



Contents lists available at ScienceDirect

International Journal of Mining Science and Technology

journal homepage: www.elsevier.com/locate/ijmst

Comparative studies on phosphate ore flotation collectors prepared by hogwash oil from different regions

Yu Huimei^{a,b}, Wang Huajun^{a,b,*}, Sun Chuanyao^c

^a School of Civil and Resource Engineering, University of Science and Technology Beijing, Beijing 100083, China

^b Key Laboratory of Metal Mine Efficient Mining and Safety Education Ministry, University of Science and Technology Beijing, Beijing 100083, China

^c State Key Laboratory of Science and Technology of Mineral Processing, Beijing General Research Institute of Mining and Metallurgy, Beijing 100070, China

ARTICLE INFO

Article history:

Received 16 January 2018

Received in revised form 18 March 2018

Accepted 1 April 2018

Available online xxxx

Keywords:

Hogwash oil

Flotation collector

Saturated fatty acid

Unsaturated fatty acid

Adsorption

ABSTRACT

In order to compare the differences between variations of phosphate ore flotation collectors prepared by hogwash oils, JZQ-F collectors were prepared by the hydrolysis method using four types of hogwash oils from different regions in China. The components of the hogwash oils were determined using Fourier transform infrared spectroscopy (FTIR) spectra analysis, while the components of the JZQ-F were determined through gas chromatography–mass spectrometry (GC–MS). The flotation effects of the JZQ-F collectors were investigated by flotation experiments and the adsorption characteristics were determined through the quartz crystal microbalance (QCM-D). Results show that the JZQ-F collector from the Ordos area has 27.43% unsaturated fatty acids. JZQ-F collectors from Beijing, Qingdao, and Dongguan areas contain over 62% of unsaturated fatty acids, which can acquire phosphorous concentrates with a the grade of P_2O_5 above 31.96% and the recovery higher than 91.52%. These three collectors have a larger adsorption capacity, faster adsorption rate, and stronger adsorption structure on apatite surface, which contributed to a good adsorption performance and a favorable flotation effect.

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1. Introduction

Due to the growing awareness of hazardous hogwash oil in the current way of being re-used or disposed, the public has paid increased attention to the utilization of hogwash oil [1]. Recently, hogwash oil is mainly used to prepare biodiesel, produce phosphor free washing powders and liquid soaps, prepare stearic and oleic acids, and prepare grease and release agent for concrete products [2–6]. The study on flotation reagent prepared by hogwash oil in recent years focuses on subsequent processing methods, including direct use method, hydrolysis method and ester exchange method. The hogwash oil needs to be purified before the use in such methods [7]. Yang directly used hogwash oil in the flotation process for Huangling coal and found it played a collecting role. Cui et al. synthesized a new collector with hogwash oils, rosin, alkali, surfactant and diesel oil at a certain proportion and achieved chemical emulsification [8,9]. It had an effect on the collection of coal flotation. In a study conducted by Tian et al., the hydrolysis method was used and hogwash oil was stratified, refined, sulfonated and saponified

to prepare anionic collectors, which are used in flotation tests for iron, fluorite and phosphate ore. Sun et al. used hogwash oil to prepare mixed fatty acids [10,11]. It can be changed to mixed fatty acid after edulcoration, acid hydrolysis, sulfonation, and vacuum distillation. After that, a separating flotation test on artificial mixed ore of diaspora and kaolinite was conducted. Zhang used hogwash oil as raw materials to produce mixed fatty acid by using the process of saponifying, salting, washing, acidifying, re-washing and drying. An α -alpha fatty acid derivative flotation reagent M-105 was prepared after 8 h under reaction condition of 120 °C, using red phosphorus as catalyst, and passing M at a steady rate [12]. This mixture was used to conduct a reverse flotation test for an iron concentrate prepared by magnetic separation from Jianshan. An outstanding flotation index was achieved with a concentrated Fe grade of 70.26% and recovery of 83%. Li used ester exchange method to prepare collector under reaction conditions of 60 °C, 6 h reaction time, and using acid as a catalyst for dolomite flotation [13]. The optimum recovery was 55.3%, and the separation efficiency between dolomite and gangue minerals was obviously improved.

In summary, currently the studies on flotation collectors prepared using hogwash oil are heavily focused on the subsequent processing methods and the applications of these collectors. The

* Corresponding author at: School of Civil and Resource Engineering, University of Science and Technology Beijing, 30 Xueyuan Road, Haidian District, Beijing 100083, PR China.

E-mail address: wanghuajun@bjjzq.com (H. Wang).

<https://doi.org/10.1016/j.ijmst.2018.04.010>

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collectors prepared by the direct method have a certain function to flotation. However, their use is less effective compared to traditional collectors such as kerosene and diesel fuel. The ester exchange method is affected by disadvantages such as a long reaction time. There are few studies investigated the characteristics and components of hogwash oil and the components and action mechanisms of the collectors prepared by hogwash oil.

This paper selected four types of hogwash oil from different areas in China as raw materials. The characterization of the hogwash oil was determined and fatty acid JZQ-F collectors were prepared using the hydrolysis method. The components and flotation differences of the JZQ-F collector were investigated. The adsorption characteristics of the JZQ-F collector on apatite surface were also discussed.

