ASSESSMENT OF PLACER ILMENITE: A CASE STUDY FROM CHAVARA DEPOSIT, KERALA STATE

THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS
FOR THE DEGREE OF MASTER OF PHILOSOPHY
IN GEOLOGY

By

C. RAMAKRISHNAN

Under the Guidance of

Dr. R. MANI, B.Sc. (Hons), M.Sc., F.M.S., F.G.S.I.
(Professor and Head)

DEPARTMENT OF GEOLOGY

ANNAMALAI UNIVERSITY

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CERTIFICATE

This is to certify that the thesis entitled **"ASSESSMENT OF PLACER ILMENITE: A CASE STUDY FROM CHAVARA DEPOSIT, KERALA STATE"** is a bonafide record of research work done by **Mr.C.RAMAKRISHNAN** during 1993-94, in the Department of Geology, Annamalai University and that it has not previously formed the basis of any degree, diploma, associateship, fellowship or other similar title.

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This is to certify that **Shri C. Ramakrishnan** has worked on **Assessment of Placer Ilmenite: A Case Study from Chavara Deposit, Kerala State**, and carried out laboratory work under my guidance for the partial fulfilment of the requirements for the Degree of **Master of Philosophy in Geology**. The present work or any part thereof has not been submitted to any other University or Institution for the award of any degree, diploma, associateship, fellowship or other similar title.

10/12/94
(P.N. Mohan Das)

DEDICATED TO MY
BELOVED
FATHER AND
MOTHER

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ABSTRACT

Iron and titanium contents of over thirty seven samples of ilmenites from eight cross-sections of Chavara deposit stretching about 22 km in the coastal zone have been determined by standard wet chemical methods. Based on ferrous - ferric and Ti/(Ti+Fe) ratios, the intensity of alteration suffered by ilmenite could be studied. It is understood that a geochemical boundary exists in the southern portion of the deposit. The generated data suggest that Chavara ilmenite corresponds to an alteration stage bordering between hydrated ilmenite and pseudorutile of Frost et. al., 1983. It further reveals that the ilmenite alteration progressively increases from the western margin of the deposit towards east. From the systematic compositional analysis, it is also obvious that the Chavara deposit, in general, shows a uniform compositional behaviour. This point out that the deposit has originated from a pile of ancient sediments and suffered a specific degree of weathering in total. The variation noticed in the MgO content of ilmenite samples can either be due to the differential geikelitic substitution in the primary crystals or due to the differential leaching which took place later in the ilmenite grains. The powder X-ray diffraction studies have shown the co-existence of ilmenite, pseudorutile and rutile phases in most of the samples studied. TGA experiements on some samples have explained the relationship between the weight change and iron content on heating the samples. SEM observations have revealed the nature of alteration in ilmenites.

INTRODUCTION

CHAPTER - I

1.1 Study Area

The study area falls in the southwestern fringe of south Indian Precambrian shield, forming a part of Kerala state, which is flanked by the Arabian Sea in the west and Western Ghats in the east. The study area ie. Chavara is located approximately 10 km north of Kollam. The Chavara beach placer deposits are seen spread over a distance of 22 km long with an average width of about 200 metres. It is a barrier bar seperated from the mainland by an inland canal connecting the Ashtamudi estuary in the south and Kayamkulam lagoon in the north. (Fig.1)

1.2 Location

The study area falls between east longitudes 76° 27' 36" and 76° 33' 44" and north latitudes 9° 8' 24" and 8° 56' 3". It is included in the Survey of India toposheet numbers 58 D/9, 58 C/8 and 58 C/12 in 1:50,000 scale.

1.3 Scope of the present work

The prime objectives of this research work are :

(i) To evaluate the compositional, mineralogical and surficial changes associated with ilmenite concentrate and

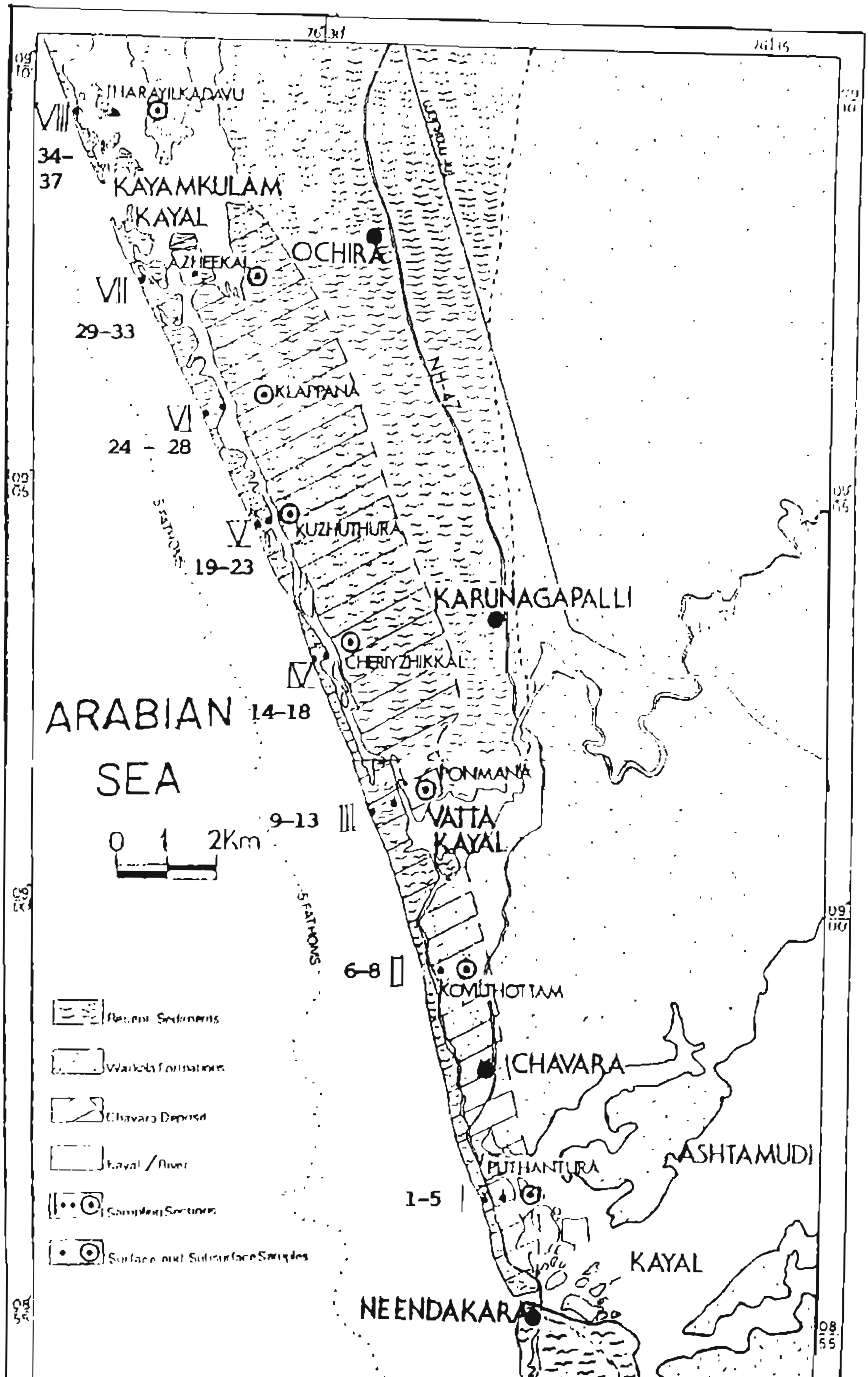


FIG 1 MAP SHOWING SAMPLING LOCATIONS

(After Prabhakar Rao, G. 1968).

(ii) To understand the degree of weathering sufferred by ilmenite and discuss the genetic aspects of placer deposit. Ilmenite is the chief source of titanium and its compounds since rutile is scarce in nature. India earns good hard currency income from the export of this valuable mineral and also its internal demand is being increased day by day. In this background, any further understanding on the quality of this placer mineral would only be complementary to our knowledge. Though several publications are available in relation to the provenance, mineralogy, sediment parameters and reserves of Chavara placer deposit, no systematic analysis on lateral and vertical changes in composition has been carried out so far. Similarly, it is reported that the processing behaviour of raw ilmenite concentrates differ depending on the zones of exploitation. This also points to the urgent need for full scale characterisation of individual minerals in the deposit.

In this broad spectrum of placer geology, my M.Phil. dissertation is mainly confined to some of the mineralogical and geochemical aspects of ilmenite, taking Chavara placer deposit as the model area. Many scientists have carried out extensive work on bulk sediments. But no serious attempt has been made on the study of monomineralic fractions especially on ilmenite. Taking into account the growing demand of ilmenite for the extraction of TiO_2 in the titanium based industry, an extensive study on the ilmenite fraction has been proved inevitable. Such a study not only helps us to adopt better methodologies for industrial processing but also aids in isolating grades of ilmenite for it's effective utilization.

CHAPTER - II

2.1 Geomorphology

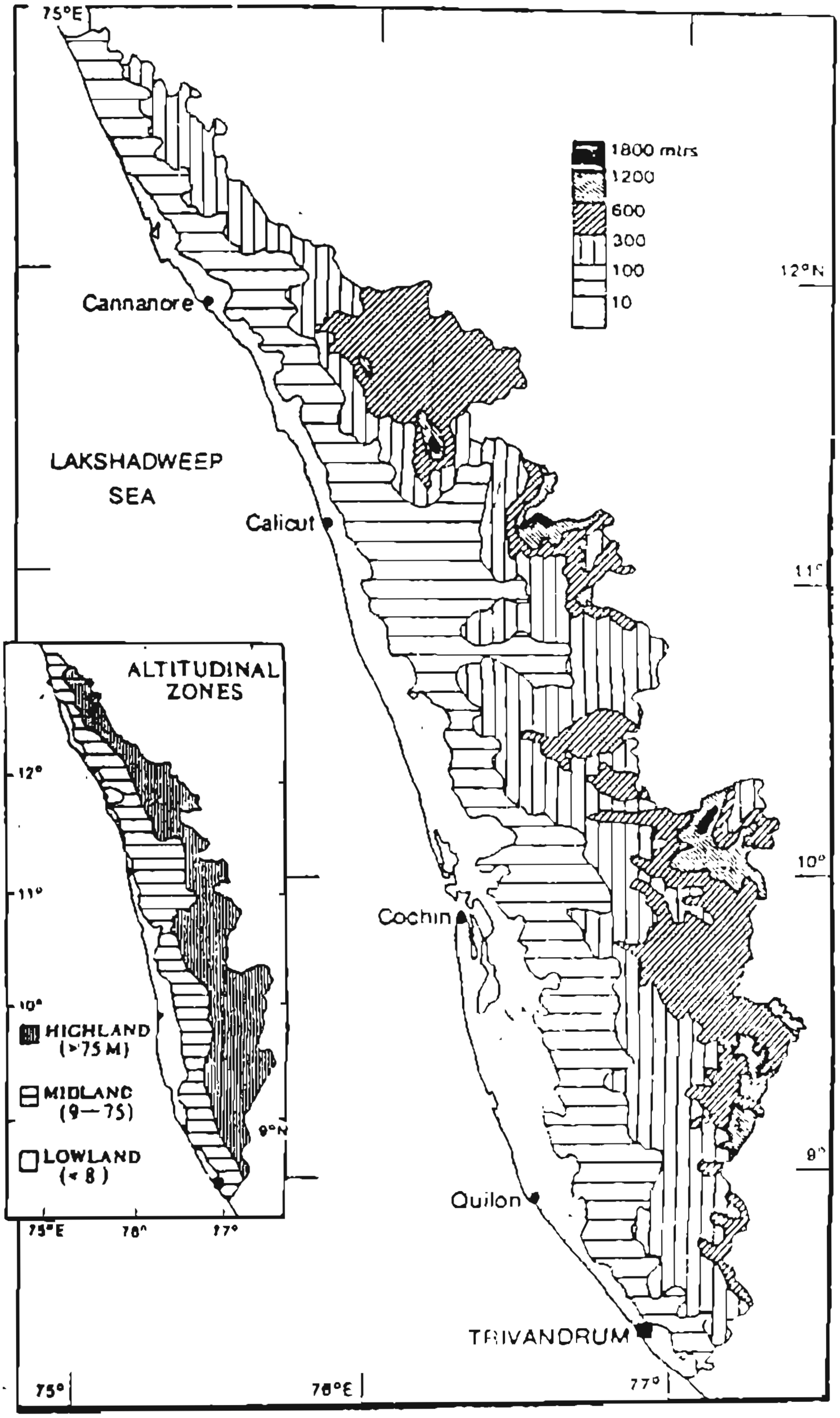
Kerala state has an area of about 38,863 Sq.km. of which nearly 6000 km. is covered by sand and alluvium. The region may be divided into four longitudinal physiographic zones namely, the highlands, the midlands, the lowlands and the coastal plains. The highlands range in altitude from nearly 600 m to 2500 m above mean sea level and form the catchment area of the major rivers of the region. The midlands at an elevation of 300 to 600 m above mean sea level, constitute the foothill and the piedmont areas that are dissected by the drainage network. The lowlands range from an altitude of 30 m to 300 m and constitute isolated areas of moderately high relief surrounded by laterite piedmont. The raised beaches and the sand dunes are superimposed on the geomorphic form of the lowlands. The coastal strip is characterized by the estuaries and lagoons. Each of these physiographic zones has characteristic fluvial and landscape morphology (Sinha Roy & Mathai, 1979). The Kerala P.W.D (1974) has identified 3 physiographic zones in the state, namely

i) The highlands above 76 m. (250 feet)

ii) The midland 7.6 to 76 m. (25 to 250 feet) and

iii) The lowland below 7.6 m (25 feet).

The physiographic map of Kerala is shown in Fig. 2.



E.SARVANAN, CESS

Fig. 2 Physiographic map of Kerala.

The most prominent geomorphologic feature of the state, and one of the important orographic features of Peninsular India is the Western Ghats, rising to more than 1500 m above mean sea level. The highest peak in the Western Ghats, the Anamudi hill, with a height of 2817 m is one among fourteen peaks of over 2000 m situated in Kerala. The Palghat Gap in central Kerala is the most conspicious geomorphic break in the otherwise continous Western Ghats. The Munnar plateau (1200 - 1500 msl.), Nilgiri massif (1850-2500 msl) and the Wynad plateau (750-1000 msl) are the other major geomorphological features of the region.

Geological formations of all ages are not represented in the Kerala region and so, might have undergone prolonged periods of weathering and erosion. A number of workers have revealed the polycyclic development of planation surfaces which roughly fit in the scheme of major landforms in the Peninsular India identified by King (1950) viz. the early Gondwana (2580 m), Gondwana (1950 m), Indian (180 - 600 m) and Godavari (180-540 m). Vaidyanathan (1967) identified erosional surfaces at 2400m, 1800m, 900m, 600m and 360m in areas south of 18°N latitude. One more surface which is the youngest is identified in the coastal tract by Subramanyan et.al (1980) to the south of 15°05'N latitude. According to Demongect (1975), there are only two surfaces in the Palghat area of Kerala, the upper one sloping from 350m at the Palghat Gap upto 75m towards the coast with a lateritic cover and the lower one representing the coastal plain. Sinha Roy (1979) has identified four geomorphic surfaces in the Karamana drainage basin of southern Kerala, the elevations of which are 1200-1300 m, 150-200 m, 60-100 m and 20-50 m. These surfaces have variable angle of slope towards west and their hinge zone is located roughly in the coastal strip which points to the periodic uplift and erosion of the onshore areas. Each of these surfaces forms part of the major physiographic zones and the medium and low level surfaces are characterised by the profuse development of laterites.

Geomorphological studies of central Kerala by Sambandham (1980) have revealed the polycyclic nature and led to the identification of four erosion surfaces. They are at an elevation of (i) 550 m, (ii) 395 m, (iii) 160-230 m, and (iv) 60-150 m. The first two surfaces had a former laterite cover which had since been stripped off and thus poorly preserved. In addition, the current cycle has advanced to the stage of early maturity and dissected the (60-150 m) erosional surface for about 45 m. forming pediments with maximum width of 1.5 km. The coastal plain 3-5 km. wide has also been developed in this cycle. Polycyclic landform dissection has also been observed in the area between Kasargode and Manjeswar.

Attempts have been made to compute the rate of erosion tentatively. According to Prabhakar Rao (1968) the sedimentation rate in the Warkalli formation was $3.96 m/10^3$ years which coincides with the sedimentation rates of $2\text{-}4 m/10^3$ years in humid regions, in bays and estuaries. Closs et.al, (1979) reported that in deeper parts of the Arabian Sea, sedimentation rate is $1 cm/10^3$ year, and near the coast it averages greater than $35 cm/10^3$ years with a maximum of $1.5 m/10^3$ years. From these (Babu 1979) assumed an erosion rate of 0.1 to 1 mm/year which may lead to a reduction of surface by 100-1000 m/ 1 million years.

An attempt was made by Sambandam to date the erosion surfaces of central Kerala. The IV erosion surface, in northern parts of Kerala are developed over Warkalli sediments of Mio-Pliocene age. By hypothetical calculations the date of erosion surface IV has been estimated as Upper Pliocene (Sambandham, 1975). Tentative ages, deduced by comparative dating methods for erosion surfaces I, II and III are Middle Miocene, Upper Miocene and Lower Pliocene respectively (Sambandham, 1980).

The highland region of Western Ghats comprises the principal sediment source zone basins while the midlands and parts of lowland areas acts as the transfer zones of the 44 drainage basins in the region. Prominent knick points are characteristic of these rivers. There is a knick point at an elevation of 500-800 m which corresponds to the boundary between the highland and midlands and another one at an elevation of 90-150 m corresponding to the boundary between the midlands and the lowlands; of which the latter one is more prominent. These factors are indicative of block movements in the area. Rivers of Kerala show almost straight courses especially in the highlands and in parts of the midlands. This feature together with the nature of river profiles indicate tectonic control of the river courses to a great extent. The drainage is of the dendritic type but a trellis pattern is superimposed by the dendritic type marked in the hill ranges.

