

# The fate of chromium impurities during acid sulphate digestion of ilmenite concentrates



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## ARTICLE INFO

### Article history:

Received 9 April 2014

Accepted 30 September 2014

Available online 7 October 2014

### Keywords:

Chromium

Spinel

Ilmenite

Acid sulphate digestion

Murray Basin

## ABSTRACT

Ilmenite concentrates from the Murray Basin region of southeastern Australia are contaminated with chromium impurities that must be removed for the ilmenite to become a satisfactory feedstock for the sulphate route to titania pigment production. The chromia is present primarily as discrete, compositionally variable, chrome-rich spinel grains with a smaller amount as intra-grain chromia distributed as coatings in fractures and pores of weathered ilmenite grains. Characterisation of chromia deportment through a simulated acid sulphate digestion process showed a small but non-negligible solubility of the spinels. Most spinels were resistant to dissolution with the exception of those containing high  $\text{Fe}(\text{Al,Cr})_2\text{O}_4$  and magnetite ( $\text{Fe}_3\text{O}_4$ ) components. Intra grain chromia was highly soluble. Processing to achieve low bulk chromia using a magnetising roast procedure must ensure that well crystallised rutile is not produced because of its insolubility in the sulphate process. It is also important that the roast conditions do not substantially increase the magnetite content of the spinels making them more susceptible to dissolution. This work highlights the importance of characterising all spinel composition types within ilmenite concentrates in addition to the level of intra-grain chromia associated with the ilmenite when considering the suitability of Murray Basin primary ilmenites as a feedstock to sulphate route titania pigment plants.

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## 1. Introduction

Australia is one of the largest producers of mineral sands in the world with most of the current production coming from deposits in Western Australia. The position of Australia as a continuing long-term supplier of mineral sands, and their upgraded products, has been reinforced with the discovery and development of extensive mineral reserves in the Murray Basin, an area covering parts of South Australia, New South Wales and Victoria (Fig. 1). Deposits in the Murray Basin are of two general types, fine-grained (40–80  $\mu\text{m}$ ) offshore sheet-like deposits, and coarse-grained (90–300  $\mu\text{m}$ ) beach facies strandline deposits (Roy et al., 2000). The latter deposits have size ranges comparable to Western Australian deposits and are the target for current development in the Basin. Both deposit types have ilmenite as the major heavy mineral component. Ilmenite, however, has the lowest commercial value of the heavy minerals and to realise the full economic value of the deposits, processing of the ilmenite to a marketable product is essential.

Primary ilmenite concentrates (i.e. those containing 45–55 wt.%  $\text{TiO}_2$ ) produced from the Murray Basin deposits are potential feedstocks for sulphate-route titania pigment plants. The requirements for such feedstocks (Harben, 2002), include; a high ferrous iron content to

react with the sulphuric acid, a low rutile content since rutile is largely insoluble in sulphuric acid, low calcium and phosphorus which impede crystallisation, and low levels of elements that could impart a colour to the pigment. Chromium is a particular problem in this latter regard and the acceptable chromia level for further processing of ilmenite is of the order of ~0.1 wt.%  $\text{Cr}_2\text{O}_3$  or less (Beukes and van Niekerk, 1999). Murray Basin primary ilmenite concentrates generally meet the first three requirements, but are limited in their suitability as sulphate-route feedstocks because of elevated chromia contents, typically >0.5 wt.%  $\text{Cr}_2\text{O}_3$  (Pownceby et al., 2003; Pownceby, 2005). Previous mineralogical investigations have shown the chromia is present predominantly within discrete spinel grains so in theory it should be possible to make a clean separation of the spinels from the ilmenite using physical separation methods such as gravity and magnetic techniques. In practice, however, separation is difficult as the spinels have variable compositions as a result of solid solution and/or weathering, thereby providing correspondingly variable physical properties (Pownceby et al., 2001, 2003; Pownceby, 2005).

