4 DISCUSSION

4.1 Development of techniques to upgrade ilmenite ore

The ilmenite leaching reaction can be described by the chemical reaction in equation (1) and it was reported that titanium ions was readily precipitated in diluted and concentrated hydrochloric acid solution. The precipitation of titanium ions in diluted and concentrated hydrochloric acid solution can be expressed by equation (2) (Girgin, 1990).

$$FeTiO_3 + 4HCl \rightarrow TiOCl_2 + FeCl_2 + 2H_2O$$
 (1)

$$TiOCl_2 + H_2O \rightarrow TiO_2 + 2HCl$$
 (2)

The soxhlet extraction was the heart of this leaching study. At first, acid in the round bottom flask was heated to boiling. The vapors rose through the outer chamber and into the condenser. Then the vapors condensed into liquid and fell back into the bottom of the soxhlet chamber. The acid extracted the soluble parts and left the solid mass behind. The extraction was followed by observing that the acid had different color from its pure form in the flask. As the acid level rose, the solution was forced through the small inner tube, and the chamber was flushed due to a siphoning effect. After that the flushed solvent returned to the flask taking the extracted compounds with it. The acid was redistilled from solution in the flask and condensed in the chamber, repeating the extraction with fresh acid. The process could be repeated as many times as necessary. The more the process repeated, the more concentrated the solution in the flask became because more material was being extracted from the solid mass. The acid extraction was usually completed when the solution in the soxhlet chamber was the same color as the pure solvent. This indicated that nothing more is being extracted from the ore by the acid.

Table 15 Composition of ilmenite ore from different locations.

Location	Composition	Reference
Egypt	42.00 % TiO ₂ , 24.79 % FeO, 28.81 % Fe ₂ O ₃ ,	El-Tawell, et al.,1996
	2.98 % SiO ₂ , 0.40 % Cr ₂ O ₃ , 0.16 % MnO ₂	
Australia	59.10 % TiO ₂ , 36.50 % Fe ₂ O ₃ , 1.48 % Mn ₃ O ₄ ,	Lanyon, et al., 1999
	0.66 % Al ₂ O ₃ , 0.55 % SiO ₂ , 0.17 % Nb ₂ O ₅ ,	
	0.16 % V ₂ O ₅ , 0.04 % Cr ₂ O ₃ , 0.01 % CaO, 0.02	
	% ThO ₂ , 0.16 % ZrO ₂ , 0.04 % P ₂ O ₅	
Thailand	35.22 % TiO ₂ , 44.65 % Fe ₂ O ₃ , 4.08 % MnO,	Wongnawa, et al., 1999
	419 ppm Y ₂ O ₃ , 529 ppm ZrO ₂ , 0.21 % Nb ₂ O ₅ ,	
	1.91 % SnO ₂ , 1.07 % WO ₃	
Brazil	47.04 % TiO ₂ , 41.30 % FeO, 0.32 % V ₂ O ₅ ,	Ogasawara, et al., 2000
	4.53 % Al ₂ O ₃ , 6.81 % SiO ₂	
India	60.30 % TiO ₂ , 9.70 % FeO, 24.80 % Fe ₂ O ₃ ,	Geetha and Surender,
	1.00 % Al ₂ O ₃ , 1.40 % SiO ₂ , 0.15 % CaO, 0.65	2000
	% MgO, 0.40 % MnO ₂ , 0.14 % Cr ₂ O ₅ , 0.26 %	
	V ₂ O ₅ , 0.17 % P ₂ O ₅ , 0.60 % ZrO ₂	
Sri Lanka	53.61 % TiO ₂ , 20.95 % Fe ₂ O ₃ , 20.67 % FeO,	Ismail, et al., 1983
	0.38 % SiO ₂ , 0.95 % MnO, 0.92 % MgO	

The reactivity of ilmenite towards hydrochloric acid depends on the nature of the mineral (Olanipekun, 1999). The composition of ilmenite ore in this study were 35.22 % TiO₂, 44.65 % Fe₂O₃, 4.08 % MnO, 419 ppm Y₂O₃, 529 ppm ZrO₂, 0.21 % Nb₂O₅, 1.91 % SnO₂, 1.07 % WO₃ (Wongnawa, *et al.*, 1999). The composition of ilmenite ore in other location in the literatures are shown in Table 15. To compare the composition of ilmenite ore in each location, it was found that the Fe and Ti were the main elements

in ilmenite in each location. The other impurity elements were different in each location.