Estuaries and lagoons are characteristic features of the Kerala coast. There are 27 estuaries and 7 lagoons within the region. The largest among the estuaries is the Cochin-Vembanad estuary. The entire stretch of Kerala coast, extending for 560 km. is also noted for the most well developed spits. Occurrence of mud banks is another peculiar feature of the Kerala coast. (Soman, K. 1980).

2.2 Climate and Rainfall

The climate is moist and hot in the coast and it is slightly cooler and drier in the interior of the district. The average monthly temperature of this district is approximately 25°C.

As in the case of other parts of the state, this district also get the benefits of the two prominent monsoons ie. SW and NE. The average rainfall exceeds 300 cms. with the highest precipitation taking place during southwest monsoon which is from June to September (Ananthakrishnan et.al. 1979). The annual rainfall of Kayamkulam for the period 1983 to 1993 are given in Table 1.

TABLE - 1 : ANNUAL RAINFALL DATA OF KAYAMKULAM
FOR THE PERIOD 1983 - 1993.

Year	Rainfall (mm.)
1983	2435.2
1984	2312.2
1985	3179.0
1986	2074.3
1987	3749.0
1988	3109.3
1989	2602.5
1990	2176.6
1991	2594.8
1992	2695.3
1993	2543.7

Source : IMD, Trivandrum.

2.3 Wind, Surface Current and Tide

There is a seasonal variation of wind circulation pattern. The wind is weakest in November and blows in a northerly direction. During June to July the wind direction changes from north to west and the prevalent wind direction is west to east with progressive increase in speed (velocity). In September and October the wind is directed from SSE. In July, the surface current run in a SSE direction following the coast with a velocity of 25-40 cm/sec. A strong southerly directed surface current with speed upto 2 knots is established from January to August. Current weakens significantly after peak monsoon and move northward in October. From November to January coastal currents flow northward. June-July are the roughest time for Kerala coast. Strong swells with periodicity 5-18 seconds and heights 2.4 to 6 m are common. The wave system approaches the shore from WSW and WNW. Tides are semidiurnal without much significance. The annual sediment discharge through streams is high along the coast. (Mallik et.al, 1987).

2.4 Vegetation

The lowlands or the coastal area constituting the backwaters, kayals/lagoons and the shore of the Arabian Sea is essentially a land of coconut trees (Cocos nucifera). The other major vegetation in the inland area is the paddy (Oryza sativa) cultivation.

2.5 Human Geography

The area is thickly populated. The major occupation is fishing and apart from that agriculture and coir based industry are the other major pre-occupation of the people.

2.6 Accessibility

The area is fairly accessibly by NH-47 and by a network of mining and metalled roads. The barrier bars are easily approachable with the help of country boats.

GEOLOGICAL SETTING

CHAPTER - III

3.1 Geology

The Kerala region, which forms the south western fringe of the south Indian Peninsular shield, is a geologically significant narrow strip of land bounded by the Western Ghats in the east and the Arabian Sea in the west. It consists of two major terrains (Ramakrishnan, 1988) :

1) The cratonic part lying north of Palghat-Cauvery shear zone and

2) The mobile belt-the Pandyan mobile belt formed of gneiss, charnockite and khondalite. The rock types falling within the Kerala region can be classified into four major age groups as belonging to Archaean, Proterozoic, Cretaceous and Cenozoic (Table 2). In Fig. 3 geological formations belonging to these age groups are shown. (Ravindra Kumar et. al., 1990). The geological map of southern Kerala is shown in Fig. 4.

In Kerala, crystalline rocks of Archaean age, sediments of Tertiary age, laterite cappings on crystallines and sediments of Subrecent to Recent age are exposed. The crystalline include charnockite and khondalite group of rocks, granite gneiss and granites traversed by basic rocks. Charnockite rocks constitute the hill ranges in the Western Ghats and are wide spread in occurrences in Cannanore, Kozhikode, Palghat, Trichur, Kottayam, Ernakulam and Quilon districts.

Table 2 Geological formations of the Kerala region.

Geological Formations	*Age*
Beach sands and soils; aeolian deposits including white and red (teri) coastal sands; shell limestone and loose shelly sands	Recent to sub Recent
Laterite	
Warkalli beds; sandstones and clays; lignite	Upper Miocene
Quilon beds; fossiliferous limestones, sands and clays	Lower Miocene
Basic dykes	
Granite, pegmatite and quartz veins	Late Proterozoic
Basic dykes	
Vengad Group; quartz mica schist, conglomerates	Proterozoic
Khondalite Group; meta sediments of garnet-biotite-sillimanite-cordierite-graphite gneiss, leptynite, calcsilicates, quartzites.	Late Archaean Early Proterozoic
Charnockites, migmatitic ortho-gneisses	
Wynad Group: (= Sargur Group) kyanite-sillimanite schist, calcareous bands, quartzite, banded iron formations, hornblende-biotite gneisses.	Archaean

Ravindra Kumar, G.R., et. al., (1990).

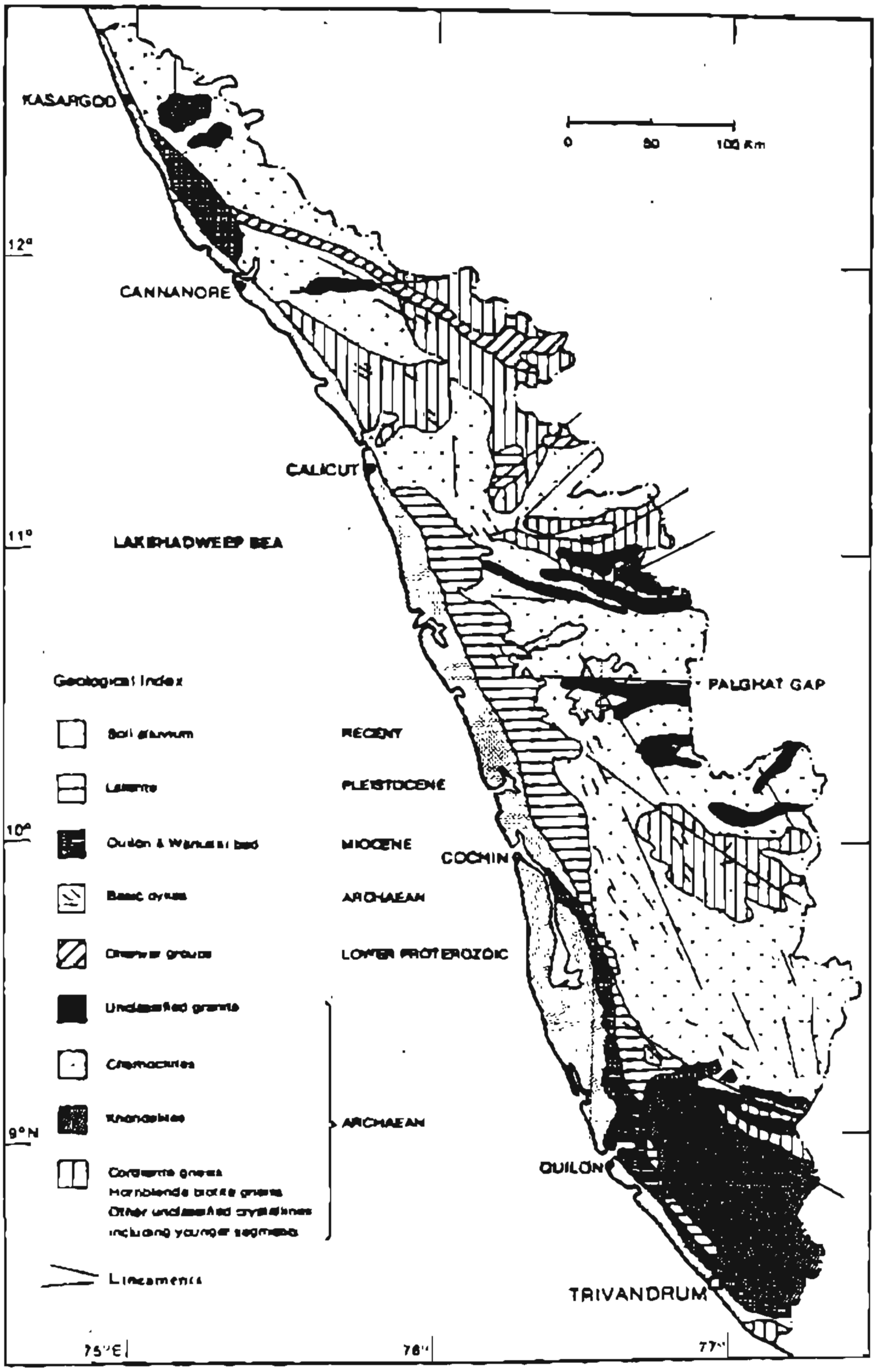
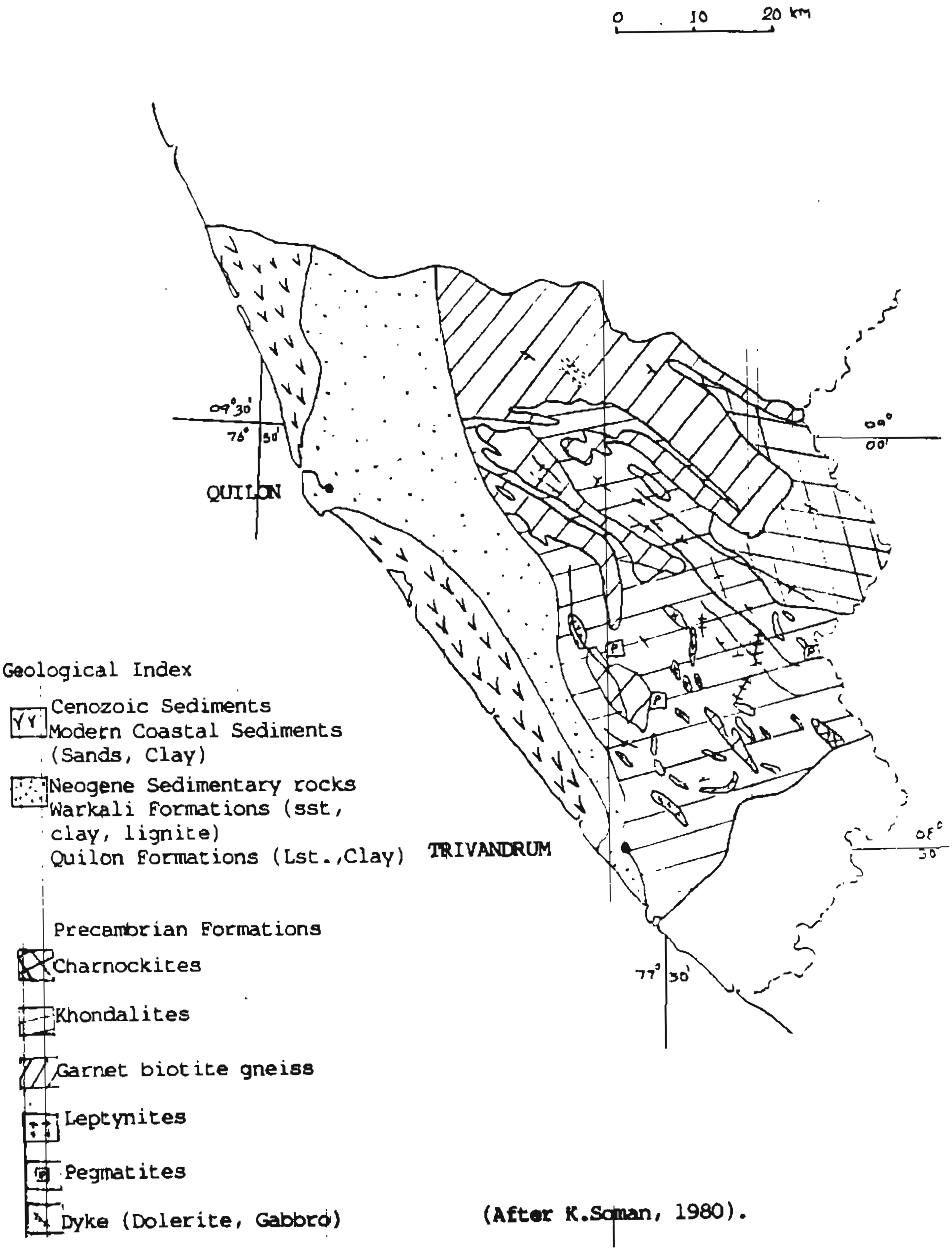


Fig. 3 Geological map of Kerala (after GSI, 1976).

Fig.4 GEOLOGICAL MAP OF SOUTH KERALA



(After K.Soman, 1980).

Table 3 Stratigraphic classification of the Cenozoic sediments of Kerala.

King (1882)	*Nair and Rao (1980)*		*Raha et al.*, (1983)	
	Unit IV Beach sand and alluvial clays		*Vembanad Formation* Beach sand, sandy clay with peat beds. Alluvium, soil and gravel bed	*MALABAR SUPERGROUP*
			––Unconformity––	
Warkalli Beds (Sandstones, arokose, clays)	*Unit III A* Laterite, ferruginous sandstone and clay	*WARKALLI BEDS*	*Ambalapuzha Formation* Fluvial sandstone clays and lignite	
	Unit III Arkosic sand, clays and lignite			
Quilon Beds Limestone	*Unit II* Limestone with sand and clays		*Quilon Formation* Limestone clac. sands and clays (marls) (0.5–130 m)	
	Unit I Pebbly sands and sandy clay with black clays lignite indistinct when Unit II is not well marked		*Mayyanad Formation* Fluvial sandstones clay, lignite	

Ravindra Kumar, G.R., et. al., (1990).

Hornblende and biotite gneiss are seen at places and derived by retrograde metamorphism and migmatisation of biotite gneiss. Khondalitic group are exposed in south Kerala (Fig. 4) and Palghat districts. Pink and grey grains with quartz and pegmatite veins are intrusive in charnockites and schists. Dolerite, basalt and gabbro cut across the crystalline rocks. Variegated sandstones and clays with lignite grains (Warkalli formations) of Miocene age are underlain by compact sands and clays with shell fragments and thin beds of limestone (Quilon formations). Laterite is the product of residual chemical weathering of both crystalline rocks and Tertiary sediments and form flat topped hills and ridges between foot hills of Western Ghats and Arabian Sea. (Mallik et.al., 1987). The geological and stratigraphic sequence is given in Tables 2 & 3.

3.2 Tectonic History

The tectonic history of the crystalline rocks of Kerala is complex. The regional structural and tectonic map of Kerala (after Rao, 1978) is given in Fig. 5. Based on the analysis of various fold patterns and metamorphic facies, Rao (1976) recognised five major phases of tectonogenesis.

The first phase of deformation is associated with the 'blanket' granulite facies metamorphism and produced E-W. trending isoclinal overturned folds affecting the charnockites, khondalites and

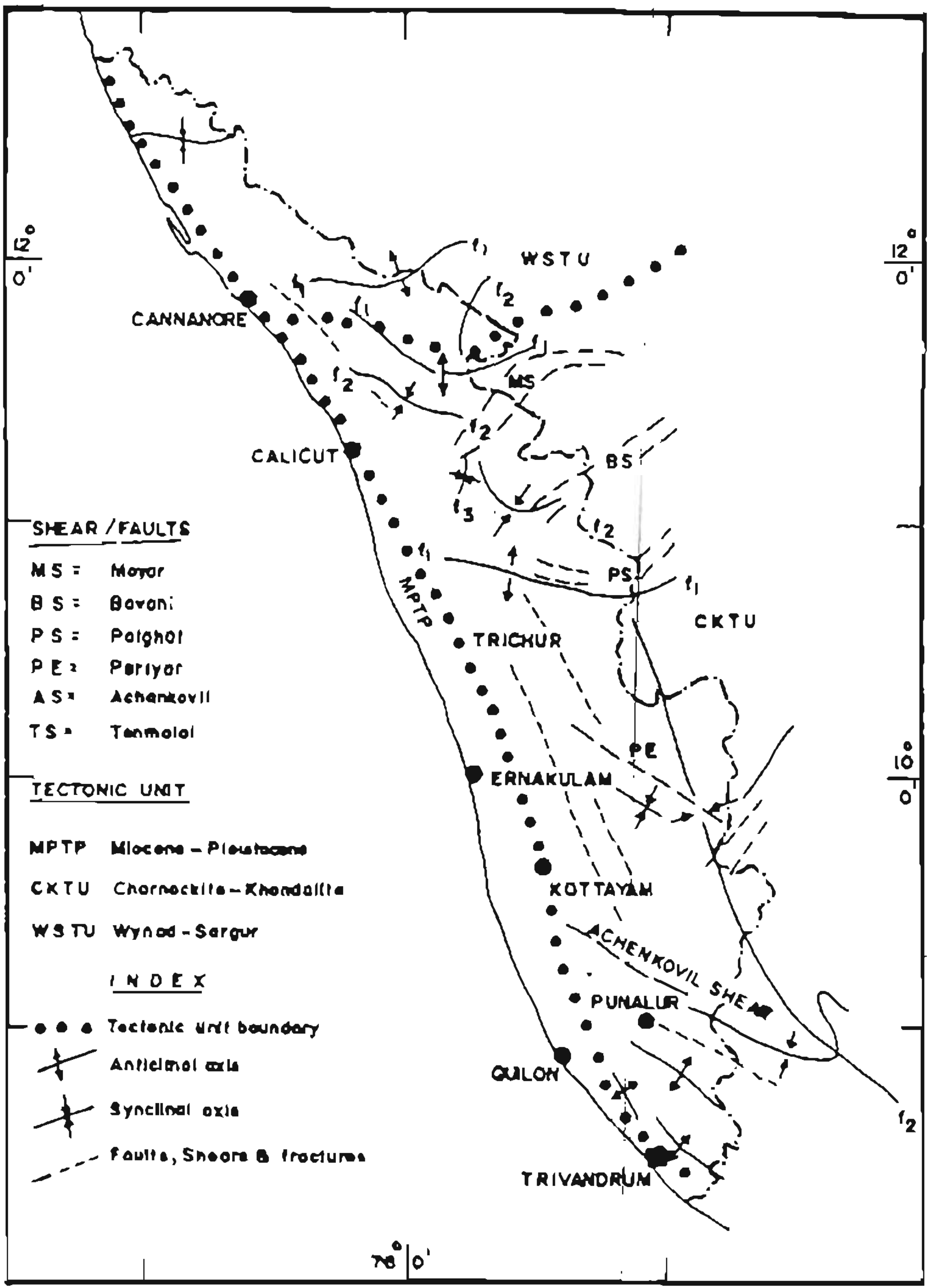


Fig. 5 . Regional structural and tectonic map of Kerala (after Rao, 1978).

gneisses. It is accompanied with the emplacement of a few basic and ultrabasic rocks like pyroxenites and anorthosites. The second phase of deformation was linked with the amphibolite facies metamorphism and affected the above groups along with the Sargurs folding them into E-W trending normal folds. It is also marked by migmatisation now represented by the grey grantitic gneisses. The third phase of deformation is associated with the Greenschist facies of metamorphism which folded the Dharwars and refolded the earlier folds into NNW-SSE trending normal folds. Three sets of fractures and shear zones trending N-S, WNW-ESE and ENE-WSW were developed during this phase and the major uplift of the Western Ghats also is assumed to be occurred during this phase. The fourth phase of deformation which is seen most conspiciously in Tamil Nadu seems to have produced only minor folding on a NNE to NE axis and is perhaps correlatable to Eastern Ghat orogeny. The fifth phase of tectonogenesis is related to the development of western coast of India during Cretaceous-Eocene times (Rao, P.S. 1976). The uplift and formation of the Wynad plateau can be attributed to this phase of deformation. The Upper Cretaceous faulting separated the SW part of Indian subcontinent from Madagascar. From the absence of Cretaceous strata in Travancore and Cochin, the venue of faulting is believed to have occurred at some distance west from the present coast. The final stage of uplift of the Western Ghats occurred during this period and the Kerala coast line runs almost parallel to the escarpment of Western Ghats (Rao, P.S. 1976).

