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The changes of surface properties and enhancement of B_2O_3 leaching ratio of boron concentrate via wet ball milling☆

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ABSTRACT

With the ascharite used up in China, boron concentrate was used as raw material for extracting B_2O_3. In this study, an environmental wet ball milling (WBM) method was proposed to enhance the B_2O_3 leaching ratio, and the effect of liquid medium, rotational speed and milling time on the properties of boron concentrate in WBM process was discussed. This surface properties of milled samples was characterized by BET, PSD, SEM, XRD, FTIR, TG-DSC and XPS analyses, and their surface free energy was calculated by the measurement of contact angle. The role of liquid medium and the relationship between the surface properties was also explored. Our research shows liquid medium helps avoid the agglomeration and plays a dispersing role in the process of milling, thereby promoting the increasing of the specific surface area from 16.65 m^2/g to 35.84 m^2/g after milling for 60 min. Besides, the B_2O_3 leaching ratio can be enhanced from 67.52% to 86.24% under the same conditions. The increased amorphous extent of crystal structure and the weakening of chemical bonds caused by the milling process helps produce an increasing in the number of reactivity points, making chemical reactions easier. With the increasing of surface free energy from 26.88 mJ/m^2 to 39.55 mJ/m^2 after milling, the hydrophilicity of the sample is enhanced, as well as the absorption of solvent and OH^- in the leaching solution, thus promoting the leaching reaction and the enhancement of B_2O_3 leaching ratio.

1. Introduction

Boron resources are widespread in China but are of low quality, about only 8.4 wt% B_2O_3 [1]. Boron minerals mainly exist in the form of ascharite and ludwigite in Liaoning province. Ludwigite is the paragenetic ore of boron, magnesium and iron and is mainly composed of magnetite, camsellite, ludwigite and serpentine minerals [2,3]. Moreover, the average boric content of ascharite used in processing enterprises is less than 10%. It is difficult for processing because of its complex structure and more associated mineral. The expedite development and utilization of ludwigite resource is of important practical significant in the short supply of boron resource in our country. Boron concentrate is a product of the combined magnetic and gravity separation of ludwigite and has gradually begun to be used as a substitute material for ascharite in the Chinese boron industry [4]. But the B_2O_3 leaching ratio of raw boron concentrate was only 67.52% without any activation methods because of its low boron grade. Given this, boron concentrate cannot be used directly unless its reactivity is enhanced.

Various pretreated methods can enhance the reactivity of minerals [5], but for boron concentrate or ludwigite, roasting activation is a traditional method for the enhancement of their activity. However, the traditional roasting method for boron concentrate cannot achieve the expected activation efficiency and usually fails to meet the demands of practical production of boron industry [6,7]. And the demand of high temperatures [7,8,9] costs fossil fuel resources and produce environmentally damaging effluent streams [8] such as SO_2 and NO_X [2]. The mechanical activation (MA) of minerals makes it possible to reduce thus harmful to the environment that the thermal activation may be omitted entirely [8,9].

MA is an environmental friendly method and seems to be a favorable process for the treatment of minerals prior to leaching to enhance the reactivity of minerals [7,8,9,10], and it prefers to the use of mechanical actions to change the crystalline structures and physicochemical properties of the solids carried out by high energy ball-milling [9]. Ball milling is considered as an efficient pretreated method for various minerals to enhance their leaching ratio in hydrometallurgy [5,8,9,10], but few paper has been reported the influences on boron concentrate. There are wide differences in the properties of various raw materials, resulting in such widely differing effect of MA on the surface properties of minerals and mechanism of activation. It has been reported that the agglomeration of particles was a major factor when the treated particles

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were ground to less than 10 μm, especially with a high surface energy of particles [11]. Wet ball milling (WBM) is an efficient way to avoid the agglomeration phenomenon and help achieve the high specific surface of minerals. As results, in previous studies, a large number of research groups have been focus on the application of WBM method, however, this method was traditionally used to obtain ultrafine powder or the synthesis of nano-materials [11,12,13,14]. The application of wet milling in the enhancement of the B_2O_3 leaching ratio from boron concentrate helps to establish the relationship between specific surface area of boron concentrate and the leaching characteristics of B_2O_3, and it will be helpful to physicochemical modification of boron concentrate by MA, thereby improving the B_2O_3 leaching ratio. Besides, the experimental results can be used to investigate the mechanism of MA by comparing with the properties of dry milled samples as well.

Therefore, in this study, we explored the influences of WBM on the surface properties and the B_2O_3 leaching ratio of boron concentrate. As for the reactivity of minerals is closely related to their surface properties, including the specific surface area, particle size, microstructures, surface free energy, crystal structure, surface functional group, thermal behavior and so on, all of them have been measured by appropriate methods. Our efforts focused on optimizing the WBM process parameters for higher B_2O_3 leaching ratio and providing a reasonable explanation for the enhancement of the B_2O_3 leaching ratio and also finding the roles of liquid medium played in this milling process.