4.1.1 Main procedure (method A)

EDXRF spectra of the ilmenite ore and the residue are shown in Figures 12 and 13. In Figure 12 the three most intense lines, Fe Kα, Ti Kα and Mn Kα, are readily detected. There are other elements but these are at much lower concentrations (i.e. Y, Zr, Nb, W, and Sn). The same spectrum has to be enlarged by 64-fold to see the characteristic x-ray lines of these elements clearly in Figure 12. Since the energy of the Sn K\alpha line is 25.27 keV, Sn is not seen in Figure 12. To see this line the spectrum must be acquired using a different set of parameter. In Figure 13 as shown, the major elements present in the residue from method A are Ti, Fe, Mn and W. The amount of Fe and Mn in the residue were less than that originally present in ilmenite ore before leaching, Figure 12. The quantitative analysis by WDXRF in Table 14 also reported that the leachant work well in removing Fe and Mn from the ore. Concentration of Fe and Mn drop from 36.10 % and 5.10 % to < 8.42 % and < 1.55 %, respectively. The leachant is not capable of leaching out the other metals (Y, Zr, Nb, W and Sn). A similar result was reported for the leaching ilmenite ore with HCl-ethylene glycol by Wongnawa, et al. (1997). The EDXRF spectrum of the precipitate obtained form reaction between acid solution after leaching and 1 M. NaOH in Figure 18 also indicated that Fe and Mn were the main elements dissolved in hydrochloric acid with negligible Ti dissolution. The most striking appearance is the complete absence of peaks from Y, Zr, Nb, Sn, and W, which would appear in the region 8-20 keV, compared to Figures 12 and 13, and Sn at 25.27 keV. These elements (Y, Zr, Nb, Sn and W) will remain in the residue after reaction. Most of the mass of Fe, Mn, and a small amount of Ti, were removed from the total mass of the ore, so the concentrations

of these elements appear to increase, as shown by the data in Table 14 and Figures 32 - 35.

The solubilities of the traces elements in ilmenite as present above are understandable from the chemical properties of the elements and their compounds. These elements, when co-existing in ilmenite, will be in the form of oxides or oxymetallates. Iron (III) oxide is soluble in acid and strong base. Manganese dioxide is inert to most acid except when heated. In the leach solution at boiling point of hydrochloric acid Fe and Mn oxide or oxymetallates are readily soluble. The oxides or oxymetallates of the other elements (Y, Zr, Nb, Sn, and W) are considered refractory and resist attack by acid (Cotton and Wilkinson, 1972). For example, SnO₂ is insoluble in water and acid or alkali but dissolves readily in fused alkali hydroxides. The pentaoxide of niobium does not attack with acid except conc. HF. The trioxide of tungsten is acidic and dissolves in aqueous alkali and it is insoluble in water (Greenwood and Earnshaw, 1984). This would explain the phenomena observed in this study.

4.1.1.1 Temperature

From the data in Table 3, it was found that use of low temperature took longer leaching time and the residue after leaching was still black. The intensity of the color of the beneficiated product is related to the residual iron content in the product. The higher the residual iron content, the darker the color, indicated that it had the amount of Fe remain in the residue more than that in the residue leached from higher temperature. Similarly, Olanipekun (1999), van Dyk, et al., (2002) and Amer (2002) concluded that the dissolution rate of Fe was significantly influenced by the temperature of the acid solution. Lanyon, et al., (1999) also reported the rate of iron

extraction was increased by raising the leach temperature until to the azeotropic boiling temperature (108° C).

