INDIAN JOURNAL OF SCIENCE AND TECHNOLOGY

RESEARCH ARTICLE

FT-IR, XRD, EDX-SEM and Radiological Fingerprints of Sedimentary Rocks of Bodamalai Hills in Tamil Nadu, India

K Surendar[1,2]*, P Rajkumar[3], V Balachandran[1], B Arun[4]

1 Department of Physics, Arignar Anna Government Arts College (Affiliated to Bharathidasan University), Musiri, Tiruchirappalli, 621211, Tamil Nadu, India
2 Department of Physics, Urumu Dhanalakshmi College, (Affiliated to Bharathidasan University), Tiruchirappalli, 620019, Tamil Nadu, India
3 Thiruvalluvar Government Arts College, (Affiliated to Periyar University), Rasipuram, Namakkal, 637401, Tamil Nadu, India
4 Radiation Application and Technology Division, Safety, Quality and Resource Management Group, Indra Gandhi Centre for Atomic Research, Chennai, 603102, Tamil Nadu, India

OPEN ACCESS

Received: 28-08-2024
Accepted: 13-12-2024
Published: 11-01-2025

Citation: Surendar K, Rajkumar P, Balachandran V, Arun B (2025) FT-IR, XRD, EDX-SEM and Radiological Fingerprints of Sedimentary Rocks of Bodamalai Hills in Tamil Nadu, India. Indian Journal of Science and Technology 18(1): 59-71. https://doi.org/10.17485/IJST/v18i1.2783

* **Corresponding author**.

surenkphy@gmail.com

Funding: None

Competing Interests: None

Published By Indian Society for Education and Environment (iSee)

ISSN
Print: 0974-6846
Electronic: 0974-5645

Abstract

Objectives: To elucidate the minerals and elements available in sedimentary rocks of Bodamalai hills which is located in Namakkal district, Tamil Nadu state, India. To examine the radiological fingerprints of the rock samples. **Methods:** Fourier Transform Infra-Red (FT-IR) Spectroscopy method was carried out to identify the minerals, X-Ray diffraction (XRD) techniques were used to confirm the presence of minerals and identify the new minerals, and to reveal the chemical composition and morphological studies as well. The Scanning Electron Microscope along with Electron Dispersive X-ray Analysis (SEM-EDX) utilized to detect the presence of elements along with their percentage of weight. The concentration of radionuclides like ^{40}K, ^{232}Th and ^{238}U was determined using High Purity Germanium (HPGe) detector and their amount expressed in Bq kg^{-1}. **Findings:** The minerals Quartz, Calcite, Cristobalite, Gibbsite, Goethite, Halloysite, Hectorite, Illite, Kaolinite, Lepidocrocite, Microcline Feldspar, Montmorillonite, Nacrite, Organic Compound, Palygorskite, Sepiolite, and Siderite are identified through FT-IR and XRD. The nature of the crystanillity of quartz was not uniform from top to bottom, it was randomly present in elevation wise with a range from 1.07 to 1.44. The elements Carbon, Oxygen, Sodium, Magnesium, Aluminium, Silicon, Potassium, Calcium, Iron and Copper found their percentage of weight with atomic percentage. Silicon and Oxygen were dominant elements in rock material. The amount of radiation exposure of radioactive elements ^{40}K, ^{232}Th and ^{238}U was found. **Novelty:** The availability of minerals and elements was identified in Bodamalai Hills. The metals Al, Fe, Cu, Mg present in this rock were found and their percentage of availability is Al-5.46%, Fe-1.54%, Cu-0.14% and Mg-0.18%. The radiological results indicate that the radioactivity shows only in ^{40}K (58.8$\pm$21.1 Bq kg^{-1} to 264.1$\pm$26.2 Bq kg^{-1}) other elements ^{232}Th, ^{238}U were shown below the detection limit (less than 1 Bq kg^{-1}).

Keywords: FTIR; XRD; EDX with SEM; Crystallinity Index; Radiological Fingerprint

1 Introduction

The Rocks are the natural sources of most minerals. The bunch amount of minerals and elements plays a vital role in construction materials, industrial and technology applications [1–3]. Some phosphate rocks minerals were used in necessary nutriments for the growth of cultivated plants, and some important uses in the production of chemicals, disinfectants, biomaterial applications, and different food products as well [4]. The spectroscopic studies will give chemical and morphological information about the minerals present in the hills [5]. A physic-chemical and geomechanical investigations were used in petroleum, mining and civil engineering applications [6]. The artificial exploration or natural degradations the concealed minerals could be exposed. These exposed rocks as in the form of rock crystals.

