

Received July 23, 2013; reviewed; accepted November 18, 2013

EFFECT OF OXIDATION ROASTING ON ILMENITE FLOTATION

Akbar MEHDILO, Mehdi IRANNAJAD, Bahram REZAI

Department of Mining and Metallurgical Eng., Amirkabir University of Technology, Tehran, Iran.
iranajad@aut.ac.ir

Abstract: In this study, the oxidation roasting was used to modify the ilmenite chemistry to improve the collector adsorption and ilmenite flotation behavior. The results indicated that the increase of the roasting temperature up to 600 °C increased the ilmenite flotation recovery. The maximum collector adsorption density occurred on ilmenite roasted at 600 °C which resulted in the increase of ilmenite flotation recovery from 73.5% to 91% at pH 6.3. This improvement was attributed to a greater conversion of Fe²⁺ to Fe³⁺ ions and significant decrease in the zeta potential of ilmenite. At the roasting temperatures of 750 °C and 950 °C, ilmenite was converted to rutile and some trivalent iron containing phases such as hematite and pseudobrookite. Under these conditions, the collector adsorption and hence ilmenite flotation improved, however the flotation recovery decreased after the roasting at 950 °C.

Keywords: *flotation, ilmenite, oxidation roasting, surface chemistry, zeta potential*

Introduction

With the consumption of rutile resources, ilmenite plays an increasingly important role in the titanium industry (Zhong and Cui 1987). Ilmenite as a titanate of ferrous iron mineral (Fe²⁺Ti⁴⁺O₃) is one of the major TiO₂ containing minerals from which titanium dioxide and titanium metal is produced (Song and Tsai 1989). Ilmenite structure is somewhat similar to that of hematite but with some distortion in the oxygen layers. Along the direction of the triad axis, pairs of Ti⁴⁺ ions alternate with pairs of Fe²⁺ ions; thus each cation layer is a mixture of Fe²⁺ and Ti⁴⁺ as shown in Fig. 1 (Zhong and Cui 1987; Deer et al. 1991; Fan et al. 2009).

The conventional methods used in the processing of ilmenite ores are gravity separation, high-intensity magnetic separation (HIMS), electrostatic separation or a combination of them. In some ores, ilmenite is freely disseminated in the gangue, and is not effectively separated from the associated gangue minerals using these separation methods. Froth flotation as a physico-chemical separation process is an effective tool

for separating fine disseminated particles (Song and Tsai 1989; Fan et al. 2009; Pownceby et al. 2008; Fan and Rowson 2000a; Fan and Rowson 2000b). However, ilmenite displays poor flotation under conventional flotation conditions in comparison with other oxide minerals such as magnetite and rutile. The poor flotation of ilmenite requires a large dosage of flotation reagents to promote ilmenite surface hydrophobicity and attachment to air bubbles. Even a large dosage of collectors gave flotation recovery of less than 80% for ilmenite (Zhong and Cui 1987; Pownceby et al. 2008; Fan and Rowson 2000a; Fan and Rowson 2000b).

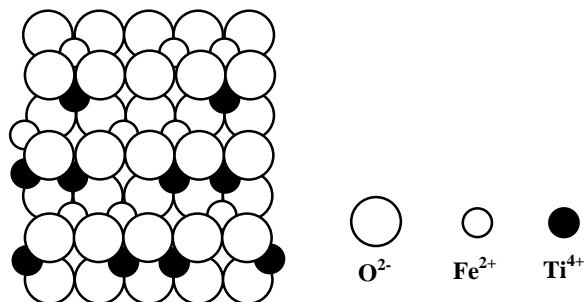


Fig. 1. The arrangement of ions in ilmenite structure on the plane (2110)

Liimatainen and Techn (1977) reported the increase of ilmenite flotation recovery with raising pulp temperatures. Runolinna and Rinne (1960) suggested agglomeration flotation for improvement of fine ilmenite recovery. Gutierrez (1976) offered aeration into ilmenite pulp as an effective method to improve ilmenite flotation recovery. Reverse flotation methods were applied for upgrading of massive ilmenite ore by Behera and Mohanty (1986). Fan and Rowson used the pre-treatment by microwave radiation to modify ilmenite surface properties prior to flotation and to improve ilmenite recovery (Fan et al. 2009, 2000; Fan and Rowson 2000a, 2002). The modification of ilmenite surface properties and improvement of its flotation using lead ions (lead nitrate) were also investigated by Fan and Rowson (2000b). The acid surface dissolution is another method which was used for improvement of ilmenite flotation behavior (Zhu et al. 2011; Bulatovic and Wyslouzil 1999). After the microwave radiation treatment and surface dissolution, Fe^{2+} ions on ilmenite surfaces are converted into Fe^{3+} ions. This conversion reduces the zeta potential of ilmenite and increases the chemical adsorption of oleate ions significantly (in the presence of sodium oleate as collector) (Fan et al. 2009, 2000; Fan and Rowson 2000a, 2002; Liimatainen and Techn 1977; Zhu et al. 2011).

