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Full Length Article

Synthesis of high purity rutile nanoparticles from medium-grade Egyptian natural ilmenite

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ABSTRACT

The Egyptian magmatic ilmenite is classified as a medium-grade ore. The present work is an attempt to produce a high-quality TiO_2 that can be used in several industries from this medium-grade raw material using the mechanical activation, carbothermic reduction, hydrochloric acid leaching and calcination. A mixture from the ilmenite ($FeTiO_3$) and activated carbon was milled for 30 h. This mixture was annealed at 1200 °C for one hour and the product was leached by hydrochloric acid and calcined at 600 °C for two hours. The role of the ball milling was to grind the raw ilmenite to obtain the nano size, and the carbothermic reduction was to reduce all the Fe-Ti phases to a mixture from Fe metal and TiO_2. Leaching procedure was carried out to remove all the Fe metal and obtain a high-grade TiO_2. After leaching and calcination of the milled and annealed mixture of $FeTiO_3$/C under the optimal conditions, TiO_2 nanoparticles with a size of 10–100 nm and purity more than 95% were obtained. The qualifications of the synthesized high purity rutile nanoparticles from the Egyptian natural ilmenite match the conditions of many industrial applications.

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1. Introduction

The mineral ilmenite (nominally $FeTiO_3$) is a huge resource of rutile (TiO_2), which can be used directly as pigment or for the manufacture of welding-rod coatings, ceramics, papers, and in other areas of chemical industry (Diebold, 2003; Mahmoud et al., 2004). White titanium dioxide pigments have been produced by two processes, namely the sulfate process, and the dry chlorination process (Mahmoud et al., 2004). In the sulfate process the ilmenite ores are reacted with concentrated sulfuric acid; it is well known and widely applied, but it is lengthy and costly, and the by-product ferrous sulfate is less marketable (Abdel-Aal et al., 2000; Afifi, 1994), while in the chlorination process the ilmenite ores are chlorinated to form titanium tetrachloride, which then re-oxidized to form pigments; it presently enjoys more favorable economics and generates less waste materials (Mackey, 1994). In recent years many researchers' efforts have been

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directed to produce the synthetic rutile (TiO_2) from the natural ilmenite using the chlorination processes (Mahmoud et al., 2004; Tao et al., 2012) due to the shortage in the natural rutile.

There are several processes for the production of synthetic rutile from ilmenite, most of which fall into one of the following categories:

A. Reduction of the iron content of the ilmenite to the metallic iron followed either by corrosion with oxygen and ammonium chloride (Becher, 1963; Farrow and Ritchie, 1987) or by leaching with sulfuric and hydrochloric acid at elevated temperatures (Natziger and Elger, 1987).
B. Pre-oxidation and reduction of ilmenite followed by hydrochloric acid leaching, Becher process (Sinha, 1972, 1979).
C. Processing of the natural ilmenite by roasting and magnetic separation followed by hydrochloric acid leaching, ERMS process (Walpole, 1997).
D. The reductive leaching for the natural ilmenite using the hydrochloric acid as a leaching reagent and the iron metal as a reductive reagent (Mahmoud et al., 2004).
E. Reduction of the mechanically activated natural ilmenite to metallic iron followed by acid leaching and calcination (Tao et al., 2012).

The last process which includes mechanical activation followed by acid leaching and calcination is the most favorable for the author as it provides high-quality synthetic rutile nanoparticles that have wide industrial applications. Mechanical activation has a very important role in increasing the efficiency of carbothermic reduction and accelerating dissolution process due to several reasons, such as extension of crystal defects and lattice distortions, decreasing the particle size, and increasing the specific surface area (Chen et al., 1997 and Wei et al., 2009). The reduction is employed to reduce the ilmenite completely or partially into iron metal and TiO_2. Acid leaching has an important role in the leaching of the metallic iron from the reduction products; the hydrochloric acid is preferred in leaching as it has some advantages, such as fast leaching, excellent impurity removal and acid regeneration technology (Demopoulos et al., 2008), which can efficiently remove residual iron and other impurities from ilmenite to form synthetic rutile (Mahmoud et al., 2004). Calcination processes have been employed to obtain a highly crystalline product and to remove any carbon impurities from the reduction processes. Only a few works have been carried out on the Egyptian magmatic ilmenite ore to produce the synthetic rutile for the chlorination processes (e.g. Mahmoud et al., 2004). Mechanical activation, carbothermic reduction, hydrochloric acid leaching and calcination methods were applied for the first time on the Egyptian ilmenite ore to obtain high-quality synthetic rutile nanoparticles that can be directly used in industry.