These rock materials are subjected to the proper investigation of various spectroscopic techniques like FTIR, XRD and EDX with SEM used to identify the elements and minerals. Infrared spectra act as "finger print" technique and yield information about the atomic grouping present in the rock samples. IR tool in mineralogy is a most powerful tool in conjunction with XRD. Using FTIR, remarkable information about the group of minerals in which the specimen belongs, the degree of Crystalline and Nature of Crystinallity also inferred. The absorption spectra in FTIR played a vital role to investigate type of bonding, structure of the crystalline minerals and some chemical information [2].

Energy Dispersive X ray Spectrometry was chosen for the quantitative analysis of potsherds because of its precise, relatively cheap and easy to handle. It has limit processing times and very low detection limits. The basic principle of EDX is that the electrons in particular elements are excited by X-rays they emit or fluorescence a spectrum of X-ray that is specific to that element. In the present study, the Bodamalai hills from Namakkal district, Tamil Nadu state, India are subjected to several analytical methodologists.

Every living organism in our earth exposed the natural radiation due to the concentration of primeval concentration of ^{232}Th, ^{238}U and their decay products, natural radio nuclide ^{40}K present in earth curst (UNSCEAR). In earth environment the natural radioactivity exist in various geological forms like soils, rocks, sand, air, water, plants etc., The human beings must aware about the health effects of these radiations. Acute leucopoenia, chronic lung diseases, anaemia could occur to human beings if there is in more harmful radiation. Rock sediments contents rich amount of minerals as well as radioactive elements. Humans should be aware of their natural radiations effects due to radioactive elements [7].

2 Methodology

2.1 Samples Collection

Bodamalai hill (11°32'31" N 78°14'37" E – Longitude and Latitude) is a part of Eastern Ghats in Rasipuram Thaluk in Namakkal district in Tamil Nadu, India. It has Elevation of 1,100 m (3600 feet) with a wide area of 180 km^2. Its length is 19.3121 km (12.0000 mi) East – West side. The location of the Bodamalai hills is shown in Figure 1.

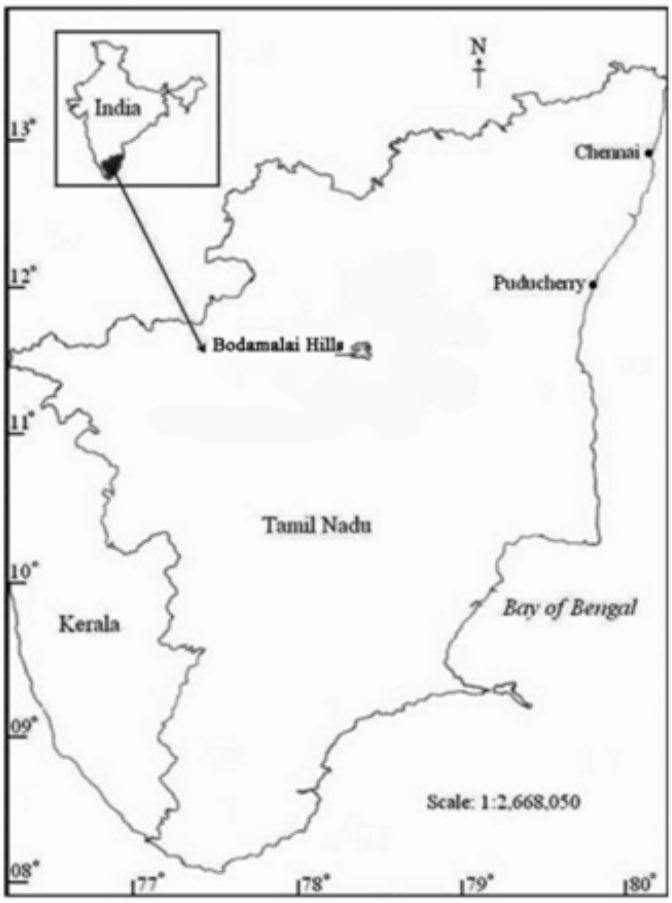

Fig 1. Location of Bodamalai Hills, Tamil Nadu

2.2 Sample Preparation and Instruments used

For Mineral analysis, the sediments were collected from various 15 locations of hills. Depending upon the elevation, the hills sample locations are divided into 15 places. The hills have no proper way to reach top locations. Their foot paths were approximately divided by 15 locations and collected sufficient rock samples for spectroscopic analysis. The samples were collected from the Elevation of the Hill from Top to Bottom. In all locations, 1 kg of rock samples were collected in airtight Polythene bags.