In this paper, the occurrence of chromia in both coarse- and fine-grained Murray Basin primary ilmenite concentrates is discussed, as are the effects of a laboratory-based, sulphate-route digestion procedure on the solubility of the contained chromia. Since the bulk of the chromia is known to be associated with the presence of individual chrome-bearing spinel grains, the aim is to characterise a series of ilmenite concentrates before treatment, followed by characterisation

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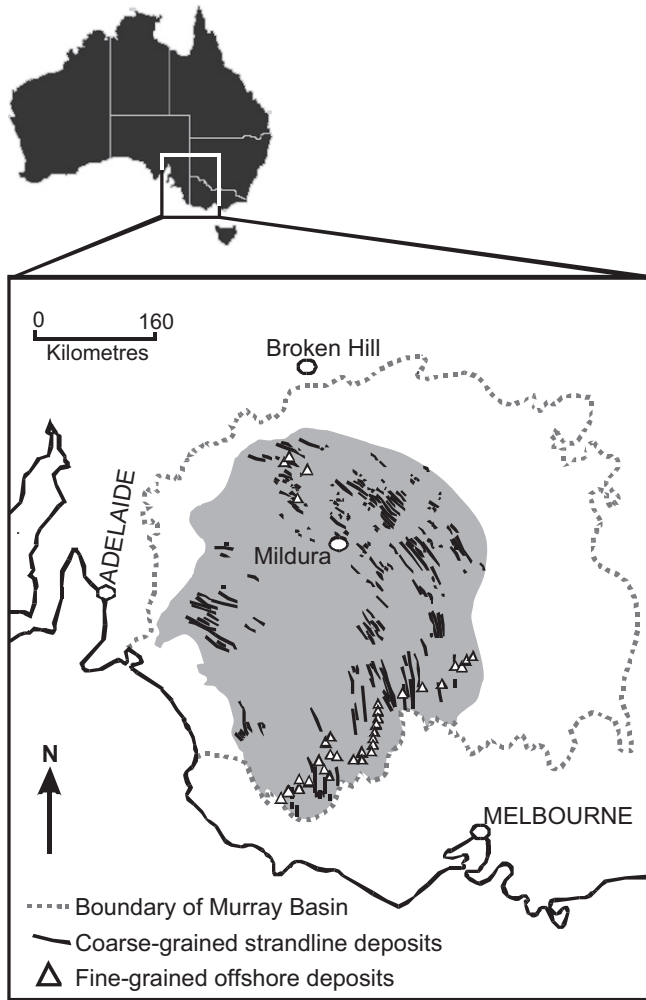


Fig. 1. Map showing the location of the Murray Basin region in southeastern Australia. The boundary of the Murray Basin is indicated by the dashed line. The host unit for the coarse- and fine-grained mineral sand deposits is the Loxton–Parilla sands (shaded region).

of the products and residues at various stages throughout the digestion process. Characterisation includes bulk chemistry determinations (solids and liquids) in association with quantitative electron microscopy of individual spinel grains within feedstocks and residues. Finally, the implications of the results for the future processing of Murray Basin primary ilmenite concentrates are discussed.

## 2. Murray Basin spinel compositional variation

Spinel contains two differing cations, or at least two different valences of the same cation, in the ratio 2:1. This gives the general formula  $AB_2O_4$ . In general, the spinel types commonly associated with ilmenite concentrates are dominated by compositions containing the cations Cr, Mg,  $Fe^{2+}$ ,  $Fe^{3+}$ , Al and Ti. The major cations substituting into the A site are the divalent cations Mg and  $Fe^{2+}$  whereas substitution within the B site involves the cations Al, Cr,  $Fe^{3+}$  and Ti. Aluminium, iron and chromium are each trivalent. However, substitution of  $Ti^{4+}$  into the octahedral B site may also occur (as in the case of ulvöspinel –  $Fe_2TiO_4$ ) giving rise to a range of spinel solid solutions within the system  $(Fe^{2+}, Mg)(Al, Cr, Fe^{3+})_2O_4 - (Fe^{2+}, Mg)_2TiO_4$ .

