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Effects of microwave heating on microstructures and structure properties of the manganese ore

Guo Chen [a,*], Lei Li [a], Changyuan Tao [b], Zuohua Liu [b], Nanxiong Chen [c], Jinhui Peng [a,**]

[a] Key Laboratory of Resource Clean Conversion in Ethnic Regions of Education Department of Yunnan, Joint Research Centre for International Cross-border Ethnic Regions Biomass Clean Utilization in Yunnan, Yunnan Minzu University, Kunming 650500, PR China
[b] School of Chemistry and Chemical Engineering, Chongqing University, Chongqing 400044, PR China
[c] CITIC Dameng Mining Industries limited, Nanning 530028, PR China

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ABSTRACT

The present work attempts to investigate the formation–decomposition reaction of the manganese ore. Thermodynamic analysis indicates that the decomposition reaction of the manganese ore was feasible by adjusting the roasting temperature and duration time. The peak intensity of Mn_3O_4 phase of the microwave heated products increases with the increase of microwave heating temperature. The microwave treated manganese ore appears irregular with numerous small cracks and pits, which was caused by the decomposition reaction of $MnCO_3$ phase and $CaCO_3$ phase under microwave heating. It was concluded that microwave heating can be applied effectively and efficiently to the calcination processes of the manganese ore.

1. Introduction

Manganese is essential to iron and steel production by virtue of its sulfur-fixing, deoxidizing, and alloying properties [1]. Meanwhile, manganese is also an alloying element in the preparation of special alloys in nonferrous metallurgy, and manganese has no satisfactory substitute in its major applications, which are related to metallurgical alloy use [2,3]. The main consumer of the manganese ores is ferrous metallurgy, which consumes up to 95% of world output [4]. The addition of manganese in the smelting of iron ore facilitates the desulfurization of pig iron and the liquefaction of slag, which removes up to 60% of the sulfur in pig iron [5]. Therefore, an exploration of a new technology, with less energy consumption and higher minerals recovery, and is very suitable for application in commercial scale operation, which is necessary for sustainable development [6].

Microwave heating operating at 915 MHz and 2450 MHz are common appliances in the household and industry around the world [7–9]. In the case of polar solutions like water, the molecules try to align themselves with respect to the microwave field. Microwave energy can penetrate the poor thermal conductors of materials to heat rapidly using convection or thermal conduction, and provide rapid volumetric heating [10–12]. The list of advantages for using microwave energy is expanding daily. Recently, microwave energy has been widely used in several fields of applications on both research and industrial processes. Microwave heating belongs to innovative processes to improve technological methods by decreasing the temperature of reaction and increasing the rate of chemical reaction [13].

In the present work, the thermodynamics graphs of the formation–decomposition reaction of the manganese ore were firstly assessed. Then, the decomposition reactions of the manganese ore using microwave heating were systematically investigated. The phase transformation and the surface morphology of the manganese ore before and after microwave heating were investigated using XRD and SEM, respectively.

2. Experimental

2.1. Materials

The raw material, manganese ore, was obtained from Wenshan city, Yunnan province, China. The chemical compositions of the manganese ore were as follows (% (w/w)): TMn, 30.00; TFe, 1.50; CaO, 14.97; SiO_2, 12.87; Al_2O_3, 1.80; MgO, 1.76; P, 0.07, respectively.

* Corresponding author.
** Corresponding author.
E-mail addresses: guochen@kmust.edu.cn (G. Chen), jhpeng@kmust.edu.cn (J. Peng).

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It was characterized by high manganese grade (30.0%) and low iron content (1.50%). The product was analyzed by the method in accordance with the recommended methods of National Standard of the People's Republic of China (GB/T).

2.2. Procedure

The manganese ore was milled and sieved into different particle size fractions, the sieved fractions (5 mm–20 mm) were collected for further studies. The manganese ores were dried at 110 °C for 10 h before each experiment. Oxygen was passed through the microwave heating device to increase the condition of reaction, and expel the gas when it was preheated at a rate of 10 °C/min to the required temperature. Then a corundum crucible loading manganese ore sample was introduced into the microwave heating cavity. The manganese ore was heated at an established temperature for a certain time period. Once roasting processing was completed, the microwave heated samples were taken out and cooled down in air. The cooled sample was then used for analysis.

3. Results and discussion

According to the standard Gibbs free energy equations of the decomposition reaction of $MnCO_3$ phase and $CaCO_3$ phase, the thermodynamics graphs of the formation–decomposition reaction of the manganese ore are investigated, and the calculation results are plotted in Fig. 1. It is observed from Fig. 1 that the temperatures for the reactions of $MnCO_3$ and $CaCO_3$ occur at 176–550 °C and 522–868 °C, respectively. Thermodynamic analysis indicates that the decomposition reaction of the manganese ore is feasible by adjusting the roasting temperature and duration time.

