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# Results in Physics

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### ABSTRACT

The detrimental effects of slimes on the flotation of rutile from eclogite ore have been studied in this work in order to improve the separation efficiency of the flotation. The study was performed through particle size distribution measurement and mineralogical analysis. The results have demonstrated that the slimes were enriched in the concentrates, which became seriously with the increase of slime contents in feed ore. The presence of slimes resulted in the involvement of a large amount of omphacite and garnet into the concentrates, deteriorating the flotation selectivity in both of fine and coarse size fraction. Accordingly, de-sliming pretreatment was applied to the flotation, which significantly improved the flotation efficiency of rutile from eclogite ore for increasing TiO<sub>2</sub> grade and recovery of concentrates.

#### Introduction

Rutile is one of the favored minerals for manufacturing titanium white pigment [\[1\],](#page--1-0) high quality welding electrodes [\[2,3\],](#page--1-1) photocatalyst in solar cells  $[4]$ . It is also a good material for bone grafting  $[5]$ .

In world, the major rutile deposits may be divided into four types, including metamorphic, igneous-related, sedimentary and weathered. In China, the sedimentary deposits are economically most important, followed by metamorphic deposits such as the eclogite type. Rutile in sedimentary deposits accounts only 14% of the Chinese reserves of economic sources [\[6\].](#page--1-4) Due to the depletion of easily processed rutile from sedimentary deposits and the increasing demand for titanium, particular focus has been given to rutile-bearing eclogites. The eclogites originated by high-pressure and temperature alteration of Proterozoic, Fe-Ti rich, gabbroic rocks. They were strongly deformed under eclogitefacies metamorphism and thereafter variably affected by retrograde processes [\[7\].](#page--1-5) Eclogite ore is a type of rutile resource with high economic value due to its large-scale reserves, good continuity of ore deposits and easy for exploitation [\[5\]](#page--1-3).

The mineral fines are produced inevitably in the grinding process for adequate liberation of the particles [\[8\];](#page--1-6) mineral fines (slimes) are known to have a detrimental effect on minerals flotation. Slimes could increase the reagent consumption and pulp viscosity. In addition, slimes are liable to get into froth phase via composite particles, slime coatings as well as froth entrainment [\[9\]](#page--1-7). It was reported that the medium density fraction which mainly contained clay-coal composite particles greatly decreased the flotation recovery of the pure coal, but the heavy fraction which mainly comprised clays only depressed the flotation to a much less extent. The authors proposed that the great depression caused by the medium density fraction was due to the partial hydrophobicity of the clay-coal composite particles. They argued that the hydrophobic side of the composite particles could adhere to the coal surface by hydrophobic force, exposing the hydrophilic side to the water [\[10\].](#page--1-8) Due to electrostatic attraction between opposite charged coarse and fine particles [\[11,12\]](#page--1-9), slimes coating on value mineral surface formed a hydrophilic "armor", which reduced the efficiency of bubble–coal attachment [\[10\]](#page--1-8). Thus, the presence of clay slimes decreased the recovery and flotation rate of coarse coal [\[13\]](#page--1-10). In microcrystalline graphite flotation, majority of sericite fines was recovered into concentrates as a result of froth entrainment, partly recovered by entrapment, affecting the flotation selectivity [\[14\].](#page--1-11) The presence of gangue slimes would cause a low flotation separation efficiency in

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#### <span id="page-1-0"></span>Table 1

Chemical composition of the ore sample (wt%).

Component	Na <sub>2</sub> O	MgO	$Al_2O_3$	SiO <sub>2</sub>	$P_2O_5$	SO <sub>2</sub>	K <sub>2</sub> O	CaO
Content	2.03	5.83	12.55	45.84	0.58	0.13	0.22	11.63
Component	ZnO	BaO	Fe <sub>2</sub> O <sub>3</sub>	MnO	SrO	TiO <sub>2</sub>	LOSS	
Content	0.014	0.041	16.51	0.24	0.013	396	0.42	

ilmenite flotation [\[15\].](#page--1-12) A good result can be obtained in rutile flotation when the gravity separation was carried out in advance  $[16]$ . Thereby, the presence of slimes led to low recovery and grade of flotation concentrate [\[17,18\].](#page--1-14)

The objective of this study is to investigate the specific effect of slimes on flotation of rutile from eclogite ore and to approach into the mechanisms of the detrimental effect. Also, a de-sliming pretreatment for improving the flotation efficiency is presented.

# Experimental

#### Materials and reagents

The rutile ore used in this study was obtained from Donghai, Jiangsu province, China. The raw ore was blended and riffled thoroughly to prepare sub-samples for mineralogy analysis. The results of an X-ray fluorescence chemical analysis of the ore sample are presented in [Table 1.](#page-1-0) According to [Table 1](#page-1-0), the raw ore is extraordinarily rich in the elements of iron and silicon, and low in titanium. The  $TiO<sub>2</sub>$  content of this sample is 3.96%, which reveals that it belongs to a low-grade ore.

