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Comparative Evaluation of High Pure Mn3O4 Preparation Technique between the Conventional Process from Electrolytic Manganese and a Sustainable Approach Directly from Low-Grade Rhodochrosite

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Abstract : Up to now, electrolytic process is a popular way to prepare Mn and MnO2 (EMD) with high purity. However, the conventional preparation process of manganese oxide such as Mn3O4 with high purity from electrolytic manganese metal is characterized by long production-cycle, high-pollution discharge and high energy consumption especially initially from low-grade rhodochrosite, the main resources for exploitation and applications in China. Moreover, Mn3O4 prepared from electrolytic manganese shows large particles, single morphology beyond the control and weak chemical activity. On the other hand, hydrometallurgical method combined with thermal decomposition, hydrothermal synthesis and sol-gel processes has been widely studied because of its high efficiency, low consumption and low cost. But the key problem in direct preparation of manganese oxide series from low-grade rhodochrosite is to remove completely the multiple impurities such as iron, silicon, calcium and magnesium. It is urgent to develop a sustainable approach to high pure manganese oxide series with character of short process, high efficiency, environmentally friendly and economical benefit. In our work, the preparation technique of high pure Mn3O4 directly from low-grade rhodochrosite ore (13.86%) was studied and improved intensively, including the effective leaching process and the short purifying process. Based on the same ion effect, the repeated leaching of rhodochrosite with sulfuric acid is proposed to improve the solubility of Mn2+ and inhibit the dissolution of the impurities Ca2+ and Mg2+. Moreover, the repeated leaching process could make full use of sulfuric acid and lower the cost of the raw material. With the aid of theoretical calculation, Ba(OH)2 was chosen to adjust the pH value of manganese sulfate solution and BaF2 to remove Ca2+ and Mg2+ completely in the process of purifying. Herein, the recovery ratio of manganese and removal ratio of the impurity were evaluated via chemical titration and ICP analysis, respectively. Comparison between conventional preparation technique from electrolytic manganese and a sustainable approach directly from low-grade rhodochrosite have also been done herein. The results demonstrate that the extraction ratio and the recovery ratio of manganese reached 94.3% and 92.7%, respectively. The heavy metal impurities has been decreased to less than 1ppm, and the content of calcium, magnesium and sodium has been decreased to less than 20ppm, which meet standards of high pure reagent for energy and electronic materials. In compare with conventional technique from electrolytic manganese, the power consumption has been reduced to ≤2000 kWh/t(product) in our short-process approach. Moreover, comprehensive recovery rate of manganese increases significantly, and the wastewater generated from our short-process approach contains low content of ammonia/ nitrogen about 500 mg/t(product) and no toxic emissions. Our study contributes to the sustainable application of low-grade manganese ore.
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Keywords : leaching, high purity, low-grade rhodochrosite, manganese oxide, purifying process, recovery ratio
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