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Note

Differential Alteration of Ilmenite in a Tropical Beach Placer, Southern India: Microscopic and Electron Probe Evidences

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Abstract: The study based on microscopic and microprobe techniques reveals that ilmenite of Manavalakurichi deposit has generally reached the alteration phase of 'leached ilmenite'. The XRD and bulk chemical analysis confirm the limited alteration undergone by ilmenite grains. Ilmenite alteration has been found to be a process of continuous leaching of iron from the mineral lattice and hydroxylisation. The enrichment of trace elements with progressive alteration is discussed. Si and Al are enriched by more than 100 fold. The prevalence of reducing environment at present in the deposit indicates that the oxidation of ferrous iron leading to pseudorutile formation would have occurred during transportation of sediments.

Keywords: Manavalakurichi, placer, alteration, ilmenite, pseudorutile, leucoxene

1. Introduction

The ilmenite deposit at Manavalakurichi (lat. 8°08'05" –8°10'05" and long. 77°15'50"–77°18'15") extends to about 7 km along Arabian Sea coast, with heavy mineral reserves of 8.30 million tonnes falling in the indicated category. The heavy mineral content of the deposit is as high as 48 % with ilmenite forming its bulk (Chandrasekharan and Murugan, 2001). The chemical and mineralogical studies of ilmenite in the deposit have been previously investigated (Viswanathan, 1957; Nair et al., 1995; Nair, 2001). Mössbauer techniques were applied and found suitable to estimate the pseudorutile formation in ilmenite grains (Subrahmanyam et al., 1982; Nair, 2001). Magnetic fractionation of ilmenite and the study of its qualitative variation are helpful in delineating the general progressive alteration of the mineral (Suresh Babu et al., 1994).

However all studies mentioned so far, have been confined to the bulk ilmenite samples. Studies on microprobe analysis of Manavalakurichi ilmenite and its alteration phases are rather limited in published literature (Chaudhuri and Newesely, 1990) and no attempt has been made, so far, on detailed microscopic studies on minerals of this deposit. While bulk chemical analyses of minerals are useful in deciphering the general chemistry and alteration trends of a mineral like ilmenite, microprobe testing of specific grains can reveal the chemical and mineralogical nature and changes of each alteration phase. An attempt is made in this paper to comment on the composition profiles and depositional environments of Manavalakurichi ilmenite, based on preliminary EPMA and mineralogical data.

2. Materials and Methods

Representative beach samples were collected after removing a few cm of the surface sand at each location (Fig.1). In the laboratory the samples were repeatedly washed and dried. After removing the ferromagnetic fractions using a hand magnet, the samples were fed into a Frantz Isodynamic Separator to obtain pure crop of ilmenite. Selected grains were mounted using polymer resin and observed under scanning electron and ore microscopes.

X-ray powder diffraction methods were used to identify the mineral phases in the samples using a Phillips Diffractometer (CuKα and Ni filter). The X-ray data were obtained with an accuracy of ±0.0005 Å. Thermogravimetric analysis was conducted using a Shimadzu TGA 5OH at a heating rate of 10°C/minute to a maximum temperature of 1000°C. Atomic Absorption spectrometry was used to determine the minor elements as described by Darby and Tsang (1987). The samples were analysed to

