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Enhancing titania value of a weathered ilmenite by reduction roasting – acid leaching

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Abstract

In the present work, the titania (TiO_2) content of a weathered ilmenite was enhanced by an improved reduction roasting process incorporating a flux. Addition of flux in to ore-charcoal pellets enabled the complete conversion of iron oxide to metallic iron at relatively lower temperature. The reduced ilmenite was leached using hydrochloric acid at 80 °C for 3 h. Results showed that reduction with charcoal yielded 70.4 wt.-% TiO_2 in the product whereas addition of soda ash along with charcoal enhanced the TiO_2 to 91.25 wt.-%. Investigations using X-ray Diffraction (XRD) provided information on presence of mineral phases during the reduction-roasting process.
Keywords: Ilmenite; Charcoal; Titanium dioxide; Catalyst; XRD

1. Introduction

Titanium dioxide (TiO_2) or titania is commonly used in the manufacture of paints, pigments, ceramics and welding rod coating. Due to its good semiconducting and photo sensibility behavior it is also used in the solar cell (Ray et al., 1985). TiO_2 is commercially produced from the Ilmenite ($FeO.TiO_2$) ore for the extraction of titanium and production of other chemicals. A number of processes have been developed to enrich the TiO_2 content of the ilmenite (Diebold, 2003; Kucukkaragoz and Eric, 2006) and some of them have been commercially exploited as well (Habashi, 1997). Iron oxide being the main impurity has been removed using several methods including reduction in presence of an appropriate reductant. Other than carbon as reducing agent, hydrogen has also been used owing to better kinetics of iron oxide reduction (Chen et al., 2013). The reduced iron is then leached in hydrochloric acid to produce TiO_2 rich residue. During reduction various other additives viz. sodium carbonate and lime have been also used (Abdel-al et al., 2000; Sankar et al, 2006). Among the several processes, reduction roasting followed by leaching is an efficient way to produce titanium dioxide from ilmenite ore. However, the ilmenite lying under natural condition undergoes weathering and generates altered mineralogy. The weathered ilmenite possessing altered mineralogy may exhibit different reduction behavior. The present investigation is based on a route where studies have been done with sodium carbonate and charcoal addition. The reduction behavior of ilmenite under the effect of temperature and time has been described. Role of sodium carbonate during reduction process is investigated using XRD.

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2. Experimental procedure

The ilmenite ore used in the present studies was obtained from Tata Steel, Jamshedpur. The ore contained 51.16% TiO_2, 20.80% Fe_2O_3, 25.08% FeO, 1.15% Al_2O_3, and 1.14% SiO_2. The charcoal used was of commercial grade with more than 90% fixed carbon. The sodium carbonate was of analytical grade. The reduction experiments were done in a raising hearth furnace where ilmenite pellets mixed with additives were dipped in a coal bed. This coal bed made of jhama coal acted as a reductant. The proximate analysis of jhama coal used was, 48.71% fixed carbon, 13.26% volatile matter, and 36.44% ash. After dipping the ilmenite pellets in coal bed the crucible was kept in the furnace. The reduction was done for a predetermined temperature and time. After reduction the pellets were separated from the coal and ground to 100 mesh size. The powder mass was then leached in hydrochloric acid at 20 wt% concentration for 4 h at the temperature of 90°C to produce TiO_2 rich residue. The ore, reduced product and leach residue were characterized by X-ray diffractometer (XRD) using Brüker D-8 Discover XRD instrument with Cu target employing Cu-Kα rays (1.54 Å wave length) at scan speed of 0.5 sec/step with increment of θ by 0.02° from 10 to 90°. The d-values were matched with standard JCPDS file data for identification of phases.

3. Results and discussion

3.1. Thermal analysis of ilmenite roasting

Thermal analysis of the ilmenite pellet was carried out to predict the thermal behavior with/without flux. Significant loss in the mass was seen in the TG/DTA plot of 5% charcoal mixed ilmenite as shown in Fig. 1a. The DTA plot did not show any marked drift. Whereas, in Fig. 2 the addition of 5% sodium carbonate along with 5% charcoal showed two drifts in the DTA plot. The first drift at about 800°C indicated the decomposition of sodium carbonate. Another drift at about 1000°C indicated chemical reactions between ilmenite ore and additives including the reduction of iron oxide as given in reactions (1-5).

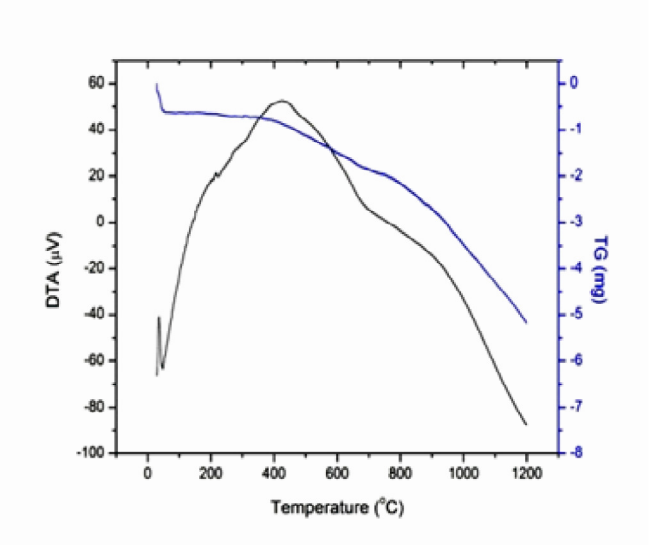


Fig. 1 TG/DTA plot of ilmenite with 5% charcoal.