2. Methodology

2.1. *Ore materials characterization*

The magmatic Fe-Ti oxides deposits of Egypt are present in the South Eastern Desert. Among these deposits, there are considerable reserves of ilmenite ore in Abu Ghalaga region. The estimations of the reserves of ilmenite ore in Abu Ghalaga area are variable, ranging from 3 million tons (Holman, 1956), through 10 million tons (Moharram, 1959) to 50 million tons (Mahmoud et al., 2004).

The Abu Ghalaga ilmenite ores can be classified into black (fresh) ore and red (oxidized) ore but our experiments were carried out on the black ore because this ore represents the major reserves in Abu Ghalaga ilmenite open-pit mine.

Phase identification for the ilmenite ore was carried out using the reflected-light microscope and X-ray diffraction. Under the reflected-light microscope, it can be seen that ilmenite grains enclose exsolution bodies of hematite. It also reveals that minute hematite lamellae are segregated within the ilmenite host (Fig. 1a). Small grains of hematite are exsolved and segregated at the borders of ilmenite grains (Fig. 1b). The XRD showed two mineral phases present; these are ilmenite and hematite (Fig. 2). Thus, the ore is mainly formed from ferri-ilmenite together with small quantities of titano-hematite.

a

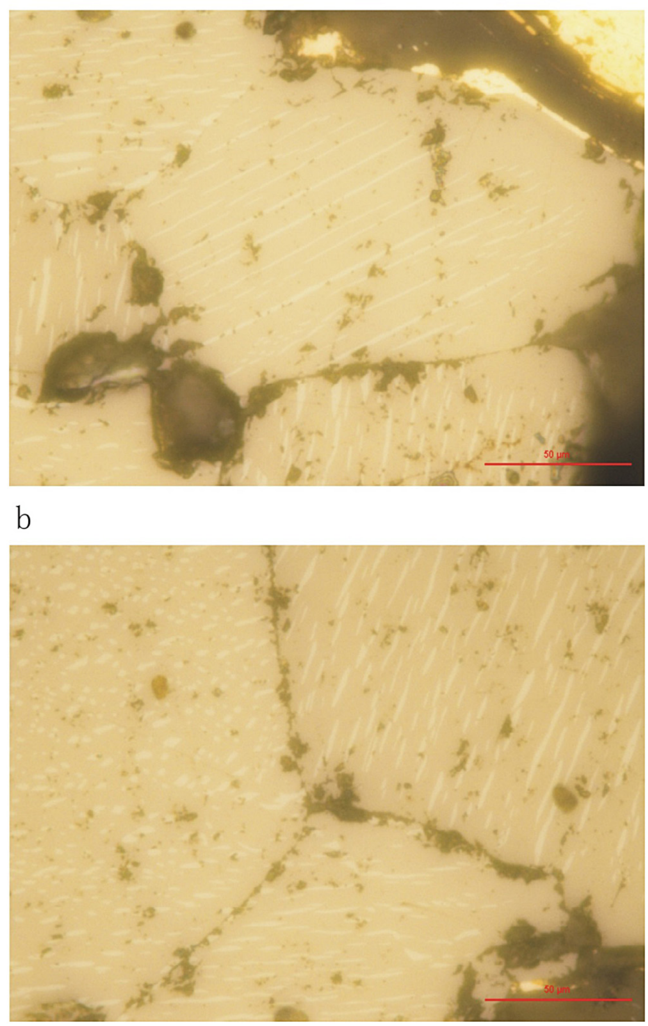

Fig. 1 – Reflected light Microscopic photographs for Abu Ghalaga ilmenite ore showing (a) Minute hematite lamellae segregated within the ilmenite host, and (b) Small grains of hematite are exsolved and segregated at the borders of ilmenite grains.

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