In this paper, the pre-treatment by oxidation roasting was used to modify ilmenite surface properties prior to flotation and to improve ilmenite recovery. For this purpose, the physical and chemical properties of ilmenite surfaces were investigated before and after the roasting at different temperatures.

Materials and methods

Materials

The samples were taken from the Qara-aghaj hard rock deposit located at north-west of Iran. The samples were first crushed and ground under 150 μm , and determined as ilmenite liberation degree. The sample was prepared using sieving and several stages of tabling, and low and high intensity magnetic separation methods. The pure ilmenite sample was washed several times with distilled water, and dried at room temperature. The examination under Scanning Electron Microscopy (SEM) showed ilmenite grains with clean surface and almost free from gangue minerals. The chemical analysis of purified ilmenite is presented in Table 1. The XRD pattern suggests that the purified sample was essentially composed by ilmenite.

Sodium oleate (with 95% purity), and the analytical grade H_2SO_4 (97%) and NaOH (98%) were used as a collector and pH adjustment chemicals, respectively. Additionally, double distilled water was used throughout this study.

Table 1. Chemical composition (wt %) of studied ilmenite samples

composition	TiO ₂	Fe ₂ O ₃	MnO	V ₂ O ₅	P ₂ O ₅	CaO	MgO	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	L.O.I	Total
(Wt %)	46.2	48.6	1.04	0.29	0.24	0.38	2.53	0.19	0.44	0	0.006	0	99.92

Methods

Materials characterization

The chemical composition of the samples was determined using X-ray fluorescence (XRF, Philips X Unique2). The phase composition was analyzed with XPERT MPD diffractometer employing Cu Ka radiation. The microscopic studies for evaluation of textural and morphological features were performed using Philips XL30 model Scanning Electron Microscopy (SEM). The electron microprobe (EMP) analysis was carried out using Cameca SX 100 equipped with five wavelength dispersive (WD) spectrometers. Accelerating voltage of 15 kV and 100 s counting time were used to perform analysis in 25 μm^2 areas. The SEM images were taken under accelerating voltage of 25 kV.

Roasting

The oxidation roasting was performed in a laboratory muffle furnace at 250, 500, 600, 750, and 950 °C for 45 min. The furnace was heated to a predetermined temperature before the sample was introduced. For each temperature, 30 g of ilmenite sample was placed in a ceramic crucible. The oxidation roasting was conducted in the crucible open to air. At the end of the roasting process, the crucible was removed from the furnace, and cooled slowly in air at room temperature.

Flotation experiments

The flotation tests in this study were carried out in a 300 cm³ Hallimond tube. A 2 g of material (unroasted or roasted ilmenite) with a size of 45–150 µm was used for each test. The sample was added to double distilled water and conditioned for 5 min. After this period, the collector was added to the suspension, and the pH was adjusted to the desired value, and a second conditioning stage of 8 min was given to the suspension. The prepared pulp was then transferred to the Hallimond tube where flotation was carried out for 4 min. After the flotation tests, the concentrate and tailing were filtered, dried, and weighed.

Zeta potential measurements

The zeta potential of minerals was measured using a Malvern instrument (UK). First, the samples were ground under 15 µm. Then, the suspension was prepared by adding 50 mg of mineral sample to 100 cm³ of double distilled water containing 2·10⁻³ M KCl as a supporting electrolyte. In order to determine the zeta potential of the sample after the collector interaction, 3.65·10⁻⁴ M sodium oleate was used in the presence of 2·10⁻³ M KCl as an indifferent electrolyte. The resultant suspension was conditioned for 15 min during pH was measured. The pH was adjusted using either NaOH or H₂SO₄ over the pH range of 2–11. The repeating tests showed that a measurement error was about ±2 mV.