2. Experimental

2.1. Reagents

Hogwash oil was collected in different cities in China, including Beijing, Ordos in Inner Mongolia, Qingdao in Shandong, and Dongguan in Guangzhou. They represents locations where people have different dietary characteristics.

Analytical reagents (AR) used for preparing the JZQ-F collector including ethyl alcohol, ethyl alcohol, n-hexane, and petroleum ether. Trichloromethane, cyclohexane, glacial acetic acid and boracic acid used to analyze the fatty acid content were also of AR.

The detailed information about the reagents used in this paper are listed in Table 1.

2.2. Mineral samples

Phosphate ore samples were obtained from an overflow product of a primary grinding classifier from Fanshan Phosphate Ore in Hebei, China. The grinding fineness is -200 mesh = 43.25%. After ambient air drying, mixing and dividing, the phosphate ore was kept in self-sealing bags as experimental samples. The phosphorus pentoxide (P_2O_5) grade of the raw ore is 7.55% and the quartz content of the main gangue mineral is 31.04%. Results of the chemical analysis of the phosphate ore sample are shown in Table 2.

Apatite pure mineral samples were collected from a mining site in the Fanshan Phosphate Ore in Hebei, China. The high-grade apatite rich ore blocks, whose impurities were further removed by weak magnetic separation and high intensity magnetic separation, were crushed and milled with ceramic ball mill to obtain particles with a size less than -0.074 mm. The particles were further sieved

Table 1
Reagents.

Reagent	Molecular formulas	Molecular weight	Manufacture
Methyl alcohol	CH_4O	32	Sinopharm Group, China
Ethyl alcohol	C_2H_5OH	46	Sinopharm Group, China
N-hexane	C_6H_{14}	86	Sinopharm Group, China
Petroleum ether			Beijing Chemical Works, China
Trichloromethane	$CHCl_3$	119.38	Sinopharm Group, China
Cyclohexane	C_6H_{12}	84	Xilong Chemical Co., Ltd, China
Glacial acetic acid	$C_2H_4O_2$	60	Xilong Chemical Co., Ltd, China
Boracic acid	H_3BO_3	61.83	Sinopharm Group, China

to -0.074 mm + 0.038 mm size and stored in self-sealing bags. The P_2O_5 grade of the apatite single mineral was 39.78%. Results from the chemical analysis of these apatite pure mineral samples are shown in Table 3.

2.3. Instrumentation

A flotation test was conducted in a XFD-0.5 flotation cell (Exploration Machinery Factory Changchun, China). The characteristics of the functional groups in the hogwash oil were measured by an infrared spectrometric analyzer NEXU-670 (American Thermo Nicolet Co., USA). It has a wave length determination range of $500\text{--}4000\text{ cm}^{-1}$.

In order to analyze the fatty acid content for collectors, a gas chromatograph and a mass spectrograph (GC-MS) were employed. A GC (GC-2014; manufactured in Japan) with a $+10$ to 420°C operating temperature range and -250 to $250^\circ\text{C}/\text{min}$ rate range was used. The MS model was DSQ.

The measurement process for adsorption characteristics was performed in real-time using a Q-Sense E4 quartz crystal microbalance imported from Sweden.

2.4. Methods

2.4.1. Characterization of the hogwash oil

The specific density and the melting point of the hogwash oil was determined through a gravity flask method and the capillary method, respectively [14]. The iodine value of the hogwash oil was determined according to the national standard GB/T5538-2010 [15].

2.4.2. Preparation of JZQ-F collector

The fatty acid JZQ-F collector was prepared by the hydrolysis method. The preparation flowsheet is presented in Fig. 1. The four types of collectors (JZQ-FB, JZQ-FQ, JZQ-FO, JZQ-FD) correspond to the JZQ-F collector prepared using hogwash oil from the four different dietary characteristics cities (Beijing, Qingdao, Ordos, and Dongguan, China).

- (1) Physical refining. The hogwash oil samples of 100 g and the active white soil samples of 20 g were putted in a conical flask. The mixture was stirred with a magnetic heated stirrer during continuous heating until after it was boiled for 20 min. When the samples were cooled, the hogwash oil and active white soil were separated in a centrifugal machine, and then the refined hogwash oils were obtained.
- (2) Saponification. The refined hogwash oils were putted in a conical flask, and the sodium hydroxide-ethyl alcohol solution (150 mL of 1 mol/L sodium hydroxide-ethyl alcohol solution for 40 g of refined hogwash oil) was added. After that, 5 drops of phenolphthalein were added as the indicator to keep the solution red. The heating time was 1 h and the temperature was kept within the range of 70 to 80°C until the oil droplets were completely disappeared.
- (3) Extraction. After the saponated solution was cooled to the room temperature, a proper amount of water was added to absolutely dissolve it. $1/3$ volume of petroleum ether was added for extraction. The substance was separated through a separating funnel, and the lower part was the fatty acid sodium.
- (4) Acidification. The fatty acid sodium was transferred to another separating funnel, and then excessive of 10% sulfuric acid solution was added to make the acidosis reaction between the fatty acid sodium and sulfuric acid. The solution was reacted completely when the pH was 1 to 2. The lower water layer was removed by the separating funnel. The

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