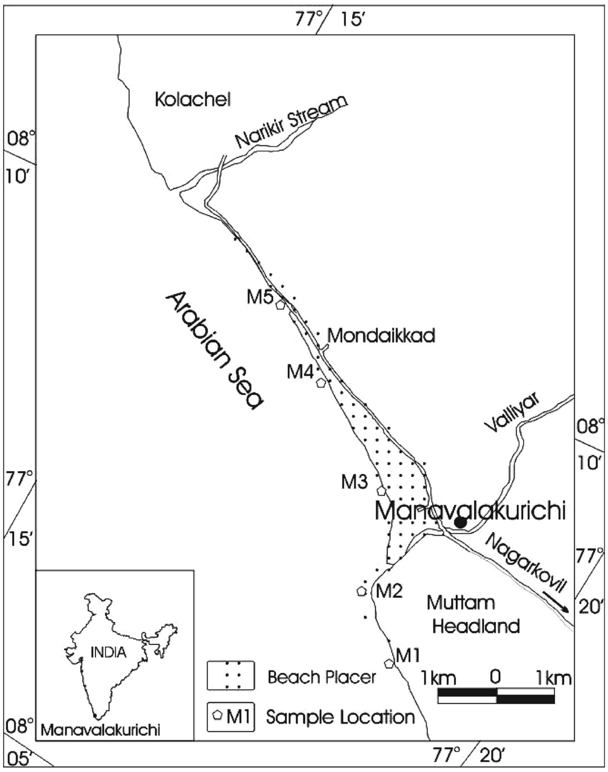

Fig. 1 Map showing location of representative samples collected from the study area.

determine the contents of total iron, ferrous iron and titanium dioxide following standard wet chemical methods (Vogel, 1961). The microprobe (model JEOL-8600) analysis was carried out under accelerating voltage of 15-20 kV, current 20-40 nA and beam diameter of 1-5 μm in the Department of Geosciences of University of São Paulo, Brazil. The error values in the EPMA measurements are in the range of 1-2 % for major elements and 5-10 % for minor and trace elements.

3. Results and Observations

3.1. Mineral phases under microscope

It is seen that in most of the grains a complete spectrum of alteration phases are observed. However, the dominant phase is that of leached ilmenite (Fig. 2a). Even in those grains the pseudorutile and leucoxene phases are seen, albeit restricted to the limited cracks and pores and the pockets of the perimeter of the grains. Intergrowths are generally found absent in the deposit under consideration and the absence of such high temperature mineral associations has been confirmed using XRD and compositional analyses in the samples investigated. Ilmenite is purplish grey in colour, anisotropic with good reflectivity under reflected light.

Leached ilmenite refers to the intermediate phase having compositions between ilmenite and pseudorutile. The optical properties are similar to those of ilmenite but are distinguished from the former by their grey bluish colour and weaker anisotropism. Reflection pleochroism has been observed in this phase. Limited occurrence of cracks is observed. This phase is generally seen along grain boundaries or along the regions of weakness on the grains intergrown with ilmenite (Fig. 2b).

Pseudorutile is distinguished by its greyish blue colour compared to ilmenite. The reflectance of this phase is in general at a higher range than shown by ilmenite. Frequently, this phase is observed to be isotropic with decrease in iron content. This is characterised by internal reflections of brown to reddish brown depending on the content of iron present. The optical properties show variation with the difference in its range of composition. Generally, pseudorutile develops from the grain boundaries and along fractures (Fig. 2c) and also parallel to crystallographic directions. Shrinkage cracks and pores are sometimes observed in this stage of alteration. This phase could be distinguished from the neighbouring ilmenite/leached ilmenite stages by the weaker anisotropism of the latter. Leached pseudorutile is a term reported elsewhere (Mücke and Chaudhuri, 1991; Chernet, 1999) which represents iron-poor members of pseudorutile phase. This phase is markedly metastable and breaks down to form leucoxene (Frost et al., 1983). In fact, it is difficult to distinguish between iron-poor members of pseudorutile and leucoxene. It has been observed here that alteration phases apparently belonging to leached pseudorutile was found to be chemically iron-poor pseudorutile.

Leucoxene is characterised by sugary internal reflections ranging from brownish through reddish brown, reddish orange, yellow and whitish yellow to white, with depletion of the iron content. Shrinkage cracks are a common characteristic of this phase. This phase has been identified as essentially microcrystalline rutile (Temple, 1966; Frost et al., 1983; Mücke and Chaudhuri, 1991). Leucoxene replaces pseudorutile and sometimes ilmenite/leached ilmenite either from the rim of grains, along cleavage lines or as patches within the grains (Fig. 2d). The leucoxenisation is also observed in the grains, in the form of pits or even from the core. In addition, the presence of leucoxene with ilmenite separated by sharp boundaries (Fig. 2e), or the occurrence of fingerprint like textures are noted. The cavities are often observed to contain flaky structures associated with altered products of ilmenite and suspected to be clay minerals (Fig. 2f).

