

# Ilmenite from the Chavara deposit, India: a critical evaluation

C. RAMAKRISHNAN, R. MANI\* AND D. S. SURESH BABU

Centre for Earth Science Studies, P.B. No. 7250, Thuvikkal P.O., Trivandrum-695 031, India

## Abstract

The mineralogical status of ilmenite from a prominent modern placer deposit of India is illustrated using different techniques. Compositional, powder XRD, TGA and SEM analyses prove that consistent low temperature alteration took place in Chavara ilmenite leading to the formation of a pseudorutile–hydrated-ilmenite stage. Except for the indication of a geochemical zonation at the southern tip of the deposit, the Chavara ilmenite points to a common origin throughout its 22 km stretch along the coast. We advocate a major segregation activity by marine factors to the accumulated sediments in the shelf zone in order to originate such a huge deposit of homogeneous composition.

KEYWORDS: ilmenite, weathering/alteration, placer, Chavara, Kerala.

## Introduction

THE Chavara beach placers (Kerala state, India) have been studied by many authors as they contain significant quantities of valuable minerals, mainly ilmenite (Tipper, 1914; Gillson, 1959; Prabhakara Rao, 1968). Several workers have also made characterization analyses on ilmenites of world-wide deposits to assess the qualitative variation from deposit to deposit (Bailey *et al.*, 1956; Temple, 1966; Wort and Jones, 1980). There are many recent publications on ilmenite alteration (Frost *et al.*, 1983; Hugo and Cornell, 1991; Mitra *et al.*, 1992). Although the mineralogy, provenance sediment parameters and micromorphology are reported extensively from the Kerala placers (Mallik *et al.*, 1987), a detailed characterization analysis on ilmenite, which forms the chief component of the placers (>70%), is lacking. Ilmenite characterization has been carried out for the Manavalakurichi placers, another west coast deposit of India, which has a limited fluvial source unlike the Chavara placer (Subramanyam *et al.*, 1982; Suresh Babu *et al.*, 1994; Nair *et al.*, 1995). Here, the compositional, mineralogical and surficial changes of ilmenite from Chavara, the single largest deposit of its

kind in the world, are reported to study the degree of alteration undergone by this mineral.

## Background information

The study area, Chavara, falls between E longitudes 76°27'36" and 76°33'44" and latitudes 9°8'24" and 8°56'3" in the SW fringe of south Indian Precambrian shield. Geographically it lies within the state of Kerala, which is flanked by the Arabian Sea in the west and Western Ghats in the east. The Chavara beach placer deposit, consisting of ilmenite, leucoxene, zircon, rutile, monazite, garnet, sillimanite and kyanite, is spread over an elongated distance of 22 km, with an average width of 1.5 km in the coastal zone of Neendakara–Kayamkulam area. It has alternating bands of light and heavy minerals up to a depth of about 15 m. These are reported to be derived from the country rocks, viz. khondalites and charnockites lying in the eastern portion of the deposit, by the fluvial transportation of at least nine rivers in the area and subsequent differentiation into black sand and glass sand deposits (Suresh Babu and Thirvikramaji, 1993). At present the enrichment of heavy minerals is taking place by wave action from the sea and modern rivers bringing sediments towards this area are discharging into the coastal back waters bordering the eastern side of the placers and thereby not contributing to the deposit zone.

\* Department of Geology, Annamalai University, Annamalai Nagar-608 002, India.

### Sampling and analytical techniques

Sampling was done along eight traverses, i.e. along three longitudinal directions parallel to one another in the Neendakara-Kayamkulam sector. The samples of the first stretch belong to the modern beach (the BS series); the second and third stretches are from the western (WS series) and eastern (ES series) sides of the kayal respectively (Fig. 1). The subsurface samples from the eastern side are represented as ESS samples. In total, thirty seven samples have been collected, of which fourteen samples were scooped out from a depth of about 1 metre beneath the ground level using an auger hand drill.