4.1.1.2 Ilmenite to acid ratio

From the results in Table 4, it was found that the leaching efficiency depended on initial ilmenite to acid ratio. When lower amount of ilmenite was used, i.e. lower ilmenite to acid ratio, the residue after leaching was lighter in color. From the result of quantitative analysis by WDXRF in Table 14 at 5 g and 10 g ilmenite, Am5 and Am10, both residues had less amount of Fe remaining in residue than that of 15 g ilmenite, Am15 (as shown in Figure 30). On the other hand, between Am5 and Am10, the latter (Am 10) had less amount of Fe remained in the residue than that in Am5. Accordingly, in the experiments conducted by Jackson and Wadsworth (1976, quoted in van Dyk, et al., 2002) at initial acid to ilmenite mole ratio ranging from 300:1 to 700:1 both iron and titanium tended to go into solution, while in the experiments conducted by Sinha (1984, quoted in van Dyk, et al., 2002) at initial acid to ilmenite mole ratios of 1:1 to 1.1:1, mainly iron was dissolved. van Dyk, et al., (2002) concluded that at higher initial acid to ilmenite mole ratios will allow much more titanium to dissolve from ilmenite. The initial acid to ilmenite mole ratios used in this study were shown in Table 16.

4.1.2 NaCl effect (method B)

The purpose of adding NaCl was to increase Cl ions in the reaction medium. The Cl ions should combine with Fe²⁺ or Fe³⁺ in the ore and, therefore, help removing iron from the ore. From the results shown in Table 5, it was found that leaching ilmenite ore by method B1 and B2 could reduce leaching time from method A

if the same initial amount ilmenite used. The EDXRF spectrum of residue after leaching by method B1, Figure 14, showed that the major elements present in residue were Ti, Fe, Mn and W which were the same as found in the residue from method A. The amount of Fe in residue from method B1, however, was less than that in method A as shown in Figure 36.

Table 10 1110 illiant acts to illimite light 14000.			
Wt. ilmenite	Acid volume	Acid to ilmenite	
(g)	(mL)	mole ratio	
5	600	~ 219 : 1	
10	600	~ 110 : 1	
15	600	~ 73 : 1	
20	600	~ 55 : 1	

Table 16 The initial acid to ilmenite mole ratios.

The results of quantitative analysis by WDXRF in Table 14 showed that method B and method A worked similarly in removing Fe and Mn from the ore. However, concentrations of Fe and Mn remaining in residue from method B were less than that from method A.

In mixing ilmenite ore with NaCl (B1), increasing NaCl from 5 g, B1Na5, to 15 g, B1Na15, decreased leaching time from 48 to 42 hours and the concentration of Fe in the residue dropped from 6.25 % to 5.70 %, respectively.

^{*} The mole of acid that is introduced into the experiment is calculated from the acid concentration and the volume of acid that used in the experiment. In ilmenite calculation is assumed that the whole sample consists of FeTiO₃ (molar mass 151.75 g/mol). The mass of ore and the molar mass of FeTiO₃ are then used to calculate the moles of ilmenite.

Adding 15 g NaCl directly into hydrochloric acid leachant (B2), although the same amount of NaCl with B1Na15, the iron content in the residue was less than in B1Na15 as shown in Figure 36. To explain this result, it was possible that, in the mixing ilmenite ore with NaCl, some NaCl inside ilmenite ore might not dissolve completely into the leachant. In the case of adding NaCl directly into hydrochloric acid (B2), as shown in equation (1) at the end of leaching, there were FeCl₂ and free HCl equilibrium in the flask. Thus, increasing Cl would help force the reaction to the right hand side of equation (1) yielding more FeCl₂.

