



The dissolution behaviour of titanium oxide phases in synthetic Bayer liquors at 90 °C

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ABSTRACT

Many of the bauxites currently being processed to alumina contain titanium minerals, yet little fundamental knowledge is available regarding their dissolution behaviour in Bayer liquors. In this paper the dissolution of various titanium oxide phases (anatase, rutile, sodium titanate and calcium titanate) are presented. It is shown that rutile, calcium titanate and sodium titanate have the expected dissolution behaviour, which increases with free caustic concentration. Anatase, too, generally increases in dissolution with free caustic concentration. However, anatase is shown to have an anomalous behaviour when gibbsite co-precipitates. In this case, the dissolution of anatase does not follow the expected free caustic trend. According to the data presented, the most probable cause is an impervious aluminium/sodium titanate surface layer hindering further dissolution.

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1. Introduction

In the processing of bauxite ore to alumina (Al_2O_3) different conditions are used, depending on the main aluminium-bearing phases. A low temperature digest ($\sim 150^\circ\text{C}$) is used when gibbsite is the main component while higher temperatures are required when boehmite (or diaspore) is present ($\sim 250^\circ\text{C}$). Alumina production is essentially the same in both cases, being a dissolution–precipitation process where the aluminium bearing phases of the ore are dissolved in concentrated caustic solution, the waste then removed and aluminium trihydrate (gibbsite) precipitated in pure form before calcining to obtain alumina. Different challenges are encountered in high temperature processing when titanium is present in the bauxite, including the negative impacts of scale formation, titanium in the alumina product, and inhibition of boehmite extraction (Malts et al., 1985; Malts, 1992; Prakash and Horvath, 1979). TiO_2 dissolution/reactivity is believed to be minor at 'lower' temperatures, although data demonstrating this fact is noticeably lacking in the literature. There is also anecdotal evidence (from alumina plant operators) that the mineral form of the titanium phase is important. Recently, an *in-situ* XRD study demonstrated that anatase, and not rutile, inhibited boehmite dissolution (Loan et al., 2005), confirming this long-held

belief that the presence of anatase in bauxite ores limits the extraction of Al in the high temperature process (Malts et al., 1985; Malts, 1992).

The literature on titanates in Bayer liquors has focussed almost exclusively on their impact on extraction and reaction with lime during digestion, or the final precipitation products. Studies have found that sodium titanates are formed at lower temperatures and higher caustic concentrations (Shultze-Rhnhof and Winkhaus, 1972) and that sodium titanate (all sodium titanate materials, regardless of composition) has a low solubility in aluminate liquors (Wefers, 1971). The solubility of anatase was found to be dependant on the free caustic concentration and the time allowed for dissolution to occur (Shultze-Rhnhof and Winkhaus, 1972). In the presence of calcium, both perovskite and tricalcium aluminate can form (Malts et al., 1985; Prakash and Horvath, 1979; Loan et al., 2005).