Ilmenite concentrates were separated with the help of a Frantz Isodynamic separator followed by wet panning and sieving of the samples. Pure ilmenite concentrate is obtained by hand picking under stereo microscope. Chemical analysis was carried out by standard wet chemical methods, aluminium reduction method for  $\text{TiO}_2$  and dichromate titration for iron (Vogel, 1961). AAS was used to detect the amount of three trace elements, viz. Mg, Mn and Zn. X-ray powder diffraction (XRD) studies were made using a Philips diffractometer (Cu- $K\alpha$  radiation, Ni-filter). The determination of the lattice constants  $a$ ,  $c$  and  $V$  was done with the help of a LCLSQ programme using  $d$  values measured from powder XRD reflections. The semiquantitative XRD phase analyses for the amounts of ilmenite and pseudorutile/rutile/anatase were determined using a Planimeter. Thermogravimetric analysis (TGA) experiments of eight selected samples was carried out using a Shimadzu TGA 50H at a heating rate of  $15^\circ\text{C}/\text{min}$ . Some samples were mounted using polyester cold setting resin for observation in a scanning electron microscope.

### Results and discussion

It is apparent that there is a systematic lowering of ferrous and total iron contents in ilmenite samples eastwards from the shore line (i.e. from BS to ES), especially in the case of V-VIII traverses (sampling sections) (Figs. 2 and 3a). The WS set of samples generally occurring between the ES and BS sample sets show intermediate values in the case of ferrous and total iron contents. The subsurface (ESS) samples do not significantly vary with the corresponding surface (ES) samples in terms of major element content. Compared with this lateral variation in the east-west profile, the major element changes in the longitudinal direction is minimum. Except for the southernmost traverse (I traverse), no significant variation is noticed in other sample sets in  $\text{FeO}$ ,  $\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$  content in the N-S direction. While all the samples have values of  $\text{FeO}$  between 5 and

10%, the samples of cross section I contain approximately 20%  $\text{FeO}$  (Tables 1 and 2).

In general the  $\text{Fe}_2\text{O}_3$  contents do not vary significantly (not more than 2%) whereas  $\text{FeO}$  and  $\text{TiO}_2$  show much higher variations (up to 6%) among the samples of the individual sections. This clearly shows the difference in the ferrous-ferric oxidation rates and the iron-removal rates (or the  $\text{TiO}_2$  enhancement rates) due to differential alteration of BS, WS and ES sample sets. Therefore, it is assumed that the internal variation in the east-west direction within each traverse, as noticed above for  $\text{FeO}$  and  $\text{TiO}_2$ , is systematic and would not interfere in the composition between any northern and southern traverse. Thus the major element data based on eight traverses prove that the  $\text{TiO}_2$  value for Chavara ilmenite varies between 61.21% and 63.59% except for the first section (57.5%). This disparity in the southern end of the deposit is clear in Fig. 3b. The insignificant variation between ES and ESS samples shows that the accumulation seen up to a depth of one metre belongs to a single phase of deposition. Thus, it can be seen that the Chavara ilmenite deposit is relatively homogeneous with respect to its 22 km lateral expansion and up to a depth of one metre.

Further, the parameters  $\text{FeO}/\text{Fe}_2\text{O}_3$  (Morad and Aldahan, 1986) and  $\text{Ti}/(\text{Ti}+\text{Fe})$  (Frost *et al.*, 1983) strongly support the earlier observations regarding the persistence of material with the composition of ilmenite (Fig. 4). Ilmenite occurs in four stages depending on the degree of weathering, namely ferrian ilmenite, hydrated ilmenite, pseudorutile and leucoxene (Frost *et al.*, 1983). The pseudorutile is a combination of goethite and rutile as an intergrowth structure (Grey *et al.*, 1983). Leucoxene contains more than 70%  $\text{TiO}_2$  and may consist almost entirely of crypto- or microcrystalline rutile and/or anatase in the final stages of alteration (Hugo and Cornell, 1991). Subsequent to the intermediate stages, ilmenite alters into either rutile (Temple, 1966) or anatase (Anand and Gilkes, 1984) and hematite. In this spectrum of ilmenite weathering, the observations show that the samples of the first section belong to the hydrated ilmenite type and the rest of the samples fall well within the lower limit of the pseudorutile stage in the case of the Chavara deposit. This assessment of all the 37 samples is made on the basis of  $\text{Ti}/(\text{Ti}+\text{Fe})$  ratio (Table 2). This consistent compositional behaviour indicates that the modern placer deposit originated from a pile of already accumulated sediments and underwent a specific degree of weathering in total to attain the pseudorutile stage of ilmenite alteration at Chavara. The slight but systematic increase in alteration eastwards from the shoreline side can be compared with the successive formation of the placer deposit from the shallow sea. The replenishing activity towards the