In the same way, sodium chloride assisted the leaching of a pre-reduced ilmenite and allowed Ramakrishma et al., (1988, quoted in Ogasawara, 2000) to obtain their synthetic rutile. Accordingly, Ward, et al., (1999) reported the catalytic effect the chloride ion, in the form sodium chloride, had on the leaching ability at any given pH. Sinha (1980, quoted in Lanyon, et al., 1999) proposed that adding ferrous chloride to the liquor had several advantages in hydrochloric acid leaching processes for upgrading ilmenite. One of these is that ferrous chloride increases the rate of extraction of iron by raising the chloride activity, although the high concentrations of ferrous chloride would be expected to decrease the rate of leaching because it is one of the products of the leach, previous studies have observed only the beneficial effect on the rate of extraction.

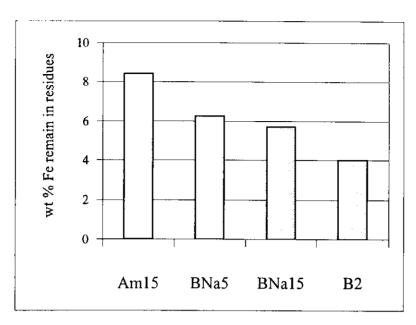


Figure 36 Concentrations of Fe remaining in residues from method A and B. (Data taken from Table 14).

4.1.3 H₂O₂ effect (method C)

The effect of H_2O_2 on the leaching of ilmenite ore by hydrochloric acid was studied by using H_2O_2 solution to oxidize the metallic iron in the porous ilmenite to iron(II)ions (Ward, *et al.*, 1999). It was well known that H_2O_2 is strong oxidizing agent. In addition, H_2O_2 solution also oxidized Fe^{2+} to Fe^{3+} .

Pearson's principle, hard acids prefer to bind to hard bases and soft acids prefer to bind to soft bases, was used in this experiment. Fe³⁺ and Fe²⁺ were classified as hard acid and borderline acid, respectively, while Cl⁻ was classified as hard bases (Huheey, 1978). Then Cl⁻ prefers to bind to Fe³⁺ more than Fe²⁺. In the opposite, many processes roasted ilmenite before leaching to reduce Fe³⁺ to Fe²⁺. The reason for converting the ferric oxide in the ore to ferrous oxide was that the latter is known to be

more easily removed than the ferric oxide in the subsequent acid leaching step (Baldwin, et al., 1988).

From the results in Table 6, it was found that increasing the infusion times in H_2O_2 solution from 1 hour (Ci1) to 24 hours (Ci24) yielded the product in difference colors, the Ci24 was lighter in color than Ci1. As seen here, infusion in H_2O_2 solution for 24 hours helped lower residual Fe content in the product. So we can see that oxidizing metallic iron to iron (II) ions and iron (II) ions to iron (III) ions help increase leaching efficiency. From the WDXRF results in Figure 30 and Table 14, it was seen that this method works in the same way as method A and method B in removing Fe and Mn from the ore. Figure 37 compared the residual Fe contents in the residues from method A, B, and C. We can see that method C was the best among of three methods.

In addition, from the results in Figure 31 and Table 14, it was found that the amount of Mn remained in the residue was lower than the residues from method A and B. This is because manganese dioxide was well dissolved in acid in the presence of hydrogen peroxide (Jiang, et al., 2003).

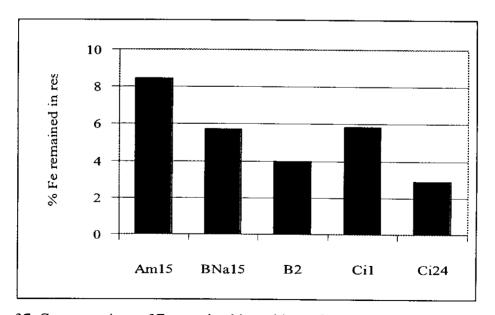


Figure 37 Concentrations of Fe remained in residues after leaching by method A, B and C. (Data taken from Table 14).