Lineament analysis in the Kerala coastal belt (Varada Rajan and Balakrishnan, 1980) reveals three distinct sets of geofractures trending in (1) NW-SE to WNW-ESE (2) NNW-SSE and 3) NE-SW. These lineaments represent deep seated fractures and extend into the sedimentary terrain with a slightly subdued depression. Of these three sets, the NW-SE to WNW-ESE ones appear to have significantly controlled the sedimentation resulting in different facies in different blocks. NE-SW trending lineaments are seen both in the hills and in the coastal belt. Movement along these could have continued upto Recent times which is evidenced by the fact that even recent landscapes including laterite have been affected. The origin of Ashtamudi kayal near Quilon is assigned to subsidence along NE-SW trending faults. The major shear zones continue into the offshore zones also. A number of offsets towards east can be seen along the Kerala coast at several places. The major off sets include the one at Kottekkunnu (north of Azhikkal) and Kadalur (south of Badagara) which are due to faults (Soman K, 1980).

Evidences of uplift are seen along the coast at several places. The Warkalli rocks occurring at a height above sea level indicate post-Warkalli uplift approximating to 60-900 m. The maximum uplift in the region of Calicut and Cannanore as indicated by the flat topped lateritic ridges and shell deposits of marine and estuarine conditions have been estimated at about 150 m from sea lvel. Recent and subrecent raised beaches and sand bars along the coasts, the raised old river terraces and submerged laterites in the coastal plains at several places indicate repeated changes in the sea level (Rao, P.S. 1976). Low intensity earth tremors of local nature were recorded at several places in the recent times confined mainly to the 20 km long strip along the coast with Calicut in the centre, and for about 32 km eastward from Calicut to the interior. These tremors are considered to have originated from movements along weak planes or faults in the offshore areas, being caused by minor coastal adjustments (Karunakaran and Mahadevan, 1968).

BACKGROUND INFORMATION

CHAPTER - IV

4.1 Placer Geology

Heavy mineral placers are surficial mineral deposits that have been formed by the mechanical concentration of detrital mineral particles due to weathering, transportation and deposition by natural agents in mostly subaqueous environments and can occur in rivers, lakes and on sea floors. Derivation of the term 'Placer' is uncertain but it denotes sand bank in Spanish and is reported to have been used by the early Spanish miners in U.S.A. Important examples of placer minerals include elements in their native state such as gold, platinium, and diamond and resistant minerals such as cassiterite, ilmenite, rutile, zircon, monazite, garnet, magnetite and corundum.

The formation of placer mineral deposits requires a primary source and involves processes of erosion, transportation and concentration. The concentration of the placer minerals into a deposit takes place in two stages. In the first stage, the stable minerals are liberated by weathering of the parent rock and in the second stage their concentration takes place. These are possible only when the minerals have the following properties :

(i) Resistance to chemical weathering and mechanical abrasion,

(ii) Durability (malleability and hardness) and

(iii) High specific gravity.

In published reports and papers by many authors placers are classified based on, the mode of concentration (alluvial, eluvial, eolian, coastal/beach); important component (gold-bearing, platinium-bearing, gem-bearing, zircon, ilmenite, cassiterite sands etc.); geological age (modern, middle and early-Tertiary etc.); relative age and relief (modern, submerged); degree of sorting and weathering (unconsolidated, consolidated); location (valley, terrain etc.); and degree of transportation of detrital material (local and distant placers).

Eluvial Placers.

The eluvial placers are formed upon slopes of the hills, by the material released from the weathered lodes that outcrop above them. eg:-eluvial gold, cassiterite, coulombite and tantalite placer deposits.

Alluvial Placers.

The next stage in the placer formation is the concentration of heavier minerals when they are transported by rivers or streams. eg:- The xenotime occurrence of Singhbhum shear zone, Closepet granites of Hassan and Mandya district, Karnataka.

Eolian Placers.

These are formed when wind instead of water acts as the agent of concentration. eg:- Gold placers from Australian deserts, the heavy mineral bearing inland placer deposit (Teris) of Tirunelveli and Ramanathapuram districts of Tamil Nadu.

Beach Placers.

Rich deposits of beach placers originate under optimum conditions of interaction along the interface between the geological environments on land and the dynamics of wave action and other coastal processes. These are deposited along the sea shore by wave and wind action. The factors controlling the formation of beach placers are geological, climate, drainage pattern, coastal processes, coastal geomorphology, neotectonics and continental shelf morphology. Most of the well known placer deposits of ilmenite, zircon, rutile, monazite, garnet and sillimanite are found associated with the beach placers. Though, all modes of placer formations are important, beach placers are significant by virtue of their extent and large tonnage. India has some of the richest and the largest placer deposits along its eastern and western coastal tracts.

Local and Distant Placers.

Placer deposits of local sources are described as large geological assemblage of placers with minerals not having the capability to persist over a long distance. Generally they are formed at a distance not more than 15 km from the source area. They are otherwise called autochthonous placers. Placer deposits of distant source are described as huge geological assemblage of placers not having any apparent connection with the primary sources and formed due to multiple transportation and distribution processes. They are otherwise called regional/aerial/allochthonous placers.

Petrogenic and Oregenic Placers.

On the basis of petrologic characteristics of source rocks of heavy minerals and their origin, two genetic types of beach placers are distinguished namely petrogenic and oregenic placers. (Suresh Babu, 1990). Petrogenic placers are formed from stable accessory and rock forming minerals as a result of the breaking of country rocks. eg. ilmenite, zircon, rutile, monazite, sillimanite, garnet, magnetite, chromite and others. Locations of petrogenic beach placers are connected with those modern continental margins where mainly Precambrian rocks are exposed for exogenic processes. Ilmenite, zircon placer deposits of south west coast of India form one such petrogenic placers. Oregenic placers are formed from stable minerals due to erosion of ore bodies, ore deposit or abandoned mines. eg. diamond, gold, platinium, cassiterite, ilmenite and chromite. Such zones are located at some places of West African coast, Pacific margins of Asia and America, British Islands, Tansmania and New Zealand coasts.

The placer deposits of India can be broadly grouped into two categories depending on their occurrence.

1) Coastal deposits including the dunes and

2) Inland deposits including the eluvial and older alluvial deposits. Almost all the deposits are composed of the light heavy minerals though a few fluvial occurrences of heavy heavy minerals, mostly gold, are known but are not economically important

4.2 Heavy Minerals

Heavy minerals are volumetrically minor constituents in terrigenous rocks. They are characterised as having a specific gravity greater than 2.85. Until the 1950's the heavies were studied as a tool for stratigraphic correlation. This application has long since been replaced by microfossils and geophysical methods. Heavy minerals are now studied as a guide to source rock lithologies and dispersal patterns. They are also useful in evaluating diagenetic history as well as pre-erosional weathering and tectonic history of the source area. Age dating of source heavy mineral species can provide important information regarding paleogeographic reconstruction. Apart from these their chief important lies in their economic and commercial uses. Various heavy minerals are used for different purposes, ranging from refractories to paints and pigments.

Multidimentional studies are being carried out all over the world in connection with heavy mineral formation and particularly on Fe-Ti oxides. Concentration and distribution of heavies in the coastal zone forms the subject of research to different scientists. Many scientists have classified heavy minerals based on the source rock composition, specific gravity as well as mineral stability during weathering and diagenesis. Folk (1954) attempted to relate and classify heavy minerals on the basis of the texture of sediments, provenance and tectonism.

Emery and Noakes (1968) have classified heavy minerals according to their specific gravity as heavy heavy minerals, light heavy minerals and gem placers. In accordance to their physical and chemical nature, heavy minerals may be divided into four groups, opaques, micas, ultra-stables and meta-stables, Folk (1974). Based on the characteristics of source rock, a classification of detrital heavy mineral suite was carried out by Pettijohn (1975).

The origin of many heavy mineral species is limited to rather specific rock types and these provides valuable information concerning provenance, which describe the character of source terrain, especially the lithologies present (Roy Lindholm, 1987). The role of heavies in provenance studies were stressed by Mortan (1985).

Much work have been carried out by various scientists on palaeogeographic reconstruction and dispersal patterns of heavy minerals. The work carried out by Luepke (1980) showed that in beach sand mineralogical studies of the heavies aid in comparing the source and sorting effects. The processes of selective grain transport and placer formation were studied in detail by Komar et. al., (1984). Slingerland and Smith (1986) discussed the conditions required for placer development and gave the mathematical explanations for mechanisms of hydraulic sorting and settling. A comparative study of the placer mineral distribution of the modern and ancient deposits were conducted by Luepke and Clifton (1985).

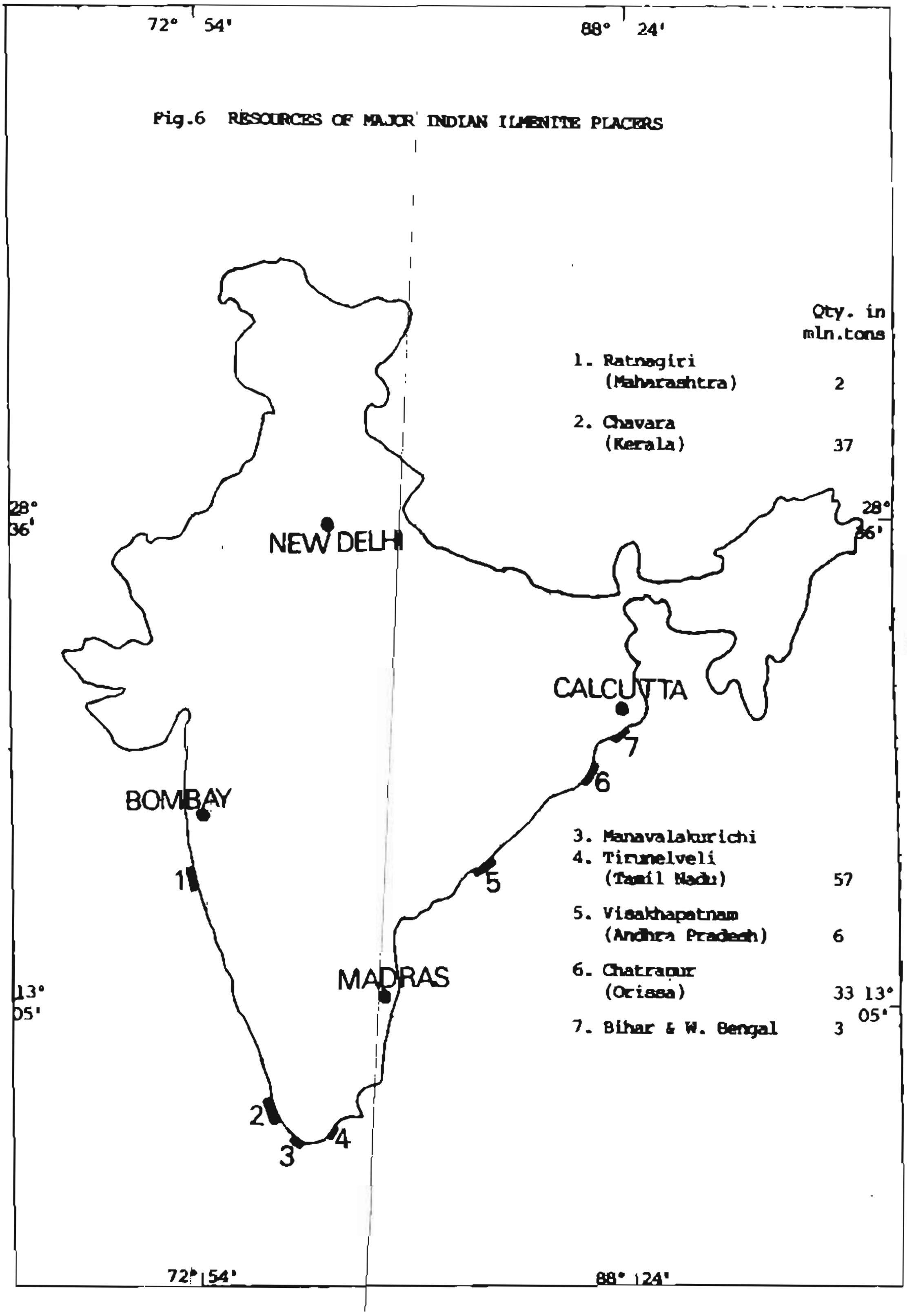
The world occurrences of important heavy mineral deposits have been reported by many authors. Beach sand heavy mineral placer deposits of Queensland have been studied by Connah (1961). An extensive study regarding the nature,occurrence, origin and exploitability of marine mineral deposit was done by Cronan (1980). Ilmenite and zircon placers from Zambezi shelf has been described by Beiersdorf et.al., (1980). Titanium and zircon placer deposits of Sri Lanka have been described by Meyer (1983). The magnetic surveys off Oregon coast were carried out by Peterson (1985). The distribution and textural characteristics of the heavy minerals in the beach and dune sands of Cox's Bazar, Bangladesh were carried out by Mitra & Ahmed (1990).

4.3 Indian Placers

India hosts rich resources of modern (coastal) and submerged (inland) placer concentrations. (Fig. 6). Placer deposits containing minerals of industrial and strategic importance like ilmenite, rutile, sillimanite, zircon, monazite etc. occur along the eastern and western sea-board of Peninsular India in discontinuous patches. They vary in size, grade, mineralogical and chemical composition and in mode of formation. Three of them namely Chavara (Kerala), Manavalakurichi (Tamil Nadu) and Chatrapur (Orissa) are of commercial grade and economically viable. The Chavara and Manavalakurichi deposits have been exploited since six decades. In the complex mineral assemblage of the Indian placer deposits, ilmenite is the major ubiquitous mineral having huge potential for the titanium based industry. In India the ilmenite reserves are of the order of 188.4 million tonnes with about 12.7 million tonnes in Chavara area.

Details regarding the occurrence of ilmenite sand bodies spread along the west and east coasts and continental slope of India have been reported and critically studied by many scientists and researchers. The occurrence of monazite bearing black sands along the southern and south-western coast of India was discovered by Shomberg, a German national in 1909. Tipper (1914) was the pioneer as far as the study of heavy minerals in India is concerned. He made an introductory study of monazite sands of Travancore. Mahadevan and Sriram Das (1948) reported for the first time, black sand concentration along

Fig.6 RESOURCES OF MAJOR INDIAN ILMENITE PLACERS



	Qty. in mln.tons
1. Ratnagiri (Maharashtra)	2
2. Chavara (Kerala)	37
3. Manavalakurichi 4. Tirunelveli (Tamil Nadu)	57
5. Visakhapatnam (Andhra Pradesh)	6
6. Chatrapur (Orissa)	33
7. Bihar & W. Bengal	3

Visakhapatnam-Bhimunipatnam coast, Andhra Pradesh. Ilmenite and garnet sands of Chowghat, west coast, Tirunelveli, Ramnad and Tanjore coasts were recorded by Jacob (1956). Viswanathan (1957) studied the Travancore beach sands and discussed in length about the prevalent separation practices and compares the mineral contents of Quilon and Manavalakurichi deposits. Ilmenite sands along Ratnagiri coast, Maharashtra were discussed by Roy (1958). The origin of heavies in beaches of south-west coast of India was explained by Aswathanarayana (1964). Setty (1972) worked on the problem of distribution of heavies in the shelf sediments off Madras coast and tried to explain them in relation to palaeogeography of the region. Mallik (1974) gave an outline about the nature and origin of heavies in beaches and offshore areas. He explained their origin and studied their economic potential and exploration patterns and methods. Prabhakar Rao (1973, 76, 77) gave details of heavy mineral reserves of beach placers of Neendakara-Kayamkulam (Kerala), Manavalakurichi (Tamil Nadu) and Chatrapur-Gopalpur deposit of Orissa.

