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Morphological and Mineralogical Characterization of Coastal Soil Samples of Kanyakumari District by FT-IR, XRD, SEM/EDAX

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Abstract
Qualitative analysis was carried out to determine the major and minor constituent minerals present in coastal soil samples collected from west cost of Kanyakumari District, Tamilnadu by FT-IR, XRD and SEM/EDAX techniques. From the IR absorption band and the locations of the different peaks, the minerals were identified with the available literature. In addition to the band positions, the sharpness of bands was helpful in the identification of mineral components. Identification and estimation of mineralogical composition were carried out by XRD. XRD technique to yield more information about the minerals. FT-IR and XRD methods were non destructive and can be used in the identification of mineralogical composition. SEM analysis showed the coastal soil samples have different morphological structures like spherical (Al-Si-O), tubular (Si-O), triangular (Fe-Al-Si-O), platy (Ca-Si-O), nearly spherical (Fe-Ti-O) and irregular (Ca-Al-Si-O) shapes. Elemental analysis (EDAX) confirmed the presence of silicon in large quantities than the other oxides such as Al, Ca, Fe and Ti.

Keywords: Mineralogical composition, FT-IR, XRD, SEM and EDAX techniques.

1. Introduction

Kanyakumari is situated in the southernmost district of Tamilnadu. The south eastern boundary is the Gulf of manner. On the south and the southwest, the boundaries are the Indian ocean and the Arabian sea. Costal soil is one of the most important resource of the nature. Soil formation is a constructive as well as destructive process [1]. Soil has a complex function which is beneficial to human and other living organism [2]. The climate and other factors largely affect the costal soil formation. The mineral composition of soil, the organic matter within it and the environment all are determined by the chemical properties of soil [3]. Understanding of soil chemical reaction and process is essential for developing innovative resource management strategies and regulating the behaviour of the terrestrial ecosystem at regional and global scale [4].

The mineral analysis gives prompt area of research and gives an important tool in economic scale also. There are number of methods are used to identify the minerals by the conventional techniques such thin section analysis, X-ray diffraction (XRD) and FT-IR spectroscopy. Many workers applied the FT-IR spectroscopy for the mineral identification [5-11]. Infrared spectroscopy as an analytical tool presents a lot of advantages as it is accurate, inexpensive and reliable, the necessary sample amount is in the sub milligram range and sampling procedure is simple. As a major advantage, infrared spectroscopy permits the identification of the alternative method for acquiring quantitative mineralogy [5].

XRD method is the best one for mineral analysis as it is rapid, cheap, time saving and nondestructive. X-ray diffraction pattern gives more information about the minerals present in soil or

sedimentary samples [12][13]. SEM is highly qualified for the identification and the quantification of different elements in various samples of geological, biological and environmental importance [14][15][16]. EDAX was performed to know the chemical composition of the minerals present in the soil.

2. Materials And Methods

2.1 Study Area

Kanyakumari District is situated at the extreme south of the Indian subcontinent; the coast line is formed nearly by three seas namely Arabian Sea, Indian Ocean and Bay of Bengal. But the main part of coast faces the Arabian Sea. The study area chosen for the present work is Kanyakumari District (Fig.1) which is covering a distance of 75km from Vattakottai to Vallavilai of Tamilnadu coast. These places are catching center of fishery activities in Tamilnadu and also plays a major role in major commodities. Numerous industries, chemical factories and aquaculture farms are also developing along these coastal areas and it makes much attention for present and future research.