The crystalline structures of the manganese ore before and after microwave heating are characterized by X-ray diffractometer (D/Max 2200, Rigaku, Japan) at a scanning rate of 0.25°/min with 2θ ranging from 5° to 100° using CuKα radiation (λ = 1.5418 Å) and a Ni filter. The voltage and anode current operated are 35 kV and 20 mA, respectively. After scanning, the mineral peaks of the manganese ore are identified. The XRD patterns of the manganese ore and the microwave heated products are shown in Fig. 2. It can be seen from Fig. 2(a) that the raw materials consisted of Mn_2O_3 (JCPDS card No. 65-7467), $MnCO_3$ (JCPDS card No. 44-1472), $CaCO_3$ (JCPDS card No. 70-0095), Ca(Mn, Mg)$(CO_3)_2$ (JCPDS card No. 83-1531) and SiO_2 (JCPDS card No. 86-1560). By comparing with Fig. 2(a), the peak intensity of Mn_3O_4 phase (JCPDS card No. 80-0382) and CaO phase (JCPDS card No. 77-2010) of the microwave heated products increases with the increase of microwave heating temperature, and the peak of $MnCO_3$ phase (JCPDS card No. 44-1472) and $CaCO_3$ phase (JCPDS card No. 70-0095) disappeared. The results of XRD analysis indicates the decomposition reaction of $MnCO_3$ phase and $CaCO_3$ phase as follows,

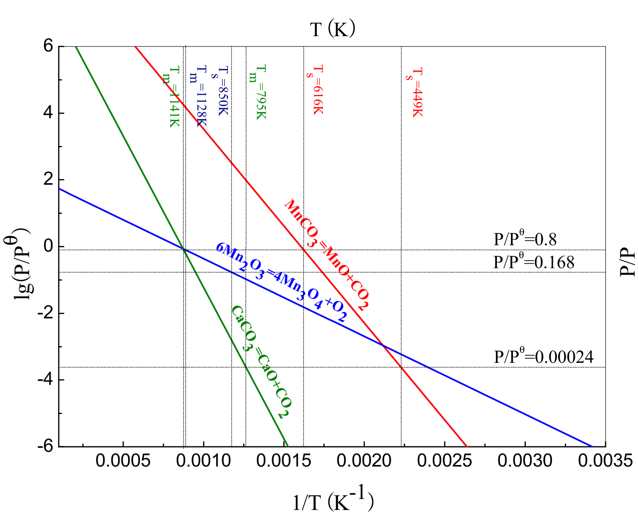

Fig. 1. Thermodynamic analysis of decomposition reaction of manganese ore.

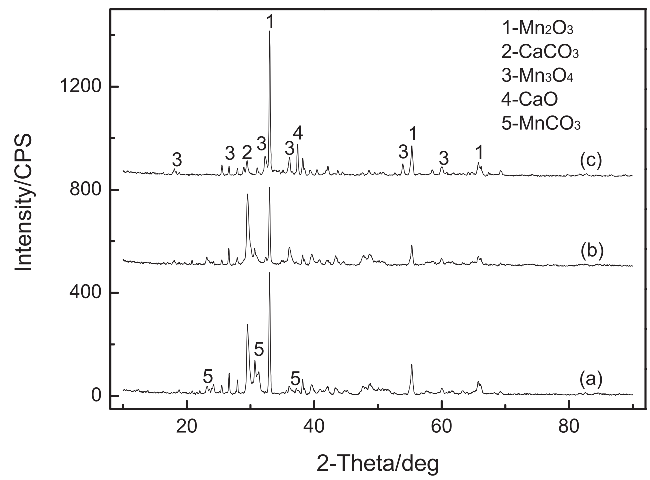

Fig. 2. XRD patterns of raw materials and microwave heated products, (a) the manganese ore; (b) microwave heating at 650 °C for 30 min; (c) microwave heating at 850 °C for 30 min.

$$MnCO_3 = MnO + CO_2 \quad (1)$$

$$6MnO + O_2 = 2Mn_3O_4 \quad (2)$$

$$CaCO_3 = CaO + CO_2 \quad (3)$$

It can be found from Fig. 2(b) and (c) that the diffraction peaks of Mn_3O_4 phase gradually broadened and their intensities increased under microwave heating temperatures up to 650 °C, and then strengthen at 850 °C. The Mn_3O_4 phase has the strongest preferential orientation of (2 1 1) plane at 2θ = 36.084°, and the second and third strong preferential orientation of (1 0 3) and (2 2 4) planes of Mn_3O_4 phase prepared are observed at 2θ = 32.383° and 2θ = 59.910°, respectively. The presence of these characteristic peaks corresponding to Mn_3O_4 phase is difficult to confirm with absolute certainty due to the close proximity of the characteristic peaks of Mn_2O_3 phase and the weak intensity of the above peaks. Microwave heating of the manganese ore has been found to microwave selectively heating the mineral matter, based on differences in microwave dielectric properties.

The morphological aspects of the raw materials and the microwave heated products are also investigated by scanning electron microscope (SEM). SEM is utilized in evaluating the surface morphology of the manganese ore during the microwave heating process. The SEM instrument (XL30ESEM-TMP, Philips, Holland) is operated at 20 kV in a low vacuum, while the energy dispersion scanner spectrometer (EDAX, USA) attached to the SEM is used for semi-quantitative chemical analysis. It is characterized by SEM and EDAX techniques, and the results as shown in Fig. 3, respectively. From the SEM in Fig. 3, the fine cracks are observed on the manganese ore surface, which appears elongated powders with small pits, and the results indicated that the size distribution of the microwave treated samples is wide (100–150 um), and the microwave heated product is uniform, loosen and un-sintered. The microwave treated manganese ore appears irregular with numerous small cracks and pits (Fig. 3(b) and (c)), was treated at temperature of 650 °C and 850 °C, and the duration time of 30 min, which is caused