Gundu Rao (1975) analysed the interspecific association of heavies and their role in provenance studies. The heavy mineral distribution pattern in the Arabian Sea was studied by Mallik (1976) and based on the mineral assemblages, he recognized four distinct provinces. The off-shore extension of the heavy mineral beach occurrence was established by Siddique et. al., (1979, 1982) along the

Konkan coast. Studies based on grain characteristics were primarly carried out by Sinha Roy (1982). The geophysical exploration for ilmenite placers were carried out off the Konkan coast by Siddique et. al., (1982). A.M. Nair and Narayanaswamy (1984) compared the morphometric characters of zircon along the Manimalai river and linked the inferences with the petrochemical data of the river basin and host rock. Soman (1985) linked the ilmenite, sillimanite and zircon formation to the migmatisation phase associated with the metamorphism.

Studies regarding the micromorphological features of heavies were carried out by Mallik (1986). Geophysical investigations were carried out off the Vijaydurg Bay, Maharashtra by Gujar et.al., (1986). Sastry et.al., (1987) conducted heavy mineral investigations along the Vishakapatnam coast. In India, the ilmenite reserve accounts to about 13.2 million tons in the onshore. The offshore potential is much greater than this. The ilmenite placer extends to 9-12m depth and 2.5 km off shore. Textural characteristics of beach sands along Fort Kochi were investigated by Purandara (1987). Unnikrishnan (1987) studied the texture, mineralogy and provenance of beach sands from Azhicode to Manavalakurichi. Sasidaran and Damodaran (1988) were of opinion that the present distribution of the heavy mineral sands in the Malabar coast is, to a large extent controlled by the longshore current. A detailed investigation and study of heavies were carried out in the

Paravur coast by Senthiappan (1989). An extensive work on distribution, paths of migration and prognostic assessment of heavy mineral concentrations in the beach placers of southern Kerala, was carried out by Suresh Babu (1990). A detailed work on paleogeographic interpretation of Kerala beach placers was made by Suresh Babu and Thrivikramaji (1993).

Although extensive research is being carried out on bulk sediment samples, the systematic study of individual fractions have till now been confined to isolated attempts. Government agencies like the Geological Survey of India, Atomic Minerals Division and National Institute of Oceanography are engaged in the assessment of ilmenite placers.

4.4 Chavara Deposit

Kerala is singularly fortunate being endowed with one of the richest placer deposit in the world namely Chavara deposit (i.e. the Neendakara-Kayamkulam deposit). The Chavara deposit with its landward extension rank as one of the richest and largest ilmenite deposits of the world. The deposit explored down to a depth of 7.5. m. is estimated to contain about 12.7 million tonnes of ilmenite, 1 million tonne of rutile, 0.9 MT of zircon, 0.17 MT of monazite and 2.0 MT of sillimanite. The average heavy mineral content of this deposit (upto an average depth of 7.5 m) is 50%, with ilmenite 35%, rutile 2.5%, zircon 2.5%, leucoxene 1.5%, monazite 0.5%, sillimanite 7% and granet 0.35% in the raw sand.

Several facets of geology of Kerala placers have been published earlier in the form of papers and reports by many. Different aspects regarding evolution of Indian west coast, transport and distribution of coastal sediments, mineralisation in the western shelf and provenance of heavy minerals were discussed by several scientists. Description of occurrence of beach placers along the southwest coast of India are found in Tipper (1914), Mahadevan and Rao (1950), Jacob (1956), Viswanathan (1957), Roy (1958), Gillson (1959), Rao (1962), Aswathanarayana (1964), Mallik (1974), Siddique et.al., (1979), Soman (1985), Mallik et.al., (1987) and Suresh Babu (1990).

Prabhakar Rao (1962) is of the opinion that the placer deposits on the west coast of India have formed by one or all of the following three processes.

i) deposition on an earlier strand line and transport to the present position by rain wash and rivers;

ii) reworking of the Warkalli sediments; and

iii) derivation of present day sediments from the hinterland.

The minerals are derived mainly from the Precambrian metamorphic rocks viz. charnockites, khondalites, granite gneisses, pegmatites, retrograde charnockites which are exposed in the drainage basin of the different rivers flowing towards west in to Arabian Sea. (Mallik et.al., 1987). They have also suggested a three stage depositional model for the heavy mineral placer sands.

i) Initial stage - supply of heavy minerals through rivers and deposition of sands containing disseminated placers by waves, currents etc.

ii) Transgressive stage - erosion and reworking of beach ridges.

iii) Regressive stage - formation of placers in beach due to waves and longshore current activity giving rise to present configuration.

The heavy mineral sands of the Kerala coast have a plural provenance which include the Precambrian crystalline rocks, the Warkalli sediments and the sediments from the palaeoberms. The final enrichment of heavies is brought about in several stages. (Rajagopal et.al., 1985).

i) Chemical weathering and lateritisation of crystalline rocks under tropical climate wherein the unstable ferromagnesian minerals like amphiboles and pyroxene are totally destroyed along with feldspar resulting in the enrichment of the resistant minerals like ilmenite, rutile, zircon, monazite etc. Lateritisation of the Warkallis.

ii) Weathering of the laterites and liberation of the heavy minerals, transportation of the liberated heavy minerals, along with the sediments by the streams and rivers draining the hinterland to the ultimate site of deposition.

iii) Direct derivation of the heavy minerals from the Warkallis by wave action and undercutting of the formation.

iv) Heavy minerals reconcentrated from former basin along the paleo - strand line.

v) Submarine erosion of the sea-bed itself in the continental shelf region (Prabhakar Rao, 1974).

The heavy minerals thus liberated are transported by the river system descending from the hinterland and are sorted and concentrated along the beach by the normal process of wave action and deposition (Rajagopal et.al., 1985). Soman (1985) linked the ilmenite, sillimanite and zircon formation to the migmatisation phase associated with the metamorphism. The presence of promontaries/sea cliffs, shore processes especially longshore currents, and typomorphic characteristics of minerals are responsible for the formation of modern beach placers. (Suresh Babu, 1990).

4.5 Status on Ilmenite Characterisation

Ilmenite is being subjected for research for different purposes. One group of scientists work on the solid solution series, exsolution/intergrowth textures, crystal structure, magnetic structure variations and high temperature alteration phenomena associated with ilmenite. Another class of researchers concentrate on weathering and diagenetic changes (low temperature processes) in ilmenite. Several publications reporting on the trace element characteristics and physical properties of ilmenite are available. Yet, a lot of literature on metallurgical/beneficiation studies to process ilmenite for extraction of value-added products are also available.

Exsolution and intergrowth textures of iron and titanium oxide have been studied by Basu and Molinaroli (1989), Buddington and Lindsley (1964). Structural characteristics of ilmenite and associated minerals relating to cation positions have been reported by Shirane et.al. (1959), (1962). The magnetic structure of the heamatite-ilmenite series were described by Hoffman (1975) and Lindsley (1976).

Temple (1966) suggested that the alteration of ilmenite in igneous and metamorphic rocks probably of hydrothermal origin. Buddington and Lindsley (1964) observed that in some Adirondack rocks, ilmenite commonly, get slightly hydrothermally altered to a mixture of heamatite, rutile, anatase and relic-ilmenite. They called the aggregate as "meta-ilmenite". Wilkinson (1957) reported a deuteric alteration of titaniferous magnetite to sphene or leucoxene. The alteration of ilmenite and titaniferous magnetite to leucoxene in granulite facies of rocks was noted by Ramberg (1948), who related it to regional metamorphism of extrusive rocks. Elsdon (1972) reported a hydrothermal decomposition of ilmenite to rutile.

The alteration of ilmenite in beach sands has been extensively studied due to its economic importance. (Hartman, 1959), Van Houten, 1968). Alteration of detrital ilmenite has also been studied by Hoffman (1975) and Morad (1986). It has been reported that ilmenite alters into products like anatase (Anand & Gilkes, 1984) rutile, a non crystalline iron titanite (Bailey et.al., 1956); arizonite (Palmer, 1909); (Karkhanavala, 1959); hydroilmenite (Flinter, 1959); Pro-arizonite (Bykov, 1964); and pseudo-rutile (Teufer and Temple, 1966); (Grey and Ried, 1975).

Fe^{2+}/Fe^{3+} ratios in ilmenite of intermediate composition have been studied in detail by Lopez Carranza & Cox (1979), Shirane et. al. (1962) and White (1990). The alteration of ilmenite is accompanied by the removal of structural iron, which results finally in an almost pure TiO_2 phase. Lynd and others (1954) concluded that in this alteration, the iron (mostly Fe^{2+}) is removed by leaching. In the case of progressively altered beach sand ilmenite, Flinter (1959) showed that the increase in TiO_2 content is associated with an increase in Fe^{3+} content. He concluded that the removal of structural iron occur in response to oxidation of iron in the ilmenite lattice. Temple (1966) suggested that oxidation in ilmenite grains is initially restricted to interfaces along structural dislocations and that partial removal of iron creats voids which allows further and deeper oxidation until the ilmenite lattice is completely decomposed.

Three successive stages of alteration of ilmenite were recognised by Bailey et.al. (1956) as patchy-ilmenite, the first stage, which consists of a patchy intergrowth of altered and fresh ilmenite, the second stage as amorphous iron-titanium oxide phase and finally to leucoxene (TiO_2) in stage three. Ramdohr (1941) and Karkhanavala and Momin (1959) observed that oxidation of ilmenite in air results in the formation of rutile and pseudorutile. Temple (1966) and Grey and Reid (1975) identified pseudorutile as an intermediate phase in the alteration of ilmenite. They reported that the alteration of ilmenite is a 2 stage process.

First, all of the iron oxidizes and some diffuses from the ilmenite leaving pseudorutile in which closely packed oxygen layers remain intact. This stage of alteration is represented by the following reaction.

$$3Fe^{2+}TiO_3 \longrightarrow Fe_2^{3+}Ti_3O_9 + Fe^{3+}$$

In the second stage of alteration pseudorutile dissolves, iron is removed by solution, and TiO_2 precipitates rapidly. The second stage involves a dissruption of the oxygen packing as both iron and oxygen are removed and is represented by the following reaction.

$$Fe_2^{3+} Ti_3O_9 \longrightarrow 3TiO_2 + Fe_2O_3$$

Grey and Reid (1975) considered that epitaxial growth of TiO_2 (rutile) crystals occur on a pseudorutile substrata so that the primary ilmenite grain is replaced by a highly oriented array of rutile microcrystals. Haggerty (1976) found that the alteration of ilmenite may include an intermediate ferrian rutile. According to one school of thought the crystalline hexagonal ilmenite on weathering passes through an amorphous stage and finally TiO_2 crystallizes as rutile in tetragonal system (Barksdale, 1966) while the other supports conversion of ilmenite first to hexagonal pseudorutile and with further loss of iron to tetragonal rutile (Parkharykov et.al., 1976). Frost et.al., (1983) has identified four stages of ilmenite alteration depending on the degree of iron removal as ferrian ilmenite, hydrated ilmenite, pseudorutile and leucoxene.

Low temperature oxidation is the major chemical change taking place in the ilmenite after its release from country rocks (Akimoto et. al. 1984). Fe-Ti oxide grains may alter diagenetically

(Sun and Allen, 1967; Ixer et. al., 1979, Morad and Aldahan, 1982, 1986). or prior to burial diagenesis. Canon (1949) believed that the alteration occurs mainly by weathering in the source rocks and during transportation. The important changes in physical property during weathering include decrease in magnetic susecptibility and specific gravity (Granin et. al., 1980). Pseudomorphs are porous and have a density of 3.3 as against 4.26 for rutile crystals (Barksdale, 1956). Trace element characteristics prove to be good indicator for distinguishing ilmenite samples belonging to spacially different zones, Darby (1984) and Darby and Tsang (1987). Grigsby (1992) reported that detrital ilmenites carry unique chemical fingerprints that can be used in provenance research.

METHODOLOGY

CHAPTER - V

5.1 Field Work & Sampling

Field survey and sampling were carried out during October to November, 1993 for a period of 2 months. Mainly road transport was sought for during the field work as the study area is fairly accessible by NH-47 and by a network of mining and metalled roads, which runs almost parallel to the coast.

The equipment used for sampling was mainly an auger drill which consists of a handle with Tee and a 2 metre extension rod attached to the auger for collecting subsurface (pit) samples, a shovel for taking surface samples and an aluminium pan for preliminary wet panning in the field itself. (Plates I & II)

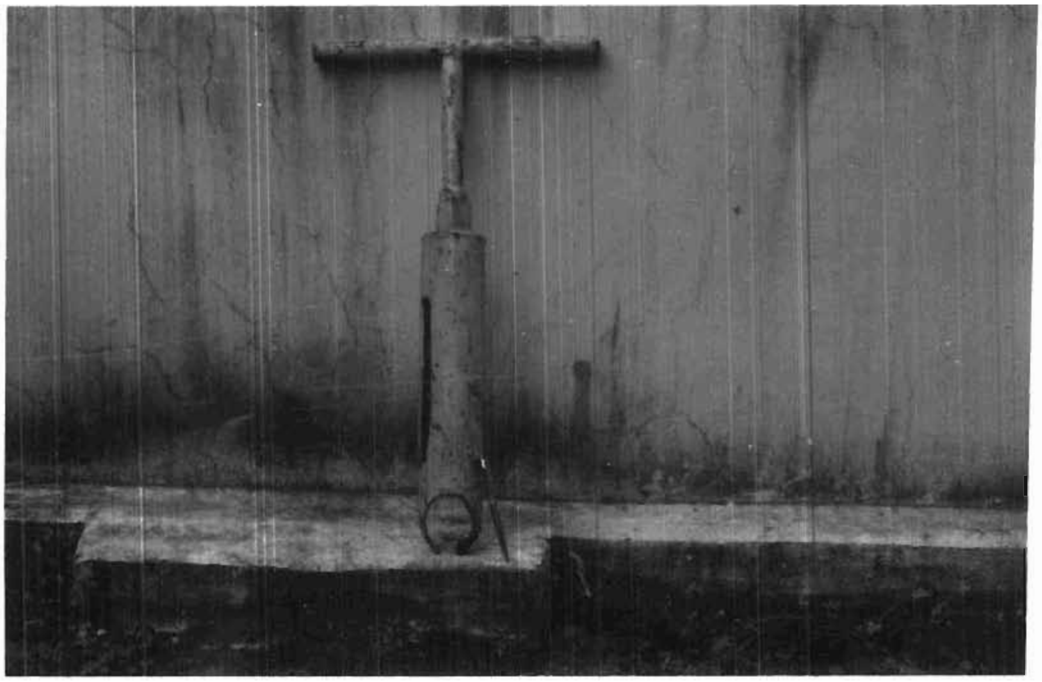
After the initial collection of samples from the beach, samples from western and eastern margin of kayal/lagoon were also collected. Likewise rest of the sampling has been done along eight cross-sections of the Neendakara-Kayamkulam sector (Fig. 1) along a pre-determined grid pattern. The sampling area as a whole cover a distance of approximately 28 km^3. Total 37 samples have been collected out of which 14 samples were scooped out from a depth of about 1 m. beneath the ground level using auger drill and rest of the samples from the beach and from the western and eastern margin of kayal/lagoon.

PLATE - I

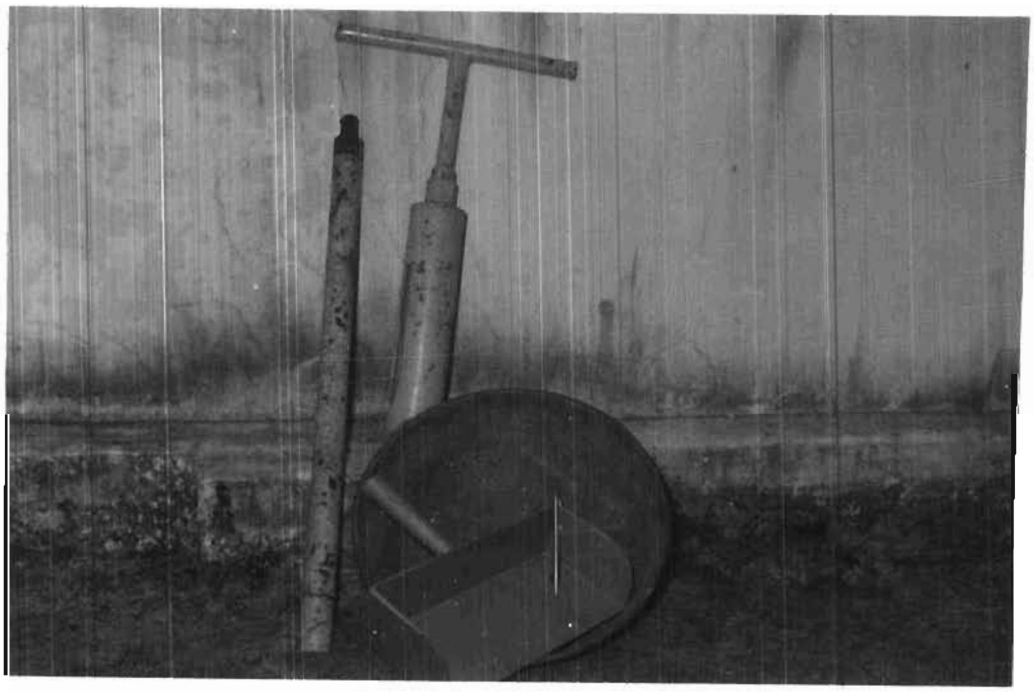
A Auger drill consisting of handle with Tee.

B Auger drill with 1 metre extension rod, aluminium pan and shovel.