2.3 Experimental Methods

All the samples were cleaned with clear water and weathered on the surface for an hour in order to remove moisture. The samples are taken 30-50 mg and grinding in an agate mortar for 15-20 minutes till to get expected size of around 60-70 mm. By pellet techniques, the grinded samples were mixed with KBr with the ratio of 1:40. The mixed materials were made like a transparent disc by using a high pressure technique. Using 'Perkin Elmer' makes 'Spectrum RX I' Model FTIR spectrometer the maximum transmittance and peaks were observed. The resolution of the instrument is ± 4 cm^{-1} and an accuracy of ± 0.01 cm^{-1}.

2.4 FTIR Spectroscopic Technique

The Fourier Transform Infrared Spectroscopic Technique has been vast used in the detailed characterization of Molecular Structure and it is used as an identical tool since every species for which the molecular motion causes a change of dipole moment. One of the most important applications of FTIR studies is the identification of minerals in rock samples.

To provide a good characterization of a mineral by IR spectroscopy the spectrum should be recorded in the range of 4000 – 400 cm^{-1}. Such coverage ensures that most of the useful vibration active IR will be included [4]. The Nicolet-Avatar 330 series FTIR Spectrometer is used in this present work to obtain FTIR spectra of the rock samples at room temperature. This device scans the spectra 16 times in one minute. A standard polystyrene film is used to calibrate the accuracy of the instrument at every time before taking the readings.

2.5 XRD Technique

X-Ray Diffraction analysis is a non-destructive method to gather the detailed information about the chemical composition and the crystallographic structure [6]. At room temperature the X-ray diffraction patterns were recorded using Siemens D500 X ray diffractometer having a curved graphite crystal diffracted monochromator, CuKa radiation used as a source and NaI (TI) scintillation counter. The derived peaks and corresponding Minerals are given in Table 2. The diffraction patterns were revealed over the 2q values in the range of 20º to 80º. The lattice parameter is the order of 0.005 Å is the estimated error of the device. Using the values of 2-Theta in degree and d-spacing in Å of XRD spectrum for various minerals have been identified from JCPDS database, 2000 and reference journals as well. The experiment XRD pattern was compared with patterns obtained from JCPDS database.

The minerals such as Quartz, Sepiolite, Goethite, Albite, Microline Feldspar, Orthoclase Feldspar, Kyanite, Calcite, Zircon, Monazite, Aragonite, Magnetite and Hematite are identified and tabulated. The above mentioned minerals are confirmed by XRD technique earlier identified from FTIR graphs. The crystallinity index and their Nature of crystallinity of the sample of minerals are also tabulated.

2.6 Radiological Fingerprints Measurements

2.6.1 High Purity Germanium Detector

The P type coaxial based 50% relative efficiency High Purity Germanium (HPGe) detector is used in this study. The detector is enclosed in cylindrical shielding with thickness of 10 cm lead, 2 mm cadmium and 1 mm copper to reduce the background counts. Detector parameters (resolution & counts) and electronic gains are periodically verified using ^{60}Co gamma source (1332 keV energy). All samples undergone the studies at Radiation Application and Technology Division, Safety, Quality and Resource Management Group IGCAR (Indra Gandhi Centre for Atomic Research) – Kalpakkam, Chennai, Tamil Nadu, India.

2.6.2 Powdered Rock Sample Pre-treatment and Analysis

The collected soil samples were dried at 110°C and sieved using 2 mm sieve. The geometry matrix used for the present study is 250 ml bottles and kept for 30 days for achieving the equilibrium between the parent and daughter radionuclides for estimation of ^{238}U and ^{232}Th activity concentration. The packed samples are measured using HPGe detector for the estimation of naturally occurring radioactivity materials (^{238}U, ^{232}Th & ^{40}K). The activity of ^{232}Th in the samples was estimated by 2614 keV gamma rays emitted from ^{206}Tl whereas ^{238}U activity was measured using 1764 keV gamma peak of ^{214}Bi. Photo peak at 1460 keV was used in determination of ^{40}K activity concentration. All the samples were counted for 86400s.

The specific activity of radionuclide in the environmental samples is estimated using equation [7].

$$Activity\left(\frac{Bq}{Kg}\right) = \frac{N}{\varepsilon * t * m * \gamma}$$

Where N is sample net counts, ε is the efficiency of the system, t is counting time, m is sample mass and γ is gamma emission yield.

