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# Study on kinetics of thermal decomposition of low LOI goethetic hematite iron ore

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#### ABSTRACT

In the present study, the kinetics of thermal decomposition of hydrated minerals associated in natural hematite iron ores has been investigated in a fixed bed system using isothermal methods of kinetic analysis. Hydrated minerals in these hematite iron ores are kaolinite, gibbsite and goethite, which contribute to the loss on ignition (LOI) during thermal decomposition. Experiments in fixed bed have been carried out at variable bed depth (16, 32, 48 and 64 mm), temperature (400–1200 °C) and residence time (30, 45, 60 and 75 min) for iron ore samples. It is observed that beyond a certain critical bed depth (16 mm), 100% removal of LOI is not found possible even at higher temperature and higher residence time. Most of the solid-state reactions of isothermal kinetic analysis have been used to analyze the reaction mechanism. The raw data are modified to yield fraction reacted " $\alpha$ " versus time and used for developing various forms of " $\alpha$ " functions.  $f(\alpha)$  is the inverse of first derivative of  $g(\alpha)$  with respect to  $\alpha$ . The study demonstrates that decomposition of hydrated mineral situations are found to high, of the order of 60 kJ/mol, reinstating that the reactions are indeed controlled by moving phase boundary and random nucleation.

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#### 1. Introduction

Indian iron ore resources are mostly hematite. High grade hematite iron ore is depleting very fast. The low grade resource has become the major resources to produce iron and steel in India. The majority of low grade hematite iron ore resource at present is fragile in nature because of its association with good amount of goethite, kaolinite, and gibbsite. As per iron ore formation, goethite generates from hematite due to weathering. The goethite associated with hematite is called vitreous goethite which is hard and crystalline. The goethite associated with clayey materials i.e., kaolinite and gibbsite is called ochreous goethite [1]. The liberation of iron phase minerals in this type of ore is at finer sizes. During the beneficiation of these low grade hematite iron ores, vitreous goethite comes with hematite and partly ocherous goethite along with kaolinite and gibbsite which contribute to the loss on ignition (LOI) in iron ore concentrate. The generation of fines is more during size reduction because of its fragile nature that leads to high Blaine number of the concentrate [2].

The utilization of low grade iron ore is need of the hour in respect to meet the high quantity of production of steel in India as per the steel policy of government of India. In order to meet the future demand of steel, low grade iron ore fines need to be beneficiated to provide raw materials for steel plants in form of pellet. The presence of goethite, gibbsite, and kaolinite leads to high LOI and high Blaine number of the iron ore concentrate after beneficiation. The presence of high LOI in the final concentrate deteriorates the quality of pellets [3]. Due to the presence of chemically bound water in the matrix of goethite, kaolinite and gibbsite, high pressure steam is released at high temperature during induration process resulting in cracks inside the pellets thereby reducing the strength. The production of pellets and its use in blast furnace has increased largely over the past decade and hence there is considerable amount of research interest to remove LOI from the iron ore sample before making the pellet. Gibbsite and goethite release their water molecules within the temperature of 300-400 °C whereas kaolinite releases its water molecule after the temperature of 850 °C.

The kinetics of many solid-state reactions can be represented by Eq. (1):

(1)

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 $f(\alpha) =$ 

where  $\alpha$  is the conversion factor (fraction of LOI removed) in time *t*, and the function  $f(\alpha)$  depends on the thermal decomposition mechanism of the iron ore with respect to temperature and pressure and the physical properties of the particle, i.e., grain size, shape, and crystallinity.

The activation energy during the transformation of goethite to hematite has been well documented by many researchers. Limade-Faria found that the activation energy for a crushed single crystal goethite is 82.8 kJ/mol [4]. Pollack et al. quoted 121  $\pm$  13 kJ/mol activation energy of two natural limonite (amorphous goethite) [5]. Thrierr-Sorel et al. reported activation energy of 88 kJ/mol for a fibrous goethite [6]. Keller also gave activation energies in the range of 96–167 kJ/mol for different variety of goethite types [7]. Goss reported the activation energy of 154  $\pm$  15 kJ/mol for the sedimentary goethite [8]. Prasad et al. examined the in-situ FTIR study on dehydration of natural graphite and reported that activation energy for pure goethite is 85 kJ/mol [9].