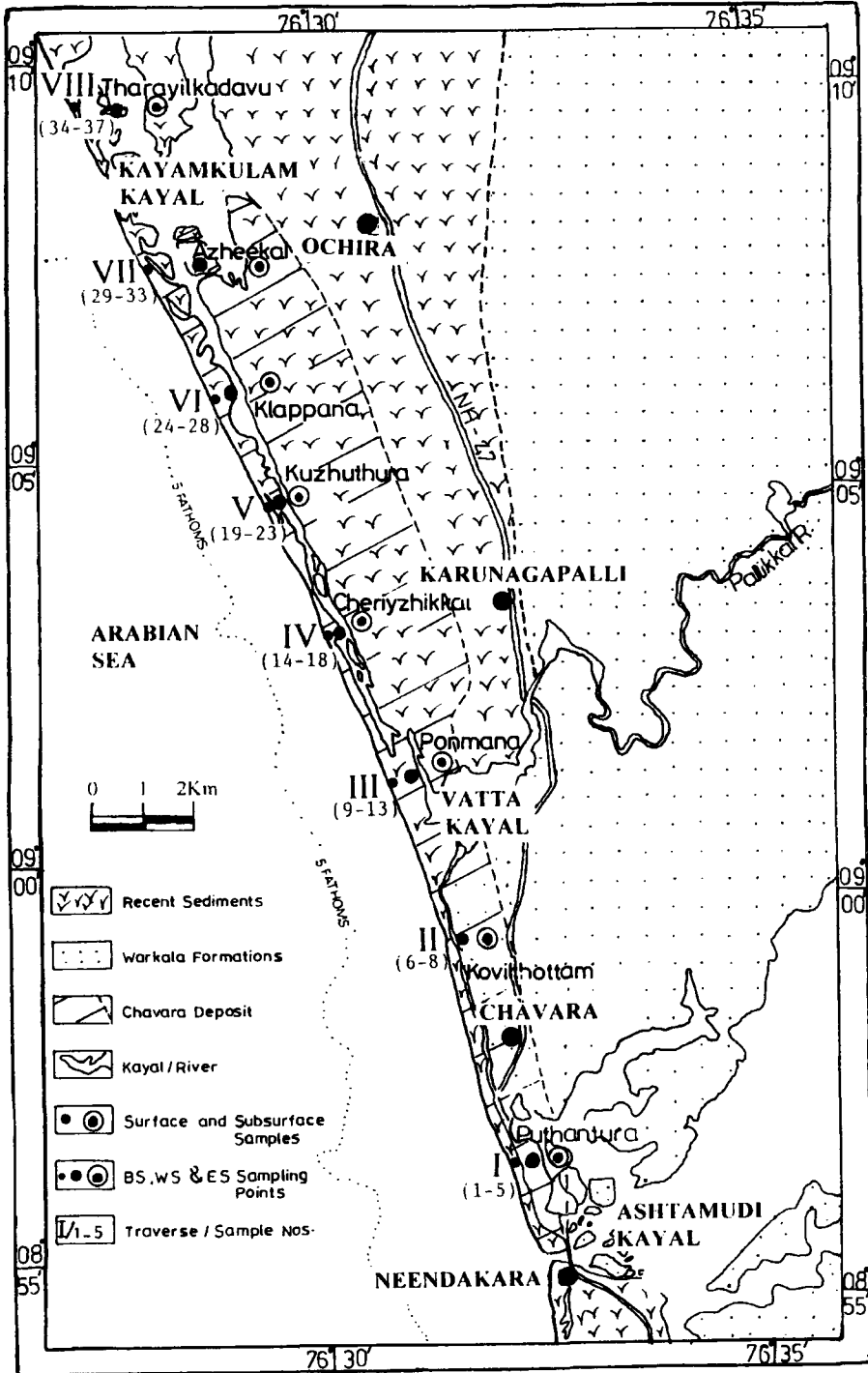


FIG. 1. Map showing sampling locations.

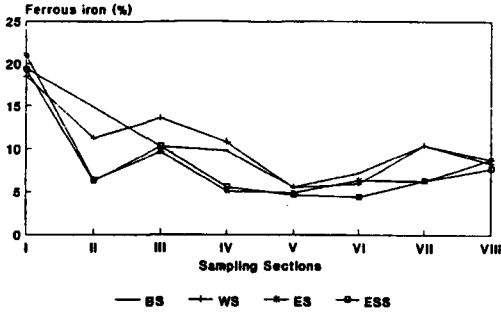


FIG. 2. Distribution of ferrous iron content in ilmenites along eight traverses of the Chavara deposit.

coast is being carried out by waves during every monsoon to this day.