3 Results and Discussion

3.1 Identification of minerals through FTIR Analysis

The absorption frequencies of all spectra are tabulated in wave number (cm^{-1}) in Table 1. The observed wave number compared with available literature the minerals such as Burnswigite, Organic Carbon, Gibbsite, Quartz, Aragonite, Sepiolite, Calcite,

Cristobalite, Hectorite, Kaolinite, Montmorillonite, Hallyosite, Nacrite, Goethite, Microcline Feldspar, Megnetite, Hematite and Pyrophyllite are identified and tabulated for Bodamalai Hills. The FTIR Spectrum is given in Figure 2.

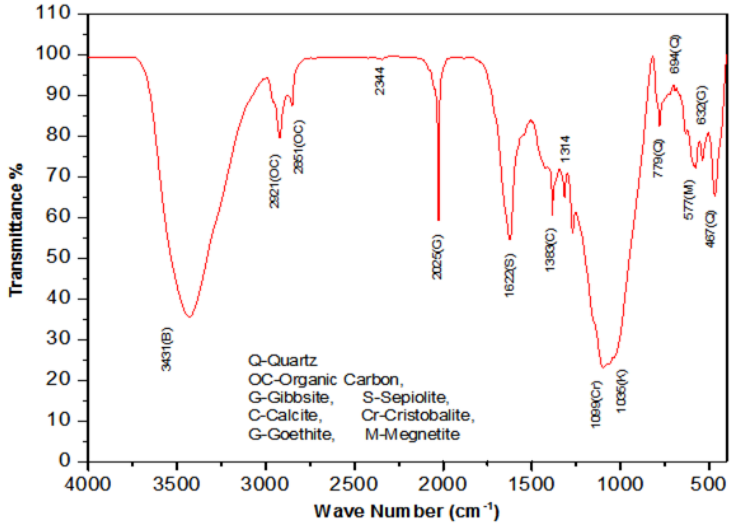

Fig 2. FTIR Spectrum – Bodamalai hills – Site Number 09

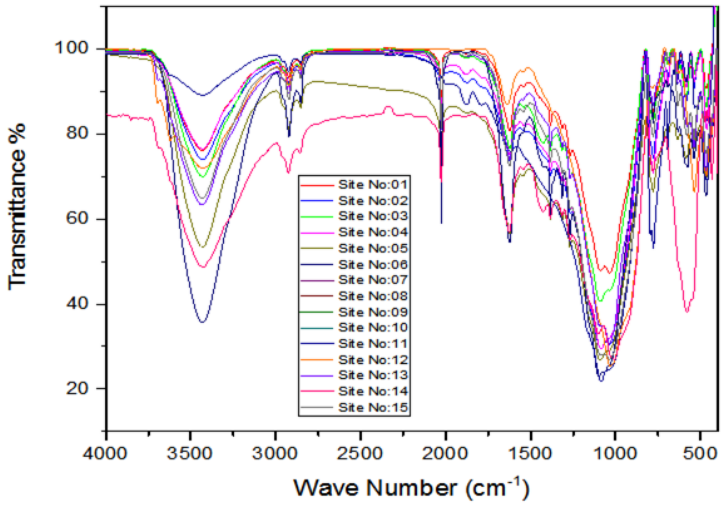

Fig 3. FTIR Spectra – Bodamalai hills – Site Numbers 1 to 15

Table 1. The observed absorption wave numbers and corresponding minerals from FTIR Spectra with Band Assignment [1–3,6,8–18]

Sl. No.	Name of the minerals	Chemical Formula	Site number	Observed wave numbers (cm^{-1})	Band Assignment
1	Quartz	SiO_2	1,3,5-7,10 & 14	1880 - 1876	Si–O -band
			3,5,6 & 10	1084, 1083	Si–O–Si symmetrical stretching
			1 to 15	780 - 778	Si–O symmetrical stretching