PLATE I



A



B

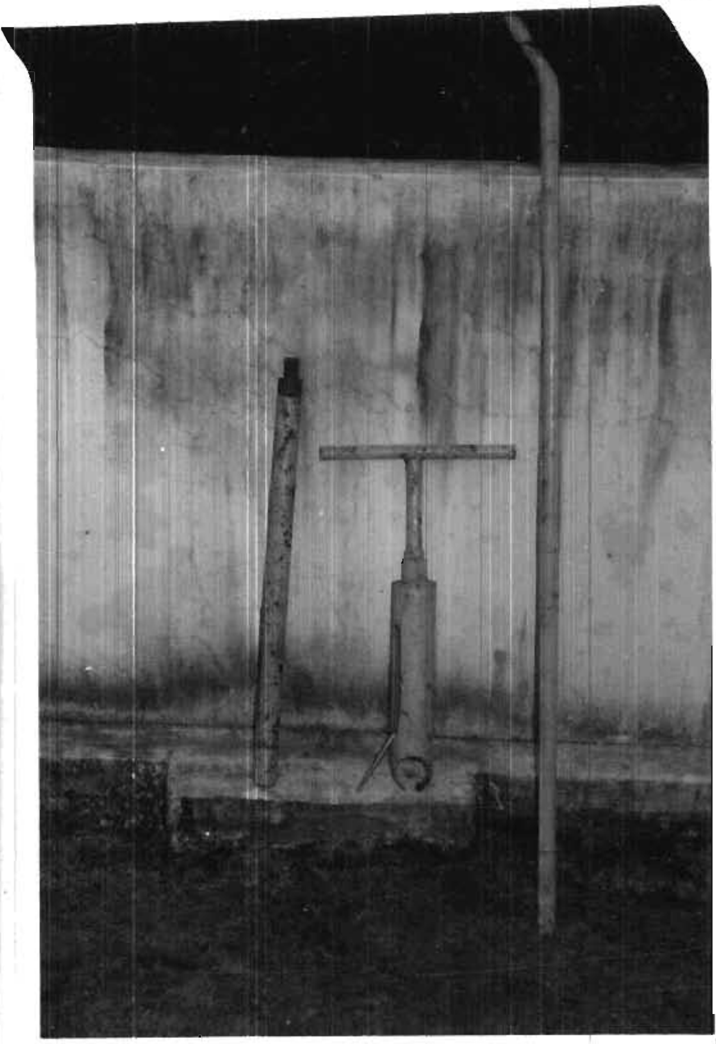
PLATE - II

A Auger drill with 2 metre extension rod.

B Collection of subsurface samples at Ponmana.

B

PLATE II



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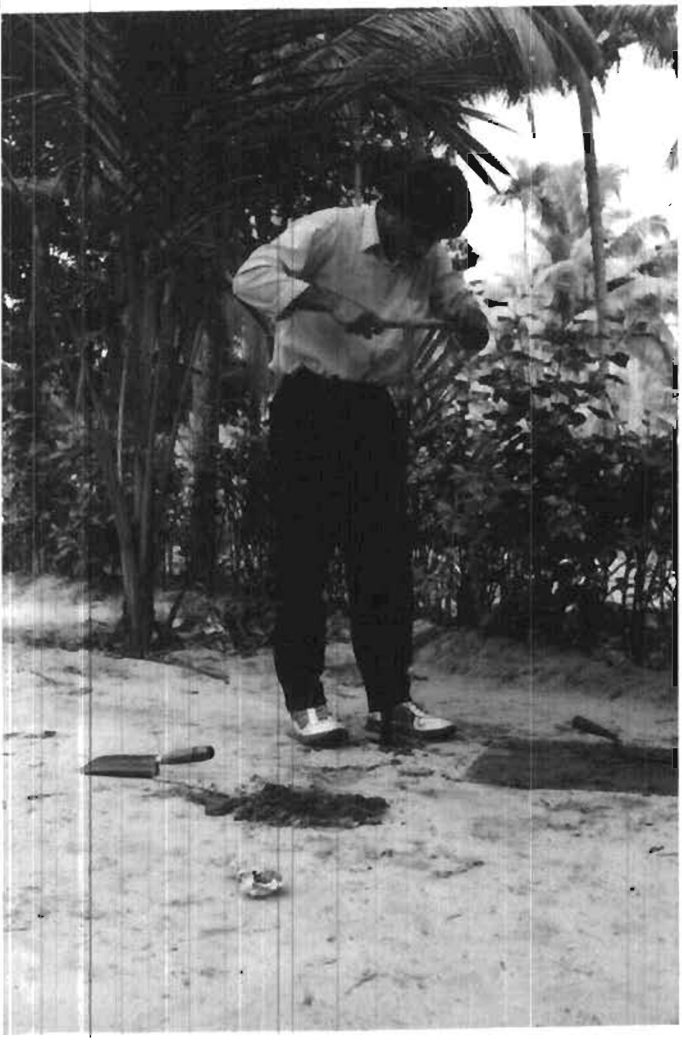


B

The sampling was done approximately at a distance of 3 kms. interval from Putantura in the south to Tharayilkadavu in the north. Wet panning of samples was carried out at each location so as to obtain a heavy mineral concentrate. An outline of the field data were noted down on a field book. The sampling locations were marked on a base map of 1:50,000 scale.

5.2 Mineral Separation

The collected samples were repeatedly wet panned in the laboratory and washed thoroughly. After drying, each sample was spread on a sheet of paper and ferromagnetic portion was removed using a horse-shoe hand magnet. The samples were then sieved in order to get uniform size fraction before feeding into the Isodynamic Separator. Ilmenite concentrate were separated from the magnetite-free samples by passing them several times into the Frantz Isodynamic Separator. The separator was kept with a foreward slope of 25° and side slope of 15°. Varying currents of 0.1, 0.2, 0.3, 0.5 and 0.6 amperes were applied inorder to collect the ilmenite grains fairly well from each sample. The ilmenite rich concentrate separated by Isodynamic separation was then purified to 100% by hand-picking. This was done under the optical microscope by removing the non-opaque grains manually. A portion of each of the purified sample was ground in an agate mortar to a very fine powder.

5.3 Chemical Analysis (Conventional and AAS)

Chemical analysis of 37 samples was carried out by standard wet chemical methods, aluminium reduction method for TiO_2 and dichromate titration for iron as formulated by Vogel, (1961). The following procedures were used for total iron, ferrous iron, and titanium dioxide determination.

a) Estimation of Total Iron

Weigh out accurately about 0.5 gms of the finely ground sample and fuse it with about 10-15 gms. of potassium hydrogen sulphate in a silica crucible. First melt the potassium hydrogen sulphate to get a clear solution. Then allow to solidify. Add the powdered sample and heat at low temperature first. During heating swirl the contents of the crucible carefully. Then increase the temperature to maximum. Keep the crucible at this temperature (900°C) for 20-30 mts. Then allow the crucible to cool and place inside a beaker containing 20% H_2SO_4 and heat till the dissolution is complete. Cool the solution and make up to 250 ml.

Pipette out 25 ml. of this solution in a conical flask and add 5 ml. of concentrated HCl and heat to about 80° C. Reduce the iron solution by adding freshly prepared $SnCl_2$ (Stannous chloride) solution dropwise until the yellow colour of the solution disappears. Add one drop excess. Cool the solution rapidly to 20°C. After cooling dilute the solution to about 100ml. The excess $SnCl_2$ present is removed by adding 10 ml of saturated $HgCl_2$ rapidly in one portion and with thorough mixing. A slight silky white precipitate of mercurous cholride should be obtained. After the addition of $HgCl_2$ allow to stand the whole solution for about 5 mts. and add 5 ml. of concentrated H_2SO_4 and 10 drops of N-phenyl anthranlic acid indicator, and titrate against standard potassium dichromate solution. The end point is the appearance of permanent pink colour throughout the solution.

The percentage of total iron present in the sample can be calculated from the titration data as follows :

$$\text{Percentage of total iron} = \frac{\text{Volume of } K_2Cr_2O_7 \text{ solution} \times \text{factor of } K_2Cr_2O_7 \text{ solution} \times 100 \times 10}{\text{Weight of the sample}}$$

$$\text{Factor of } K_2Cr_2O_7 \text{ solution with respect to iron} = \frac{55.84 \times \text{Normality of } K_2Cr_2O_7}{1000}$$

b) Estimation of Ferrous Iron

Take about 0.3gm of sample (weigh out accurately) in a 100ml conical flask fitted with two curved glass tubes using a cork. Add 100 ml. of 80% concentrated H_2SO_4 and 5 ml. HF. Close well and immediately pass CO_2. Keep on a hot plate for about 5 minutes under

CO_2 atmosphere. Cool the flask well in ice. Add 15% boric acid solution for removing excess HF. Then add 5 ml. of phosphoric acid. This is followed by the addition of 10 drops of sodium-diphenylamine sulphonate indicator. Titrate the solution against standard dichromate solution. The end point is the appearance of purple colour.

The percentage of ferrous iron in the sample can be calculated from the titration data as follows :

$$\text{Percentage of ferrous iron} = \frac{\text{Volume of } K_2Cr_2O_7 \times \text{factor} \times 100}{\text{Weight of the sample}}$$

$$\text{Factor of } K_2Cr_2O_7 \text{ solution with respect to ferrous iron} = \frac{55.84 \times \text{Normality of } K_2Cr_2O_7}{1000}$$

c) Estimation of Titanium Dioxide

Method : (Aluminium Reduction)

Titanium IV in an acid solution (containing HCl) is reduced to titanium III by aluminium and then titrated with a standard oxidant solution.

Apparatus

The apparatus consists of a 500 ml Erlenmeyer flask with a two-hold rubber stopper. A pointed glass rod nearly touching the bottom of the flask is placed in one hole of the stopper and the short end of the delivery tube is placed in the other hole. The other end of the delivery tube is placed into a 400 ml. beaker containing saturated sodium bicarbonate solution. The aluminium foil is attached to the glass rod fixed with the stopper.

Procedure

Weigh out sufficient air dried and powdered sample so as to contain 75 to 100 mg. of titanium and fuse with 5 gm. of fused potassium bisulphate. Cool the cake and dissolve it in 15 ml. concentrated H_2SO_4 and 150 ml. distilled water. Add 30 ml. HCl and take it in the 500 ml. Erlenmeyer flask. Boil the solution. Remove from the burner and attach 2 gms. of high purity aluminium foil to the end of the glass rod of the reductor. Immediately insert the rubber stopper carrying the glass rod with aluminium and the delivery tube into the flask. Place the other end of the delivery tube below the level of sodium bicarbonate solution taken in the 400 ml. beaker. The reaction between the aluminium foil and the solution is rapid. Towards the end of the reactions swirl the flask to ensure complete mixing and reduction. When all the aluminium appears to be dissolved, gently boil the solution for 3-5 minutes keeping the delivery tube still immersed in the sodium bicarbonate solution.

Cool the sample to less then 60°C. As the sample cools, the sodium bicarbonate solution is drawn into the Erlenmeyer flask and the carbon dioxide evolved gives the necessary protective atmosphere. When the solution is cooled remove the stopper and revise the glass rod and the delivery tube with distilled water. Add 2 ml. of 24% ammonium thiocyanate indicator. Titrate with standard ferric-ammonium sulphate solution.

Preparation of Ammonium thiocyanite indicator

Dissolve 24.5 gms. of ammonium thiocyanite in 80 ml. of hot distilled water. Filter through two Whatman 42 filter paper using vaccum. Cool, dilute to 100 ml. and store in a dark coloured bottle.

Standardisation of Ferric Alum

Dissolve 30.16 gms. of ferric ammonium sulphate in 800 ml. distilled water and 15 ml. concentrated H_2SO_4. Add H_2O_2 to ensure complete oxidation of the iron. Boil the solution to remove excess H_2O_2 and dilute to 1 litre. Standardise the solution against standard TiO_2 reduced in the same way as described for the sample.

Estimation of Ferric Iron

The % of Ferric iron can be calculated indirectly as follows :

% of Ferric Fe = % of total Fe - % of Ferrous Fe.

The Atomic Absorption Spectrometer (AAS) was used to detect and calculate the amount of three trace elements namely Mg, Mn & Zn present in the sample. The solution of the samples are prepared as in the determination of total iron and made upto 250 ml. The standard solutions of the various concentration of trace elements to be determined (Mg, Mn and Zn) are prepared. They are fed to the AAS and the values are noted. These represent the absorbance of the solution. The instrument is set in characteristic wave lengths depending on the trace element to be detected. Then the concentration in 250ml of solution containing A grams of sample is calculated using the formula

$$\frac{\text{Concentration of the std.} \times \text{Absorbance of the std}}{\text{Absorbance of the std.} \times \text{A gms. of sample}} \times \frac{100}{10^6} (\%)$$

5.4 X.R.D., T.G.A. and Microscopic Studies

Finely powdered fractions of selected 8 samples were subjected to X-ray diffraction analysis. The analysis was done by Philips diffractometer using Cu K α as target and Ni filter at 20° to 60° 2θ angle. The reflections caused to the X-rays are recorded in the form of a series of peaks of different heights in the recording strip chart. The height of the peaks are proportional to intensity of reflection. The individual peaks were identified by comparing the 'd' values with the standard JCPDS files.

TGA experiments were done for selected four samples, for which XRD analysis have also been carried out. The analysis was done by SHIMADZU TGA 50H at a heating rate of 15° C/minute.

Inorder to carry out ore microscopic and SEM observations two samples were mounted using polyster cold setting resin and polished in the alumina medium.

RESULTS AND DISCUSSION

CHAPTER - VI

RESULTS AND DISCUSSION

A total of 37 samples have been subjected to chemical analysis, XRD, TGA and microscopic studies to evaluate the ilmenite concentrate of Chavara placer deposit.

Table 4 and 5 shows the major element contents of analysed samples. From the tabular data and Fig.7 and 8, it is obvious that there is a systematic lowering of ferrous and total iron contents in ilmenite samples towards east from the shore line especially in the case of V to VIII sections. An exception to this tendency is noticed only for the Ist cross-section of samples and no beach sample has been collected from the Ist set. Though there is no significant variation in the iron content among the samples of IIIrd section, the TiO_2 content in them confirms that no antipathetic variation exists. The samples of cross section I, ie. the southern most traverse (Fig.1) are strikingly different from the other seven sets in the FeO content as well. While all the samples correspond to a value between 5 and 10% of FeO, the samples from southern side of the deposit contain approximately 20% FeO.

In this study, the sampling has been done along three longitudinal directions parallel to one another. While the first stretch

TABLE : 4 MAJOR ELEMENT DATA OF ILMENITE SAMPLES

		CHEMICAL CONSTITUENTS (%)						
Traverse No.	Sample No./ Location*	Total Iron	Fe^{2+}	FeO	Fe^{3+}	Fe_2O_3	$FeO + Fe_2O_3$	TiO_2
I	1. BS-1	26.16	14.89	19.47	11.27	16.11	35.58	60.37
	2. WS-1	28.54	14.17	18.53	14.37	20.55	39.08	56.53
	3. ES-1	29.09	16.03	20.96	13.06	18.67	36.63	56.39
	4. ESS-1a	28.61	14.98	19.58	13.63	19.48	39.06	56.21
	5. ESS-1b	27.87	14.53	19.00	13.34	19.07	38.07	58.03
II	6. WS-2	26.78	8.59	11.23	18.18	25.99	37.22	57.89
	7. ES-2	23.47	4.90	6.40	18.57	26.54	32.94	62.20
	8. ESS-2b	23.62	4.77	6.23	18.85	26.95	33.18	63.54
III	9. BS-3	25.02	7.91	10.34	17.11	24.46	34.80	61.95
	10. WS-3	26.24	10.43	13.63	15.81	22.60	36.26	61.35
	11. ES-3	23.94	7.43	9.71	16.51	23.60	33.31	62.36
	12. ESS-3a	24.74	7.67	10.03	17.07	24.40	34.43	62.38
	13. ESS-3b	24.62	8.10	10.59	16.52	23.61	34.20	62.40
IV	14. BS-4	25.46	7.48	9.78	17.98	25.70	35.48	62.04
	15. WS-4	25.52	8.25	10.78	17.27	24.69	35.47	60.94
	16. ES-4	22.36	3.92	5.12	18.44	26.36	31.48	64.27
	17. ESS-4a	22.57	4.12	5.38	18.45	26.37	31.75	64.26
	18. ESS-4b	22.49	4.42	5.78	18.07	25.83	31.61	64.53

(Contd...)

V	19. BS-5	24.16	4.24	5.54	19.92	28.48	34.02	62.38
	20. WS-5	23.51	4.21	5.50	19.30	27.59	33.09	63.33
	21. ES-5	23.23	3.76	4.91	19.80	28.31	32.79	63.86
	22. ESS-5a	23.07	3.43	4.48	19.64	28.07	32.55	64.22
	23. ESS-5b	23.33	3.63	4.74	19.70	28.16	32.91	64.01
VI	24. BS-6	25.37	5.54	7.24	19.83	28.35	35.59	64.22
	25. WS-6	23.07	4.53	5.92	18.54	26.56	35.48	63.70
	26. ES-6	23.42	4.87	6.36	18.55	26.52	32.88	62.06
	27. ESS-6a	22.51	3.40	4.44	19.11	27.32	31.76	63.48
	28. ESS-6b	23.14	3.35	4.39	19.79	28.29	32.68	64.50
VII	29. BS-7	25.75	7.99	10.44	17.76	25.39	35.83	61.23
	30. WS-7	24.68	7.97	10.42	16.71	23.89	34.31	61.27
	31. ES-7	22.85	4.83	6.31	18.02	25.76	32.07	64.01
	32. ESS-7a	22.68	4.61	6.02	18.07	25.83	31.85	62.82
	33. ESS-7b	23.01	4.99	6.52	18.02	25.77	32.29	63.30
VIII	34. BS-8	24.79	6.78	8.86	18.01	25.75	34.61	63.02
	35. WS-8	24.07	6.41	8.38	17.66	25.24	33.62	62.41
	36. ES-8	23.94	6.74	8.81	17.20	24.59	33.40	61.23
	37. ESS-8a	23.54	5.93	7.75	17.61	25.17	32.92	62.69

*BS - Beach Sample WS - Western Side of Kayal/Lagoon

ES - Eastern Side of Kayal/Lagoon ESS - Eastern Sub-surface Sample

TABLE : 5 CHEMICAL ANALYSIS OF ILMENITES (AVERAGE) FROM EIGHT CROSS SECTIONS

	CHEMICAL CONSTITUENTS (%)							
Traverse No.	Total Iron	FeO	Fe_2O_3	FeO + Fe_2O_3	FeO/Fe_2O_3	TiO_2	Fe_2O_3/TiO_2	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

of samples are from 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 (Fig. 1). In this background, it is observed that the WS samples generally occupy an intermediate position between the BS and ES sets of samples (Fig.7 and 8) in the northern sector based on the ferrous and total iron contents. The subsurface samples (ESS) do not significantly vary with their corresponding surface (ES) samples. Therefore it is assumed that the accumulation seen upto a depth of 1 m from the surface might have been occurredin the recent past only. The total iron content as well as the ferrous iron content give specific clues for measuring the degree of alteration in ilmenite grains (Wort and Jones, 1980).