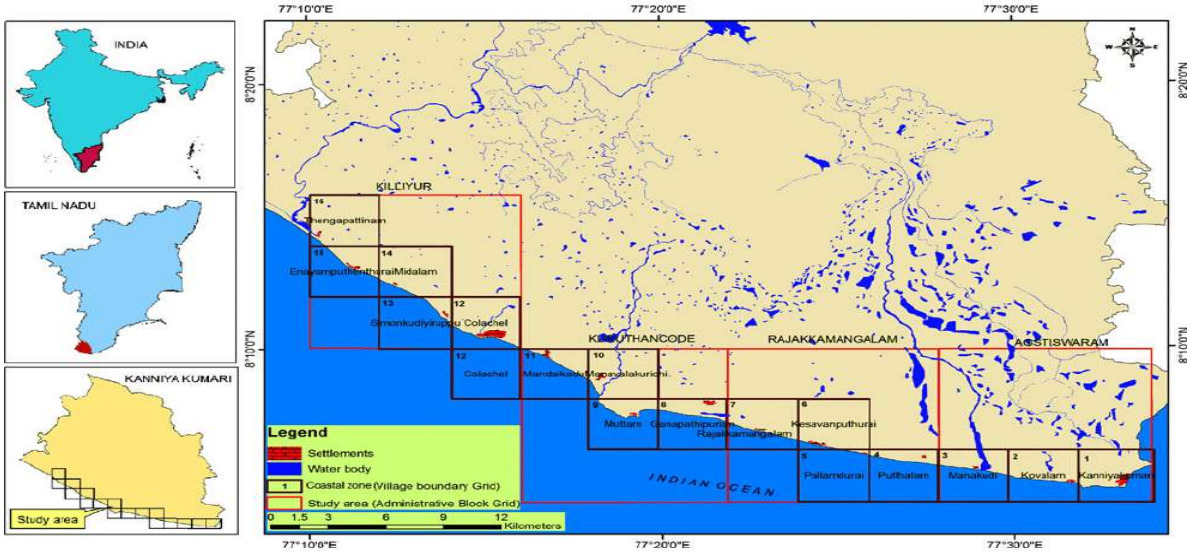


Fig .1 Study area Location

The present work encompasses the mineralogical findings of coastal soil samples through FT-IR, XRD, SEM and EDAX techniques.

2.2. Soil Samples Collection And Preparation

Coastal soil samples were collected using Peterson grab at all the designated locations during low tide. The samples collected from ten different sites under study were labeled as K1, K2, K3, K4, K5, K6, K7, K8, K9 and K10.The distance between each site falls around 5kms.

The collected soil samples were initially sundried for seven days followed by drying in hot air oven at 383 +- 1K for two days. The dried soil was crushed, sieved and stored in sterile closed glass bottles till further investigation [17].

2.3 mineralogical and chemical analyses

The infrared spectra were recorded in the mid IR region 400-4000 cm^{-1} using Shimadzu Fourier Transform Infrared Spectrometer (IR-Affinity-1). The KBr pressed pellet was used to record the spectrum. The crushed samples were grounded before making the KBr pellet. The samples were mixed with KBr in the proportion of 1:20 and pressed to 5 tons for one minute in preparing the disc. To identify the mineral phases in the samples, X-ray diffractograms for the shreds in the powdered form recorded using Schimadzu XRD 6000 25^{o}c of y = 1.5405 A^{o}. Microstructures were examined by Scanning Electron Microscope (SEM) with JEOL JSM 6390 model. The chemical composition was determined by an Energy Dispersive X-ray Spectroscopy (EDAX) attached to SEM.

3. Results and Discussion

3.1. Characterization of Soil Particle

For understanding the nature of coastal soil samples, FT-IR, XRD, SEM and EDAX were performed, which are described in the following sections.

3.2. FT-IR Characterization

The FT-IR spectrum was used to determine the nature of functional groups which could possibly influence the adsorption of the soil. The FT-IR spectrum of the soil is shown in Fig.2 and FT-IR observed frequencies (cm^{-1}) of coastal soil samples of Kanyakumari District with mineral identification are reported in Table 1.

By comparing the observed frequencies with available literature [11, 18-24], the minerals such as quartz, microcline, orthoclase, albite, kaolinite, montmorlinite, calcite and aragonite have been identified. The mineral wise discussion is outlined is given below.

Quartz

The silicate mineral quartz is the most abundant mineral available in the coastal soil samples of Kanyakumari district. The presence of IR absorption bands at 695-700, 795-810, 1050-1150, 1600-1800 cm^{-1} indicates quartz in all samples. It is included in Table 1. The symmetrical bending vibration occurs at a frequency 695 cm^{-1} in the samples K1, K3, K5 and K6. The symmetrical stretching vibration occurs at a frequency 775 cm^{-1} in the samples K3, K4 and K8. The symmetrical stretching vibration occurs at a frequency 795 cm^{-1} in the samples K1, K7 and K10.