Though this relatively uniform ilmenite concentrate slowly merges in the north with the Cherthala glass sands having syngenetic significance (Suresh Babu and Thirivikramaji, 1993), a marked geochemical variation is noticed in the southern segment of the deposit. This has been explained earlier as the I traverse of samples differ in composition from the rest and it can be linked to an accretion phase of second generation heavy minerals from the southern extremity towards the previously formed placers. The southern side of the deposit is bordered by a coastal promontory of crystallines at the Quilon area. The ultimate source rocks of placers is the charnockite-khondalite belt of south Indian Precambrian shield which outcrops widely in the south Kerala region (Soman, 1985). Therefore, the compositional and quality changes noticed in the ilmenite could only be due to exogenic processes undergone by the mineral after its release from crystalline rocks. In this background, the immediate source of less altered ilmenite noticed in the southern part of Chavara deposit (I traverse) can be attributed either to the

disintegration of Quilon promontory or to the modern sediments of R. Kallada being deposited near the southern side of the deposit (Ramakrishnan *et al.*, 1995). It is to be mentioned that Chavara ilmenite has undergone a higher degree of weathering compared with other placer deposits seen along the Indian coast. The substantial quantities of leucoxene fraction (400–500 tonnes per annum) being separated from this deposit support this view. Detailed analysis of the rutile component from the deposit would further reveal the intensity of ilmenite alteration. However, the systematic alteration of this huge deposit points to a large physical force like marine activity facilitating the accumulation of heavy minerals at a faster rate on the shore. The palaeogeographic considerations in this respect are discussed elsewhere (Suresh Babu and Thirivikramaji, 1993).

The trace element data (Mg, Mn and Zn) have revealed that Mn and Zn do not vary from place to place (Fig. 5). The Zn shows a value of 0.04 to 0.06 in all the analysed samples and hence is not plotted in the figure. It may be due to the "maturity" level of leaching that took place during the alteration stage. However, the MgO content shows higher variations from the mean value and it is either due to the differential geikeliitic substitution in the primary state or to the differential leaching which took place later. It is also noticed that the sample ESS-6b, which is the significantly altered one (TiO<sub>2</sub> is 64.5%), shows maximum values of MgO and MnO compared with the other samples. This may be due to the relative enrichment of these trace elements as alteration increases.

The XRD patterns of eight samples are given in Fig. 6. It shows that all the samples of ilmenite concentrates contain significant formation of pseudorutile and rutile phases barring the samples of the first two traverses. The XRD patterns of samples from traverse I show that the ilmenite peaks are sharp and the formation of pseudorutile and rutile is

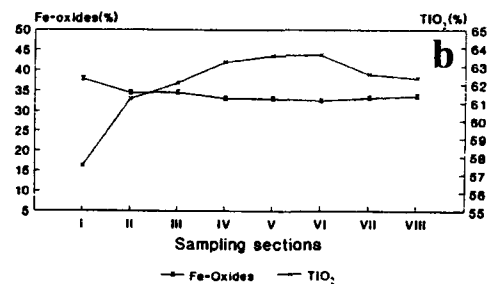
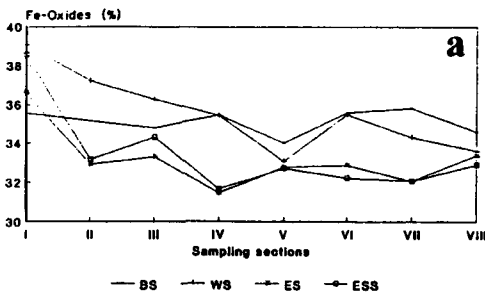


FIG. 3. Distribution of FeO+Fe<sub>2</sub>O<sub>3</sub> in BS, WS, ES and ESS samples of I - VIII sections (a) and FeO+Fe<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> (average) in I - VIII sections (b) in ilmenites.