2. Materials and methods

2.1. Materials

Details of the preparation and characterization of boron concentrate used in this study have been reported earlier [15]. The boron concentrate used in this research contained 11.89 wt% of B_2O_3. Details characterization using X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier Transform Infrared (FTIR) and thermal analysis studies has shown that the compositions and morphology of the test sample are extremely complex. The results of XRD, SEM and FTIR of the raw boron concentrate are presented in Figs. 1–3. As shown in Fig. 1, the mineral compositions of boron concentrate contained mainly szaibelyite ($MgBO_2(OH)$), serpentine ($Mg_3Si_2O_5(OH)_4$), biotite ($K[Mg,Fe]_3[Al,Fe]Si_3O_{10}(OH,F)_2$) and phlogopite ($(Mg,Al)_3[(Si,Fe)_2O_5](OH)_4$). The SEM image demonstrates strip, granular, needle-shape, and fibrous patterns in the raw mineral with various particle size. From Fig. 3, it is clearly that so many types of functional groups existed in boron concentrate, including hydroxyl groups (—OH), C—O functional groups, Si—O functional groups and B—O functional groups.

As previous reported [15], the median particle diameter (d_{50}) and specific surface area of the boron concentrate are 13.2 μm and 15.65 m^2/g, which were analyzed from particle size distributions (PSD) and Brunauer-Emmett-Teller (BET) results, respectively.

Alcohol ($C_6H_{14}O_6$) was used as the liquid medium for WBM process. The water used in this research was ultrapure and KCl was spectral purity, and other reagents were all of analytical grade.

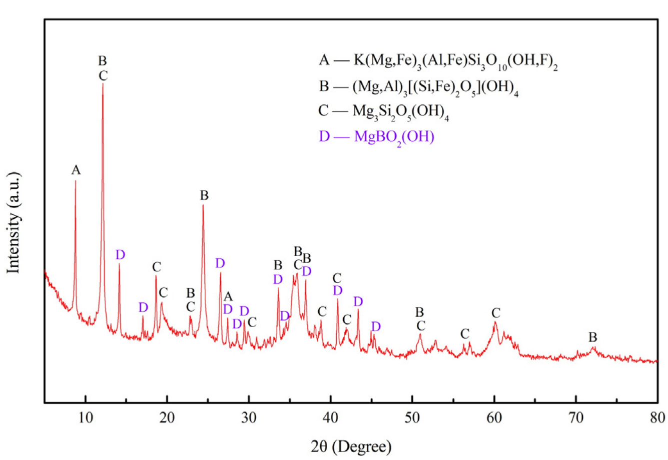

Fig. 1. X-ray diffraction pattern of boron concentrate.

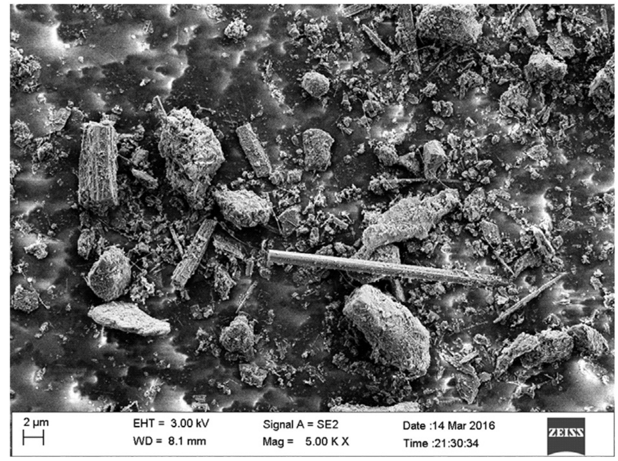

Fig. 2. SEM image of boron concentrate.

2.2. Methods

The overall experimental flow sheet is presented in Fig. 4. After crushing, sieving and drying, the boron concentrate sample used for WBM, then extracting the useful composition (B_2O_3) from the milled samples by an alkaline leaching method.

2.2.1. Wet ball milling

To investigate the effect of WBM on the properties of boron concentrate, the sample (24.0 g) and zirconia milling balls (288.0 g, 8 mm diameter) were added to a zirconia chamber and ground for 60 min in a Fritsch Pulviresette-4 planetary mill. A schematic illustration of the planetary mill is shown in Fig. 5. In the milling instrument, one of the milling chamber was used for milling samples, the other was for mass balance. These two chambers were revolved around their vertical center axes, and rotated around the central axis of the wheel in the opposite direction at the same time, thereby driving the samples and milling balls in the zirconia chamber to do

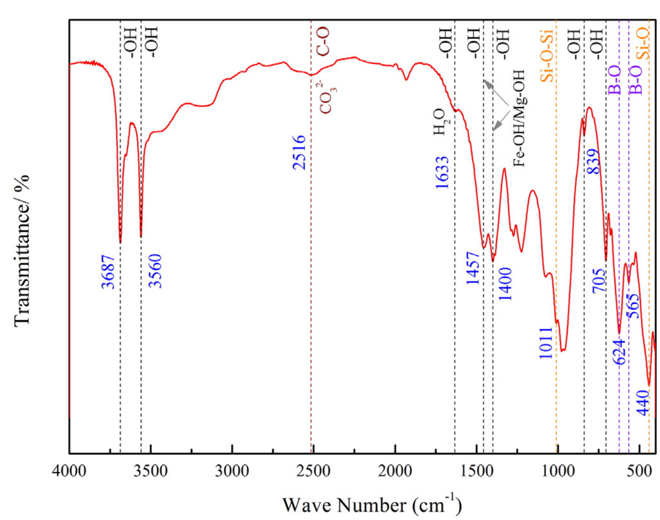

Fig. 3. FTIR spectra of boron concentrate.