In addition to the above mentioned regional trends, the distribution of contents of Fe-oxides and TiO_2 also points to the absolute degree of weathering taken place in the Chavara ilmenite. Though physical weathering promotes chemical changes and vice versa, a dominance of chemical action is attributed to the alteration of detrital minerals in the study area due to the surrounding tropical environment. Therefore change in chemistry becomes more important for characterisation of minerals like ilmenite owing to its industrial implications. With respect to the chemical changes in ilmenite associated with weathering, the low temperature oxidation of iron is identified as the major change in chemistry after its release from the

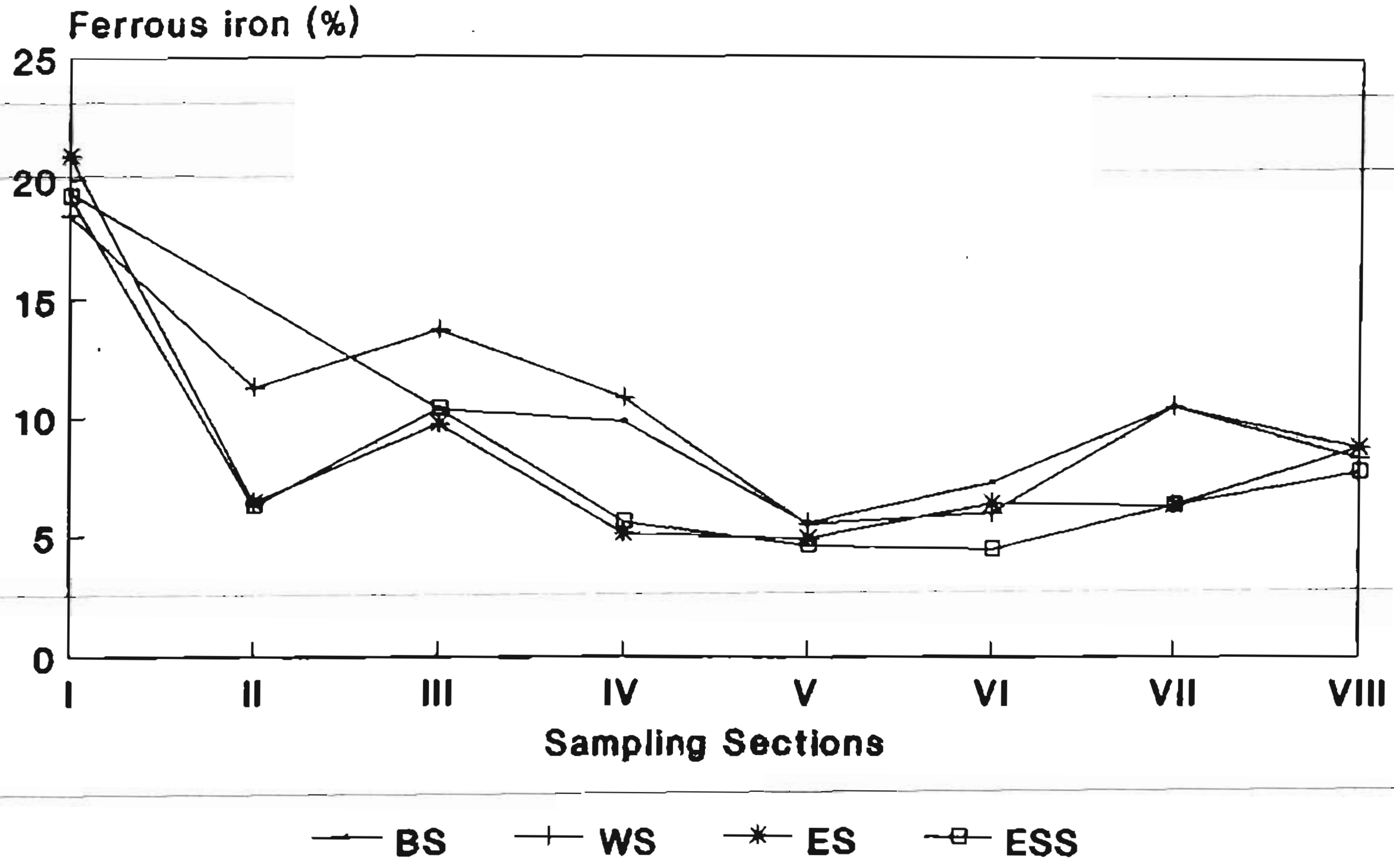


Fig. 7 - Distribution of ferrous iron content in ilmenites

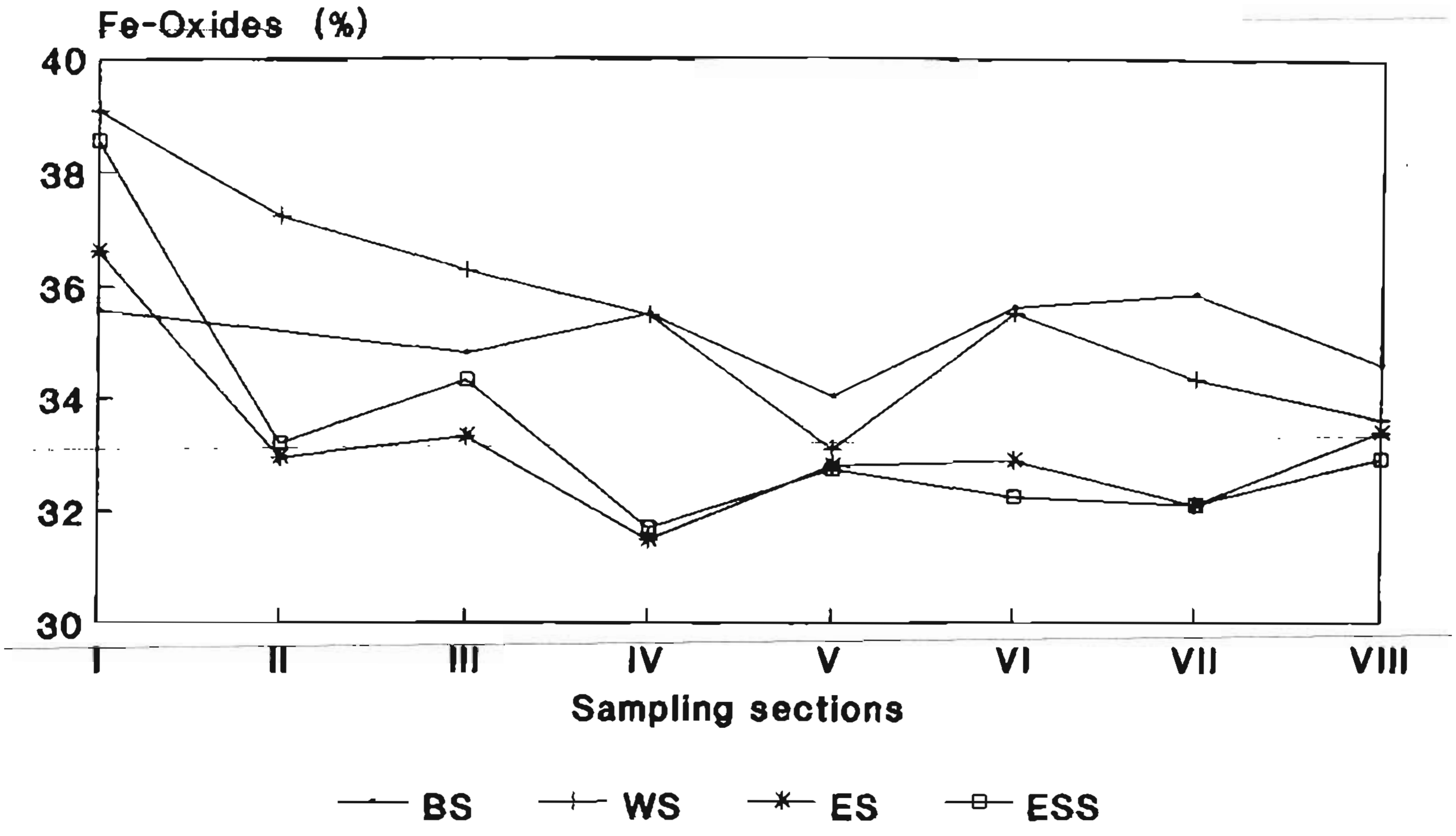


Fig. 8 - Distribution of total iron oxides in ilmenites along eight cross-sections of Chavara deposit

country rocks (Temple, 1966; Akimoto et.al., 1984). Subsequent to ferrous-ferric conversion, iron oxide removal takes place to form the intermediate phase called pseudorutile (Grey and Reid, 1975). The iron removal, in turn, is exhibited by relative increase in TiO_2. Taking into consideration such findings, Frost et. al., (1983) have classified the ilmenite-alteration into four stages. These inferences have been used in the following discussion to assess the Chavara ilmenite.

Total iron oxide contents and the TiO_2 values of ilmenites are illustrated in Fig.9. Fig. 10 shows the trend of FeO, Fe_2O_3 and TiO_2 in the eight cross-sections. Average values from each cross sections are plotted for clarity. Here, the Fe_2O_3 contents do not vary significantly (not more than 2%) among the samples of individual sections. But FeO and TiO_2 show much higher variations (up to 6%) among the samples of individual sections. This clearly shows the difference in ferrous-ferric oxidation rates and also the iron-removal rates (or the TiO_2 enhancement rates) due to the differential alteration of BS, WS and ES sample sets (Fig.8). Therefore it is assumed that the internal variation in the east-west direction within each cross section, as noticed above for FeO and TiO_2, is systematic and would not interfere the comparison between any northern and southern cross-sections. Thus the major element data based on eight cross-sections prove that the TiO_2 value for Chavara ilmenite varies between 61.21% and 63.59% except for the first section (57.5%). This disparity in the

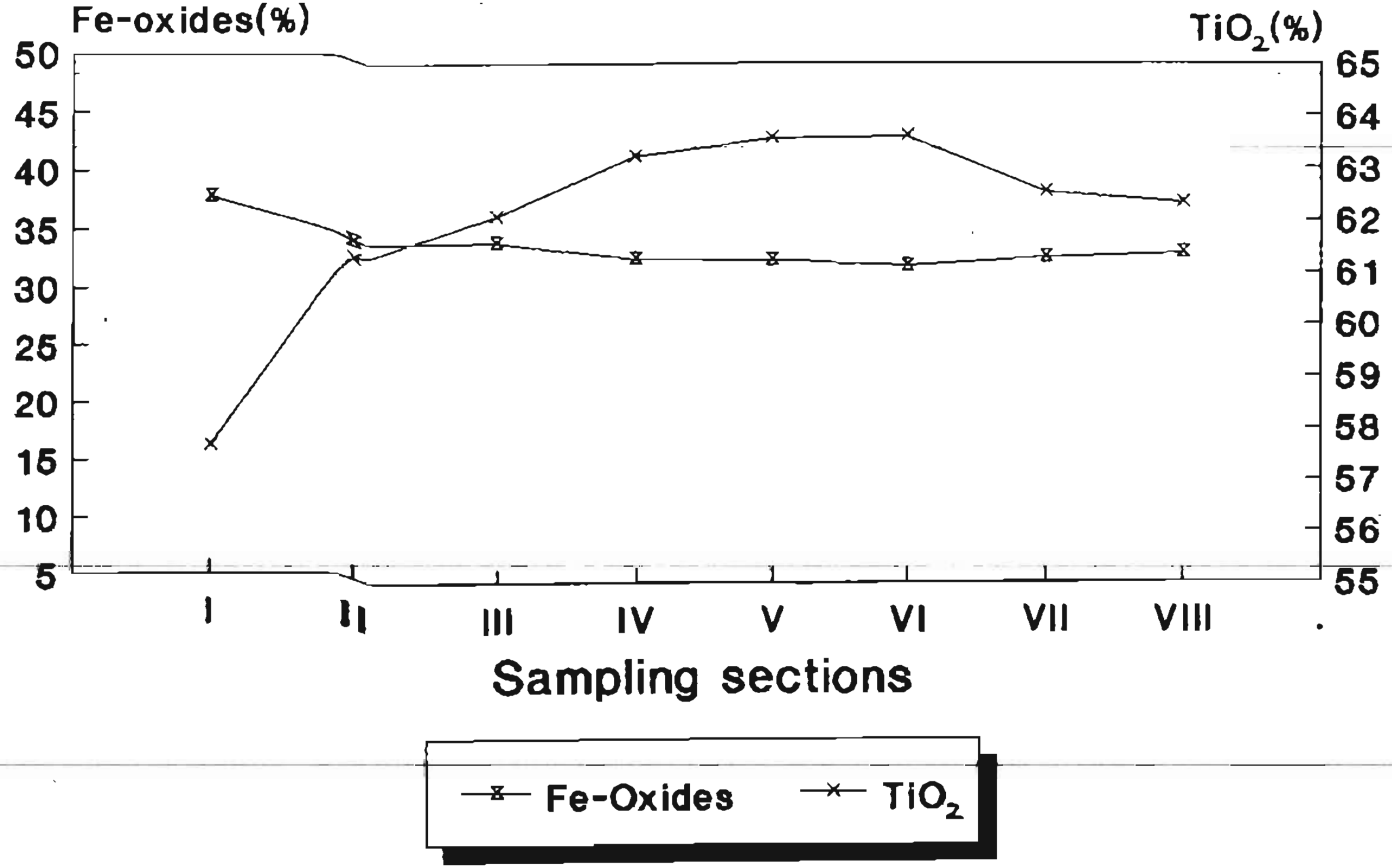


Fig.9- Distribution of Fe-Oxides and TiO_2 in ilmenites along eight cross-section of Chavara deposit.

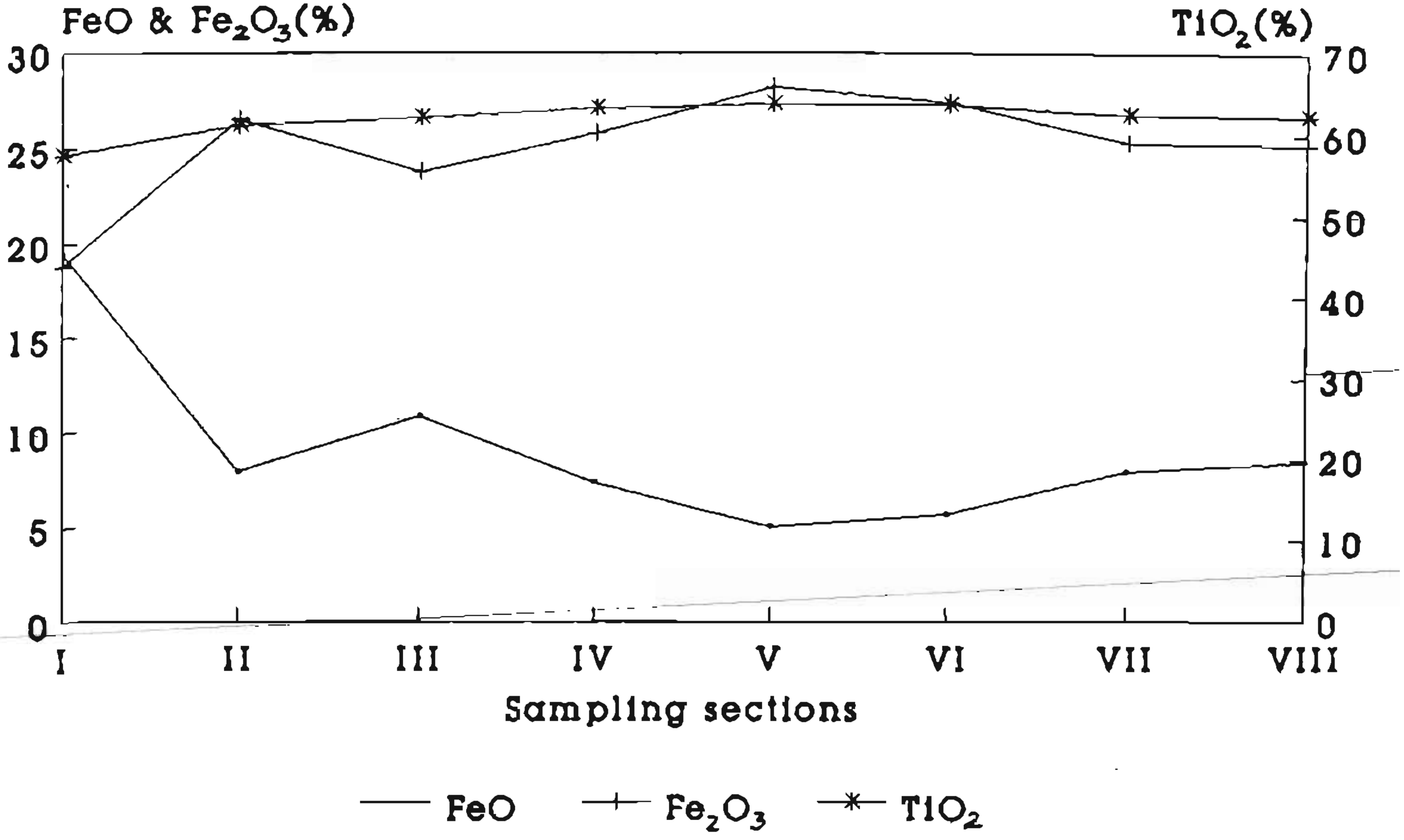


Fig.10 - Distribution of FeO, Fe_2O_3 and TiO_2 in ilmenites along eight cross-sections of Chavara deposit

southern end of the deposit is distinctly clear in Fig.9, 10 and 11. In short, the Chavara ilmenite deposit is relatively homogeneous barring the samples of Ist section.