TABLE 1. Major element data of ilmenite samples

Traverse no.	Sample no/ location*	Total iron	Chemical constituents (%)		
			FeO	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>
I	1. BS-1	26.16	19.47	16.11	60.37
	2. WS-1	28.54	18.53	20.55	56.53
	3. ES-1	29.09	20.96	18.67	56.39
	4. ESS-1a	28.61	19.58	19.48	56.21
	5. ESS-1b	27.87	19.00	19.07	58.03
II	6. WS-2	26.78	11.23	25.99	57.89
	7. ES-2	23.47	6.4	26.54	62.20
	8. ESS-2a	23.62	6.23	26.95	63.54
III	9. BS-3	25.02	10.34	24.46	61.95
	10. WS-3	26.24	13.63	22.60	61.35
	11. ES-3	23.94	9.71	23.60	62.36
	12. ESS-3a	24.74	10.03	24.40	62.38
	13. ESS-3b	24.62	10.59	23.61	62.40
IV	14. BS-4	25.46	9.78	25.70	62.04
	15. WS-4	25.52	10.78	24.69	60.94
	16. ES-4	22.36	5.12	26.36	64.27
	17. ESS-4a	22.57	5.38	26.37	64.26
	18. ESS-4b	22.49	5.78	25.83	64.53
V	19. BS-5	24.16	5.54	28.48	62.38
	20. WS-5	23.51	5.50	27.59	63.33
	21. ES-5	23.23	4.91	28.31	63.86
	22. ESS-5a	23.07	4.48	28.07	64.22
	23. ESS-5b	23.33	4.74	28.16	64.01
VI	24. BS-6	25.37	7.24	28.35	64.22
	25. WS-6	23.07	5.92	26.56	63.70
	26. ES-6	23.42	6.36	26.52	62.06
	27. ESS-6a	22.51	4.44	27.32	63.48
	28. ESS-6b	23.14	4.39	28.29	64.50
VII	29. BS-7	25.75	10.44	25.39	61.23
	30. WS-7	24.68	10.42	23.89	61.27
	31. ES-7	22.85	6.31	25.76	64.01
	32. ESS-7a	22.68	6.02	25.83	62.82
	33. ESS-7b	23.01	6.52	25.77	63.30
VIII	34. BS-8	24.79	8.86	25.75	63.02
	35. WS-8	24.07	8.38	25.24	62.41
	36. ES-8	23.94	8.81	24.59	61.23
	37. ESS-8a	23.54	7.75	25.17	62.69

BS - Beach sample, WS - Western side of Kayal/lagoon. ES - Eastern side of Kayal/lagoon ESS - Eastern subsurface sample.

insignificant. In sample ES-3, other than the significant formation of pseudorutile/rutile, anatase is only in negligible quantities along with broad ilmenite peaks. The formation of anatase as a product

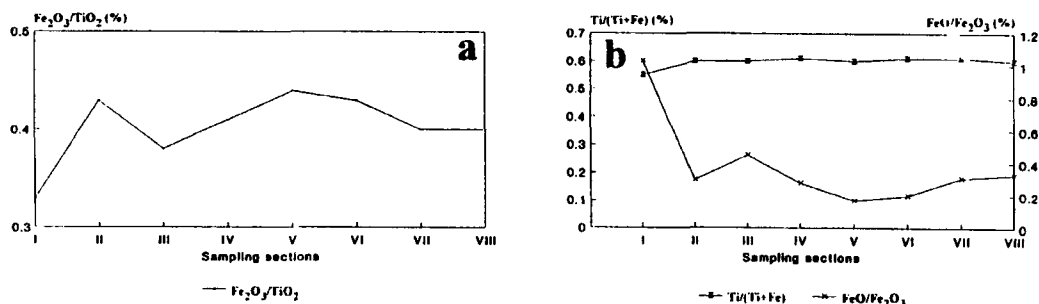
of ilmenite alteration was reported by Anand and Gilkes (1984). In samples ESS-6b and ES-7 the pseudorutile/rutile phase is apparently more prominent than the ilmenite phase, indicating a possible

TABLE 2. Chemical analysis of ilmenite samples (average) from eight cross-sections

Traverse	Total iron	Chemical constituents (%)						
		FeO	Fe <sub>2</sub> O <sub>3</sub>	FeO + Fe <sub>2</sub> O <sub>3</sub>	FeO/Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub> /TiO <sub>2</sub>	Ti/(Ti+Fe)
I	28.05	19.50	18.77	37.81	1.03	57.50	0.33	0.55
II	24.62	7.95	26.49	34.44	0.30	61.21	0.43	0.60
III	24.91	10.86	23.73	34.60	0.45	62.08	0.38	0.60
IV	23.68	7.36	25.79	33.15	0.28	63.20	0.41	0.61
V	26.46	5.03	28.12	33.07	0.17	63.56	0.44	0.60
VI	23.50	5.67	27.40	32.47	0.20	63.59	0.43	0.61
VII	23.79	7.94	25.32	33.27	0.31	62.52	0.40	0.61
VIII	24.08	8.45	25.18	33.63	0.33	62.33	0.40	0.60