4.1.4 Method D

From the data in Table 7, it was found that, when compared with residues from method B and C, the residue from method D2 was buff color but the residue from method D1 was rather black. This indicated that the amount of Fe remained in residue from method D2 was less than method B and C but residue from method D1 was higher. The quantitative analysis by WDXRF (Table 14 and Figure 30) of these residues can be shown graphically in Figure 38. This result indicated that method D2 was a good method to combining both the effects of NaCl and H_2O_2 .

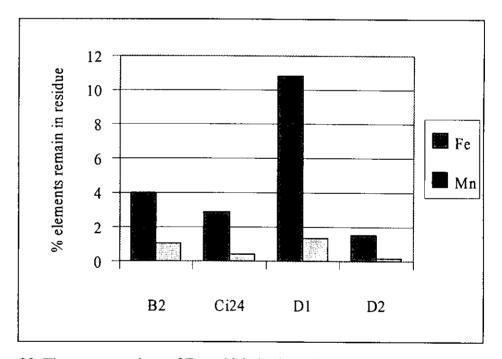


Figure 38 The concentrations of Fe and Mn in the residues from method B, C and D. (Data taken from Table 14).

All methods (A, B, C and D) had some white precipitate formed in the round bottom flask. In method D1 the white precipitate formed most. The EDXRF spectrum in Figure 17 showed that this white precipitate had Ti as the main composition with a

trace of Fe, Mn, and W. The white precipitate that formed in round bottom flask was obtained from the supply of oxygen to the aqueous solution (by bubbling air or pure oxygen) would provide titanium subsequent re-oxidation to TiO₂ while iron is kept in solution (Ogasawara, 2000). Chen. et al., (1997) and Mahmoud, et al., (2004) also reported, depending on the type of ilmenite and process conditions used, some titatium value would be dissolved in the hydrochloric acid as titanium chloride. Then under the influence of heat, the latter would hydrolyze into and precipitated out as titanium oxide hydrate fines or slime of very fine particle size in colloidal state.

4.1.5 Method E

From the data in Table 7, it was found that the residue after leaching by method E was dark fine granule indicating high residual Fe content and used a long leaching time. It could be described as the sintered glass used in this method was high, the leachant vapors condensed into liquid and slowly fell back into the sintered glass. It took long time to fill up the entire sintered glass. The concentration of leachant in the sintered glass was gradually change make it slowly extracts the Fe in ilmenite ore.

4.1.6 Double leaching with fresh acid

From the data in Table 7, it was found that the residue after leaching was lighter in color than residue from one step leaching in method A and C. This indicated that the Fe remained in the residue after leaching by method A and C could be leached out further. From the quantitative analysis by WDXRF (Table 14 and Figure 30), it can be seen that the concentrations of Fe dropped from 8.43 % (Am15) and 2.90 % (Ci24) to 1.95 % (AA) and 0.88 % (CA) as shown in Figure 39. The recent method for

producing synthetic rutile disclosed by Balderson, et al., (1999) also proceeds to a multiple stages leaching.

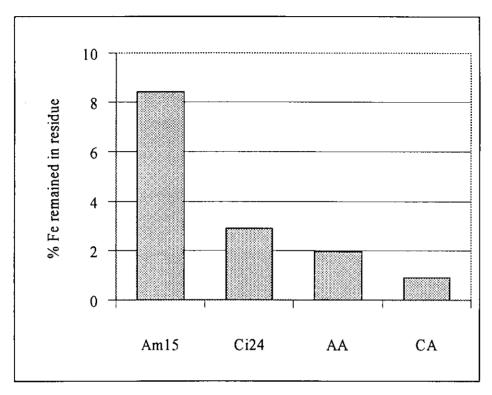


Figure 39 Concentrations of Fe remained in the residues in the double leaching with fresh acid study. (Data taken from Table 14).