3.2. XRD and TGA evidences

The XRD patterns exhibit sharp and well-defined peaks of ilmenite, while broad and diffused peaks of pseudorutile and rutile are much less obvious. The peaks of pseudorutile and rutile are rather indistinguishable

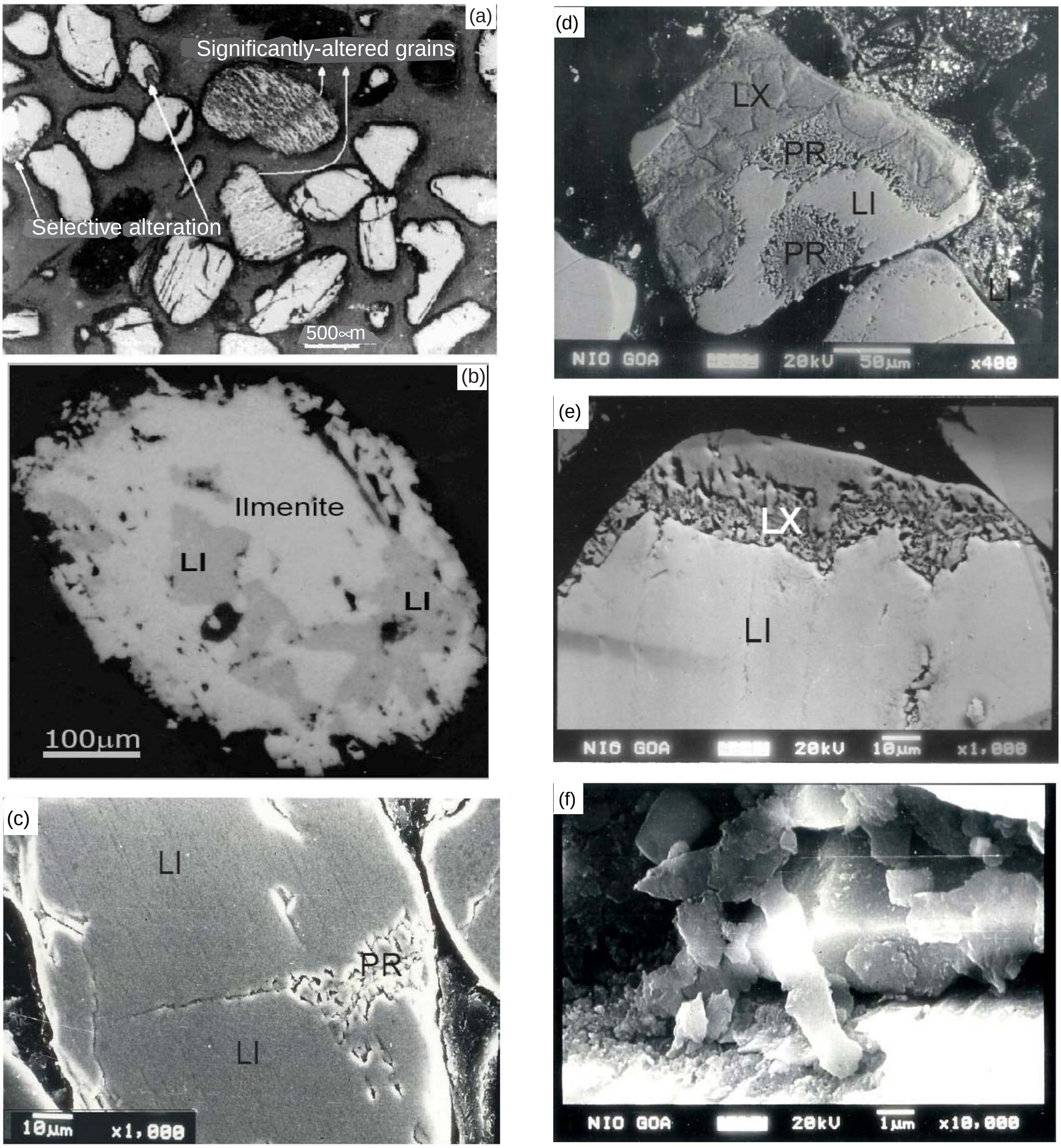

Fig. 2 a: Typical ilmenite crop of Manavalakurichi deposit with limited scale of alteration patterns, as observed under reflected light. Spatial alteration covering through out the grains and also alteration in selective regions of ilmenite are visible. b: Alteration of ilmenite (light grey) to leached ilmenite (dark grey) in sample M2 (under reflected light). Note that the phase boundaries within the grain are irregular. LI – Leached ilmenite. c: Alteration along micro fractures and pits with phases of leached ilmenite (LI) and pseudorutile (PR). d: Grain showing the gradual replacement of leached ilmenite (LI) by leucoxene (LX) through pseudorutile (PR). Note the shrinkage cracks. e: Sharp boundary between leached ilmenite (LI) and leucoxene (LX) phases exhibiting apparently 'discontinuous' alteration. f: Clay like bodies inside a fracture on the grain surface.