Location	Sample ID	Silicate Minerals	Field spar			Clay Minerals		Carbonate Minerals	
		Quartz	Microcline	Ortho clase	Albite	Kaolinite	Monte morlinite	Calcite	Aragonite
Vattakottai	K1	695, 700, 795, 800, 802	742, 1050	765	785	–	880	875, 1455	–
Chothavilai	K2	702, 797, 1087, 1092	1065	765	720, 787	–	–	1488	855
Rajakkamangalam	K3	695, 775, 1092, 1125	742	765	787	1016, 1030, 1116	–	878	1480, 1478
Muttam	K4	775, 778, 795	1050	–	785, 790	1115, 1016	–	–	1480, 1478
Manavalakurichi	K5	695, 700, 780	740, 742, 1051, 1055	1040	–	–	880,1640	875, 880, 1795	–
Kottilpaadu	K6	695, 700, 975	1051, 1120	765	785, 991	985, 1016, 1080	–	875, 1420	–
Kurumpanai	K7	698, 700, 780, 825, 795, 1622	740	–	1065	–	880,1640	1420, 1792, 1795	–
Pattanam	K8	775, 797, 1080	–	650, 765	720, 785, 990	935, 1080, 1115	–	1420	–
Thoothoor	K9	700, 780, 1080, 1622	742, 1050	765, 1040	–	–	–	1420	–
Vallavilai	K10	702, 780, 795, 1616	742, 1059	765, 1040	–	1016,1119	–	–	1478

Feldspar

Feldspar is an abundant of rock minerals, which constitute 60% of the earth's crust. They are also found in many types of sedimentary rocks. Feldspar weather to yield a large part of clay found in soils

1. **Microcline**

The microcline mineral is available in all the samples except sample site no K8. The presence of microcline is indicated by the peaks at a frequency of ranges 730-740, 1000-1050, 1060-1065, 1100-1150 and 1080-1085 cm^{-1}.

2. **Orthoclase**

Orthoclase mineral is unavailable in the samples K4 and K7. The presence of orthoclase is indicated by the peaks obtained at 765,780, 1040, 1065 cm^{-1}. The asymmetrical stretching vibration occurs at a frequency 1040 cm^{-1} in the samples K5, K9 and K10.

3. **Albite**

The mineral element albite is not available in the samples K5, K9 and K10. The presence of albite is indicated by the peaks obtained at 765, 785, 869 and 1065 cm^{-1}.

Table 1.

FT-IR observed frequencies (cm^{-1}) of coastal soil samples of Kanyakumari District with mineral identifications

