EXTENDED ABSTRACT

Solar Carbothermic Reduction of Complex Weathered Ilmenite using Biomass

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Introduction

The dominant energy source for Earth comes from the Sun with an average total radiative energy of 1361 W/m^2 to the upper Earth's atmosphere [1]. Concentrated solar energy on the Earth's surface using devices such as a solar furnace allows generation of temperatures that can reach 3500°C, which is sufficiently high enough to melt almost all types of materials [2]. Concentrated solar thermal technology (CST) therefore can provide an innovative and more environmentally friendly route for processing of metals and materials. The possible applications of CST, especially for minerals and metallurgical processing, include various thermal and chemical processes including carbothermic reduction processes [3].

Carbothermic reduction is commonly used in iron and steel manufacturing. Nevertheless, there is still very limited application of CST for this process. Purohit et al. carried out reduction experiments on composite iron ore pellets using a solar simulator at 1130°C and achieved a 55% degree of metallization after 1.5 h of heating [4]. Steinfeld and Fletcher carried out a carbothermic reduction of Fe_2O_3 using graphite at 1027-2117°C in a solar furnace, and reported a metallic iron yield of 78 percent [5]. Carbothermic reduction of hematite using carbon and concentrated solar energy at 1084-1367°C was carried out by Fernández-González et al. [6]. They reported that metallic iron (3.30-5.60%) was only detected at 1338-1367°C. No previous solar carbothermic reduction studies using ilmenite has been reported in the literature other than from the authors [7].

Solar carbothermal processing of ilmenite

Complex weathered ilmenite ($FeTiO_3$) ore is used as a secondary source to produce synthetic rutile (TiO_2), titanium metals, and alloys. Therefore, making an effective upgrade of the ore is vital. In carbothermic ilmenite reduction, the ilmenite is reduced in a smelting process to produce pig iron and a slag containing high TiO_2. The main challenges in the process of smelting ilmenite are its slow reduction time. For example, the reduction process in an electric arc furnace can take up to 8 h at 1700°C which results in a high overall energy consumption [8]. There are no previous studies on carbothermic reduction of ilmenite concentrate using biomass as a reducing agent and using solar energy as a heat source. In this paper, a solar simulator was used to simulate the use of solar energy for heating the ilmenite ore during the carbothermic reduction process. This study was coupled with similar reduction experiments carried out using a regular heat source (an electrical resistant furnace) for enabling direct comparison of results to the simulated solar energy heat source data.

Experimental Procedure

Detailed microstructural and phases characterization of a Kalimantan ilmenite, as well as the chemical composition of palm kernel shell biomass used in this study, were reported by Setiawan et al. [7]. The molar ratio of C/O, the mass of carboxymethyl cellulose, used as a binder, and distilled water in the mixture were set to 1.5, 0.5 wt. %, and ~0.8 wt.%, respectively. The mixtures were mixed homogenized and then compressed into pellets and dried at 100°C for 5 h before being used in the experiments. The two types of carbothermic reduction experiments were conducted using a horizontal tube resistance furnace and a solar simulator. In the electrical resistant furnace, the samples were reduced at temperatures between 1000°C and 1200°C with a reaction time up to 60 min, and a heating rate of 200°C/h. In the solar simulator, the samples were placed in a fused-quartz tube reactor and inserted into the main solar reactor. The solar simulator was set up using five metal halide lamps to achieve the final temperature of 1200°C for a reaction time of 60 min. The reduced samples were characterized for microstructure and phase analysis using Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Analysis (EDX), High-Resolution Electron Probe Microanalysis (EPMA), and X-Ray Diffraction (XRD).