progressive alteration towards the north-eastern portion of the deposit. The widespread co-existence of pseudorutile and rutile along with ilmenite indicate that the alteration of ilmenite to its daughter products (leucoxenisation) is intense in the study area. Pseudorutile/rutile peaks are broader compared with the sharp peaks of ilmenite, indicating the semi-crystalline nature of the altered phase. Thus the XRD analysis also explains the changes in chemistry of Chavara ilmenite, which is analogous to the intensity and amount of different phases present in the XRD reflections (Table 3). It is reported that, in addition to the phase changes, the unit cell parameters can be utilised as a tool for deciphering the degree of alteration in ilmenite (Chaudhuri and Newesely, 1990; Suresh Babu *et al.*, 1994) and the present XRD analyses are also compatible with these findings. Figure 7 represents the qualitative change in cell parameters while transformation takes place in ilmenite. It is observed that both volume *V* and *a* parameters show a general decreasing trend as alteration starts and then an increasing pattern as it proceeds. However, the lattice constant *c* depicts a reverse trend with an initial increase and then a decrease towards the weathering trend.

TGA experiments have been carried out for four samples in order to understand the high-temperature behaviour of the differentially-weathered ilmenite samples. The four samples are ES-1, ESS-1b, ES-3 and ESS-6b, having FeO of 20.96%, 19.58%, 9.71% and 4.39% respectively. An effective weight increase has been noticed in two samples (1.88% for ES-1 and 1.49% for ESS-1b as represented in Fig. 8). This could be due to oxidation of substantial FeO present in the samples. Samples ES-3 and ESS-6b show an effective decrease in weight of 0.58% and 1.17% respectively, when heated to 1000°C. The reasons for this change could be due to one or more of the following factors. Firstly, the initial ferrous iron content was too low to acquire weight gain. Secondly, the release of hygroscopic free water (~50°C) and structurally bound water (~300°C) causes loss in weight as in the case of every sample. The structural water (hydroxyls) represents the major weight loss component in ilmenite and it increases with increase in ferric iron content. Substantial ferric iron content in ilmenite is actually due to the formation of the pseudorutile phase which can be equated with FeOOH-TiO<sub>2</sub> mixtures (Grey *et al.*, 1983). Among the four samples, ESS-6b has the

FIG. 4. Distribution of Ti/(Ti+Fe) and FeO/Fe<sub>2</sub>O<sub>3</sub> in ilmenites.

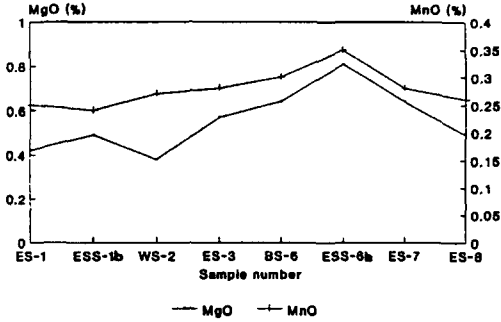


FIG. 5. Distribution of MgO and MnO.

maximum content of  $Fe_2O_3$  (28.29%) which, in turn, must contain more hydroxyl molecules and hence maximum decrease in weight. However, the bound water release ceases at temperatures below  $500^\circ C$  and then attains weight gain due to  $FeO-Fe_2O_3$  conversion. The changes in weight and the amount of bound water trapped in different samples are listed in Table 4. This shows that the significantly altered

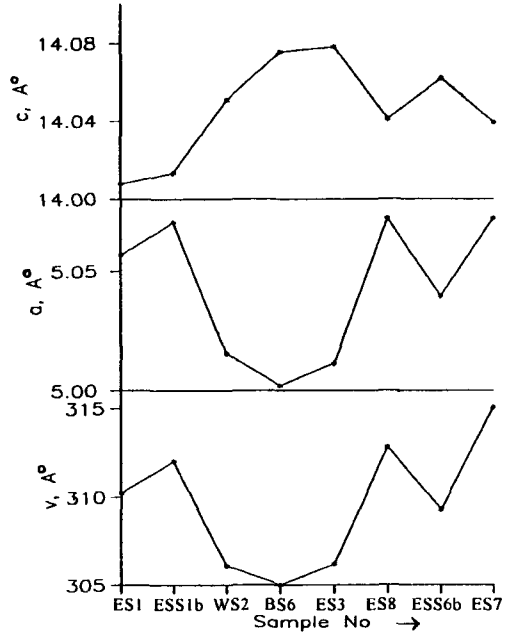


FIG. 7. Change in lattice parameters and cell volume.