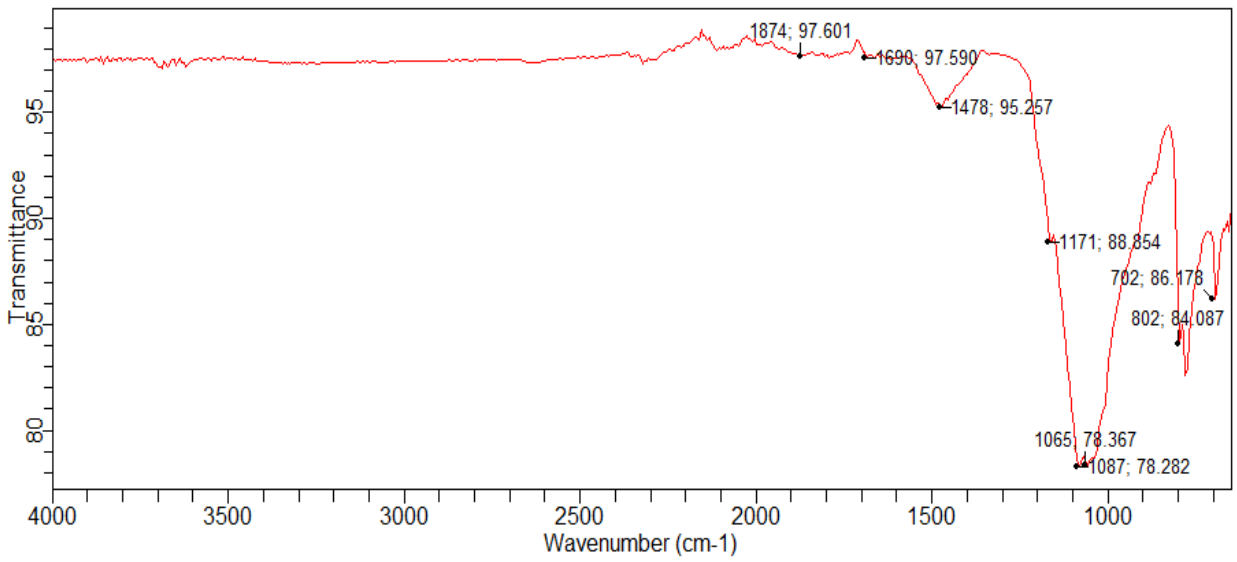


Fig.2

FT-IR spectrum of coastal soil sample of Kanyakumari district

Clay minerals

i) Kaolinite

The presence of kaolinite is indicated by the peak obtained at ranges from 905-910, 950 - 955, 1010 - 1015, 1030 - 1035, 1115 - 1120 and 1150 - 1155 cm^{-1}. Out of all the samples, kaolinite presence is identified only in the samples K3, K4, K6, K8 and K10. The O-H deformation occurs at a frequency 935 Cm^{-1} in sample site K8. The Si-O stretching at 1030 cm^{-1} is present in the sample site no K3.

ii) Montmorillonite

Montmorillonite contains the peak like 800 - 805, 860 - 865, 895 - 900 and 1650 - 1655 cm^{-1} which is shown in the spectrum Fig 2. It contains the samples sites K1, K5 and K7 respectively.

Carbonate minerals

The IR absorption bands at 875 - 880, 1420 - 1425& 1795 - 1800 cm^{-1} are found to be calcite. Aragonite is identified in the samples by the IR absorption peaks at 1785 - 1790, 855 - 860 cm^{-1}.

i) Calcite

The calcite mineral is available in all the samples sites except sample sites K4 and K10. The frequency range from 875 - 880, 1445 - 1455, and 1480 - 1490 and 1790 - 1795 cm^{-1} shows the presence of calcite. The frequency of 875 cm^{-1} seen in sample K1, K5 and K6 shows the presence of Fe^{3+} (Al - OH) reported in Table 2.

ii) Aragonite

The sample K2 contains the aragonite mineral in the peak at 855 cm^{-1} which is shown in the spectrum Fig 2 and in the Table 2 respectively. The samples K3 and K10 contain the same peak at 1478 cm^{-1}, 1480 cm^{-1}. The sample K4 contains the aragonite mineral in the peak at 1790cm^{-1}.

Organic Carbon

From the spectra of all the samples in Table 1 a very weak absorption band present at 2140-2150 cm^{-1} may suggest the presence of organic carbon. These bands are due to C-H absorption of contaminants present in the samples.

Table 2.

Band assignments of different minerals of coastal soil samples of Kanyakumari district

Sample site number	Minerals	Frequency Cm^{-1}	Tentative assignments
K1, K3, K5, K6 K3, K4 K8, K1, K7, K10	Quartz	695 775 795	Si-O symmetrical bending vibration [11] Si-O symmetrical stretching vibration Si-O symmetrical stretching vibration [5] [6]
K5, K9, K10 -K6	feldspar	1040 1120	Si-O asymmetrical stretching vibration Si-O asymmetrical stretching vibration [25]
K3, K8	kaolinite	1030 935	Si-O stretching [11] O-H deformation [26]
K1, K5	Calcite	875	Fe^{3+}(Al-OH) [26].