Further, the parameters like Fe_2O_3/TiO_2, FeO/Fe_2O_3 (Morad and Aldahan, 1986) and Ti/(Ti+Fe) strongly support the earlier observations. (Fig.12 and 13). 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% 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 these four stages, ilmenite alters into either rutile (Temple, 1966) or anatase (Anand & Gilkes, 1984) and hematite. In this spectrum of ilmenite-weathering, the observations show that the samples of first section belong to hydrated ilmenite type and the rest of the samples fall within the lower limit of the pseudorutile stage in the case of Chavara deposit. This division is possible on the basis of Ti/(Ti+Fe) ratio. The Feo/Fe_2O_3 and Fe_2O_3/TiO_2 ratios indicate the degree of oxidation and iron-removal took place in the grains.

This consistent compositional behaviour indicates that the modern ploacer deposit is originated from a pile of already accumulated

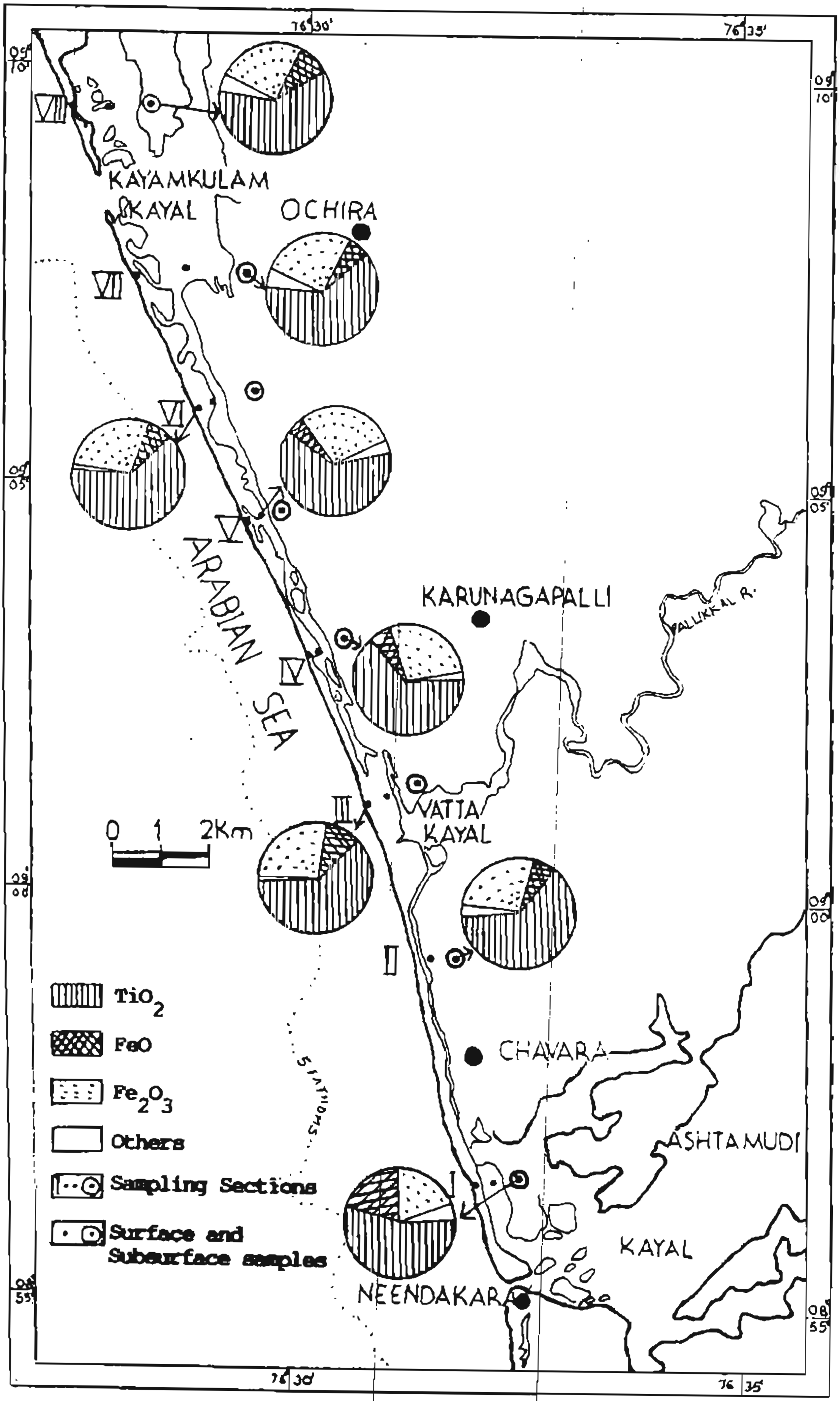


Fig.11 MAP SHOWING COMPOSITIONAL VARIATION IN ILMENITES

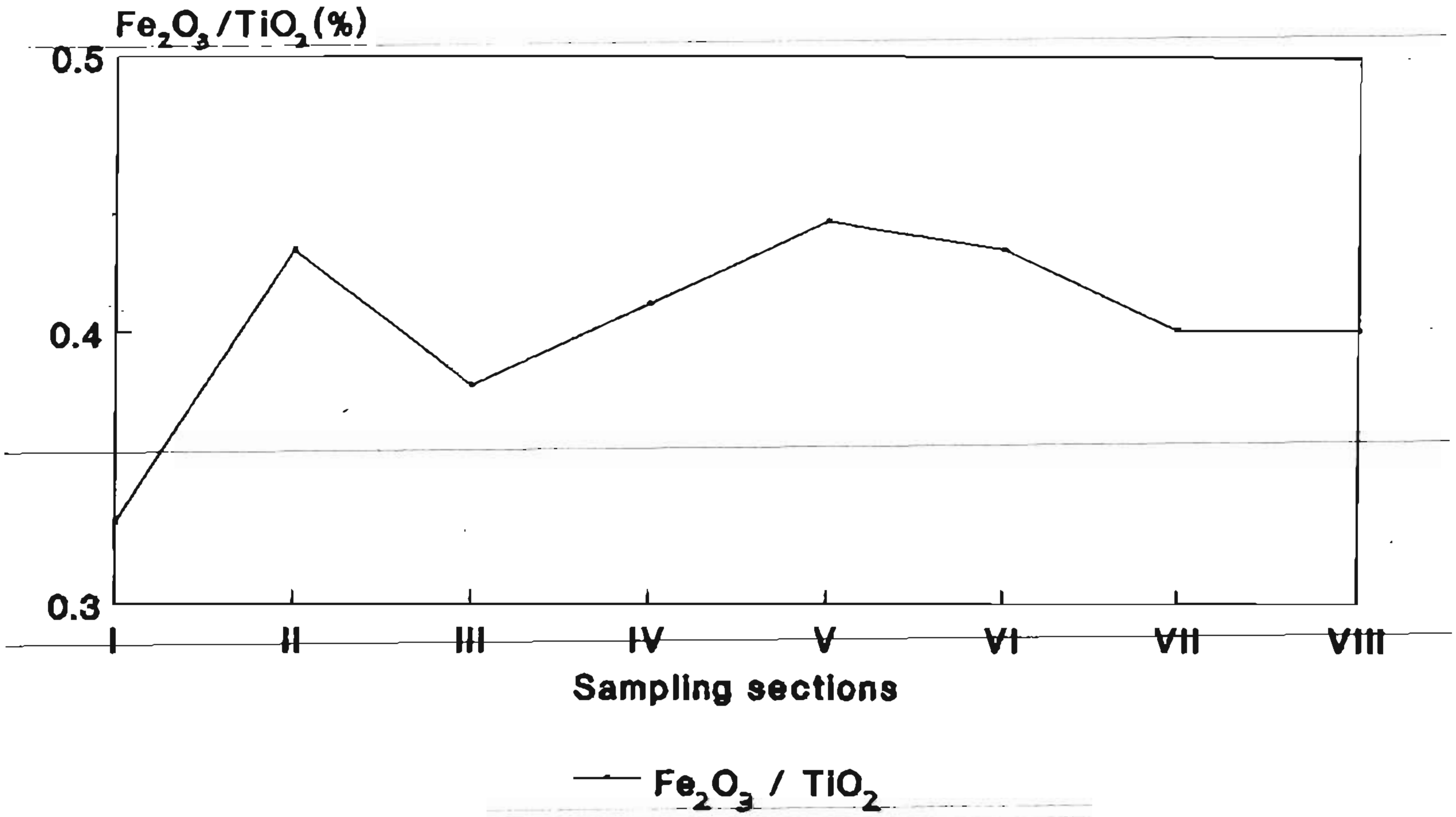


Fig.12 - Distribution of Fe_2O_3/ TiO_2 in ilmenites along eight cross-sections of Chavara deposit

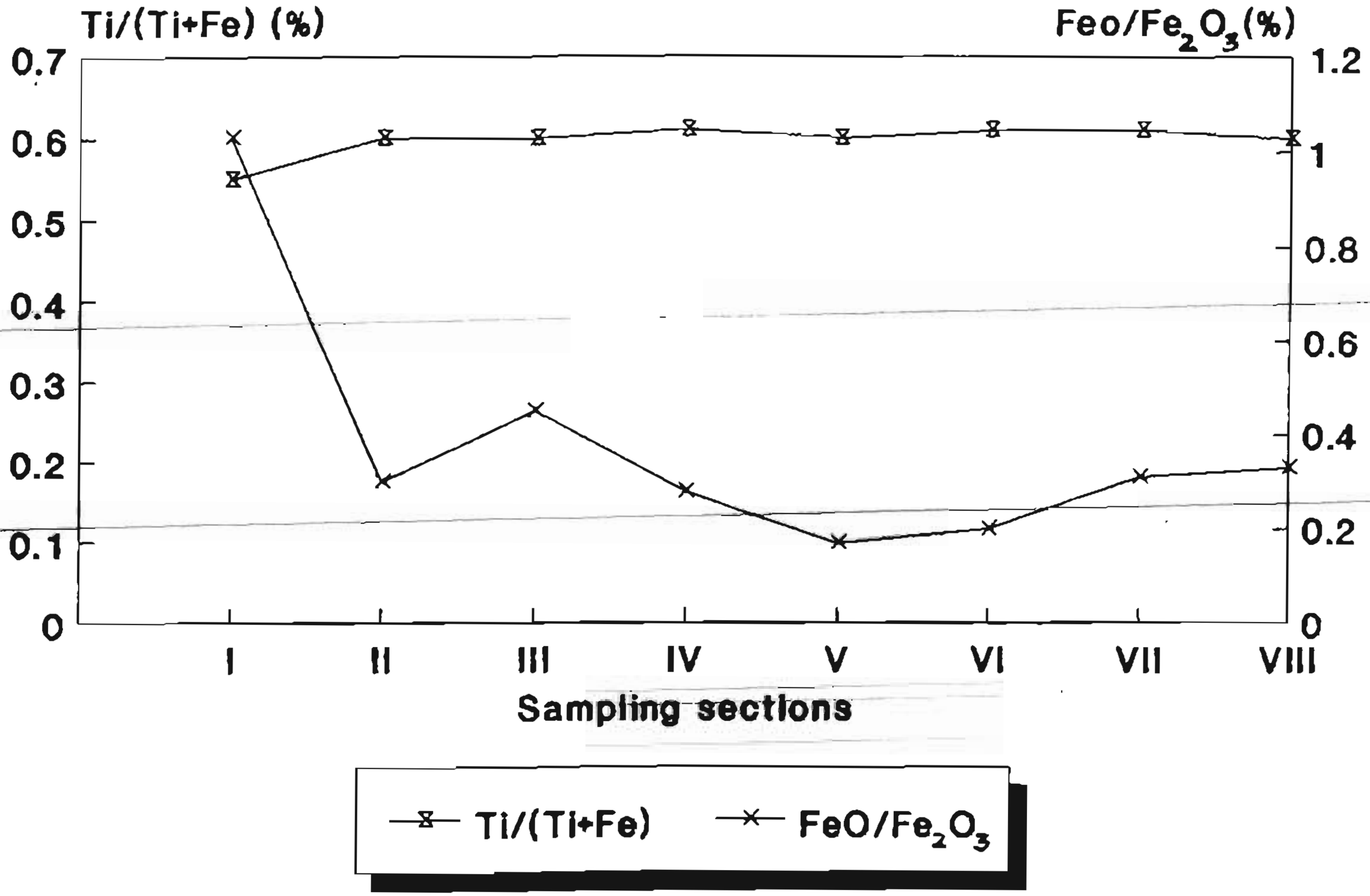


Fig.13- Ti/(Ti+Fe) and FeO/Fe$_2$O$_3$ distribution in ilmenites.

sediments and sufferred a specific degree of weathering in total to attain the pseudorutile stage of ilmenite alteration. Ilmenite is found as the major component of Chavara placers (above 65%). The slight but systematic increase in alteration towards east from the shoreline side can be compared with the successive formation of placer deposit from the shallow sea. The replenishing activity towards the coast is being carried out by waves during every monsoon even today.

Though this relatively unifrom ilmenite concentrate slowly merges in the north with the Chertala glass sands having syngenetic origin (Suresh Babu & Thrivikramaji, 1993), a sharp geochemical boundary is noticed in the southern segment as the first cross section of samples do not match with the rest. This has been explained earlier and can be connected to an accretion of second generation of heavier from the southern extremity and towards the previously formed black sand placers. The ultimate source rocks of ilmenites form the charnockite-khondalite belt of South Indian Precambrian shield and are outcropped in the south Kerala region. Therefore the compositional and quality changes noticed in the ilmenite could only be due to exogenic processes. In this background, the immediate sources for the less-altered ilmenites noticed in the southern part of Chavara deposit can be attributed to either the Quilon rock promontary or the modern sediments of River Kallada, both bordering the southern side of the deposit.

It can also be mentioned that Chavara ilmenite has suffered a significant degree of weathering. This is because of two reasons. Firstly, the carefully separated ilmenite fraction using Isodynamic separator at 0.1 to 0.3 A itself gives a pseudorutile stage to the deposit. Secondly, the available data point to an eastward progress in alteration and also a slight increasing trend in the subsurface samples. The present study has not fully covered the inland deposit and also the downward extension of the same. Similarly, the leucoxene fraction as well as the rutile fraction were not subjected for investigations. Accounting these facts, it is felt that alteration may be severe in the case of Chavara ore sands as a whole.

The trace element data (Mg, Mn, and Zn) have revealed that Mn and Zn do not vary from place to place (Table 6 and Fig.14). It may be due to the "maturity" level of leaching took place during the alteration stage. The MgO content varies between 0.38% and 0.81% and it may be either due to the differential geikelitic substitution in the primary state or due to the differential leaching took place later. However, the absolute value of these trace elements are not significant.

XRD patterns show that all the samples of ilmenite concentrates contain significant formation of pseudorutile and rutile phase except the sample of the first two traverses which from the

TABLE : 6 TRACE ELEMENT DATA OF ILMENITE SAMPLES

Sample No./ Location*	Mg %	Mn%	Zn%
1. ES-1	0.25	0.20	0.04
2. ESS-1b	0.29	0.19	0.05
3. WS-2	0.24	0.21	0.04
4. ES-3	0.34	0.22	0.05
5. BS-6	0.39	0.24	0.06
6. ESS-6b	0.49	0.27	0.05
7. ES-7	0.39	0.21	0.04
8. ES-8	0.29	0.21	0.05

*BS - Beach Sample
WS - Western Side of Kayal/Lagoon
ES - Eastern Side of Kayal/Lagoon
ESS- Eastern Subsurface Sample