Continued on next page

Table 1 continued

			1 to 15	694 - 692	Si-O-Si asymmetrical bending
			1 & 3-15	467 - 463	Si-O-Si symmetrical bending
			1,3-5,7-9 & 12-15	3433 - 3430	Si-O symmetric elongation vibration
2	Kaolinite	$[Al_4Si_4O_{10}(OH_8)]$	2,6,10 & 11	3426 - 3423	Si-O symmetric elongation vibration
			2	1017	Si–O stretching (clay mineral)
			1, 3 ,7,9,12,13 & 15	1035, 1036	Si–O stretching (clay mineral)
			14	474	Si–O stretching (clay mineral)
3	Calcite	$CaCO_3$	1,3-5,8,9 & 12-15	1385 - 1383	C=O stretching vibration
			6 & 7	1082	$(CO_3)_2$ out-of-plane bending
4	Cristobalite	SiO_2 (Polymorph of silica)	1,4,8 & 9	1099 - 1093	Si–O -band
5	Gibbsite	$Al(OH)_3$	1,3-5,8,9 & 12-15	2026, 2025	O-H bending vibrations
6	Goethite	α-FeO(OH)	2 to 15	636 - 632	Fe-O bending
			14	455	Fe-O bending
7	Halloysite	$Al_2Si_2O_5(OH)_4$	14	1101	
8	Hectorite	$Na0,3(Mg,Li)_3Si_4O_{10}(OH)_2$	15	1079	Si-O stretching band
9	Hematite	α-$\Phi\varepsilon_2O_3$	1,3-10 & 12-15	539 - 537	Si–O–Al (or) Fe_2O_3
10	Illite	$(K,H_3O)(Al,Mg,Fe)_2$ $(Si,Al)_4O_{10}[(OH)_2,(H_2O)]$	12	754	H-O-H Stretching
			7,11	1620	H-O-H Stretching
11	Lepidocrocite	FeO(OH)	2	541	
12	Microcline Feldspar	$(K)[AlSiO_3O_8]$	1 to 15	585 - 575	Al–O coordination vibrations
13	Montmorillonite	$(Na,Ca)_{0.3}(Al,Mg)_2Si_4O_{10}$ $(OH)_2$ nH_2O	6 & 10	1027	O-H stretching of absorbed water
14	Nacrite	$Al_2Si_2O_5(OH)_4$	14	1008	molecule
15	Organic Carbon	C	1 to 15	2925 - 2921	Organic matter
			1 to 15	2857 - 2853	Organic matter
16	Palygorskite	$(Mg,Al)_2Si_4O_{10}(OH)_4(H_2O)$	2	1634	stretching and bending regions of water molecules
			11	517	OH-stretching band
17	Sepiolite	$Mg_4Si_6O_{15}(OH)_26(H_2O)$	1,2-6,8-11 & 12-15	1635 - 1622	Mg_3OH bending
			2 & 14	433	vibration
18	Siderite	$FeCO_3$	7,11	1426	Fe-O bending

3.1.1 Quartz

Quartz (SiO_2) is a ubiquitous mineral and it is an abundant constituent in all the Rock sediments. It is non-clay mineral and common and invariably present in almost all the samples. The Si-O bonds are the strongest bonds in the silicate structure and it could be recognized in IR spectra. It is an important component of almost all the samples and its characteristic peaks are reported by several workers [1–6,8–24]. The presence of Quartz in Bodamalai hills is available in all the site locations.

The presence of Quartz is observed in various wave numbers and it is tabulated in Table 1. The FTIR absorption band 1880 – 1876 cm^{-1} with Si-O stretching band, 1084 cm^{-1}, 1083 cm^{-1} absorption with Si-O-Si symmetrical stretching vibration band, 780 – 692 cm^{-1} Si–O symmetrical stretching, 467 – 463 cm^{-1} Si-O-Si symmetrical bending are suggest the presence of Quartz in wave number [1,2,8,9]. The presence of Quartz in the rock samples can be explained by Si-O asymmetrical bending vibration in the range of 455-460 cm^{-1} and 470 cm^{-1}, Si-O asymmetrical bending vibrations are 693-695 cm^{-1} frequency range [1,2,9]. The frequency ranges 775 cm^{-1} and 780 cm^{-1} shows the Si-O symmetrical stretching vibration 780 cm^{-1} are symmetrical bending

vibrations. 778 cm^{-1} frequency shows the Si-O symmetrical bending vibration [1,2,6,8–10,19].

3.1.2 Feldspar (Orthoclase, Microcline, Albite)

Feldspars [$AlSi_3O_8$] are the most important mineral group in all rock sediment types which make up perhaps as much as 60% of the Earth's Crust. The general formula is WZ_4O_8. "W" may be Na, K, Ca and/or Ba, "Z" is Si and/or Al. Its group of minerals such as Orthoclase, Microcline (K)[$AlSiO_3O_8$], Sanidine (K-Feldspar), Aorthite (Ca-Feldspar), Albite [$NaAlSi_3O_8$] are identified by many researchers by analysing the FTIR spectroscopic techniques [3].

They differ in structure Orthoclase Feldspar is Monoclinic, Microline Feldspar is Triclinic and Sanidine is Tetraheral, the frequency range 435 cm^{-1} lies Si-O mixed vibration range [2] and 533-540 cm^{-1} are Si-O asymmetric bending vibration [2,10], 570 cm^{-1} shows the Si-O symmetrical bending vibration, 585 cm^{-1} is O - Si(Al) - O bending vibration [2] and 640 – 644 cm^{-1} frequency ranges are shows Al - O - Co-ordination vibration [9].