because of their similar d values and they often overlap. The most intense diffraction of ilmenite phase is formed by crystallographic plane (104) with d value of 2.74 Å. The other main peaks are (116), (204), (110), (108) and (113). The rutile (leucoxene) peaks are characterised by the planes (211), (101), (111) and (210). The intermediate phase of pseudorutile is characterised by the diffraction of the planes (102), (100) and (101). In the samples, the cell volume varies in a narrow range around 317 Å^3 except for sample M3 (313.58 Å^3) as given in Table 1.

The patterns of TGA curves depend on parameters intrinsically related to the state of alteration undergone like the content of structural water and the amount of ferrous ions. All the samples register an increase in weight at the maximum temperature of 950°C, ranging from 1.3 % (M3) to 2.15 % (M5). The weight loss exhibited due to the loss of structural water at 500–600°C is less marked varying from 0.21 % for M5 to a maximum of 0.86 % for M3 sample.

3.3. *Chemical analysis of bulk ilmenite*

The TiO_2 content is around 54 % for all ilmenite samples except for M3 (58.79 %) as documented in Table 2. The ferrous oxide content ranges from 14.95 to 23.68 %. Ferric oxide dominates over the ferrous state in samples from the southern part of the deposit (M1-M3). The content of total iron oxide shows a minimum for M3 (40.26 %) while increasing for M1 and M5 (~46 %). The Fe/Ti ratios are around 1 for samples except for M3 (0.83). Al and Si contents are highest among the minor elements, while V is the most prominent trace element (Table 2). The sample M5 displays significant difference from other samples, particularly in the amounts of Si (0.09 %), Al (0.24 %), V (1375 ppm) and Zn (99 ppm).

3.4. *Microprobe analysis*

A representative data of the microprobe spot analyses carried out on different alteration phases of seventy five grains is given in Table 3 converted to the respective molecular formulae based on the methods of Mücke and Chaudhuri (1991). The table helps to bring out a clear idea about the transformation in the mineral structure due to leaching and hydroxylisation.

Table 1 Lattice parameters of ilmenite samples from Manavalakurichi deposit.

Sample	*a* Å	*c* Å	*V* Å^3
M1	5.09	14.11	316.90
M2	5.10	14.10	317.50
M3	5.07	14.08	313.58
M4	5.10	14.10	317.50

The ilmenite phase is found to be a mixed crystal ilmenite (97-99.45 %) and haematite. Geikielitisation is prominent in many grains with $MgTiO_3$ forming as much as about 6 %. Pyrophanite occurrence is marginal (Table 3). The hydroxylisation sets in significantly in the iron-poor members of pseudorutile. In the leucoxene phases, hydroxyl content accounts for higher values in the mineral molecule. In the analysis 1-11 3(2) of leucoxene, the leaching of iron from the mineral structure is almost complete.

The concentrations of trace elements as a whole (excluding Mn and Mg) are found enriched as alteration proceeds (Table 3). However, it is seen that only Si and Al increase conspicuously. Nb and Ca generally show limited enhancement. The increase of Si and Al contents is more marked as leucoxenisation sets in. Si is enriched 100 to 400 fold and Al 100 fold. However, the content of trace elements is seen to decrease at the advanced stage of leucoxenisation.