There is maximum of six to eight peaks are observed in all samples. The characteristic feature of quartz is doublet appearing at or around 800 cm^{-1} and 780cm^{-1}. Such a clear observation of doublet was noticed in the sample sites of Vallavilai, Pattanam, Kotilpadu and Manavalakurichi. The peak appearing at 695 cm^{-1} is most useful to determine nature of the mineral with regard to the structural stability. Many workers have calculated the crystallinity index of quartz using the symmetrical bending vibration of Si-O group obtained at 695 cm^{-1}. As 695 $cm^{-1}$is present in the spectra of all the samples indicate the quartz mineral in these samples are well in crystalline form. The presence of kaolinite, illite and montmorlinte indicate clay minerals in samples. Absorbance at 1030 $cm^{-1}$is attributed to Si-O stretching of clay mineral like Kaolinite. The FTIR absorption bands at 875-880 cm^{-1}shows the presence of montmorilinite.

3.2. X -Ray Diffraction (XRD)

Qualitative mineralogy of the coastal soil samples was determined with the standard interpretation procedures of XRD. Quartz, albite, orthoclase, kaolinite, calcite, aragonite were identified from the peaks in

diffractrogram it shows in Fig 3. Major minerals in the samples are quartz, feldspar and calcite. The observed XRD patterns indicate quartz, feldspar, calcite, Kaolinite as the major constituents and other minerals as the minor constituents in coastal soil samples of Kanyakumari district. The d-values of XRD pattern of soil were estimated, compared with standard values of clay minerals supplied by crystallographic and crystallochemical Data base for minerals and their structural. Comparing the d-values it can be concluded that the soil is mainly quartz (silicon dioxide), microcline, orthoclase, albite, kaolinite, calcite and argonite.

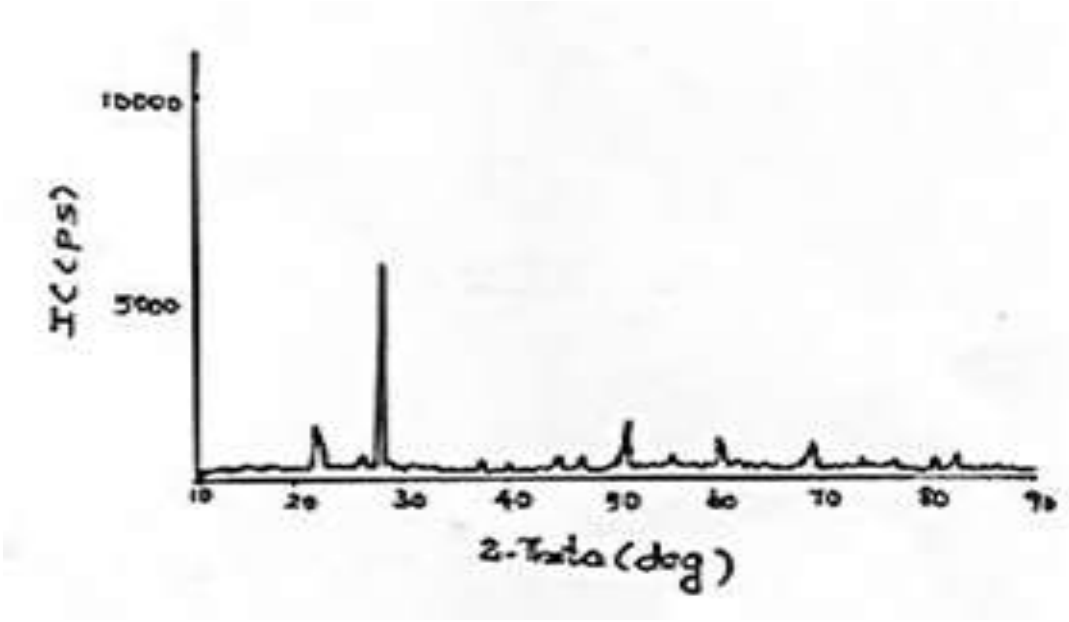


Fig.3 XRD image of coastal soil sample

3.2.1. Mineral wise analysis

Qualitative mineralogy of the coastal soil samples of Kanyakumari district is determined with the standard interpretation procedures of XRD. The mineral wise discussion is as follows