3.1.3 Clay Minerals (Kaolinite, Montmorillonite, Illite)

Kaolinite [$Al_4Si_4O_{10}(OH_8)$] is a clay mineral crystallizing the Monoclinic form and forming a major component of China clay and Kaolin. It is softy earthy, usually white mineral. This raw material is basic for ceramics and large quantities are used in fabricate the coated paper. In FTIR frequency spectrum 3426-3423 cm^{-1} shows the presence of Kaolinite in the site numbers (2,3,6,10 & 11) with symmetric elongation band vibration [2,3,14], 1017 cm^{-1}, 1037–1035 cm^{-1} shows the presence of Kaolinite minerals in site numbers (1,3,7,9,12,13 & 15) with Si-O stretching band [2,6,9].

According to N. Oumabady Alias Cannane [9], Montmorillonite [$(Na,Ca)_{0.3}(Al,Mg)_2Si_4O_{10}(OH)_2\ nH_2O$] contains both tetrahedral and octahedral isomorphism substitution, Al (and occasionally Fe^{3+}) for Si in the former case, and Fe^{3+} and Mg for Al in the latter. These substitutions of result, crystalline order are reduced. Montmorillonite is a very soft phyllosilicate mineral and it forms in microscopic crystals and forming clay. 1019 – 1027 cm^{-1} range of frequency spectrum shows the presence of Montmorillonite in site numbers 4 and 5 with the O-H stretching of absorbed water molecule band [9].

3.1.4 Carbonate Minerals (Calcite, Aragonite, Dolomite)

Calcite [$CaCO_3$] is one of the major minerals in rock sediments and it is present in abundant nature in almost all the site numbers in the Hills were examined. It contains the anions $(CO_3)^{2-}$ and includes Ca, Aragonite (both calcium carbonate), Dolomite (Magnesium/calcium carbonate), Siderite (iron carbonate). In the Bodamalai hills at site numbers 1,3,4,5,8,9 & 12-15 abosroption shown the presence of calcite with C=O stretching vibration band assignment 1383 cm^{-1} and 1384 cm^{-1} [1,3,6,9,14,15,20]. From the site numbers 6 and 7 $(CO_3)_2$ out-of-plane bending vibration has shown the calcite mineral at 1082 cm^{-1} band absorption [16–18,21,25].

3.1.5 Orgonic Carbon

These minerals are in a very weak absorption band, due to C-H absorption of contaminants present in the samples. In Bodamalai hills the wavenumber region of (2925 – 2921) cm^{-1} and (2857-2853) cm^{-1} the organic carbon are identified in all site numbers [3,18,25].

3.1.6 Other Minerals

Cristobalite is a naturally occurring mineral with Si-O vibrating band (1099-1093 cm^{-1}) present at site numbers 1,4,8 and 9 [9], Gibbsite [$Al(OH)_3$] with O-H bending vibrations shows at wave number 2026 and 2025 cm^{-1} shows their presents at site numbers 1,3-5,8,9 & 12-15 [17], Goethite [a - FeO(OH)] is a iron bearing mineral revealed at the site numbers 2 to 15 with Fe-O bending vibrations at frequency range from 636 to 632 cm^{-1} [17]. Similarly the Halloysite mineral [$Al_2Si_2O_5(OH)_4$] shows their availability at the site number 14 wavenumber 1101 cm^{-1} [17]. Hectorite [Na0,3$(Mg,Li)_3$Si4$O_{10}(OH)_2$] exhibits at the site number 15 with 1079 cm^{-1} [16,25]. Hematite [a - Fe_2O_3] is a iron oxide mineral present in almost all site numbers except 2 and 11 at frequency range of 539-537 cm^{-1} [2,3,5,8,9,12,18,25]. Lepidocrocite [FeO(OH)] is a reddish brown iron- oxide-hydroxide mineral presents at site number 2 with the frequency of 541 cm^{-1} [3].

Nacrite [$Al_2Si_2O_5(OH)_4$] is colourless transparent mineral with monoclinic structure revealed at site number 4 with the frequency of 1008 cm-1 [9]. Palygorskite, also known as attapulgite is a fibrous clay mineral with chemical formula $(Mg,Al)_2Si_4O_{10}(OH)_4$, shows its availability at site number 2 with frequency of 1634 cm^{-1} at the site location number 2 [3,17,25]. Sepiolite $Mg_4Si_6O_{15}(OH)_26(H_2O)$ is a soft white clay mineral present at site numbers 1,2-6,8-11 & 12-15 with the wavenumber 1635 – 1622 cm^{-1} [3,18,25]. Siderite made of iron carbonate [$FeCO_3$] mineral revealed at site numbers 7 and 11 with the frequency range of 1426 cm^{-1} with Fe-O bending vibration [25].