4. Discussion

Figure 3 illustrates the behaviour of ferric content against the (Fe+Mg+Mn) values of ilmenite and its alteration products. In the preliminary stages of alteration of ilmenite, oxidation of ferrous irons and leaching of iron goes hand in hand. The oxidation of iron is more or less complete in the pseudorutile phase and thereafter alteration is characterised by steady leaching of the remaining ferric ions from the lattice structure. The curve shows the gradual and progressive leaching of iron lending credence to the argument that ilmenite alteration is a continuous process. It is seen from Table 3 that hydroxylisation is very prominent in the latter stages of alteration, notably in the leucoxene phase. Our results agree with reported values for hydroxyl contents in leucoxene (Mücke and Chaudhuri, 1991).

Our observations and findings generally correspond to the alteration mechanisms propounded by Mücke and Chaudhuri (1991). According to them alteration of ilmenite is a continuous process in which water is the main agent. The cationic deficiency caused by the leaching of ferrous iron from the ilmenite structure is balanced by the oxidation of the remaining two-thirds of iron to ferric state. This process defines the first stage of alteration.

Table 2 Elemental distribution in bulk ilmenite samples of Manavalakurichi.

Sample	Fe^{2+} (%)	FeO (%)	Fe^{3+} (%)	Fe_2O_3 (%)	Total Oxide(%)	TiO_2 (%)	Fe/Ti	Mn (%)	Mg (%)	Al (%)	Si (%)	Cr (ppm)	V (ppm)	Zn (ppm)	Cu (ppm)	Ni (ppm)
M1	12.57	16.17	20.96	29.97	46.14	54.00	1.037	0.24	0.32	0.31	0.45	540	1021	500	62	43
M2	13.90	17.88	18.30	26.17	44.05	54.21	0.992	0.24	0.31	0.54	0.32	810	1042	447	78	20
M3	11.62	14.95	17.70	25.31	40.26	58.79	0.833	0.20	0.12	0.41	0.22	511	1164	396	52	144
M4	18.39	23.66	13.80	19.73	43.39	54.03	0.995	0.23	0.28	0.36	0.41	650	968	361	71	69
M5	18.41	23.68	15.79	22.58	46.26	54.72	1.044	0.22	0.23	0.24	0.09	860	1375	99	69	40

Table 3 Representative microprobe data of selected spot analysis calculated to respective molecular formula on basis of O=3 for ilmenite and Ti=3 for leached ilmenite, pseudorutile and leucoxene. Trace elements given in terms of their weight %.

Grain No		Molecular formula							Wt%					
Ilmenite	Composition of ilmenite mixed crystal	Fe^{2+}	Fe^{3+}	Mn	Mg	Tot	O	(OH)	SiO_2	Al_2O_3	Cr_2O_3	CaO	Nb_2O_5	NiO
I-11 1(1)*	Ilm –97.34% Fe_2O_3 –2.66%	0.935		0.002	0.063	0.973	3		bd**	bd	0.115	bd	0.048	bd
I-11 1(2)	Ilm –97.43% Fe_2O_3 –2.57%	0.935		0.006	0.063	0.974	3		0.012	bd	0.100	0.030	0.027	0.035
I-11 2(1)	Ilm –97.10% Fe_2O_3 –2.90%	0.974		0.005	0.021	0.971	3		0.008	bd	0.012	0.029	0.184	bd
I-11 3(4)	Ilm –97.34% Fe_2O_3 –2.66%	0.973		0.021	0.006	0.973	3		bd	0.015	bd	bd	0.128	bd
I-11 3(6)	Ilm –98.09% Fe_2O_3 –1.91%	0.99		0.001	0.009	0.981	3		bd	0.044	0.022	0.002	0.259	bd
Leached Ilmenite														
I-11 3(1)		1.775	0.795	0.012	0.02	2.602	9		0.112	0.065	0.019	0.020	0.054	0.031
I-11 4(9)		2.479	0.249	0.066	0.081	2.875	9		0.009	0.202	0.018	0.025	0.108	0.029
Pseudorutile														
I-11 3(5)		0.166	1.854	0.01	0.04	1.92	9		0.146	0.333	0.056	0.094	0.230	bd
I-11 3(8)			1.402	0.002	0.142	1.546	7.494	1.506	0.236	0.564	0.202	0.101	0.317	bd
Leucoxene														
I-11 2(2)			0.048	0.032	0.001	0.081	3.209	5.791	1.814	1.498	0.229	5.487	0.253	0.005
I-11 3(2)			0.01	0.0006	0.001	0.012	3.033	5.967	0.056	0.026	bd	0.015	0.205	0.016
I-11 3(3)			0.073	0.001	0.289	0.363	3.797	5.203	6.848	2.271	0.090	0.142	0.189	0.011
I-11 3(7)			0.099		0.035	0.134	3.369	5.631	4.344	3.986	0.112	0.128	0.439	0.034

* I-11 represents the Ilmenite from Manavalakurichi deposit and 1(1), the sample number and number of the spot analyzed.
** below detection limit.

