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Chemical and mineralogical composition of ilmenite: Effects on physical and surface properties

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ABSTRACT

Some physical and chemical properties of three ilmenite samples: IL-F (purchased from the mineral dealership of A and F Krantz), IL-Q (Qara-aghaj deposit, Iran) and IL-K (Kahnuj deposit, Iran) were studied. The substitution of different amounts of Mg, Mn and V in the crystal structure of ilmenite was identified by microprobe analysis. Different amounts of hemoilmenite exsolution were observed inside all three ilmenite samples using SEM, electron microprobe and X-ray diffraction analysis. Using these analytical techniques, titanite phase was found inside the IL-K ilmenite. The XPS analysis indicated that the contents of Fe^{3+} are 5.66, 5.33 and 3.72% for IL-F, IL-Q and IL-K, respectively which are in good agreement with the amounts of hemoilmenite exsolution. Mg and V contents in the ilmenite crystal structure have a good negative correlation with lattice constants (LC), unit cell volume, crystallinity index, specific gravity and IEP, while the Mn content correlates positively with these parameters. The IEP determined as 4.2, 5.4 and 6.25 for IL-F, IL-Q and IL-K, respectively, has a negative correlation with Fe^{3+} ions and ilmenite floatability. In comparison with magnetic susceptibility, the electrical resistivity of ilmenite has a higher correlation with surface properties (IEP and floatability), lattice constants, and unit cell volume.

1. Introduction

The transformation of a mined material into a marketable product usually requires many separation processes. The different separation processes are based on the differences between physical and chemical properties of minerals including particle size, density, shape, hydrophobicity, capability of adhesion, magnetic properties, electrical properties, ability of adsorbing chemical substances, etc. (Drzymala, 2007). These properties of minerals are used in the separation processes for the determination of a reaction completion, quantifying separator efficiencies, plant tuning, plant feed control and control of final product grade (Cavanough et al., 2006; Drzymala, 2007).

Ilmenite as a titanate of ferrous iron mineral ($Fe^{2+}Ti^{4+}O_3$) is one of the major TiO_2 containing minerals from which titanium dioxide and titanium metal is produced (Song and Tsai, 1989). The various properties of ilmenite such as high density, paramagnetism and electrical conductivity have enabled the gravity separation, high-intensity magnetic separation (HIMS), electrostatic separation or a combination of all three to be conventional methods for processing of ilmenite ores. In some ores however, fine ilmenites are freely disseminated in the gangue minerals, and are not effectively separated using the above separation methods. Froth flotation as a physico-chemical separation process is an effective tool for these cases (Fan and Rowson, 2000a,b; Fan et al., 2009; Zhu et al., 2011).

The ilmenite structure is similar to that of hematite, but with some distortion in the oxygen layers. Along the direction of the triad axis, pairs of Ti ions alternate with pairs of Fe^{2+} ions; thus each cation layer is a mixture of Fe^{2+} and Ti^{4+}. The formula of ilmenite may be more fully expressed as (Fe, Mg, Mn)TiO_3 with only a limited amount of Mg and Mn (Deer et al., 1991). The elements such as Mn, Mg, and Cr may substitute for Fe or Ti in the original ilmenite lattice, while elements like Al, Si, Th, P, V and Cr are commonly incorporated into the ilmenite grains during chemical weathering. Prolonged exposure to oxidizing and/or acidic environments may also cause changes to the chemistry of ilmenite grains with elements such as Fe and Mn being significantly leached (Pownceby et al., 2008). These impurities could have negative effects on TiO_2 production processes (Chernet, 1999a; Pistorius, 2008).

The physical and chemical properties of ilmenites from different sources may not be essentially the same, and therefore, the properties determined for a particular ilmenite mineral should be considered as pertaining only to that mineral. Thus, if the wanted properties of the ore constituents are known, it is possible to define

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the suitable condition in which specific minerals are made to selectively separate from associated gangue minerals. It is therefore of vital importance to understand the effect of chemical and mineralogical composition on the different properties of a mineral. The impurities in the ilmenite structure and/or composition can affect its different properties and consequently the different separation processes.

In this work, the relationship between ilmenite composition and its different properties is studied; and their effects on different properties such as crystal structure, lattice constant, specific gravity, magnetic susceptibility, electrical conductivity or resistivity, zeta potential, floatability and surface reaction with organic reagent is investigated.

2. Materials and methods

2.1. Materials

Three ilmenite samples from different localities were used in this study. The IL-K and IL-Q ilmenite samples were taken from the Kahnuj deposit (south of Iran) and Qara-aghaj hard rock deposit (northwest of Iran), respectively. The IL-F sample was received from a laboratory of mineralogy as a hand sample (purchased from the mineral dealership of Friedrich Krantz). The samples were crushed and ground to a size 150 μm; and were then purified using steps of sieving, several stages of tabling, and low and high intensity magnetic separation methods. The pure ilmenite samples were washed several times with distilled water and dried at room temperature. Examination under a binocular microscope showed ilmenite grains with a clean surface, and almost free from gangue minerals.