3.2 Crystallinity Index of Quartz

The Crystallinity Index could be defined as the fraction of crystalline materials in a mixture of crystalline and non-crystalline materials. It cannot be found directly and is determined from the crystallinity index which is inversely proportional to crystallinity. Quartz is the major mineral present in all the sediment samples, the crystallinity index could be found. If crystallinity is minimum, then the minerals are said to be disordered. If it is maximum then the minerals are considered to be ordered state[4] .

The Ratio of absorption band around 777 cm^{-1} (I_{777}) and 695 cm^{-1} (I_{695}) are taken to calculate the crystallinity index of the minerals. When the crystallinity index is minimum, the minerals are said to be well crystallized and if it is maximum, the minerals are considered to be in a poorly crystallized state[3,18,21,25].

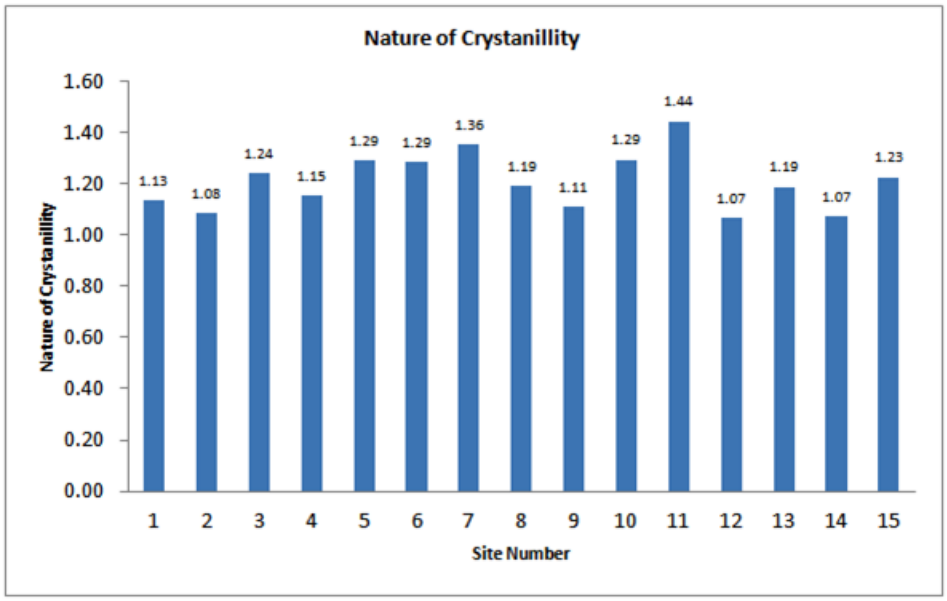

Fig 4. Nature of Crystanillity of Bodamalai hills from Top to Bottom

3.3 X-Ray Diffraction Analysis

The minerals identified by the FTIR spectrum are confirmed by XRD Technique. The different mineral phases of rock materials, the XRD method was chosen. The four location in Bodamalaihills with GPS values (Site No-2: 11°32'13" N 78°12'33" E, Site No-4: 11°31'54" N 78°12'11" E , Site No-6: 11°31'34" N 78°11'48" E and Site No-9: 11°30'49" N 78°10'59" E were selected and their mineral availabilities were analysed using 2q values, d-spacing, Relative intensity and (hkl) parameters, and those crystalline planes were confirmed with JCPDS values.

Table 2. XRD values of Bodamalai Hills at Site number 02

S. No	Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	Left	d-spacing [Å]	Rel. Int. [%]	(hkl)	Mineral Name
1	20.903	173.91	0.1476		4.24985	7.09	100	Quartz
2	22.0531	138.45	0.1476		4.03074	5.64	020	Goethite
3	23.6849	67.19	0.1476		3.75662	2.74	111	Albite
4	24.399	83.99	0.1476		3.64827	3.42	131	Microline Feldspar
5	26.7196	2453.88	0.1476		3.33644	100	101	Quartz
6	27.5832	233.72	0.0984		3.23392	9.52	002	Orthoclase Feldspar