Results and discussion

Results

Flotation experiments

The flotation recovery of untreated ilmenite and roasted ilmenite under different temperatures as a function of pH values is presented in Fig. 2. Two recovery peaks appeared on the flotation recovery curve of untreated ilmenite which is in a good agreement with other results reported in the literature (Fan et al. 2009; Fan and Rowson 2000a). The first peak is located at a pH 6.3 where the flotation recovery is 73.5%. The second peak is at about pH 2.5–3.5, which is similar to the flotation behavior of rutile (Bulatovic and Wyslouzil 1999) and the flotation recovery is 42.5%. This indicates that the titanium ions are mainly active sites for reaction with sodium oleate collector at this pH rang. When the pH values were over 8 or less than 2.5, the flotation recoveries decreased rapidly. The reason is for that is due to the strong competition between the oleate ions from the collector and OH⁻ ions present in the pulp on the ilmenite surface in the alkaline aqueous solution. Additionally, most of the active sites on the ilmenite surface should be occupied by OH⁻ ions with the increase of alkalinity. In strong acidic solution, the metallic ions dissolved from ilmenite surfaces, and there were no active sites in which oleate ions adsorb (Fan et al.

2009; Fan and Rowson 2000a). The optimum pH for the flotation was located between 6 and 7.

The results indicated that after the oxidation roasting up to 600 °C, the flotation behavior of ilmenite was greatly improved across a wide pH range. The maximum increase in ilmenite flotation recovery was about 50% at pH values ranging from 4.0 to 4.5. After the roasting of ilmenite at 600 °C, ilmenite was floated with a recovery of 91% at pH 6.3. The maximum improvement of recovery at this pH was about 17.5%. In alkaline aqueous solutions, ilmenite flotation recovery was improved slightly. The flotation behavior of ilmenite roasted at 750 °C is very similar to that of unroasted ilmenite. After the oxidation roasting at 950 °C, ilmenite floatability was improved slightly at pH values below 5 but it dropped significantly above this pH value.

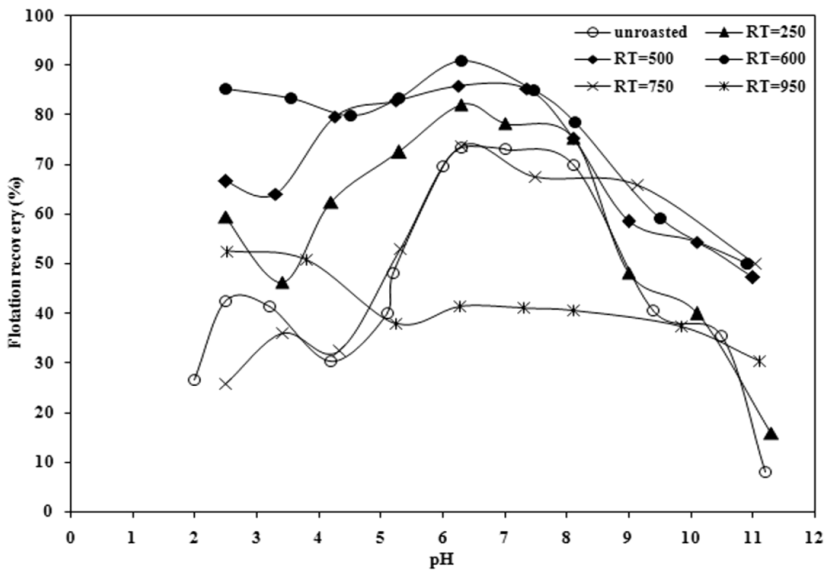


Fig. 2. The flotation behavior of unroasted and roasted ilmenite as a function of pH (sodium oleate concentration $3.65 \cdot 10^{-4}$ M)

Figure 3 shows the effect of sodium oleate concentration on the flotation of untreated and treated ilmenite at pH 6.3. As seen from Fig. 3, the increase of oleate concentration clearly promotes flotation recoveries. The maximum recovery of untreated ilmenite (73.5%) was obtained at $3.65 \cdot 10^{-4}$ M of sodium oleate. When the collector dosage was $3.65 \cdot 10^{-4}$ M, the maximum flotation recoveries of roasted ilmenite at temperatures 250 °C, 500 °C, and 600 °C reached to 82%, 85.9%, and 91%, respectively. After the heat treatment, the ilmenite flotation recovery increased dramatically with a little increase as a function of the collector concentration. Almost 83.3% and 85% of ilmenite floated in the presence of only $1.83 \cdot 10^{-4}$ M of sodium oleate after the pretreatment at 500 °C and 600 °C, respectively. These recoveries were not achieved for untreated ilmenite even at higher dosages of the collector.