Sodium oleate (with 95% purity) was used as a collector in flotation experiments and FTIR analysis. Analytical grade H_2SO_4 and NaOH were used for pH adjustment and double distilled water was used throughout this study.

2.2. Methods

2.2.1. 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 structural features were performed using the Philips XL30 model scanning electron microscopy (SEM). The electron microprobe (EMP) area and point analysis of the samples was carried out using a Cameca SX 100 instrument equipped with five wavelength dispersive (WD) spectrometers. The phases finer than 5 μm were analyzed by microprobe point analysis. In order to prepare the samples for SEM and EMP analysis, the grains of ilmenite were set into a mould (typically 30 mm diameter) with epoxy resin to form a hardened block. The block was then ground down to expose a representative cross section of particles which was subsequently polished and then coated with carbon before being presented to the SEM and EMP. Accelerating voltage of 15 kV and 100 s counting time were used to perform EMP point analysis.

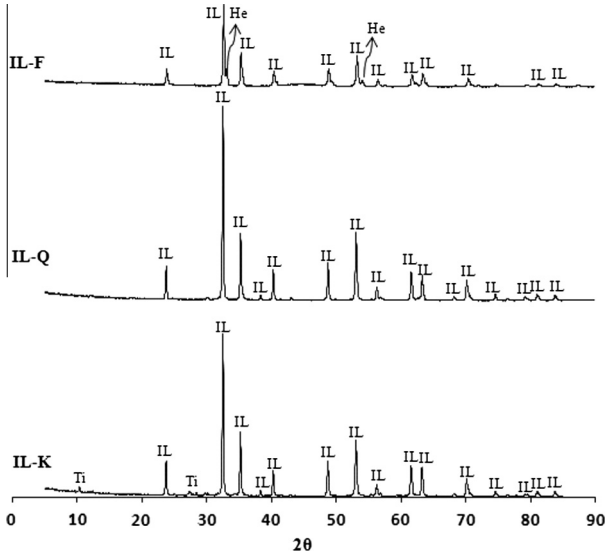

Fig. 1. XRD pattern of the samples (IL = Ilmenite, He = Hematite, Ti = Titanite).

2.2.2. Determination of specific gravity

A pycnometer with a volume of 50 cc (v) and its glass stopper were carefully cleaned with soap and water and then rinsed with a small amount of acetone. The dried pycnometer and stopper were weighed on the analytical balance (m_1). The pycnometer was filled fully with distilled water. The stopper was inserted and tapped the sides gently to remove the air bubbles. Then the sides were dried and the full pycnometer was weighed on the analytical balance (m_2). The emptied and cleaned pycnometer was filled with the determined mass (m_s) of sample material, and the rest of the pycnometer filled with distilled water and weighed again to obtain the combined mass of the sample with the water (m_3). Finally, the measured specific gravity (ρ_m-g cm^{-3}) of the ilmenite samples were determined using Eq. (1).

$$\rho_m = \frac{m_s(m_2 - m_1)}{v(m_2 - m_3 + m_s)} \quad (1)$$

2.2.3. XPS analysis

XPS spectra were measured with a Specs EA10 X-ray photoelectron spectroscope to study the distribution density and binding energy of the elements on the mineral surface. The XPS analysis was carried out with an Al Kα X-ray source at 1486.6 eV and the peaks deconvoluted using SDP software (version 4.1) with 80% Gaussian-20% Lorentzian peak fitting. The XPS measurements were performed inside the analysis chamber operating in a high vacuum of about 10^{-7} Pa. Binding energies were calibrated using characteristic C1s carbon peak (C1s = 284.7 eV).

2.2.4. Zeta potential measurement

The zeta potential of mineral suspension was measured using a Malvern instrument (UK). The samples were ground under 15 μm. The suspension was prepared by adding 50 mg of pure ilmenite to

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

Sample	Location	TiO_2	Fe_2O_3	MnO	V_2O_3	P_2O_5	CaO	MgO	SiO_2	Al_2O_3	Co_3O_4	ZrO_2	Nb_2O_5
IL-F	F. Krantz	43.5	49.8	0.26	0.50	0.018	0.17	4.11	0.03	0.84	0.064	–	–
IL-Q	Qara-aghaj	46.2	48.6	1.04	0.24	0.240	0.38	2.53	0.19	0.44	0.025	0.019	0.018
IL-K	Kahnuj	43.9	45.9	1.15	0.31	0.091	1.51	1.84	3.70	1.19	0.022	0.044	0.012