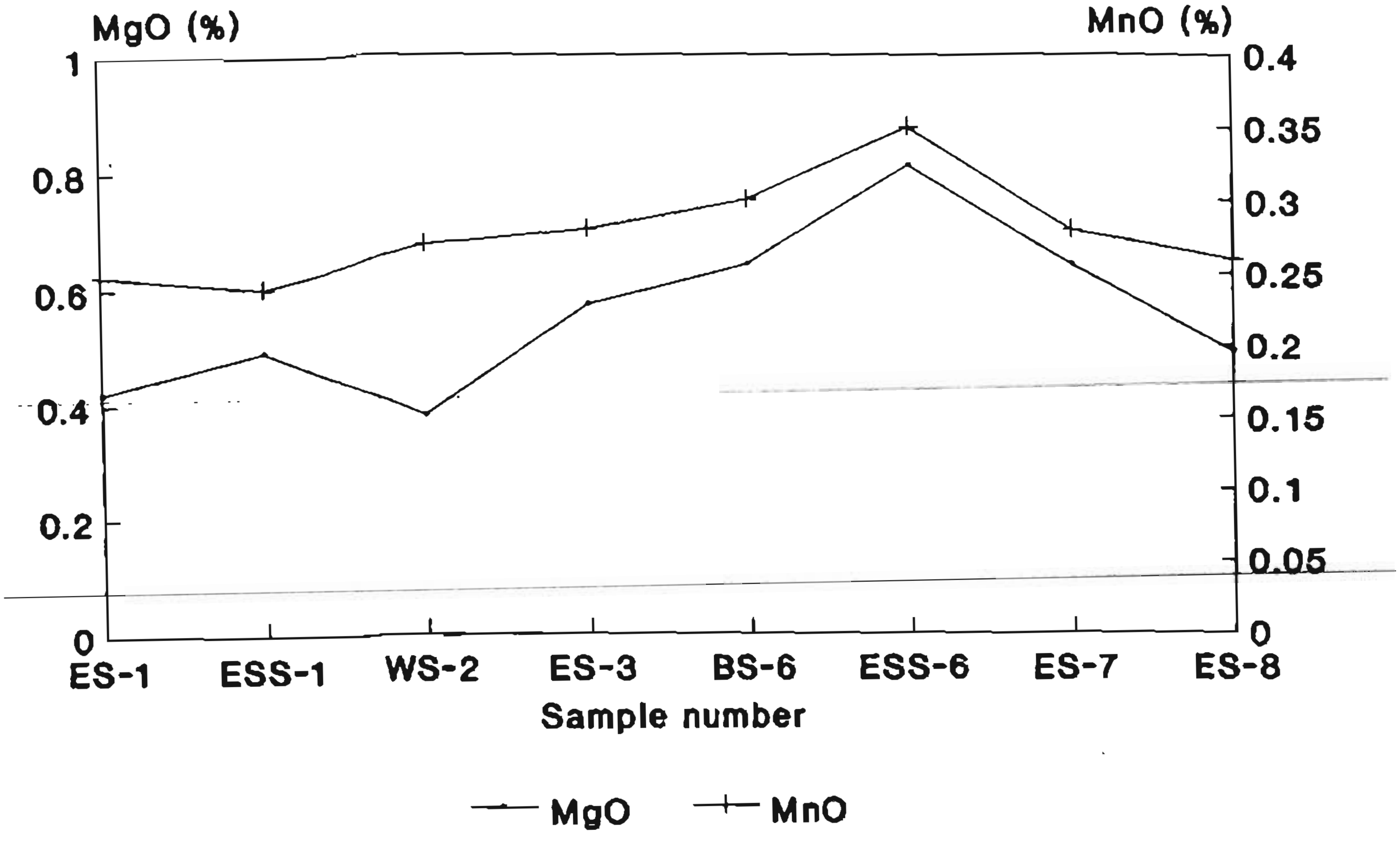


Fig.14 Distribution of MgO and MnO in ilmenites

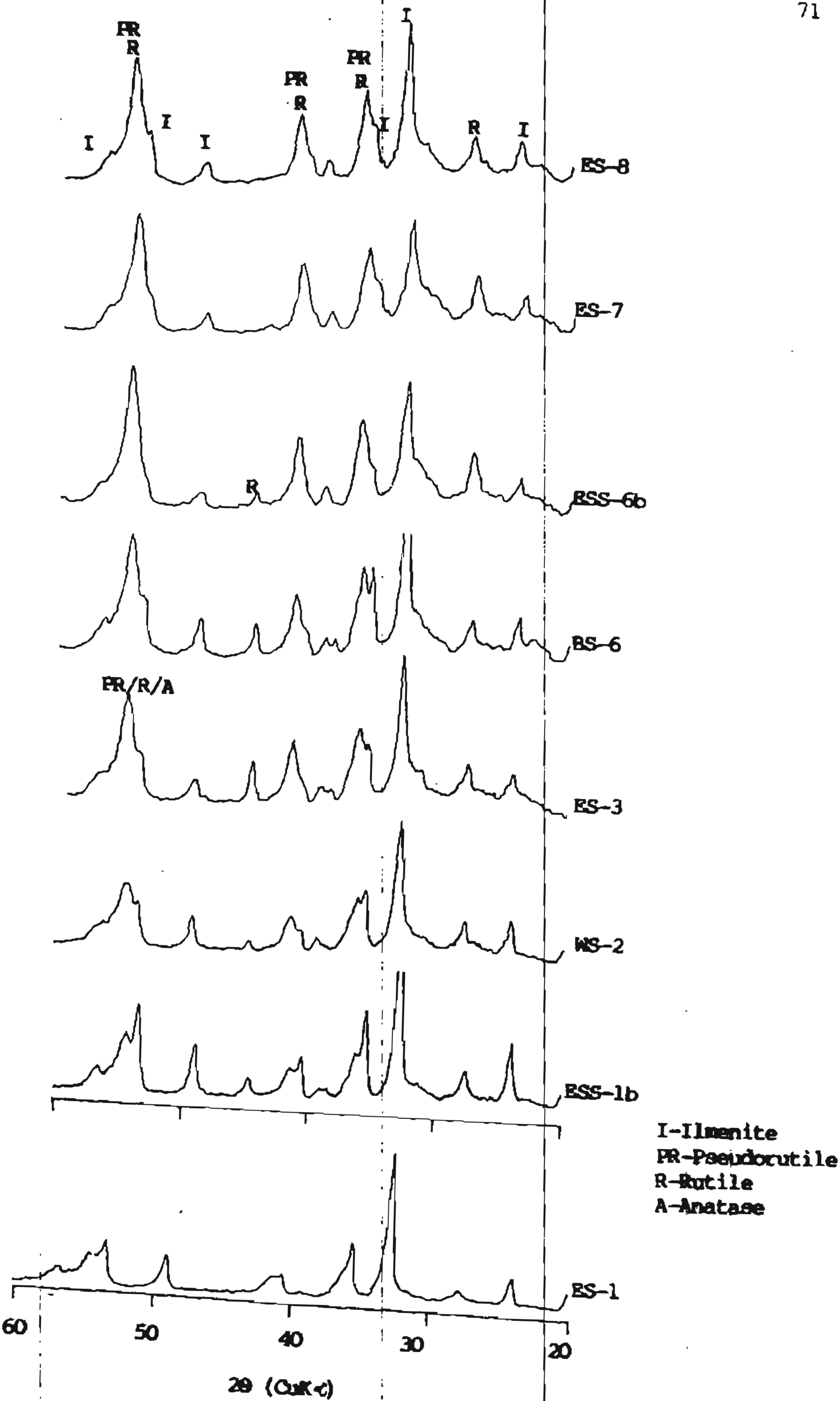


Fig.15 POWDER XRD PATTERNS OF ILMENITE SAMPLES

TABLE : 7 RESULTS OF XRD ANALYSIS

Sample No.*	Location	Inference (minerals in the order of decrease)	Remarks
ES - 1	Puthanthura	Ilmenite, Pseudorutile and rutile	Ilmenite peaks are sharp. Insignificant formation of pseudorutile and rutile.
ESS - 1b	Puthanthura	Ilmenite, rutile & pseudorutile	Ilmenite peaks are sharp. Pseudorutile/rutile peaks are prominent.
WS - 2	Sankaramangalam	Ilmenite, pseudorutile and rutile	Pseudorutile and rutile phases seems to be well developed in the ilmenite structure.
ES - 3	Ponmana	Ilmenite, pseudorutile, rutile and anatase	In addition to significant pseudorutile and rutile, anatase is noticed in negligible quantities along with ilmenite. Ilmenite peaks are broader.
BS - 6	Panakkada	Ilmenite, pseudorutile and rutile	Ilmenite phase is dominant compared to pseudorutile and rutile.
ESS - 6b	Klappana	Ilmenite, pseudorutile and rutile	Apparently ilmenite and its daughter products are equally distributed.
ES - 7	Alampidika	Pseudorutile, ilmenite and rutile	Similar to the above sample and peaks are broad.
ES - 8	Tharayilkadavu	Ilmenite, pseudorutile and rutile	Ilmenite is slightly dominating over the pseudorutile and rutile phase.

*BS - Beach Sample WS - Western Side of Kayal/Lagoon

ES - Eastern Side of Kayal/Lagoon ESS - Eastern Subsurface Sample southern portions of the Chavara deposit. The co-existence of pseudorutile and rutile phases along with ilmenite indicate that the alteration of ilmenite to its daughter products (leucoxinization) is significant in the study area. Pseudorutile/rutile peaks are broader compared to the sharper peaks of ilmenite, which shows the semicrystalline nature of the altered phase. Results of X-ray diffraction analysis are given in Table 7.

From the Fig.15 it is seen that the most developed crystallographic face of ilmenite is (104). The other prominent crystallographic phases are (116), (204), (110), (108), and (113). The pseudorutile/rutile phase is seen more conspicious in samples collected from the northern portion of the deposit than those from the southern tip. The most prominent crystallographic faces of rutile are (211), (101), (111), and (210) and that of pseudorutile are (102), (100), and (101).

XRD pattern of samples from traverse I shows that the ilmenite peaks are sharp and the insignificant formation of pseudorutile and rutile. These are well developed in the ilmenite structure of WS-2. In the sample ES-3, other than the significant formation of pseudorutile/rutile, anatase is noticed in negigible quantities along with ilmenite. Also ilmenite peaks are broader. Ilmenite is slightly dominant over the pseudorutile/rutile phase in the samples BS-6 and ES-8, whereas in samples ESS-6 and ES-7 the pseudorutile/rutile phase is more prominent than the ilmenite phase.

TGA experiments have been carried out for selected four samples for which XRD analysis were also done. The objective of thermal analysis was to understand the high temperature behaviour of differentially-weathered ilmenite samples. The four samples are ES-1, ESS-1b, ES-3 and ESS-6b (Table 4) having FeO of 20.96% 19.58%, 9.71% and 4.39% respectively. It is noted that an effective weight increase of 1.88% for ES-1 and 1.49% for ESS-1b have been noticed (Fig.16). This could be due to oxidation of substantial FeO present in the samples. While samples ES-3 and ESS-6b show an effective decrease in weight of of 0.58% and 1.17% respectively, when heated upto 1000°C. The reason for this change is possibly due to the following factors. First, the initial ferrous 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. But, for all samples the structural water (hydroxyls) represents the major weight loss component and it increases with increase in ferric iron content. Substantial ferric iron content in ilmenite can be related to the formation of pseudorutile phase. It has been reported that pseudorutile can be compared to FeOOH - TiO_2 mixtures Grey and Ried (1972). Among the four samples, ESS-6b has the maximum content of Fe_2O_3 (28.29%), which, in turn, must be containing more hydroxyl molecules. However, the bound water release ceases at temperatures below 500°C and then attains weight gain due to FeO-Fe_2O_3 conversion.

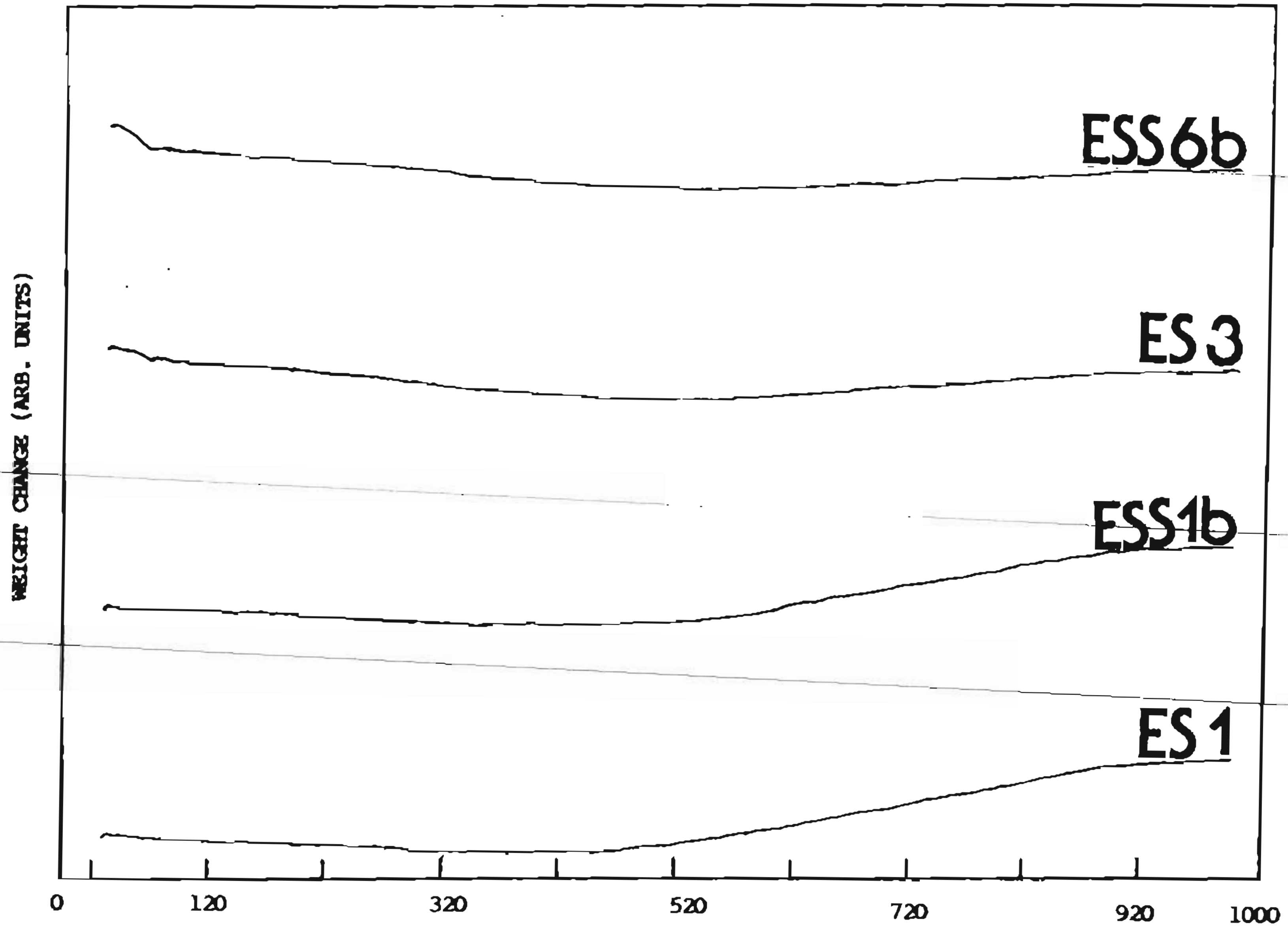


FIG.16 TGA PATTERNS OF ILMENITE SAMPLES

The release of these hydroxyl molecules explains the initial weight loss in sample ESS-6b. From the TGA data, it is concluded that the amount of FeO content left out in the ilmenite grains is crucial for the high temperature behaviour of iron-titanium oxides.

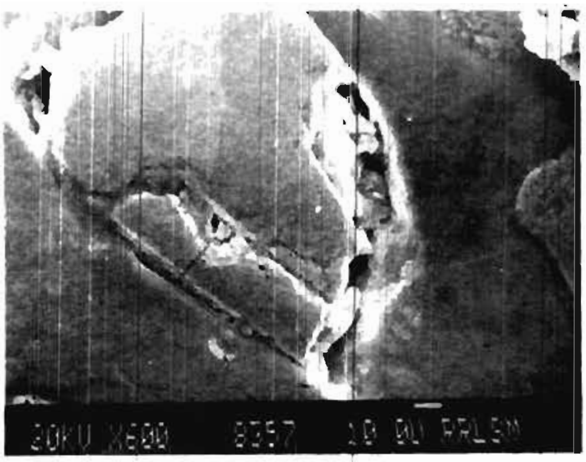
SEM observations have revealed the nature of alteration in ilmenites (Plates III & IV). Alteration seems to be initiated through microfractures as well as from the periphery.

PLATE - III

A Development of microfractures and initiation of ilmenite-alteration.

B Secondary mineral formation at the altered zone.

PLATE III



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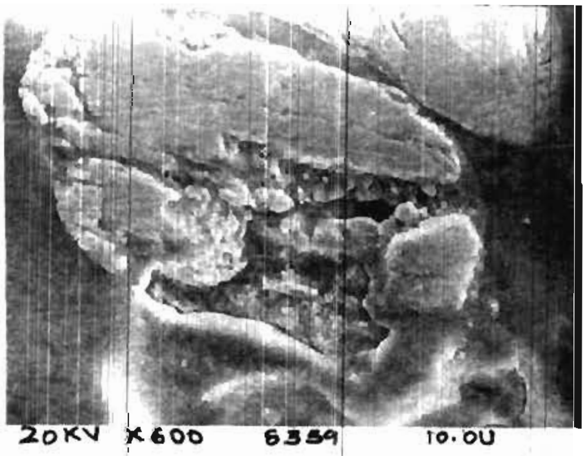
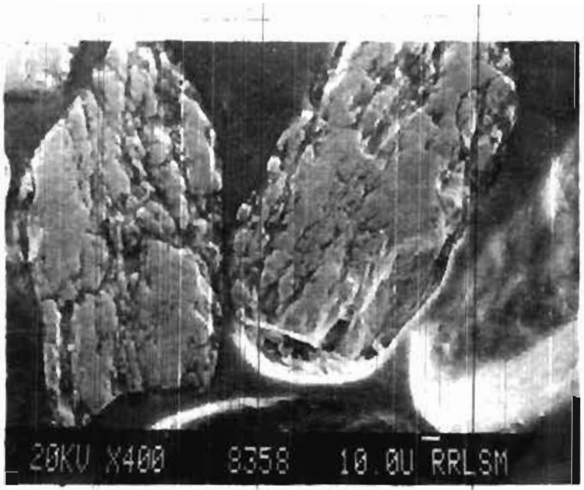


B

PLATE - IV

A **Differential relief on ilmenite grains due to surficial changes.**

PLATE IV



A

SUMMARY AND RECOMMENDATIONS

SUMMARY

Mineralogical, compositional and structural evaluation of Chavara ilmenite reveals that there is a marked geochemical boundary in the southern portion of the 22 km long Chavara deposit. The ilmenite alteration progressively increases from the western margin of the deposit towards east. Detailed characterisation of the representative samples collected from 37 stations show that Chavara ilmenite has, in general, altered to a stage bordering between hydrated ilmenite and pseudorutile of Frost et. al., (1983). The uniform compositional behaviour postulates that the Chavara deposit has originated from a pile of already accumulated sediments and suffered a specific degree of weathering in total.

RECOMMENDATIONS

In order to further understand the weathering effects on ilmenite of the area, it is necessary to study the leucoxene and rutile fractions also. Though ilmenite is a petrogenic placer mineral, it would be interesting to investigate its provenance characteristics in the subsequent research work. There is a view that source material of the present day Chavara deposit has been derived not only from the eastern side of the modern shore line but also from the western side. An attempt should be made to throw some light upon this aspect in future studies. The relationship between weathering in ilmenite and beneficiation processes is also worth to study.

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