ using a ring mill in closed circuit with an $850 \, \mu m$ aperture screen to provide material for chemical and XRD analysis. Small particles of metallic aluminium remaining on the screen were picked out, weighed in bulk, and submitted for chemical analysis. The weight of aluminium removed in this way was added to the total aluminium assay of the head sample.

2.2. Wet grinding procedure

Some leaching tests were conducted on -2 mm crushed material, but other tests were conducted on finely ground material. Grinding was accomplished in a 20 cm diameter by 17.5 cm long stainless steel ball mill charged with 7.80 kg of 25 mm diameter stainless steel balls. Usually, a 500 g charge of salt cake and 750 mL of Melbourne tap water (i.e., an initial pulp density of 40% solids or a liquid:solid ratio of 3:2) was ground for 20 min. Before each grind the mill and ball charge were cleaned by grinding a batch of quartz and water for 5 min, and then rinsing the mill and balls with water.

After grinding a batch of salt cake, the ground charge was screened on a 150 μm aperture screen to remove any relatively coarse particles of metallic aluminium, which were separately dried, weighed, and assayed. The screen undersize was then filtered on a Buchner funnel to recover the ground solids. The filter cake was washed with two 500 mL aliquots of distilled water and dried in an oven at 60 °C. The dry solids were weighed and then riffled into sub-samples for size analysis, chemical analysis, and subsequent leaching tests.

All of the liquors from each test were combined, the total volume measured, and the combined solution was sent for chemical analysis to determine the amount of material dissolved.

2.3. Aqueous leaching test procedures

All aqueous leaching tests, as distinct from the Bayertype leaching tests described below, were conducted in a 1000 mL round-bottomed Quickfit flask fitted with a top-driven paddle stirrer and a heating mantle. Feed weights of 200 g, 250 g, or 300 g were leached at either room temperature (25 °C) or elevated temperature (60 °C). For tests at elevated temperature, the solution was heated first to the required value and then the required weight of solid sample was added. All of the tests were conducted at an initial pulp density of 30% solids (a liquid:solid ratio of 7:3). Tests were conducted with either Melbourne tap water at 25 °C or with 16% w/v NaOH at 60 °C on the crushed (-2 mm) material and on the dried finely ground material. Previous, unpublished work had shown that these latter conditions could be suitable for the decomposition of aluminium nitride.

During each run a solution sample was taken for analysis at 0.5, 1.0, 2.0, and 4.0 h by stopping the stirrer, allowing the pulp to settle until a layer of clear supernatant solution was available on top of the pulp, and then pipetting out 20 mL of clear liquor. Each sample was then re-filtered through a 1.2 μ m aperture Millipore filter. The four liquid samples were sent for analysis for selected elements.

After 4 h agitation the pulp was filtered on a Buchner funnel to recover most of the pregnant liquor for analysis. The filter cake was washed with two 500 mL lots of Melbourne tap water, then dried in an oven at 60 °C, and weighed the next day when cool. A sub-sample of the dry cake was pulverised for chemical and other analyses as desired.

2.4. Bayer-type leaching procedures

Bayer-type leaches were conducted on the five washed residues from the above aqueous leaching tests and on the crushed head sample ($-2\,\mathrm{mm}$). Thus these tests involved six leaches at atmospheric pressure and $100\,^{\circ}\mathrm{C}$, followed by six leaches at $145\,^{\circ}\mathrm{C}$ and high pressure, i.e., one on each residue from the atmospheric pressure tests. These test conditions were adopted to minimise any problems from the release of any gaseous components from the salt cake during the pressure leach.

For the atmospheric pressure leaching tests, 100 mL of caustic liquor (150 g/L NaOH, or 200 g/L Na₂CO₃ equivalent) was placed in a sealed glass vessel and heated to 100 °C. A 10 g sample of salt cake or leached residue was heated in an oven to 100 °C. The preheated solid material was then carefully transferred to the hot liquor and the resultant slurry held at temperature with mixing for 15 min. The slurry was then filtered on 0.45 µm aperture Millipore filter paper and the filtrate sent for chemical analysis. The filter cake was washed by mixing with 2 L of deionised water and re-filtered. The washed cake was dried overnight in an oven at 100 °C. A sub-sample of the dry residue was sent for chemical and mineralogical analysis.

For the high pressure/temperature leaching tests, a portion of the dried residue from the atmospheric digestion was digested in caustic liquor for 15 min at 145 $^{\circ}$ C in a CSIRO-designed gas-fired reactor. Reacted slurry was then pressure-filtered on a 0.45 μ m aperture Millipore paper.

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