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Mechanochemical Conversion of Ilmenite to Synthetic Rutile: A Green Technology

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Abstract

Titanium minerals such as ilmenite ($FeTiO_3$), leucoxene (altered ilmenite) and rutile (TiO_2) are economically imported in the global heavy mineral market. In this regard, high quality ilmenite, leucoxene, and rutile concentrates can be directly used as feedstock for TiO_2 pigment production. However, lower quality ilmenite requires prior processing to titanium slag or synthetic rutile. Chemical routes such as Sulphate and Chloride processes are traditionally followed to prepare synthetic rutile from ilmenite. However, chemical methods yield environmentally unfriendly by-products such as iron(III) chloride and acidic iron(II) sulphate. Consequently, the current study is focussed on upgrading Sri Lankan ilmenite using crystalline vein graphite and commercially available sulphur as reducing agents. In this mechanochemical method, the ball milling induced Sulphurisation and carbothermic reductions were examined using X-ray diffraction (XRD), X-ray fluorescence (XRF), Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and particle size analysis. XRF results suggest that raw ilmenite was characterised by over 95% of $FeTiO_3$, ~2% of SiO_2, ~1% of Al_2O_3 and the rest compensated by other minor elements. Ilmenite was mixed with a combination of sulphur and vein graphite in three ratios by weight as 1:1, 2:1 and 4:1. These mixtures were uniquely milled for 4 hours and 6 hours each. The reduction in the intensity of sulphur and graphite peaks and the broadening of ilmenite peaks in XRD spectra obtained after milling. It indicates a possible dissolution of sulphur and graphite into the ilmenite structure through cleavage planes. The incorporation of additives into the ilmenite structure was confirmed using the SEM images. Pseudorutile peaks observed in the XRD spectra of milled samples. Each sample was separately annealed at 800^o C, 1,000^o C and 1,200^o C respectively. Consequently, the effective temperature was determined to be 1,000^o C. Each ratio produced similar results and the optimum ratio was obtained at 4:1. The pseudorutile peaks were disappeared whereas, brookite and rutile peaks appeared in the XRD spectra of the annealed samples. It was concluded that mechanical attrition applies beneficial outcome on the diminishing temperatures of ilmenite reduction. Consequently, this environmentally friendly method could be applied to convert ilmenite to synthetic rutile.

Keywords: Ball milling and isothermal annealing, Ilmenite, Sulphur and vein graphite, Environmentally friendly