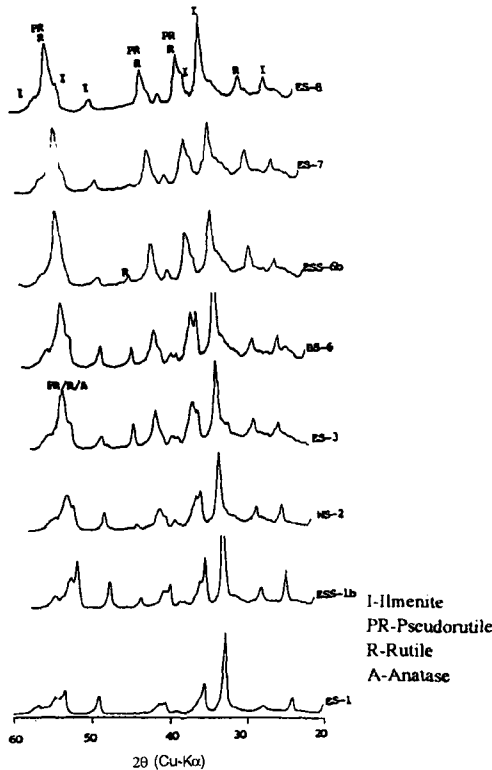


FIG. 6. Powder XRD patterns of ilmenite samples.

sample, ESS-6b (-1.122%) releases the bound water fully at  $581.4^\circ C$ . From the TGA data, it is concluded that the amount of FeO left out in the ilmenite grains is crucial for the high-temperature behaviour of iron-titanium oxides.

SEM observations of the samples revealed the nature of alteration and gave an insight into the compositional variation in ilmenites. The surficial patterns representing altered and unaltered zones on selected grains were photographed. The alteration

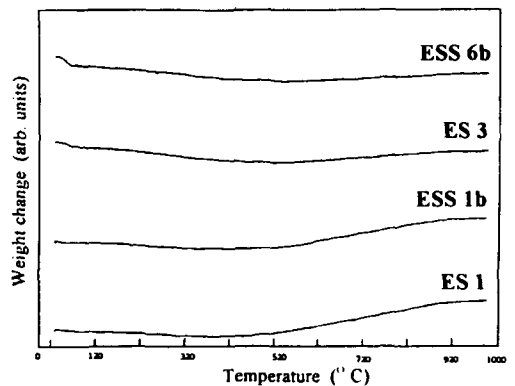


FIG. 8. TGA patterns of ilmenite.

TABLE 3. X-ray diffraction data of ilmenite samples

Sample	d	hkl	i*	Total I'	Area(%)# PR/R'	Sample	d	hkl	i*	Total I	Area(%)# PR/R	
ASTM No 3-0781	3.73	102	50			BS-6	3.71	102	w	44.71	55.29	
	2.74	104	100				2.73	104	vs			
	2.54	110	85				2.53	110	s			
	2.23	113	70				1.82	204	m			
	1.86	204	85				1.72	116	ms			
	1.72	116	100				1.63	108	mw			
	1.63	108	50									
ES-I	3.70	102	w	72.63	27.37	ESS-6b	3.71	102	ms	36.46	63.54	
	2.73	104	vs				2.73	104	vs			
	2.53	110	s				2.53	110	s			
	2.23	113	mw				1.85	204	w			
	1.86	204	m				1.72	116	m			
	1.72	116	ms									
	1.63	108	vw									
ESS-1b	3.72	102	m	62.08	37.92	ES-7	3.71	102	ms	42.45	57.55	
	2.73	104	vs				2.76	104	vs			
	2.54	110	s				2.55	110	s			
	2.23	113	mw				2.23	113	vw			
	1.86	204	w				1.87	204	mw			
	1.72	116	ms				1.72	116	m			
	1.63	108	vw				1.63	108	w			
WS-2	3.67	102	m	52.91	47.09	ES-8	3.71	102	ms	47.94	52.06	
	2.71	104	vs				2.73	104	vs			
	2.52	110	s				2.54	110	s			
	1.85	204	w				1.87	204	mw			
	1.71	116	ms				1.72	116	ms			
	1.63	108	mw				1.63	108	w			
ES-3	3.68	102	m	45.84	54.16^							
	2.71	104	vs									
	2.51	110	s									
	1.85	204	mw									
	1.71	116	ms									
1.63	108	w										

\* vs - very strong; s - strong; ms - medium strong; m - medium; w - weak; mw - medium weak; vw - very weak.

