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Ilmenite Reduction Studies Using charcoal and Petroleum Coke as Reductant

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ABSTRACT

The ferric content in the ilmenite is reduced to ferrous by carbothermic reduction using petroleum coke and charcoal as reductant. A mixing of 1:3 ratios of petroleum coke and charcoal gives the maximum reduction efficiency. The optimum time for the reduction was found to be 15 min, and the optimum temperature was 900°C. The reduction follows first order kinetics and the rate of the reaction was 0.22/min.

Key words: *Ilmenite, Reduction process, Petroleum coke, Charcoal.*

1. INTRODUCTION

The ilmenite is the major raw material for titanium industries. The "Q" grade ilmenite contains approximately 60% of titanium dioxide. The major impurities present in the ilmenite is the iron oxide in the form of ferric oxide (25%) and ferrous oxide (10%) The ilmenite is upgraded to synthetic rutile (beneficiated ilmenite) by removing the iron content and thereby enriching the titanium value to 92-95%. Ilmenite is first subjected to carbothermic reduction to convert the ferric oxide to easily acid leachable ferrous state followed by leaching with hydrochloric acid to remove the iron content in the ilmenite. The quality of synthetic rutile is governed by the titanium content, particle size, and color. The good quality synthetic rutile contains around 93-95% titanium dioxide; the particle size should be between US mesh –40 +140 and bright golden yellow in color. Different types of reductant varying from solid to gas are used for the reduction of ilmenite [1-6]. The efficiency of the reduction process depends on the ratio of ferric and ferrous present in the ilmenite, the crystalline parameters, surface roughness, and also the quality parameters of reductant. The carbaneseous type reductant are widely used due to its easy availability, easiness of handling, and cost factor. In carbon type reductant, the volatile matter present, fixed carbon value and moisture content present are playing key role for in the reduction process. As the particle size of the reductant decreases, the reduction efficiency will be improved, but very fine size reductant cannot be used due to environmental contamination and losses of the reductant. Usually, the reduction process is carried out in a rotary kiln at high temperature, where air draft will pick up the fine particles and it should be a loss through stacks. Pre-oxidation and subsequent reduction are employed for effective reduction of ilmenite, but it is not feasible in a practical scenario due to energy waste and economy. This paper deals with the reduction of ilmenite with a mixture of petroleum coke and charcoal in the varying ratios at different temperatures (700-1000°C). The individual performance of the reductant was also studied for evaluating the synergistic effect of the reductant mixture.

2. EXPERIMENTAL

2.1. Raw Material (Ilmenite)

Q grade ilmenite is selected for the study. The particle size distribution and typical composition of the Q grade ilmenite are given in Tables 1 and 2.

The Q grade ilmenite contains nearly 60% of titanium dioxide, 25% of ferric oxide, and 10% ferrous oxide.

2.2. Reductant (Petroleum Coke and Charcoal)

The petroleum coke and charcoal were selected as the reductant the particle size distribution and composition of the reductant selected for this study are given in Tables 3 and 4.

The ash content in the charcoal was on a higher side compared to petroleum coke which may cause a chance to decrease the reduction efficiency. The higher moisture content and volatile matter in the charcoal compared with petroleum coke enhance the reduction and that compensate the high ash content.

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2.3. Reduction Studies

The reduction studies were performed on SS316 cylindrical tube of capacity 100 CC closed at one end. The other end is threaded and fitted with an air tight lid which can be opened and closed, as and when required. A small pin hole is given on the second bottom thread of the tube which is used to escape the gas formed inside the tube during the reduction process. The muffle furnace is used for heating the reduction cylinder during the reduction process. Usually, 10 g of ilmenite and 1.0 g of the reductant were added to the reduction tube. The temperature of the furnace is adjusted to get suitable temperatures for the study (700°C-1000°C). The time of reduction process varied from 5 min to 30 min.

Table 1: Particle size of ilmenite

Mesh size	Retained (%)	Cumulative (%)
+40	Nil	Nil
−40 +60	6.7	6.7
−60 +80	24.0	30.7
−80 +100	42.6	73.3
−100 +140	20.0	93.3
−140 +200	3.1	96.4
−200	3.6	100

Table 2: Composition of the ilmenite

Contents	%
TiO_2	59.8
FeO	9.9
Fe_2O_3	24.9
SiO_2	1.0
Al_2O_3	1.0
V_2O_5	0.2
Cr_2O_3	0.2

Table 3: Sieve analysis of petroleum coke and charcoal

Sieve number	Charcoal (%)	Petroleum coke (%)
+5	15.4	9.7
−5 +10	39.5	35.6
−10 +80	30.1	34.4
−80 +100	14.0	18.9
−100	1.0	1.4