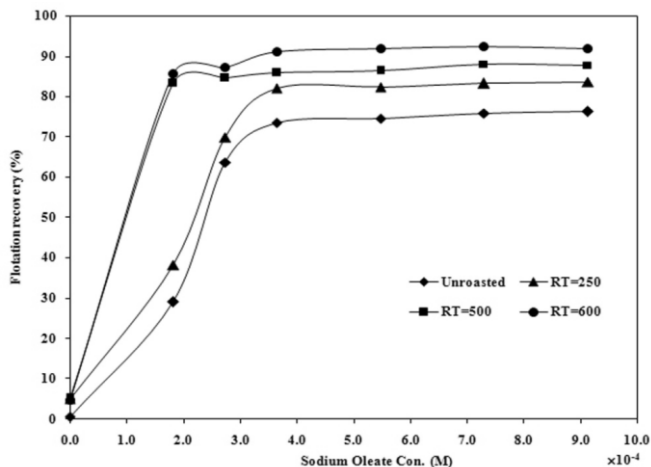


Fig. 3. Effect of sodium oleate concentration on flotation behavior of unroasted and roasted ilmenite (pH=6.3)

Zeta potential

The zeta potentials of roasted and unroasted ilmenite, measured as a function of pH in the presence of $2 \cdot 10^{-3}$ M KCl as a background solution, are shown in Fig. 4. For the unroasted ilmenite, the point of zero charge (pzc) was found to be pH 5.4. The heat treatment process caused to a decrease of ilmenite zeta potential over pH range from 2.5 to 7.5. The pzc of ilmenite shifted to about pH 4.6, 3.9, 2.9, and 3 for treated ilmenite at temperatures 500 °C, 600 °C, 750 °C, and 950 °C, respectively.

Figure 5a shows the zeta potential of different ilmenites in the presence of sodium oleate solution. Using sodium oleate of $3.65 \cdot 10^{-4}$ M, the zeta potential of ilmenite displayed a negative value over a wide pH range from 3 to 11. The pzc of ilmenite shifted to between 2.6 to 3.2. The zeta potential values for raw ilmenite, roasted ilmenite at 600 °C, and roasted ilmenite at 950 °C at pH 6.3 were measured as -66.3 mV, -73.1 mV and -55.9 mV, respectively.

The zeta potential-pH profile of raw ilmenite and ilmenite roasted at 600 °C and 950 °C in the absence and presence of sodium oleate is shown in Figs 5b-d. In all experiments, in order to maintain the ionic strength, hence the double layer thickness at constant level, the concentration of KCl was fixed at $2 \cdot 10^{-3}$ M. The zeta potential measurements in the presence and absence of the collector showed that the collector adsorption resulted in a significant change in the zeta potential. When the oleate was present between about pH 3 and pH 11, the zeta potential of raw ilmenite and roasted ilmenite at 600 °C was considerably more negative than when KCl was present only. This phenomenon took place for ilmenite roasted at 950 °C at above pH 3.2. More negative surface zeta potential is a clear indication of strong adsorption of oleate ions derived from sodium oleate as an anionic collector. The adsorption of the collector in Stern plane causes the pzc (point of zero charge) of ilmenites to shift to pzc (point of zeta potential reversal) at pH 2.5–3.5.