Spinel solid solutions may also contain some degree of non-stoichiometry associated with defects in the oxide structure. The most likely non-stoichiometry to occur in spinels associated with ilmenite concentrates is the defect spinel component  $(Fe^{3+}, Al, Cr)_{2.67}O_4$

(Pederson, 1978). This type of defect spinel is believed to occur in natural systems as a result of the chemical weathering of the spinel by the same mechanism that occurs in ilmenite alteration i.e. diffusion of iron and other divalent elements out of the spinel, with oxidation of the remaining iron to the trivalent state to maintain charge balance (Grey and Reid, 1976).

Pownceby (2005) previously characterised the range of spinel compositional variation within Murray Basin ilmenite concentrates. These data are shown in Fig. 2 plotted on a quaternary  $Fe_T$ –Mg–Cr–Al diagram which describes a compositional area enclosed by the seven spinel end member components;  $Fe_3O_4$ ,  $FeCr_2O_4$ ,  $MgCr_2O_4$ ,  $FeAl_2O_4$ ,  $MgAl_2O_4$ ,  $MgFe_2O_4$  and  $(Al, Cr)_{2.67}O_4$ . Compositions which lie outside this area are typically spinels that have either been extensively altered or leached or represent more exotic spinel compositions such as those dominated by Mn- and Zn-rich components. The bulk of the spinel data from the Murray Basin are Fe- and Cr-rich spinels such as chromite ( $FeCr_2O_4$ ), however trends towards more MgAl-rich and MgCr-rich compositions are evident. The spread of the data toward the  $Fe_T$  apex indicates spinel chemistries trending towards more MgFe-rich or more ferric-rich (magnetite) compositions.

## 3. Experimental

### 3.1. Samples

Samples used for the digestion tests comprised two primary ilmenite concentrates (Ilmenites A and B) and one ilmenite concentrate (Ilmenite C) that had been subject to procedures to lower the total chromium content by removing some of the spinels through a low temperature roast treatment followed by magnetic separation to derive a low chromia primary ilmenite concentrate. Details of the bulk chemistry, gangue mineralogy and processing conditions for each of the samples are given below.

#### 3.1.1. a) Primary unprocessed concentrates

Samples A (54.2 wt.%  $TiO_2$ ; 0.435 wt.%  $Cr_2O_3$ ) and B (53.5 wt.%  $TiO_2$ ; 0.551 wt.%  $Cr_2O_3$ ) were primary ilmenite concentrates that represented the grainsize range of deposit types within the Basin. Ilmenite A was a coarse-grained strandline concentrate sourced from the western sector of the Basin whilst Ilmenite B was typical of the fine-grained material prevalent in the south-eastern sector of the Basin (Fig. 1). The samples were supplied from commercial operations and each represented the primary ilmenite fraction only. In both samples, unweathered ilmenite

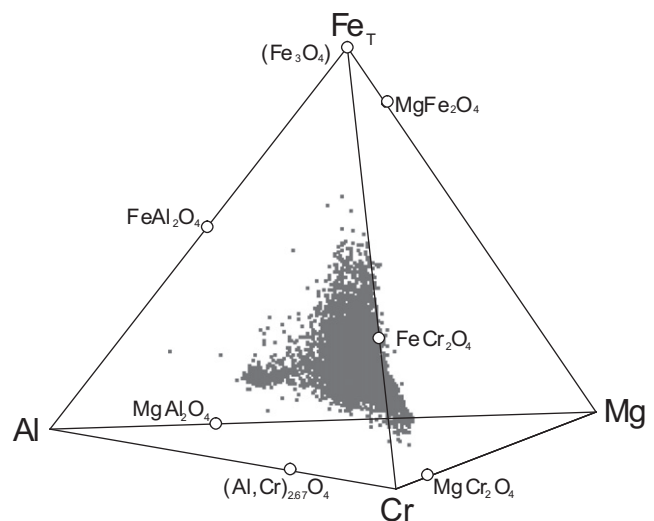


Fig. 2. Plot showing the range of spinel compositions measured in Murray Basin ilmenite concentrates (Pownceby, 2005).

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