4.1.7 Method F

From the data in Table 7, it can be seen that the residue after leaching was lighter in color than the residue from method A (Table 3). The amount of Fe remained in residue from method F was lass than those from methods A, B, and C, indicated that this method was the best method, in this work, for leaching ilmenite ore by hydrochloric acid because of its high leaching ability for removing Fe from ilmenite ore (Figure 40) and used shortest leaching time of all methods as in Table 17. It can be ascribed to the high leachant temperature that was 112°C in method F compared to

105°C in method A. Lanyon, et al., (1999) and van Dyk, et al., (2002) reported the maximum rate of iron extraction occurred near the maximum boiling temperature. Lonyon, et al., (1999) used a glass fluid bed reactor as shown in Figure 4 and the details described in page 15. van Dyk, et al., (2002) conducted their experiments in a 1-1 batch fluidized leach column, fitted with a reflux condenser. In addition, this study employed the soxhlet extraction unit to facilitate the leaching, the higher leachant temperature increased the rate of evaporation and condensation so the rate of extraction was also increased. Accordingly, in the experiment conducted by Mahmoud, et al., (2004) the optimum temperature is selected as 110°C for acid leaching of ilmenite ore in a 250 cm³ three necked glass reactor provided with a reflux condenser and a mechanical agitator with Teflon-coated stirring rod.

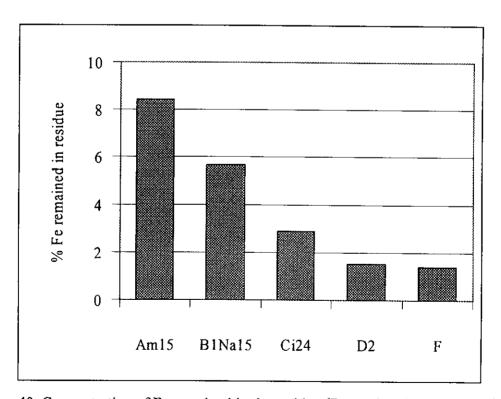


Figure 40 Concentration of Fe remained in the residue (Data taken from Table 14).

Table 17 Leaching time used in each method at the same ilmenite ore to acid ratio.

Method	Leaching time (hrs)
A	65
B1Na15	42
B2	50
Ci24	65 + 48
D1	48 + 48
D2	48 + 48
F	24

4.1.7.1 Leachant temperature

From Table 8, it was found that higher leachant temperature increased the quantity of removed Fe and Mn (as shown in Figure 41). In a similar experiment, method A (4.1.1.1) also showed the same results. Since this study employed the soxhlet extraction unit there were two crucial points to measure the temperature of the leachant, one in the round bottom flask, the other in the soxhlet unit above the flask. The increase temperature of the leachant in round bottom flask to near boiling point of leachant increased the rate of evaporation and condensation and helped increase rate of leaching.

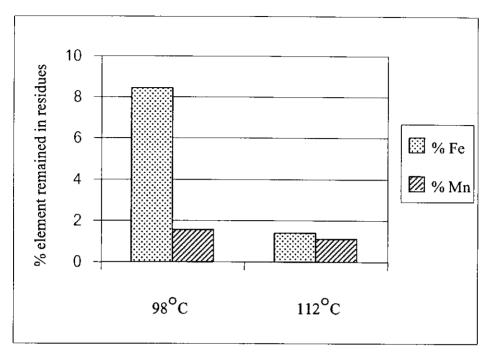


Figure 41 The concentrations of Fe and Mn remained in residues from method F in the effect of leachant temperature.

4.1.7.2 The outer side of soxhlet temperature

From the results in Table 9, it was found that the leaching efficiency of Fe depended on the outer side of soxhlet temperature as shown in Figure 42. As seen here, the amount of Fe remained in the residue first sharply decreased with increasing the outer side of soxhlet temperature until 100°C, after that it slowly decreased and remained constant at 120°C - 130°C. From this result, it can be concluded that, the appropriate temperature range for the outer side of soxhlet temperature was between 100 - 120°C, and 120°C was the most appropriate since the amount of Fe in the residue was the lowest. Other rationale would be the same as given in 4.1.1.1. The amount of Mn appeared to be less affected than Fe probably due to its low concentration originally.