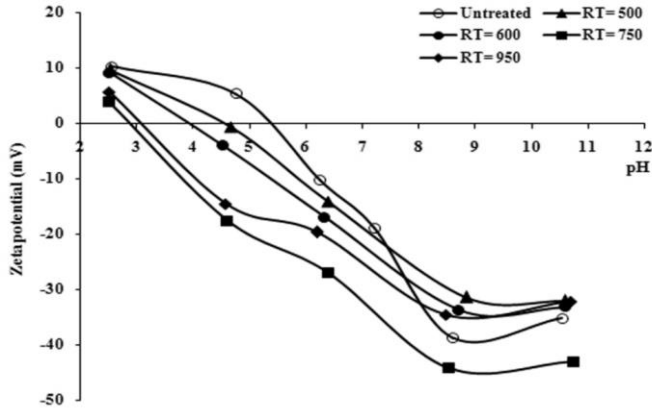


Fig. 4. Zeta potential of unroasted and roasted ilmenite as a function of pH

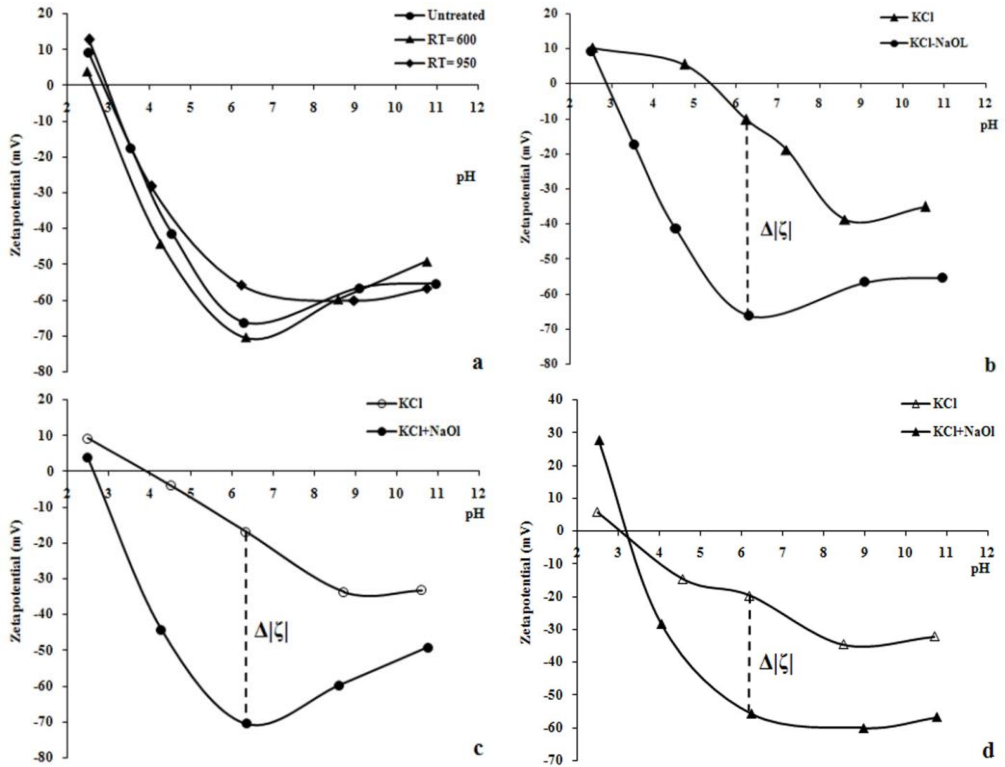


Fig. 5 Zeta potential of different ilmenites as a function of pH in the presence of KCl (2×10^{-3} M) and KCl + NaOl (3.65×10^{-4} M sodium oleate and 2×10^{-3} M KCl). (a) Different ilmenites in the presence of KCl + NaOl. (b) Unroasted ilmenite. (c) Roasted ilmenite at 600°C . (d) Roasted ilmenite at 950°C

X-Ray diffraction

The XRD patterns of ilmenite before and after the roasting at different temperatures are shown in Fig. 6. The main phase present in the sample heated up to 600 °C was ilmenite and hematite formation was not observed. It was found that some of ilmenite phase began to change into hematite and rutile via the oxidation roasting process above 600 °C. When the temperature increased the formation of hematite and rutile became more obvious. After the roasting at 750 °C, the ilmenite phase was almost completely converted into hematite and rutile. This means that the main reactions were the oxidation of ilmenite to the intermediate phase $\text{Fe}_2\text{O}_3\text{-}2\text{TiO}_2$ and decomposition to hematite and rutile at temperatures up to 750 °C. With the increasing of the temperature up to 950 °C, the most dominant phase was ferric pseudobrookite (Fe_2TiO_5) which was formed by combining of hematite and rutile. As seen from Fig. 5, most of the hematite and rutile peaks disappeared. The other weak peaks observed at this temperature are attributed to the negligible amount of metallic iron which is probably due to the local melting phenomena in the ilmenite surface.