In this paper, the most commonly methods for isothermal kinetic analysis are used in order to determine the mechanism of decomposition of hydrated minerals associated in hematite iron ore using heating cycle process in muffle furnace and thermogravimetric analyzer and evaluate the activation energy. The thermal decomposition of goethite, gibbsite and kaolinite has given in Eqs. (2)–(4) respectively:

$$2FeOOH = Fe_2O_3 + H_2O$$
 (2)

$$2AI(OH)_3 = AI_2O_3 + 3H_2O$$
(3)

$$Al_2Si_2O_5(OH)_4 = Al_2O_3 + 2SiO_2 + 2H_2O$$
(4)

#### 2. Materials and methods

The iron ore sample collected from Barbil region of Odisha, India is selected for the study, which contain LOI of 5.46%. Mineralogical characteristic are analyzed through quantitative X-ray diffraction (XRD). The XRD study was carried out by X-ray diffractometer (PANalytical, X'pert) and quantitative analysis was done through high score plus software. The mineralogical characteristic of the sample is given in Table 1. The chemical analysis of the sample was carried out through wet chemical and XRF analysis techniques. The detailed chemical analysis of the sample is given in Table 2. The particle size distribution is given in Table 3.

Initially, the iron ore samples are subjected to thermo gravimetric analysis to determine the rate of weight loss with increase in temperature by TGA (Thermo gravimetric Analysis)/SDTA (Simulation of Differential Thermal Analysis) supplied by Mettler, USA. In order to calculate the cumulative weight loss for high mass (250–1000 g) samples in fixed bed with temperature, the sample with different bed depths are taken after removal of surface moisture and kept inside the furnace for a fixed time of 30 min at a particular temperature from 200 °C to 1200 °C with an incremental increase of 50 °C to compare the TGA data.

Further, the iron ore samples are dried at about  $120 \,^{\circ}$ C for 2 hours for complete removal of surface moisture. Then the

Table 1 Mineralogical sample.	constituents of the
Details	Sample (wt.%)
Hematite	44.96
Goethite	47.41
Kaolinite	6.5
Gibbsite	0.9
Quartzite	0.1

samples are put in four different crucibles of identical size having 96 mm internal diameter and 91 mm height. All the experiments are made in batch mode in a fixed bed system. The crucibles are made of high alumina refractory to withstand high temperature. The different weights i.e. 250, 500, 750 and 1000 g of samples are taken for the experiments. The depth of the ore bed varies from 16 mm to 64 mm depending on the weight of the sample. The top of the crucibles is kept open. The samples are decomposed in the muffle furnace isothermally at different temperatures (ranging from 400 °C to 1200 °C) for a particular residence time. The heating rate of the furnace is maintained at 15 °C per minute. The temperature of the furnace is maintained precisely by PID controller. After removal from the furnace, final weight of each sample is measured to calculate the percentage of weight loss. The experiments are carried out for different residence time from 30 min to 75 min. For different experiments, fresh samples are used. In overall, the heating cycle is conducted in the muffle furnace at different temperatures (400-1200 °C) at incremental increase of 50 °C each, residence time (30-75 min) and bed heights (16-64 mm). Changes in the iron ore phases with increase in temperature were supplemented through XRD analysis.

#### 3. Results and discussion

#### 3.1. Non-isothermal condition

Initially a few experiments are carried out non-isothermally to understand the effect of increasing temperature on the LOI removal. Firstly, some low mass SDTA analysis is done to find out the temperature when the chemical water starts releasing the hydrated hematite ore. The change in LOI of iron ore sample is observed through the loss of weight at different temperatures using SDTA/TGA as shown in Fig. 1.

Fig. 1 shows that the removal of LOI starts at a temperature of 200 °C and loses around 77% and 86% at 350 °C and 500 °C, respectively. Following this some non-isothermal experiments are carried out for high mass system in fixed bed. Here, temperature is increased in step wise fashion and the sample is soaked for 30 min at each temperature.

Fig. 2 shows the cumulative loss with increase in temperature taken with different bed depths. In this case the sample is continuously roasted at temperatures ranging from 200 °C to 1200 °C with incremental increase of 50 °C at different bed depths (16-64 mm) for a residence time of 30 min at each temperature. In these experiments the sample is loaded once in the beginning and not changed after each observed temperature to know the cumulative removal of LOI. Similar experiments are carried out for different bed depths. It is observed that LOI removal follows S-shaped curve. Initially up to 300 °C, the LOI removal is insignificant and thereafter there is a steep rise in LOI removal to around 90% at temperature of 700 °C for all depths of bed. Subsequently, the LOI removal almost comes to standstill with increasing temperature in irrespective of depth. In respect to bed depth, initial percentage of removal of LOI shifts towards higher temperature with increase of bed depth in the range of 400 °C to 700 °C. This is in contrast to observe in low mass TGA experiments where LOI is removed at low temperature between 250 °C and 350 °C. The shift in temperature range between low (around 36 mg in TGA) and high (1000 g in experiment) mass experiments may be attributed to decrease in specific reaction surface area for large mass sample. In fact, the reaction surface area (cross section of the bed) remains the same irrespective of the mass in the bed. Therefore, higher temperature is required to enhance the surface reaction rate to compensate for the reduction in reaction rate due to lower specific surface area for larger mass in fixed bed. While the

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