In addition, X-ray diffraction (Bruker, Braun, Germany) was used to further analyze the composition of the raw ore, with the result shown in [Fig. 1.](#page-1-1) The main gangue minerals associated with rutile are omphacite, garnet, quartz, albite and biotite. As shown in [Table 1](#page-1-0), there are very small impurities in the ore sample, but they cannot be detected using XRD because of their very low contents.

In flotation test, composite collector (Styryl phosphonic acid and noctyl alcohol) was added to induce the hydrophobicity of valuable minerals. Lead nitrate, and sodium fluorosilicate were used as activator and depressant, respectively. The sodium silicate was used as slurry dispersant to prevent the non-selective coagulation of slimes during the flotation. And the frother was terpene. Lead nitrate, sodium fluorosilicate, n-octyl alcohol were analytical grade and were purchased from Sinopharm Chemical Reagent Co., Ltd., China. Industrial grade styryl phosphonic acid (SPA) and sodium silicate were supplied by Chengdu reagent factory, China. In terms of the sample used in this study, no additional pH regulator was added, and the experiments were

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Fig. 1. X-ray diffraction pattern of the sample ore.

performed under natural pH around 4. The reagents used for experiments were prepared daily prior to the flotation tests using tap water.

# Samples preparation

In order to increase the recovery of valuable minerals, raw ore, has to be ground to a much smaller size. However, by doing so, gangue minerals are also ground finely. And both fines (gangue and valuable mineral particles) can experience entrainment [\[18\]](#page--1-15), while the entrainment of fine gangue mineral particles (−45 μm) into the froth can be one of the reasons for the difficulty in obtaining qualified concentrates [\[19\].](#page--1-16) Thus, in this work, the fines (−32 μm) are collectively called "slimes".

## Flotation samples preparation

The raw ore with different slime contents was ground in porcelain ball mill and a sample of 500 g was used each time, and then samples with different slime contents (2.56%, 8.91% and 19.88%, respectively) were obtained. The grades of the as-prepared mineral samples were 4.05%, 4.14% and 3.82%, successively.

#### De-sliming flotation samples preparation

The rutile sample grading 3.96% used in de-sliming flotation was ground to 45% passing 74 μm using a porcelain ball mill at a pulp density of 50% (by weight). After grinding, the materials were then stirred in a container with 20 cm diameter and 2000 ml volume. The deslime yields including 0%, 4.69%, 8.4%, 12.28%, 14.35% and 16.49% were chosen for these tests. De-slime yield was the amount of slime discarded via de-sliming pretreatment.

The procedures for preparing these samples are as follows: (1) The samples were placed into the container and the slurry was adjusted to achieve calibration of 2000 ml by adding tap water. (2) An agitator was then applied to achieve homogeneous dispersion of suspension with a stirring speed of 500 rpm for 10 s. The height from the blender impeller to the bottom of the container was 400 ml. (3) After stirring, settlement is needed. The upper solution was then scraped out by siphonage and 1400 ml of upper solution was aspirated each time. (4) After siphonage, the tap water was then added to pulp and conditioned the calibration to 2000 ml.

The sample with de-slime yield 0% was the ground product of raw ore. The settling time of the remaining five samples were 3 min, 2 min, 1.25 min, 1 min and 0.5 min, respectively. Step 3 to step 4 were required to repeat three times. A flow chart of preparation process is shown in [Fig. 2.](#page--1-17)

#### Flotation tests

To investigate the effects of slimes on the flotation of rutile from eclogite ore, batch flotation tests of samples with different slime contents (2.56%, 8.91%, and 19.88%, respectively) were performed. The flotation concentrates were subjected to determine the particle size distribution, mineral composition and distribution of Ti in different size fractions.

Flotation tests were performed in a XFDII (1.5 L) mechanically agitated flotation machine with an agitation speed of 1750 rpm. First, the samples with different slime contents were conditioned for 3 min; next, lead nitrate (300 g/t), sodium fluorosilicate (800 g/t), sodium silicate (200 g/t), styryl phosphonic acid (600 g/t), n-octyl alcohol (30 g/ t) and terpene were added staged to pulp and agitated for 3 min after each addition; then, the flotation time lasted for 4 min, and the froth product was scraped out as concentrates and the pulp left in cell was tailing; finally, the concentrates and tailing were dewatered, dried and weighed. The flowsheet of the rougher flotation is shown in [Fig. 3.](#page--1-18)

The de-sliming treatment was conducted via siphonage to mitigate the detrimental effects of slimes on flotation. After pretreatment, the flotation tests were performed as described above. The flowsheet of the

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