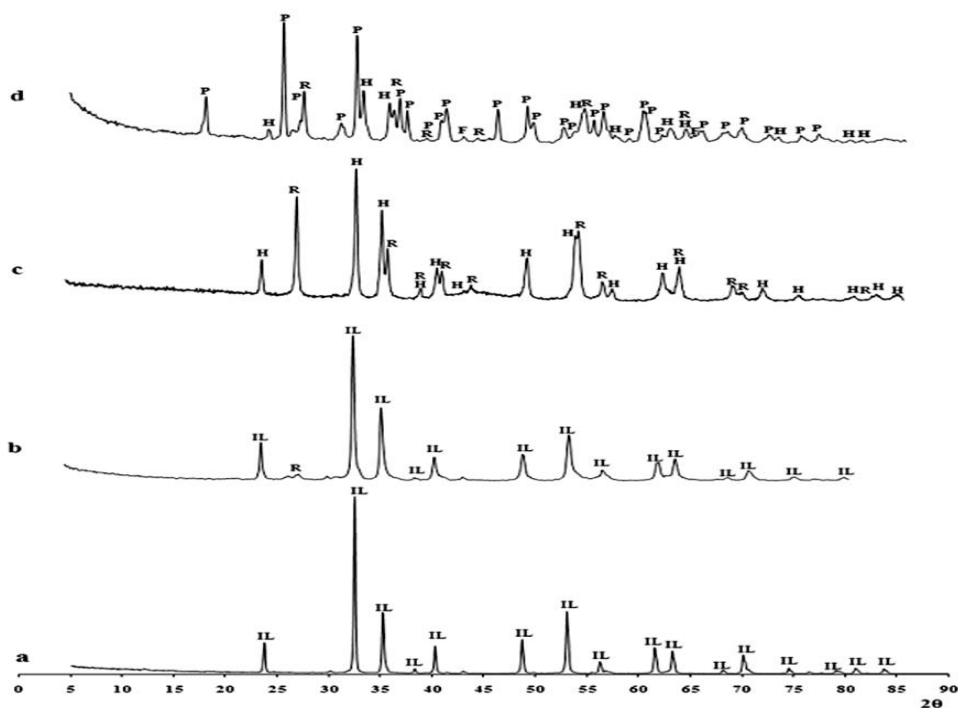
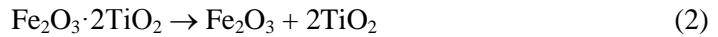


Fig. 6. XRD patterns of unroasted ilmenite and roasted ilmenite at different temperatures (a) Unroasted ilmenite (b) roasted ilmenite at 600 °C (c) roasted ilmenite at 750 °C and (d) roasted ilmenite at 950 °C. (IL: ilmenite, H: hematite, R: rutile, P: pseudobrookite, F: Fe (metallic iron))

Reactions during the oxidation of ilmenite

Oxidation of iron(III) into iron(II) occurs mainly in the surface of ilmenite. Formation of an intermediate $\text{Fe}_2\text{O}_3 \cdot 2\text{TiO}_2$ phase and its decomposition with the formation of hematite and rutile at 600–750 °C is given by:



while formation of ferric pseudobrookite (Fe_2TiO_5) from the combination of hematite with rutile at 750–950 °C by:



Scanning Electron Microscopy (SEM)

The morphology of ilmenite before and after the roasting at different temperatures studied by SEM is shown in Fig. 7. The particles of raw ilmenite are composed of clusters of fine grains which accounts for the high porosity of ilmenite. When the roasting temperature was increased, the conversion of Fe^{2+} ions to Fe^{3+} ones in the ilmenite surface increased. This conversion leads to non-uniformity in the composition and different surface appearance across the ilmenite particles. The oxidation of Fe^{2+} ions to Fe^{3+} appeared as a formation of light particles in the surface. The chemical composition of these particles analyzed by microprobe is presented in Table 2. These results indicated that these particles are mainly composed of Fe^{3+} as active sites in flotation, and the formation of these particles increased with the increasing of the temperature. At the roasting temperature of 600 °C, these particles covered almost the all surface of the grains. At temperatures of 750 °C and 950 °C where ilmenite was decomposed to hematite, rutile and pseudobrookite, the iron content decreased in the surface.

Discussion

The ilmenite surface chemistry is strongly dependent on the solution pH. At pH values less than 3 which most of $\text{Fe}(\text{II})$ ions are dissolved from ilmenite surface, titanium ions are the most dominant cations appearing as $\text{Ti}(\text{OH})^{3+}$ and $\text{Ti}(\text{OH})^{2+}$ species (Fan et al. 2009; Fan and Rowson 2000a). The recovery peak at about pH 2.5 in Fig. 2 is attributed to these titanium hydroxyl complexes as active sites on which oleate ions are adsorbed. In weakly acidic or weakly alkaline solutions titanium ions mainly existed in the more stable form ($\text{Ti}(\text{OH})_4$) while ferrous ions existed in the forms of Fe^{2+} and FeOH^+ are active sites for adsorption of oleate ions (Fan et al. 2009). Because of the solubility product of iron(II) oleate $K_{\text{spFeO}_2} = 10^{-15.5}$ and that of iron(II) oleate $K_{\text{spFeO}_3} = 10^{-29.7}$, FeO_3 is much more difficult to dissolve than FeO_2 . The

oxidation of Fe^{2+} ions on the surface of ilmenite into Fe^{3+} ions facilitates the action on oleate and consequently the increase of its floatability (Zhong and Cui 1987).

