

Up-grading of Sri Lankan Ilmenite

by

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Abstract : Ilmenite, rutile and leucoxene are the only naturally-occurring titanium bearing minerals that have been seriously considered as suitable feed stock for either the metal producing or pigment industries.

The existence in many places in the coast of Sri Lanka of natural concentrates of titanium bearing mineral sands such as ilmenite and rutile with a high degree of purity has been known as far back as 1903. These deposits are found as beach sands on the east and north-west coast and occur in association with others, namely, rutile, zircon and monazite.

The beach sand industry of the country, operated by the Ceylon Minerals Sands Corporation produces about 70,000 tonnes of ilmenite per year and about 10,000 tonnes per year of rutile.

If Sri Lanka were to convert the ilmenite produced here into a value added product, instead of exporting the raw material as such, it would enable us to make sizeable strides in industrial development.

The work carried out showed that Sri Lankan ilmenite can be converted to synthetic rutile containing about 95% TiO_2 by oxidation-reduction-leach process.

The oxidized ilmenite was reduced with carbonaceous materials to reduced rutile and iron. Iron can be leached out using dilute HCl acid. Reduced rutile phase was calcined to obtain synthetic rutile. The paper will discuss the main steps involved in this process.

Ilmenite, rutile, and leucoxene are the only naturally-occurring titanium-bearing minerals that have been seriously considered as suitable feedstock for either the metal producing or pigment industries. This is because only these minerals are found in large enough commercial concentrations; compared with other naturally occurring minerals containing titanium, to be suitable for industrial exploitation.

The existence in many places on the coasts of Sri Lanka of natural concentrates of titanium-bearing mineral sands such as ilmenite and rutile with a high degree of purity has been known since as far back as 1903. Over the last few decades systematic work on the beach sands of the islands referred to as "black sands" has been carried out by the Geological Survey Department of Sri Lanka and the distribution of these deposits, and their mineralogy is now known (Geological Survey of Ceylon, 1970: Herath, 1980).

These deposits in Sri Lanka are found as beach sands on the east and north west coast and occur in association with others, namely, rutile, zircon and monazite. The largest and most important beach-sand deposit occurs at Pulmoddai on the northeast coast of the island, 34 miles north of Trincomalee. The deposit is approximately 4 miles in length with an average width of about 200 feet. The richest part of the deposit is the southern part, where the percentage of heavy mineral amounts to 95%. The approximate composition of the sand is as follows: ilmenite ($\text{FeO} \cdot \text{TiO}_2$) 70-90% w/w; rutile (TiO_2) 8-12% w/w; and zircon ($\text{ZrO}_2 \cdot \text{SiO}_2$) 8-10% w/w. Besides the Pulmoddai deposit there are other deposits of mineral sands at several scattered points on the west coast from Kundiramalai Bay on the northwest coast to Kirinda in the south. These deposits contain heavy-mineral contents ranging from 10-20%, while occasionally the concentration rises over 80%. In all these deposits ilmenite is the main constituent with zircon next in order of importance (Ministry of Industries, 1969).

Table I gives the results of chemical analysis of ilmenite from Pulmoddai.

TABLE I
Chemical composition of Sri Lanka ilmenite (Herath, 1980)

Oxide	%w/w
SiO_2	0.38
TiO_2	53.61
Al_2O_3	0.54
Fe_2O_3	20.95
FeO	20.67
MnO	0.95
MgO	0.92
CaO	0.05
Cr_2O_3	0.05

The beach sand industry of the country, operated by the Ceylon Minerals Sands Corporation produces about 70,000 tonnes of ilmenite per year and about 10,000 tonnes per year of rutile. Due to the lack of adequate development in the field of titanium technology in Sri Lanka, there is no consumption of these titanium minerals. At present there is no single unit in Sri Lanka which makes use of ilmenite or rutile on a large scale, and the country has been exporting several thousand tonnes of titanium concentrates for the last so many years, primarily to pigment manufacturers, without any beneficiation. If Sri Lanka were to convert the ilmenite produced here into a value added product, instead of exporting the raw material as such, it would enable us to make sizeable strides in industrial development.

During the last two decades, many processes have been proposed for upgrading ilmenite, including high-temperature smelting, direct acid leaching methods, and reduction processes in which the iron content is reduced either to ferrous oxide and extracted with acid or to metallic iron and removed by acid leaching or accelerated corrosion (Geological Survey of Ceylon, 1970; Shigaki; 1976).

Having studied most of these methods, we followed the oxidation-reduction-leaching process. A systematic study of each stage was carried out to obtain the optimum conditions for Sri Lankan ilmenite.

Ilmenite from the separation plant at Palmoddai was used throughout the present work. X-ray powder diffraction analysis showed that ilmenite contains small fraction of rutile and pseudorutile $\text{Fe}_2\text{O}_3 \cdot 3\text{TiO}_2$. (Teufer and Temple, 1966). This is a transition phase between ilmenite and rutile. Results of chemical analysis are given in Table I.

Oxidation of ilmenite ore was carried out in temperature-controlled furnaces. Formation of fine pores on the ilmenite particles was observed from SEM photographs of the oxidised product. The presence of microbacks and pores in the ilmenite particles facilitates the reduction of ilmenite to rutile. Therefore this prior oxidation step is required. The major products formed by oxidation of ilmenite at temperatures greater than 900°C were ferric pseudobrookite and rutile. Oxidised ilmenite is then reduced in a closed vessel with saw dust as the reductant. Saw dust is found in large quantity as a waste material in Sri Lanka and the experiments carried out showed that the saw dust reduces the iron values in ilmenite to acid-leachable form.

A typical chemical analysis of the saw dust used is given in Table II

TABLE II

Chemical composition of saw dust

Component	%w/w
C	50.07
H ₂	6.45
O ₂	42.95
SiO ₂	0.21
CaO	0.21
MgO	0.03
Fe ₂ O ₃	0.02
Al ₂ O ₃	0.11
TiO ₂	0.00
K ₂ O	0.01
Na ₂ O	0.03
Total	100.09

The first stage of reduction is conversion of Fe^{3+} in pseudobrookite to Fe^{2+} in the form of ilmenite. The second stage of reduction is conversion of Fe^{2+} to metallic iron. Hence the ilmenite concentration progressively decreased and there was corresponding increase in the concentration of metallic iron, and reduced rutile. The optimum temperature for the reduction was found to be 1100°C .

This reduced product was leached with 15-20% HCl and the leached product was then calcined at 1000°C to get synthetic rutile having 90-95%TiO₂. The chemical composition of this product is given in Table III.

TABLE III

Chemical analysis of synthetic rutile

Component	% w/w
TiO ₂	95.97
Fe ₂ O ₃	1.57
MgO	0.86
Al ₂ O ₃	0.80
MnO	1.25
SiO ₂	0.69