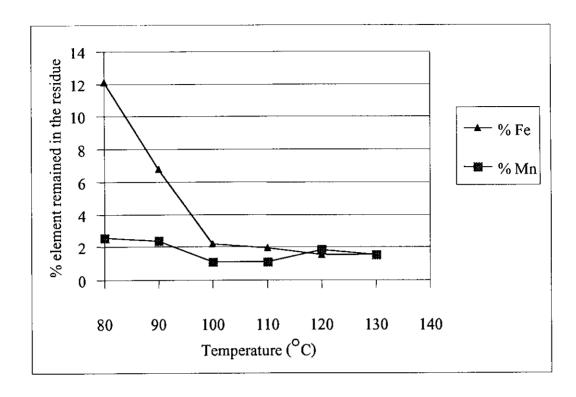


Figure 42 The relation between the amount of Fe and Mn remained in residue from method F and outer side of soxhlet temperature.

4.1.7.3 Initial ilmenite to acid ratio

From the results in Table 10, it was found that the amount of Fe remained in the residue after leaching depended on the initial ilmenite ore to acid ratio but the Mn was not dramatically change as shown in Figure 43. As seen here, the amount of Fe remained in the residue when used ilmenite ore 10 and 15 g were close together and increased when 20 g ilmenite ore was used. Other explanations were the same as in 4.1.1.2.

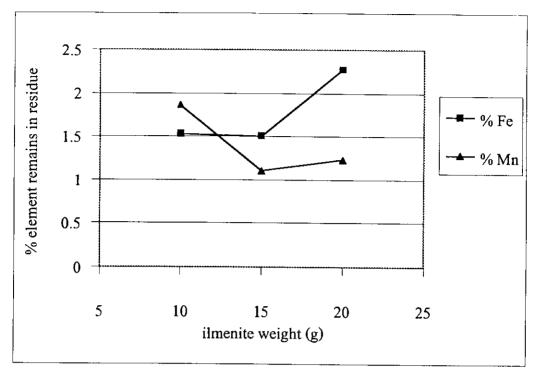


Figure 43 The relation between the amount of Fe and Mn remained in residues from method F and initial ilmenite used.

4.1.7.4 Leaching time

From the results in Table 11, it was found that the amount of Fe remained in residue after leaching depended on the leaching time as shown in Figure 44. As seen here, the amount of Fe remained in the residue first decreased with increasing leaching time up to about 24 hours after which it remained almost constant. For Mn it was rather constant with the increasing leaching time. At this time it was the same time as the solution in soxhlet turns to colorless. This indicated that nothing more is being extracted from the ore by the acid. The experiment conducted by Wongnawa, et al., (1997) were leached ilmenite with 125 mL of a mixture of concentrated HClethylene glycol at the boiling point of the leaching solution and leaching time at 70 to 120 hours. In their experiment, concentrations of Fe and Mn drop from 20.58 % and 1.57 % to <0.78 % and <0.11%, respectively. Lanyon, et al., (1999) also reported the

extraction of Fe continuously up to 72 hours and found that the rate of extraction was very slow after 24 hours.

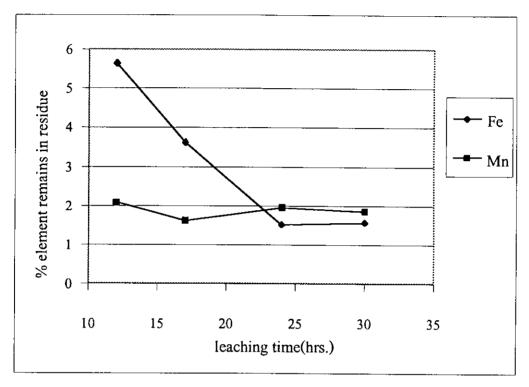


Figure 44 The relation between the amount of Fe and Mn remained in residues from method F and leaching time.