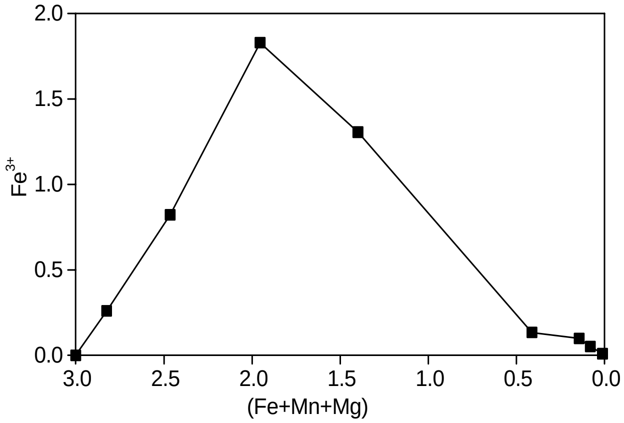

Fig. 3 Diagram showing the progressive oxidation and leaching of iron with alteration based on analytical data. The elemental data is given in terms of number of atoms on the basis of Ti=3. Oxidation to ferric state is initially simultaneous with leaching of iron. Leaching continues beyond the termination of significant oxidation. Each data point represents a cluster of similar values.

As alteration continues, ferric iron is further progressively leached out. This is balanced by the replacement of oxygen $(O)^{2-}$ by hydroxyl $(OH)^{-}$ ions. As documented in Table 3 and Figure 3, formation of leucoxene from pseudorutile stage is a gradual process of hydroxylisation and leaching of iron.

The Fe/Ti value is often used as an index of alteration indicating, the relative enrichment of iron with respect to the amount of titanium present (Grey and Reid, 1975; Frost et al., 1983). The chemical data of the bulk ilmenite samples reveal limited alteration generally suffered by the deposit (Table 2). The Fe/Ti ratios range about 1 except for M3 and alteration has succeeded only in the slight Ti enrichment from the theoretical TiO_2 value of 53 %, due to leaching of iron.

The sample M5 from the northern end of the deposit (Fig. 1) exhibits the least altered state evident by its high FeO and total iron oxide contents (Table 2). The trace element distribution of this sample also deviates from the general pattern in the deposit. Previous studies have shown that this region is a zone of prominent beach accretion (Kurian et al., 1997). The dominant coastal currents are southerly in Manavalakurichi region. The sediments contributed by rivers further north of the deposit are carried by these currents. The abrupt change in the orientation of the coast (Fig. 1) checks the current velocity yielding favourable wave diffraction patterns leading to deposition of this sediment load at the northern part of the deposit. Another source could be attributed to the disintegration of the prominent rock outcrops adjacent to the northern extremity of the Manavalakurichi deposit. The anomalous contents of elements like Si, Al, V and Zn in the sample M5 (Table 2) could also probably point to the contribution from this northerly source.

The contents of analysed trace elements, in general, increase as leaching of iron proceeds (Table 3). They augment slightly in leached ilmenite and drastically in advanced stages of alteration like pseudorutile and leucoxene. Elements like Al, Si, and V register enhanced values, partly as a result of relative enrichment due to the leaching out of iron. Frost et al. (1983) have traced the high contents of Al and Si to the dissolution-reprecipitation mechanism of leucoxene formation in the mineral structure in soil layers, leading to the co-precipitation and adsorption of such elements from pedogenic clay. In this work, clay like structures is observed in the shrinkage cracks and pores in ilmenite grains (Fig. 2f). Such accommodation of clay particles must have, partly contributed to the enrichment of the above-mentioned elements in the chemistry of bulk ilmenite. However, the minor and trace element contents significantly decrease in the highly iron-deficient leucoxene phases (as in I-11 3(2) in Table 3) due to the complete breakdown of the pseudorutile structure.