Table 4: Proximate analysis of the reductant

Parameters	Charcoal (%)	Petroleum coke (%)
Moisture	11.7	5.5
Volatile matter	16.6	16.5
Ash content	10.0	3.0
Fixed carbon	61.7	75.0

3. RESULTS AND DISCUSSSION

3.1. Reduction Efficiency with Varying Ratio of Petroleum Coke and charcoal

10 g of ilmenite is taken in the reduction tube. 1 g of reductant mixture containing varying ratio of petroleum coke and charcoal were admixed in the reduction tube. The tube is heated at 900°C for 15 min. The reduction efficiency of the ilmenite with varying ratio of petroleum coke and charcoal is given in Table 5. The reduction efficiency was calculated based on the amount Fe_2O_3 before and after reduction in the ilmenite.

The reduction efficiency increases with an increase in the proportion of charcoal in the reductant mixture. The maximum reduction efficiency was noticed using a reductant mixture containing 75% of charcoal and 25% of petroleum. Thus, 1:3 mixing of petroleum coke and charcoal is considered as the optimum reductant mixture combination and further studies were conducted with this combination only.

3.2. Reduction Efficiency with Temperature

The reduction efficiency with temperature varying from 700°C to 1000°C is given in the Table 6. Here, the reductant is the mixture of 1:3 proportion of petroleum coke and charcoal. The reduction time was set us 15 min.

The reduction efficiency increases with increasing the reduction temperature. The percentage of reduction

Table 5: Reduction efficiency with respect to a different ratio of petroleum coke and charcoal as reductant

Ratio of reductant		Reduction efficiency %
Petroleum coke	Charcoal	
100	0	71.0
75	25	73.5
50	50	78.4
25	75	81.0
0	100	80.0

Table 6: Reduction efficiency with temperature

Temperature °C	Reduction efficiency %
700	50.0
800	68.2
900	81.0
1000	82.2

efficiency above 900°C is marginal. Moreover, there will be a chance of metallization of iron and also a reduction of titanium from +4 oxidation state to +3 oxidation state occurs above 1000°C with reduced atmosphere. Hence, 900°C is taken as the optimum condition for the reduction studies.

3.3. Reduction Efficiency with Time

The reduction efficiency of ilmenite with varying the reduction time (5 min-30 min) is given in Table 7.

The reduction efficiency increases with increasing the reduction time of the process. There is not much improvement of the reduction when the reduction time goes above 15 min. Conducting reduction process above 15 min will lead to energy loss without improving the quality of reduced ilmenite considerably. Hence, 15 min of reduction of ilmenite can be considered as the optimum time for reduction. The reduction efficiency linearly rises up to 15 min, then remains almost steady values. The initial concentration of ferric oxide in ilmenite is represented as “a” and “x” is the quantity of ferric oxide converted to ferrous at particular interval of time. A graph was plotted with ln (a/a-x) against time up to 15 min, and the plot is given in Figure 1.

The graph is linear with squire of regression 0.9977 suggests that the reduction process follows first order kinetics up to 15 min. The rate of the reaction was found out from the slope of the graph, and the value is 0.22/min.

4. CONCLUSION

1. The petroleum coke and charcoal can be used alone and mixed in varying composition for reducing ilmenite in the ilmenite upgradation process. The optimum mixing ratio of petroleum coke and charcoal as reductant is found to be 1: 3.
2. The optimum temperature for the reduction process is found to be 900°C, below which the reduction is incomplete and above which the metallization started.
3. The optimum the reduction time was found to be 15 min. There is no considerable increase in reduction above 15 min rather than the wastage of energy for the reduction process. A linear line obtained by plotting ln (a/a-x) against time indicates that the reduction follows first order kinetics and the rate of the reaction was found to be 0.22/min.

Table 7: Reduction efficiency with time

Time of reduction	Reduction efficiency %
5	50.0
10	68.2
15	81.0
20	81.2
25	81.4
30	81.4

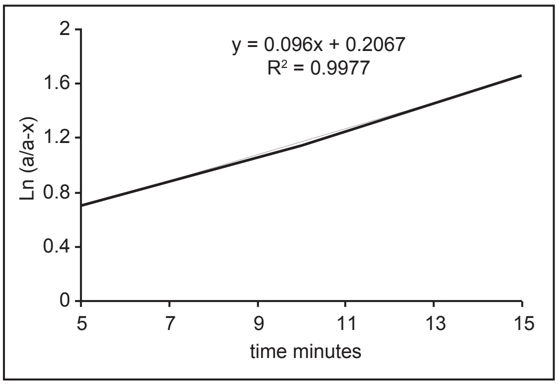

Figure 1: A plot of ln (a/a-x) against time for the reduction process.

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***Bibliographical Sketch**

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