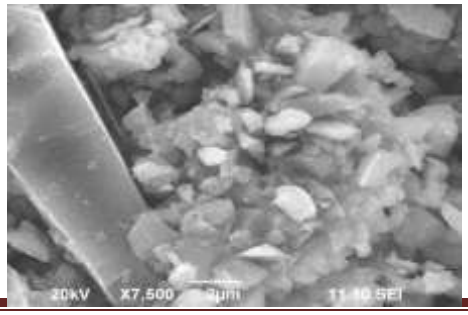
Quartz : Quartz is ubiquitous and forms one of the most abundant mineral in all the samples.It is identified by the distinctive reflection at 4.254 A^o, 3.343 A^o, 2.456A^o, 1.816A^o, 1.541 A^o and 1.374 A^o.

Feldspar : The feldspar mineral is invariably present in all samples. Albite is identified by bascal reflection at 3.196 A^o, 3.663A^o, 3.310 A^o and 2.571 A^o indicated the presence of orthoclase.

Calcite : It is identified distinct reflections in the d-spacing of 3.035A^o, 2.285 A^o, and 1.875 A^o.

3.3. Sem Analysis

Scanning Electron Microscopy gives an insight of morphological analysis of coastal soil samples.SEM picture of coastal soil sample was taken at 20 KV with different magnification and presented in Fig 4. It depicts the tubular, spherical, Platy shape, triangular, rectangular, nearly triangular and sun flower like appearance of the soil samples.



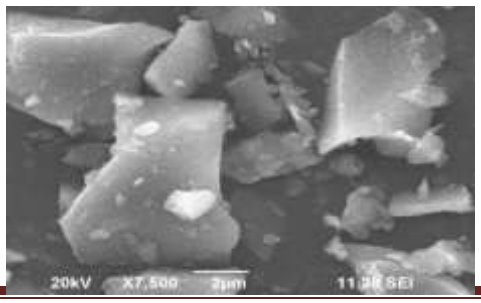
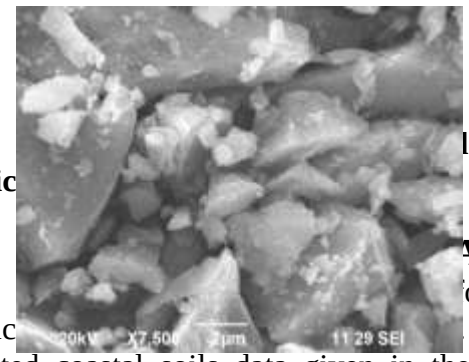
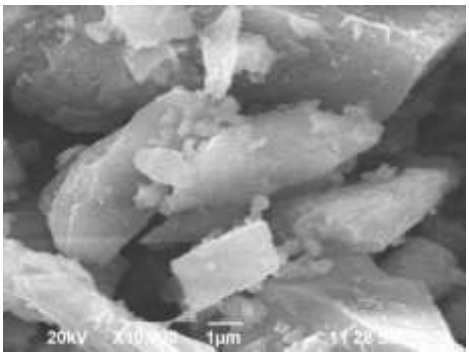


Fig 4.  **les of**  **Kanyakumari Distric**

3.4. **Analysis (EDAX)**

ormed to know the chemic . In this present study collected coastal soils data given in the table 3. From the EDAX show the different elemental composition of coastal soils in the Kanyakumari district.

From the elemental composition and EDAX spectrum represented the sample site K9 present maximum number of elements. Fig 5. and Table 3 clearly represented the results. The sample site K9 is Thoothoor coastal area. In sample site K2 contains only silicon, calcium and oxygen. The sample site K2 is Chothavilai coastal area. The strong peaks observed in the spectrum are related to silicon, oxygen and calcium. SEM and EDAX confirmation of coastal soil samples are shown in Table 4.

