

## 5 SUMMARY

Synthetic rutile was produced from ilmenite ore by a process that employing the soxhlet extraction. The process included using hydrochloric acid as a leachant, NaCl as additive to increase  $\text{Cl}^-$  ions in the reaction, and  $\text{H}_2\text{O}_2$  as an oxidizing agent to oxidize the metallic iron in the porous ilmenite to iron(II) ions and oxidize iron(II) ions to iron(III) ions. The leachant worked well in removing Fe and Mn from the ilmenite ore but was not capable of leaching out the other metals(Y, Zr, Nb, W and Sn) due to the chemical properties of the elements and their compounds. This also illustrates the use of XRF technique for studying materials that are insoluble or hardly soluble, rendering their analysis by other instruments difficulty or almost impossibility. The addition of NaCl, both the mixing with ilmenite (method B1) and adding directly into hydrochloric acid leachant (method B2) also helped increase the leaching efficiency. The iron content in residue from B2 was less than in B1. The residue from the  $\text{H}_2\text{O}_2$  infusion sample (method C) had residual Fe content in residue lower than main procedure (method A) and method B and the amount of Mn remained in residue also lower too because manganese dioxide was well dissolved in acid in the present of hydrogen peroxide. The combined effect of NaCl and  $\text{H}_2\text{O}_2$  was preferred to infuse residue after leaching in  $\text{H}_2\text{O}_2$  and then added NaCl into the leachant (method D2) than the mixing NaCl with ilmenite before leaching and then infused residue in  $\text{H}_2\text{O}_2$  solution (method D1). The double leached with fresh acid was to further leach out the iron remained in residue after first stage leaching. The optimum leaching method, in this work, was method F (similar to method A but did not flow air bubble into the leachant). In this method it showed high leaching ability for removing Fe from ilmenite ore and using shortest leaching time of all methods. Both temperature and initial ilmenite to acid ratio had significant effect on the leaching efficiency. The

optimum leaching conditions were as follows, 600 mL concentrated hydrochloric acid : 15 g ilmenite ore, leachant temperature 112°C, the outer side of soxhlet temperature 120°C, and leaching for 24 hours. The final produced residue at the optimum condition contained about 1.42 % Fe. The XRD spectrum of the residue after leaching showed the peaks of the rutile and cassiterite. This was in agreement with the WDXRF data, which showed that it had Sn in the residue. All methods in this study had some white precipitate formed in the round bottom flask. In method D1 the white precipitate formed most. This white precipitate had Ti as main composition with a trace of Fe, Mn, and W. The XRD spectrum of the white precipitate formed in round bottom flask after leaching by method D1 showed the peaks of anatase due to the influence of manganese ions induced the TiO<sub>2</sub> precipitated in anatases form. From the determination of acid concentrations data indicated that some HCl vapor had been lost during leaching processes. Because of method A resulted in lower acid concentration than method F, therefore, method A had lower leaching efficiency.