$Na_2CO_3 \rightarrow Na_2O + CO_2$ (1)

$CO_2 + C \rightarrow 2CO$ (2)

$FeO.TiO_2 + CO \rightarrow Fe + TiO_2 + CO_2$ (3)

$Fe_2O_3 + CO \rightarrow 2FeO + CO_2$ (4)

$FeO + CO \rightarrow Fe + CO_2$ (5)

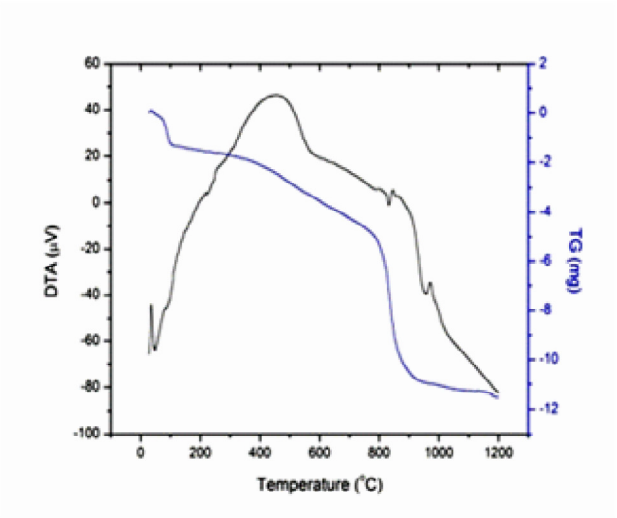


Fig. 2 TG/DTA plot of ilmenite with 5% charcoal and 5% sodium carbonate

3.2. Effect of reduction temperature with charcoal/sodium carbonate

Before studying the effect of sodium carbonate addition in pellets, the effect of only 5% charcoal was studied. It was found that the reduction of iron in ilmenite was temperature dependent. At temperature 1000 °C, the titania content in the product was 65.8%, which increased with the increase in temperature. Consequently, TiO_2 content was improved up to 78.24% at 1150°C by 5% mixing charcoal in the ilmenite pellets.

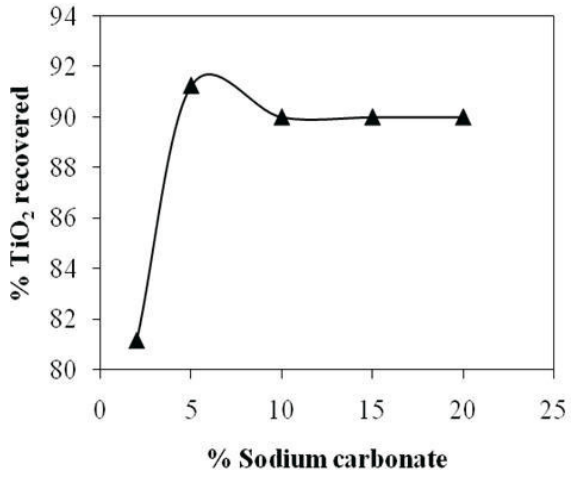


Fig. 3 Variation of sodium carbonate on recovery of TiO_2

It was also found that with the addition of 2% (w/w) sodium carbonate, the recovery of titanium dioxide increased with increase in temperature and time. Besides, comparatively lower temperature was required than that with only charcoal addition. For instance, at 1050°C, maximum 81.16% of TiO_2 was recovered in 3 h duration. The reduction with 5% charcoal and 5% sodium carbonate addition gave better result. As shown in Fig. 3, where variation of sodium carbonate was done with fixed 5% charcoal. The experiment was carried out at 1050°C temperature for 3 h duration. It can be seen that addition of sodium carbonate up to 5% (w/w) enhanced the TiO_2 content to 91.25% in 3 hr duration. Thereafter, no marked change in the recovery of TiO_2 was observed.

3.3. XRD analysis

The ilmenite ore, reduced product and leach residue were characterized by XRD. The objective was to know the phases evolved in the roasted and leached product to understand the process. The XRD phase identification of optimum condition products are depicted in Table1, where the various phases found are listed.

Table 1 Phases identified by XRD

Product	**Major phase**	**Minor phase**
Ilmenite ore	TiO_2	FeO Fe_2O_3 $Fe_2Ti_3O_9$
Reduced ore	TiO_2 Fe	$NaAl_6O_{9.5}$ $NaFeTi_3O_8$
Leaching product	TiO_2	$Ti_4O_7(H_2O)_{0.25}(OH)_2$

It can be seen from the Table 1 that the ore has TiO_2 as major phase which implies the advanced weathering of $FeO.TiO_2$ phase giving rise to rutile and hematite phase. The iron phase is mainly present as FeO and Fe_2O_3. However, pseudorutile ($Fe_2Ti_3O_9$) is also found to present as minor phase in the ore. The reduced ore shows presence of Fe metal and TiO_2 as major phase. During reduction the $Fe_2Ti_3O_9$ phase undergoes reduction and completely breaks into TiO_2 and FeO. Thereafter, FeO and the Fe_2O_3 further reduce to metallic Fe and TiO_2 remains as major phase. The minor phases in the leached product include titanium aqua oxide hydroxide. On the other hand, presence of TiO_2 as the only dominant phase confirmed the improved titania content of the leached product in the present investigation.

4. Conclusions

Studies on reduction of ilmenite by charcoal and sodium carbonate showed that the incorporation of sodium carbonate in to the charcoal mixed ilmenite produces a higher amount of titanium dioxide by reduction roasting. At 1050°C the amount of titanium dioxide formed with and without sodium carbonate were 91.25% and 70.4% respectively. The XRD analysis showed evolution of metallic iron in the roasted product which disappeared in the leached product implying removal of iron.

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