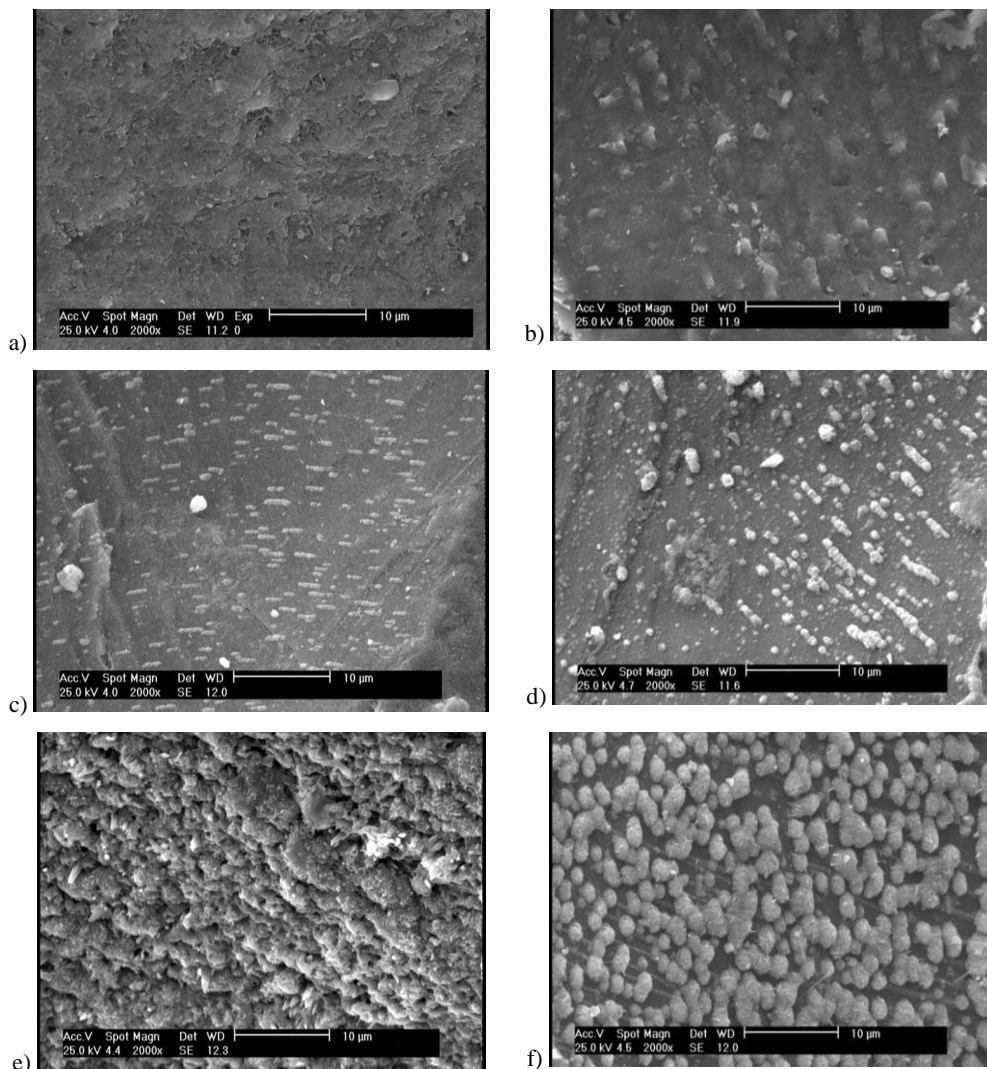


Fig. 7. SEM images of unroasted and oxidation roasted ilmenite. (a) Unroasted ilmenite (b) roasted ilmenite at 250 °C (c) roasted ilmenite at 500 °C (d) roasted ilmenite at 600 °C (e) roasted ilmenite at 750 °C and (f) roasted ilmenite at 950 °C

Table 2. Microprobe analysis of light particles appeared in the surface of ilmenite