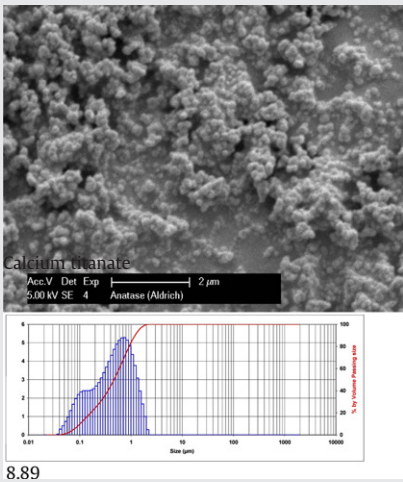
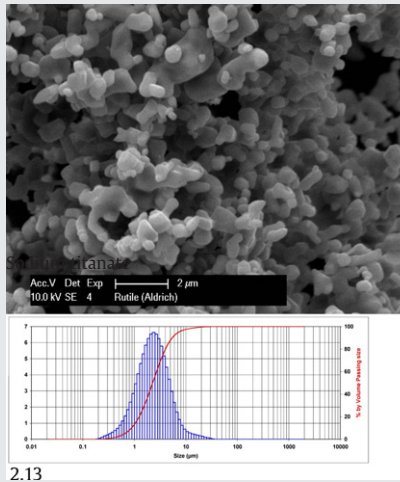
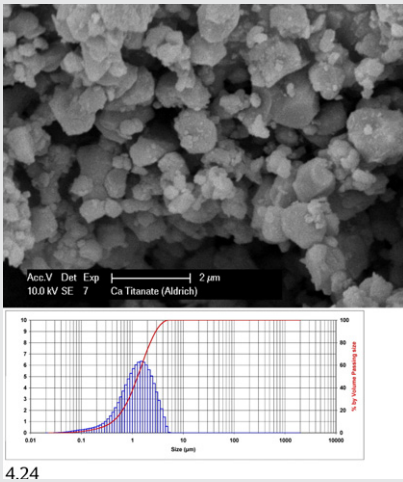
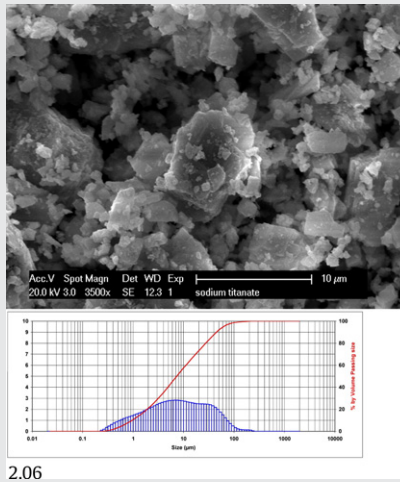
The interest in titanates is not limited to the alumina industry however, and titanate compounds are currently under investigation as a route to the formation of Ti-containing inorganic nanotubes (Wu et al., 2006; Morgado et al., 2007; Pradhan et al., 2007; Menzel et al., 2006). Results from this literature that are of relevance to the work described here include the observation that sodium titanates are formed at high caustic strengths and high temperatures (Wu et al., 2006; Morgado et al., 2007; Pradhan et al., 2007; Menzel et al., 2006). Furthermore, both rutile and anatase have been found to form sodium titanate nanotubes with the greater conversion of anatase to nanotubes related only to the higher surface area of the anatase starting material (Menzel et al., 2006). Of more interest, perhaps, is the work of Wang et al. (2006) that shows that the form of titanium oxide can be important even when adsorbing organic titanates. The

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Table 1
Sample characteristics

	Anatase	Rutile
SEM PSD surface area (m ² /g)	 <p>8.89</p>	 <p>2.13</p>
SEM PSD surface area (m ² /g)	 <p>4.24</p>	 <p>2.06</p>

organic titanate was found to react with the corundum surface, strongly adsorb onto anatase and weakly adsorb onto rutile. This is further confirmed by the work of Hotta et al. (1991), where the organic titanate was coupled to a hematite surface, which is iso-structural with corundum.

Missing from our current understanding is any fundamental information regarding the solubility of titanium (in various forms) in Bayer liquors. However, such data is difficult to obtain unambiguously when re-precipitation reactions can also occur. To this end we have begun to systematically measure the dissolution of anatase, rutile, sodium titanate and calcium titanate in synthetic Bayer liquors. Unfortunately, the measurement of dissolution under hydrothermal conditions is not a trivial exercise; the greatest challenge being to remove the excess solids without significant cooling, and subsequent re-precipitation, occurring. In the preliminary experiments we conducted, even at 90 °C we found that the measured [Ti] levels were lower when the filtered solution was left to stand undiluted or not sufficiently diluted. This highlights that re-precipitation can and does occur as the temperature drops towards ambient and the [Ti] level in solution reaches supersaturated levels, limiting experiments to lower temperatures.

In what follows we present some preliminary dissolution data for anatase, rutile, sodium titanate and calcium titanate in synthetic Bayer liquors at 90 °C. Although this data can be used to extrapolate what

can be expected under the hydrothermal conditions of the Bayer process, it is clearly well below even the low temperature Bayer process. However, collecting dissolution data at higher temperatures presents major practical difficulties under atmospheric pressure which must first be overcome.

2. Materials and methods

The solids, TiO₂ (anatase, 99% and rutile, 99% pure) and calcium titanate (CaTiO₃), were obtained from Aldrich and used as received. Sodium titanate was obtained from Strem Chemicals (95% pure) and used as received. All were found to be single phases except the sodium titanate where the XRD found a combination of two sodium titanate phases; namely JCPDF# 37-951 Sodium Titanium Oxide (Na₂Ti₆O₁₃) and JCPDF# 72-148 Sodium Titanium Oxide (Na₂Ti₃O₇). Table 1 and 2 give all the relevant physical characteristics of the solids used.

Sodium hydroxide (A.R. grade, BDH), MilliQ water, sodium carbonate (A.R. grade, BDH) and aluminium wire (BDH, 99.9% pure) were used for synthetic Bayer liquor preparation. XRD patterns were collected on a Philips Xpert diffractometer using Co K_α radiation. Scanning electron microscopy was performed on a Philips XL30 SEM and surface area was determined using the BET isotherm with N₂ as carrier gas.

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