Results

A comparison of XRD results for the reduced samples using the different heat thermal sources is shown in Figure 1. Results from the carbothermic reduction using the electrical resistance furnace at 1000°C resulted in the major phases identified, including ilmenite ($FeTiO_3$), rutile (TiO_2), chromite spinel ($FeCr_2O_4$), and metallic iron $Fe_{(m)}$. As the temperature of reduction was increased to 1100°C and 1200°C, there was a significant decrease in the intensity of the ilmenite peaks accompanied by a sharp increase in intensity of the peaks for metallic $Fe_{(m)}$ and rutile. The XRD results indicated that after 60 min of reaction, complete reduction of ilmenite had been achieved at 1200°C, but not at 1000°C and 1100°C. Complete reduction of ilmenite ($FeTiO_3$) was also achieved when the sample was carbothermically reduced in the solar furnace at 1200ºC for 60 min (similar to the case of electric furnace reduction), as no ilmenite peaks were observed from the XRD pattern. $FeCr_2O_4$ chromite spinel was also observed to form in the experiments however, this phase was not affected by the reduction process. The SEM analysis showed a clear distinction between the morphology of the metallic Fe produced by the two heating methods. When the electric furnace was used as the heat source, the particles of metallic iron formed with spherical/globular structures. In contrast, the metallic iron formed in the solar heating experiments developed a streak-like structure, and many pores formed in the reduced ilmenite. In general, the average particle size of the iron in the samples heated using solar furnace was found to be higher compared to those samples heated using the electric furnace, as shown in Figure 2.

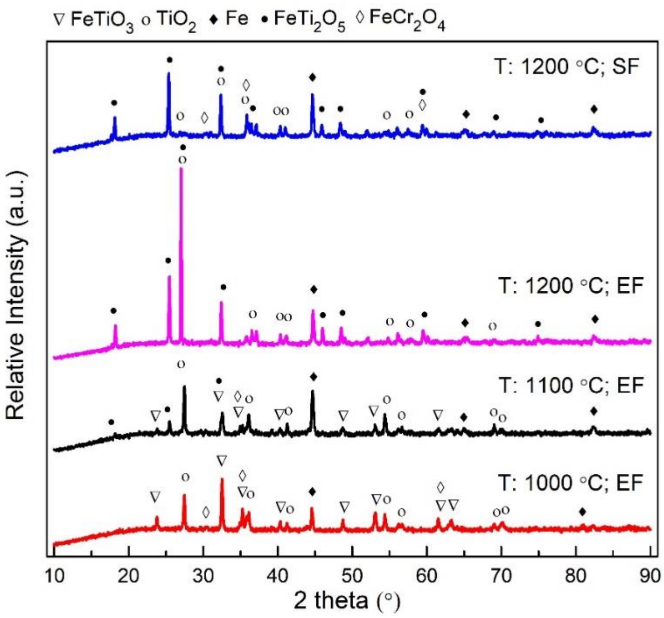

Figure 1. XRD analysis results from ilmenite samples carbothermically reduced using an electrically resistant furnace (EF) and a solar simulator furnace (SF).

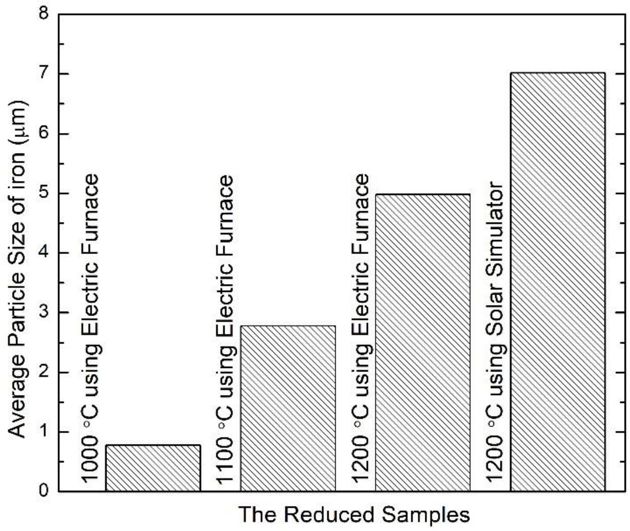

Figure 2. The average particle size of metallic iron formed after carbothermic reduction using different heat thermal sources.

Conclusions and future work

A complete ilmenite reduction was attained at 1200 °C for 60 min reaction using both electrical and solar furnace heating. When solar thermal reduction was applied, a distinct morphology of metallic iron formed, i.e., a streak structure developed as opposed to globular structure formed under electrical heating. Future work by the authors will include a more detailed study on the kinetics, the effect of additives and mixing methods, and the mechanism of carbothermic reduction of ilmenite using biomass and concentrate using solar energy.

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