Table 3. EDAX analysis of soil

Elements	***Weight %***	***Atomic %***
O	*54.55*	*69.28*
Al	*4.52*	*3.41*
Si	*32.20*	*23.30*
Ca	*4.87*	*2.47*
Ti	*2.23*	*0.95*
Fe	*1.62*	*0.59*

Fig 5. EDAX spectrum of soil

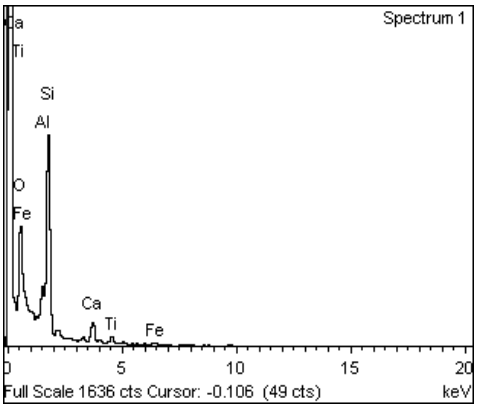


Table 4 . SEM and EDAX confirmation of coastal soil samples of Kanyakumari District

Geogenic particles	Sub groups	Possible phase/ Minerals	Morphology of particles and ample site number	Element composition of each sub type (wt%)
Quartz/Silica	Si-O	Sandy	Tubular shape (k10)	Si 40.75%,O 50%,Al 3.37%,Fe 3.98%,Ti 1.91%
Quartz/Silica	Al-Si-O	Fly ash	Spherical shape (K9)	Si 32.20%,O 54.55%,Al 4.52%,Fe 1.62%,Ti 2.23%Ca 4.87 %
Quartz/Silica	Ca-Al-Si-O	Grossular	Irregular shape(k8)	Si 36.11%,O 57.28%,Al 2.45%, Ca 4.17%
Quartz/Silica	Fe-Al-Si-O	Alamandine	Triangular (k9)	Si 32.20%,O 54.55%,Al 4.52%,Fe 1.62%,Ti 2.23%Ca 4.87 %

Alumino silicates	Ca-Al-Si-O	Ca-feldspar	Irregular shape(k8)	Si 36.11%,O 57.28%,Al 2.45%, Ca 4.17%
Calcium rich particles	Ca-Si-O	Wollasonite	Platy shape(k2)	Si11.09%,O 67.70%, Ca 21.22 %
Fe\Ti oxide	Fe-Ti-O	Iron Titanium oxide	Nearly spherical (k9 and k10)	Si 40.75%,O 50%, Al 3.37%,Fe 3.98%,Ti 1.91%K9, (K10)-Si 32.20%,O 54.55%,Al 4.52%,Fe 1.62%,Ti 2.23%Ca 4.87 %

4. Conclusion

Environmental pollution is an undesirable change in air water and land. Pollution of soil environment like coastal area due to industrial wastes and other wastes. It is an important problem faced by the developed as well as developing countries. The morphology of a beach is mainly controlled by wave, climate, tide and sediment characteristics.

The qualitative identification of minerals in coastal soil samples of west coast of Kanyakumari district was carried out by FT-IR and XRD techniques. The IR analyses of coastal soil samples from different locations indicate the presence of quartz, microcline, orthoclase, albite, kaolinite montmorillionite and calcite. Among the different minerals quartz is present invariably in all the samples. Feldspar is also present in different composition like microcline, orthoclase and albite. Clay minerals also present in the form of kaolinite and montmorillionite. Presence of calcite and aragonite conclude the soil samples contain carbonate minerals. IR concludes that all the samples have least amount of organic carbon. Mineralogical composition of the soil sample provides a fundamental step in gaining knowledge about the minerals in Kanyakumari district.

The XRD analysis indicate quartz, feldspar, calcite, Kaolinite as the major constituents in coastal soil samples of Kanyakumari district. The EDAX spectrum shows the presence of dominant elements such as silicon, oxygen and calcium.

SEM picture of coastal soil sample was taken at 20 Kv with different magnification and it depicts the tubular, spherical, Platy shape, triangular, rectangular, nearly triangular and sun flower like appearance of the soil samples. SEM and EDAX confirmed the possible phase, morphology of particles and elemental composition of each samples in Kanyakumari district.

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