The limited alteration undergone, in general, by the Manavalakurichi ilmenite, is reflected in the XRD and TGA patterns by marginal presence of pseudorutile and rutile (leucoxene) peaks. The lattice volumes calculated from the XRD data (Table 1) indicate that the most altered sample M3 present the least value of 313.58 $Å^3$, while the *V* values of other samples are similar to each other. The same trend is shown by the *a* axis with lengths of about 5.10 Å for all samples except M3 (5.07 Å). The leaching of iron from the ilmenite structure result in the reduction of the cell volume as alteration advances (Suresh Babu et al., 1994). The TGA data reveal the relatively higher structural water content and hence the higher alteration state of sample M3, as signified by the weight loss at 500–600°C. This is evidenced by shrinkage cracks and pores, which increase progressively from leached ilmenite phase to leucoxene.

The dominance of leached ilmenite as observed under microscope points to the limited scale of alteration prevalent in Manavalakurichi ilmenite. However, most of the grains have more than one alteration phase, with diffused boundaries indicating that alteration is continuous. The shrinkage cracks increase with alteration with the reduction of lattice volume consequent to the oxidation and leaching of ferric iron from the mineral structure (Frost et al., 1983). The presence of members of leucoxene phase with ilmenite/leached ilmenite separated by sharp boundaries is noted in some grains (Fig. 2e). This has been explained as due to the direct formation of leucoxene from ilmenite/leached ilmenite in near surface zones by the action of soil acids (Frost et al., 1983; Hugo and Cornell, 1991). However, Mücke and Chaudhuri (1991) argued that such structures are formed by the disintegration of the unstable iron-poor pseudorutile forming leucoxene. Further, it is observed that other parts of such grains usually show the presence of intermediate alteration phases. The occurrence of fingerprint like textures is also attributed to the primary stage of the same process.

In the Manavalakurichi deposit, marked presence of peat bands are reported pointing to the reducing environment prevalent (Murugan et al., 2000). It has been opined that ilmenite transforms to pseudorutile in oxidizing and acidic ground-water environment (Grey and Reid, 1975). The subsequent leucoxene/secondary rutile formation is thought to be occurring in near surface zones in an acidic and reducing environment (Grey and Reid, 1975; Frost et al., 1975). On the other hand, Mücke and Chaudhuri (1991) have argued that ilmenite alteration even beyond the pseudorutile phase occurs in oxidizing and acidic environment. In Manavalakurichi deposit, reducing conditions seem to prevail throughout the depth of the deposit since peat is found from just below the water table (at ~1 m depth). Though direct alteration of ilmenite to leucoxene is observed (Hugo and Cornell, 1991), the grains in the study area exhibit all the different alteration stages under the microscope. It is plausible to envisage that conditions must have changed to reducing environment (and subsequent peat formation), after the grains have been differently altered as observed. The peat beds are likely to be the result of the major transgression event that took place all along the southwest coast of India in early Quaternary (Rajendran et al., 1989). The leaching and hydroxylisation subsequent to the formation of pseudorutile could possibly take place as reported in reducing conditions. However, the initial process of oxidation of ferrous ions to ferric state could take place only during transportation processes leading to concentration and deposition in the present site at Manavalakurichi, or prior to the establishment of reducing environment.

5. Conclusions

The Manavalakurichi deposit has undergone limited alteration where the dominant phase is leached ilmenite. However most of the grains show more than one alteration phases. The iron-poor members of pseudorutile phase are unstable and disintegrate and recrystallise to form leucoxene which develops sharp boundaries with ilmenite giving the impression of direct leucoxene formation from ilmenite. Leucoxene phase is characterised by intense hydroxylisation. The trace elements like Si, Al, and V increase with alteration due to relative enrichment and, at least partly, to adsorption of clays to the leucoxene structure.

The deposit at present occurs in a reducing and acidic environment as attested by peat formation in the area. The multiphase nature dominant in the grains indicates that conditions might have changed to a reducing climate after the grains have been differently altered. The initial oxidation reaction of the alteration process would be occurring significantly during the transportation of grains or after the deposition prior to the inception of a reducing environment.

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