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Titania preparation from soda roasted slag using sulfuric acid solution

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TiO₂

Abstract This work deals with treatment of the sodium titanate cake results from roasted titania slag using 60% sulfuric acid solution. The working sample produced by roasting titania slag with NaCO₃ at 850 °C. After roasting V and Cr species as impurities in the roasted sample are converted to water soluble species as NaVO₃ and Na₂CrO₄ before acid treatment however, the insoluble sodium titanate products (NaFeTiO₄, Na₈Ti₅O₁₄, Na₆Ti₂O₇ and Na₂TiO₄) are then subjected to H₂SO₄ acid dissolution. The produced sulfate solution is subjected to hydrolysis step in presence of oxalic acid as a reducing agent. The hydrolyzed precipitate after filtration and washing with H₂SO₄ solution and warm water is dried at 100 °C and calcinated at 850 °C to prepare high pure TiO₂ (99.8%) besides removing ferrous sulfate (FeSO₄) as a byproduct from the sulfate solution.

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1. Introduction

Titania (TiO₂) is a white pigment used in paints, papers, plastics, cosmetics, coatings and many other industries. The main commercial sources used in the production of TiO₂ are natural or synthetic rutile, ilmenite and titanium slag [1]. Manufacture of TiO₂ is based on two main processes known as sulfate and chloride. In the chloride process titanium ore is converted to TiCl₄ which is subsequently oxidized at high temperatures while in the sulfate process the ore is converted into sulfate solution and then thermally hydrolyzed to hydrous titanium

then calcined to produce the pigment [2–4]. Other processes proposed to produce high grade titania slag from titanium bearing ores [5–8]. On the same context several pyrometallurgical and hydrometallurgical processes are reported to upgrade titania slag [9–14]. All the previous processes possess the disadvantages of being carried out at high temperatures and necessitate preliminary high degree of sample grinding in addition the difficulty impurities disposal. For these purposes treatment of ilmenite or its slag with alkali salts (Na and K) for high grade TiO₂ preparation was carried [4,15–20]. Other investigators indicated that titanium ores can be treated with alkali salts followed by acid (H₂SO₄ or HCl) attack for titania preparation [17,19,21–23]. The present work aims to prepare pure titania sample via intermediate product produced from slag roasting process with Na₂CO₃ using H₂SO₄ acid solution. The relevant factors (comprising time, grain size, H₂SO₄ concentration, temperature . . .) were investigated and optimized.

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Table 1 The chemical composition of slag.

Composition	TiO ₂	Cr ₂ O ₃	CaO	Na ₂ O	MgO	SiO ₂	Al ₂ O ₃	MnO ₂	Fe ₂ O ₃ ^a	V ₂ O ₅
W (%)	76.6	0.76	0.54	0.2	0.71	3.2	1.01	0.65	12.9	0.59

^a Total iron content as Fe⁺² and Fe⁺³.

2. Materials and methods

2.1. Materials

Titania slag sample is prepared from Rosetta ilmenite by its carbothermal reduction in an electric arc furnace and is kindly provided from Nuclear Materials Authority titanium project. The obtained slag is crushed, ground and sized and then subjected to wet chemical analysis (Table 1).

2.2. Experimental procedures

Finely ground slag sample ($-74\ \mu\text{m} \approx -200$ mesh) is mixed with Na₂CO₃ at the required ratio in porcelain dish. The mixture is roasted in a muffle furnace under the desired conditions (Na₂CO₃/slag weight ratio; 0.5/1 at 850 °C for 30 min). The roasted porous cake is leached using warm water (60 °C) for 20 min while stirring to leach V and Cr species as sodium salts. The residue free from V and Cr ions is subjected to leaching process using H₂SO₄ (60%) at a pulp acid/solid ratio of 6/1 for 30 min at 90 °C. The leach liquor is subjected to hydrolysis step in presence of oxalic acid solution.

2.3. Analytical procedures

For quantitative chemical slag analysis the powdered sample (0.25 g) is digested with concentrated H₂SO₄ acid (25 ml) till dryness then distilled water is added with heating until obtaining clear solution and completed up to certain volume. Titanium and total iron analysis in the titania slag feed, products and the different stream solutions are performed using colorimetric and titrimetric methods respectively. For titanium, iron as complexing agent is used and the absorbance is measured at 430 nm using Unicam UV2-100 spectrophotometer. Total iron is determined by titration against EDTA using sulfosalicylic acid indicator. Both calcium and magnesium are titrimetrically determined against EDTA using murexide and Eriochrome Black T indicators. Molybdate reagent in the presence of tartaric acid is used for silica determination whereas alumina is determined colorimetrically using Alizarine red reagent at 475 nm. Chromium is spectrophotometrically determined using 1,5 diphenyl-carbazide reagent using UNICAM (UV-VIS-100) spectrophotometer at 540 nm [24]. Vanadium (V) is determined spectrophotometrically at 595 nm using 2-(5-bromo-2-pyridylazo) 5-diethyl amino-phenol (5-Br-PADAP) dye at pH 1.0 in presence H₂O₂ [25]. The obtained analyses

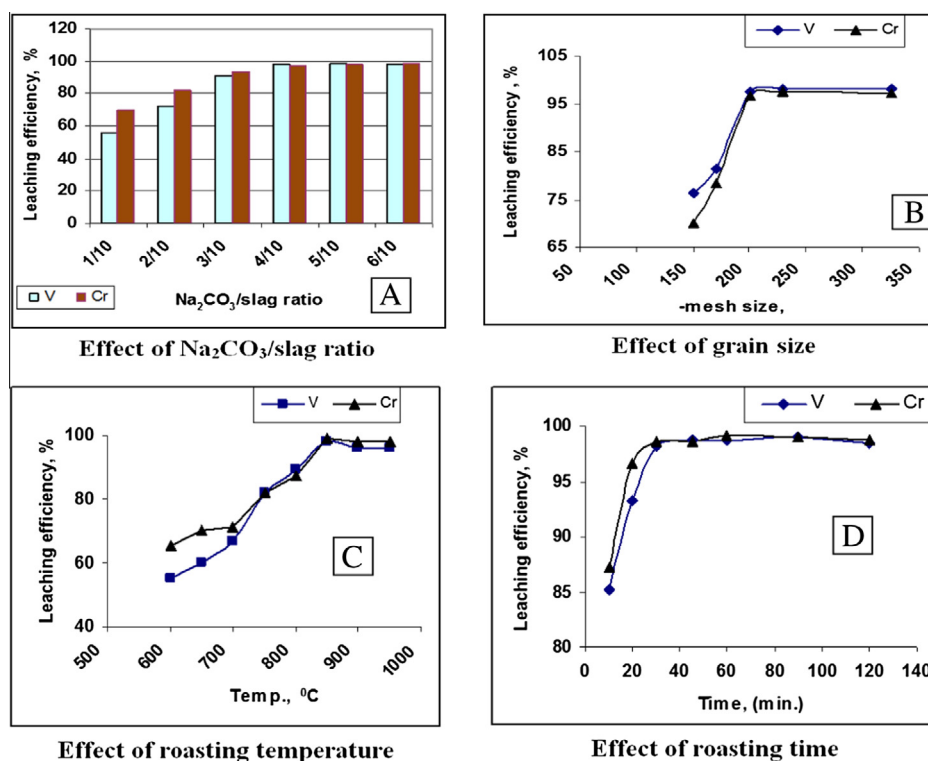


Figure 1 Factors effect on V and Cr species leaching from roasted slag using Na₂CO₃.

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