Composition (%)	Fe_2O_3	TiO_2	MnO	V_2O_5	P_2O_5	CaO	MgO	SiO_2	Al_2O_3	Cr_2O_3	SO_2	Total
$T = 250\text{ }^\circ\text{C}$	49.34	44.79	1.59	0.19	0.1	0.21	1.51	0.25	0.78	0.29	0.89	99.94
$T = 500\text{ }^\circ\text{C}$	87.13	6.45	1.75	0.58	0.08	0.23	1.42	0.25	0.64	0.36	0.96	99.85
$T = 600\text{ }^\circ\text{C}$	89.09	4.28	1.37	0.23	0.11	0.2	1.62	0.26	1.1	0.11	1.44	99.81

With the increasing conversion of Fe^{2+} into Fe^{3+} ions due to the raising roasting temperature, pzc of ilmenite decreased and consequently its floatability improved significantly. These changes in ilmenite surface properties are in a good agreement with literature (Zhong and Cui 1987; Fan et al. 2009; Fan and Rowson 2000a; Mehdilo et al. 2013). At the roasting temperature of 600 °C, where it has not been taken place any significant phase decomposition in ilmenite structure (Fig. 6b); iron(III) oxide fine particles cover almost all surface of ilmenite and the capability to chemically bind with oleate ions is significantly increased. This is clearly seen from Fig. 5c, in which the zeta potential of ilmenite after the oleate ions adsorption reaches to its highest negative value. At temperatures higher than 600 °C, despite the formation of different phases containing trivalent iron and reduction of zeta potential, the collector adsorption and flotation recovery considerably decreased. This can be due to the changes in ilmenite structure and disruption of cations arrangement on the surface of particles. The decrease of flotation recovery after the roasting at 950 °C in comparing with that roasted at 750 °C can be related to increase of zeta potential (Fig. 4).

The relative adsorption densities of collector in Stern plane can be estimated with the Stern–Grahame equation (Fuerstenau and Shibata 1999). This simplified equation (5) for the case when the collector concentration is constant and the pH or zeta potential is variable (the zeta potential is varied by pH variation, Fig. 5b, c and d) is

$$\Gamma_s = (\text{Constant})C \exp\left(-\frac{zF\Delta|\zeta|}{RT}\right) \quad (4)$$

$$\Gamma_s = (\text{Constant})C' \exp(\Delta|\zeta|) \quad (5)$$

where C is the bulk concentration, R the gas constant, T the absolute temperature, z is the valence of the adsorbing ion, F is the Faraday constant, C' is constant and $\Delta|\zeta| = \zeta_2 - \zeta_1$; ζ_1 and ζ_2 are the measured zeta potential before and after anionic collector adsorption, respectively. The difference in the two values of the zeta potential ($\Delta|\zeta|$) results from the strong adsorption of the collector in Stern plane is a necessary condition for good flotation.

Using Eq. 5 and the results presented in Fig. 5, the relative adsorption densities of oleate (Γ_s) are estimated for unroasted ilmenite and roasted ilmenite at 600 °C and 950 °C at pH 6.3 according Table 3. As seen from Fig. 5 and Table 3 that the relative adsorption densities of oleate at a wide pH range are in good agreement with flotation recoveries shown in Fig. 2.

Table 3. Zeta potential, adsorption densities of oleate and the standard free energy of adsorption at pH 6.3 for unroasted and roasted ilmenite samples

Sample	PZC	PZR	ζ_2	ζ_1	$\Delta \zeta $	Γ_s
Unroasted ilmenite	5.4	2.83	-66.3	-10.3	56.0	$C' \times \text{exp}$ (56.0)
roasted ilmenite at 600°C	3.85	2.6	-73.1	-16.9	57.2	$C' \times \text{exp}$ (57.2)
roasted ilmenite at 950°C	3.05	3.2	-55.9	-19.7	36.2	$C' \times \text{exp}$ (36.2)

Conclusions

The oxidation roasting converts the Fe^{2+} to Fe^{3+} ions on ilmenite surfaces, and enhances the adsorption of oleate, and consequently improving flotation. This conversion also reduced the collector dosage. The maximum collector adsorption density and flotation recovery were achieved after the roasting of ilmenite at 600 °C where the highest conversion of Fe^{2+} to Fe^{3+} ions took place. There was no improvement in the ilmenite flotation at the roasting temperatures above 600 °C. This is probably due to the occurrence of phase decomposition and disruption of the arrangement of metallic cations in the particle surface.

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