4.2 Measurement of temperature at the soxhlet

From Table 12, it was found that the experiment A (no cover on the soxhlet), when increases the temperature around the soxhlet to 130° C, the water temperature in soxhlet was lower than water boiling point. The heat was lost due to water evaporation. In experiment B (cover the soxhlet with beaker) the cover protected the water evaporation. From the results in Table 12, it can be seen that increasing temperature at outer side of soxhlet from 100° C to 130° C, the water temperature in soxhlet was highest at 100° C and remained constant there because it is the boiling point of water.

In the real leaching experiment the temperature of leachant in the soxhlet may be lower than what was found here due to the soxhlet reactor was joined with condenser that has cooling water and solution condensed into soxhlet chamber.

4.3 Determination of acid concentrations

From the results in Table 13, it can be seen that at the reflux time of 2 hours, the acid concentration in method A decreased more than that in method F. At the reflux time 45 hours in method A, the acid concentration in soxhlet was lower than that in round bottom flask. This indicated that some HCl vapor had been lost during it condensed back into the soxhlet so the acid concentration in soxhlet decreased. Because method A employed air flow into the reactor, the air bubble flew into leachant and carried HCl vapor out off the reactor, resulting in lower acid concentration. Therefore, method A has lower leaching efficiency, high residual Fe content in product.

4.4 X-ray powder diffraction pattern (XRD)

The XRD spectrum of starting ilmenite ore (Figure 19) shows that the most intense refractions in the diffraction pattern matched the standard data of synthetic ilmenite, $FeTiO_3$ and synthetic cassiterite, SnO_2 , indicated its main composition is ilmenite with a trace of cassiterite. This is in agreement with the WDXRF data, which show that it has Sn in ilmenite ore (Table 14). Figures 20-22 show the x-ray diffraction pattern of the residue after leaching by methods AA, CA, and B1, respectively. They show the peaks of the rutile and cassiterite. This result indicated that the mixing NaCl with ilmenite ore or infused residue in H_2O_2 solution did not affected to the yield phase.

The x-ray diffraction pattern of the white precipitate formed in round bottom flask after leaching ilmenite ore by method D1 (Figure 23) shows the peak of the anatase. Clark (1968) explained that from boiling sulphate solutions TiO₂ was precipitated in the anatase form whereas from boiling chloride solutions rutile was separated. In this study, it is possible that the influence of manganese ions induced the TiO, precipitated in anatase form. The influence of the cations on the structural and textural properties of the TiO₂ can be explained based on the changes caused by the cations on the defect structure of the TiO₂ lattices; these changes are strongly dependent on the charge and size of the cation. If a charge of cation was +4 or lower, can reduce or increase the oxygen vacancy concentration in TiO2 depending on their position in the lattice, and as such, can either go into the interstitial or substitutional positions, replacing Ti ions. If cations such as Mn²⁺ replace Ti ions, the oxygen vacancies increase, but if they are put into the interstitial position, the oxygen vacancy concentration decreases. The stabilization of anatase phase can be explained assuming that Mn2+ ions are interstitially incorporated into the TiO2 structure, promoting a decrease of oxygen vacancies (Arroyo, et al., 2002). In addition, Amer (2002) reported TiO, then reacted in the presence of sodium hydroxide to form sodium titanate and subsequently was leached with 30 % HCl also yielding the anatase.

Based on above experimentaly results, it can be explained that the process employing the soxhlet extraction was useful in upgrade ilmenite ore by hydrochloric acid leaching. The temperature, initial ilmenite to acid ratio, NaCl, and H_2O_2 were affecting the leaching efficiency. The appropriate leaching time corresponded to the point that the solution in the soxhlet unit turned to colorless. The residues after leaching from every method were mixtures consisting of rutile and cassiterite with Fe content 1.42 % in the optimum condition. The white precipitate formed in round bottom flask came from the hydrolysis of titanium species that was soluble in the

leachant. This white precipitate was anatase due to the influence of manganese ions. The process is applicable for higher grade ilmenite ores which will produce higher quality synthetic rutile.