^ Pseudorutile/rutile/anatase

# Area under the peaks were determined using planimeter

' Ilmenite/pseudorutile/rutile.

apparently initiates from the periphery and also through micro-fractures. Figure 9a shows the development of microfractures and the initiation of ilmenite alteration. The secondary mineral formation in the altered zone (Fig. 9b) and the differential relief over ilmenite grains (Fig. 9c) are some of the surficial changes commonly seen.

## Conclusions

The present study reveals that a significant amount of alteration has taken place in the ilmenites of the Chavara beach placer deposit, resulting in the decrease of FeO to values as low as 4.39 wt.%. It is possible that the underlying zone from 1 metre to



TABLE 4. TGA data of ilmenite samples

Sample No.	Weight loss at ~50°C (of hygroscopic free water) %	Weight gain at 1000°C (for the oxidation of Fe <sup>2+</sup> ) (%)	Weight loss at 1000°C (of structural water) (%)
ES-1	NIL	1.880	-0.3099 (up to 449.8°C)
ESS-1b	NIL	1.496	-0.4474 (up to 465.4°C)
ES-3	-0.2746	-0.5875	-1.0264 (50°C-523.4°C)
ESS-6b	-0.5029	-1.179	-1.1220 (50°C-581.4°C)

15 metre depth in the Chavara sands is much altered, as the top 1 metre layer of the placer body has already attained the pseudorutile stage. From the systematic chemical analysis, it is also obvious that the Chavara ilmenite, in general, shows a uniform compositional behaviour. The degree of weathering/alteration in ilmenite is an indicator of its economic value and therefore compositional characterization would not only help us to adopt better methods for industrial processing but also aids in isolating grades of ilmenites for its effective utilisation.

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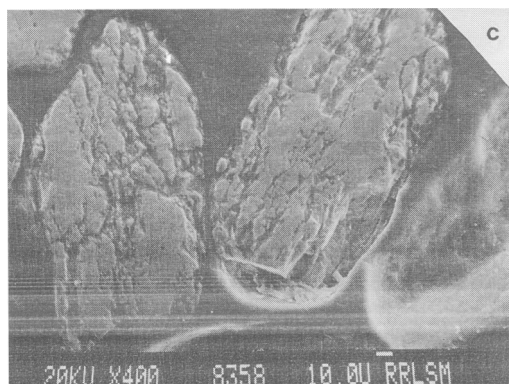
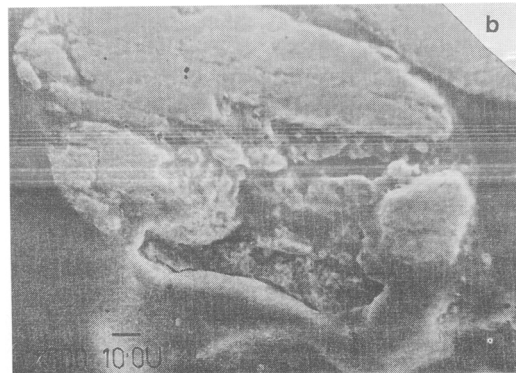
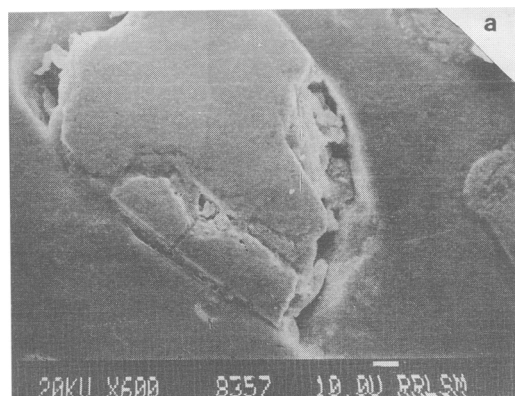


FIG. 9. SEM photographs showing (a) development of micro-fractures and initiation of alteration; (b) secondary mineral formation in the altered zone; (c) differential relief due to surficial changes in ilmenite grains.

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