Continued on next page

Table 2 continued

7	28.0992	500.76	0.1476	3.17569	20.41	121	Kyanite
8	29.8179	63.62	0.1968	2.99645	2.59	104	Calcite
9	30.4105	71.9	0.1968	2.93939	2.93	402	Zircon
10	31.4851	33.76	0.2952	2.84148	1.38	200	Monazite
11	33.8837	17.96	0.2952	2.64563	0.73	031	Aragonite
12	35.505	38.32	0.246	2.52844	1.56	011	Goethite
13	36.6231	125.42	0.1968	2.45378	5.11	240	Microline Feldspar
14	39.5014	92.95	0.1968	2.28136	3.79	012	Quartz
15	40.3569	112.81	0.1476	2.23496	4.6	200	Quartz
16	42.5201	85.21	0.1476	2.12613	3.47	200	Quartz
17	45.8655	48.28	0.1476	1.97854	1.97	200	Quartz
18	50.1841	277.32	0.1968	1.81793	11.3	354	Magnetite
19	59.9848	101.84	0.1968	1.54223	4.15	332	Orthoclase Feldspar
20	63.8237	18.62	0.3936	1.45841	0.76	311	Hornblende
21	68.3567	139.33	0.1476	1.37233	5.68	301	Quartz

From site number 02 the diffraction peaks at 2q^o : 20.90, 26.71, 39.50, 40.35, 42.52, 45.86, and 68.35 that correspond respectively to the planes of crystalline (100), (101), (012), (200), (200), (200) and (301) these XRD peaks are assigned to the Quartz mineral phase (SiO_2-Hexagonal structure) according to the JCPDS (N^o 00-046-1045) [1]. 2q^o : 24.39, 36.62 diffraction peaks show the (hkl) crystalline plane (121), (340) for Microline Feldspar and 2q^o : 27.58, 59.98 peaks with (002), (332) for Orthoclase Feldspar. The calcite mineral ($CaCO_3$) presence was confirmed from the diffraction peak 2q^o:20.81 with the (hkl) trigonal crystalline plane (104) according to the JCPDS (N^o 01-086-2344) [4,6].

The diffraction peaks at 2q^o: 20.05, 23.68, 28.09, 30.41, 31.48, 33.88, 35.50, 50.18 and 63.82 values with the crystalline plane (hkl) values (020), (111), (121), (402), (200), (031), (011), (354) and (311) corresponds to the minerals Goethite, Albite, Kyanite, Zircon, Monozite, Aragonite, Goethite, Magenetite and Hornblende respectively [9,10,12].

The second location on Bodamalai hills site number:04 X-Ray diffraction peaks 2q^o: 20.92, 26.71, 39.50, 40.34, 45.86, 50.85, 68.30, 69.99 and 73.59 with the crystalline plane (100), (101), (012), (113), (200), (200), (003), (301), (014) and (014) belongs to Quartz mineral.

2q^o: 67.45, 27.74 and 68.12 with the crystalline plane (131), (130) and (240) shows the Microline Feldspar mineral. 2q^o: 22.05, 23.80, 28.43, 29.72, 30.42, 31.50, 33.85, 35.44, 64.21 and 66.44 diffraction peaks shows with crystalline plane (330), (111), (021), (211), (104), (402), (200), (031), (011), (140) and (112) revealed the minerals Sepoilite, Albite, Kyanite, Calcite, Zircon, Monazite, Argonite, Geothite, Magnetite and Hematitie respectively.

The third location with the site number 06 with latitude and longitude 11°31'54" N 78°12'11" E revealed the diffraction peaks 2q^o: 20.81, 26.62, 39.38, 42.24 and 45.69 with the crystalline plane (100), (101), (012), (200) and (200) shows the availability of Quartz mineral.

The diffraction peaks 2q^o: 22.89, 24.36, 36.53 with the crystalline plane (111), (131) and (240) shows the availability of Microline Feldspar. And 2q^o: 27.76 peak with the plane (002) shows the presence of Orthoclase Feldspar. 29.71, 42.87 diffraction peaks give the availability of Calcite with the crystalline plane (104) and (121).

In the same location 2q^o: 23.62, 28.02, 30.24, 31.41, 33.81, 35.44, 50.11 and 67.69 diffraction peaks correspond with the crystalline plane (111), (021), (204), (200), (031), (011), (354) and (167) gives the availability of the minerals Albite, Kyanite, Zircon, Monacite, Aragonite, Geothite, Magnetite and Aragonite.

The fourth location of the same hills with the diffraction peaks 2q^o: 20.88, 26.72, 55.50 and 68.32 with the crystalline plane (100), (101), (013) and (301) shows the presence of Quartz mineral. 2q^o: 24.42, 36.05 peaks with crystalline plane (131), (130) revealed the Microline Feldspar and the peaks with 29.87, 42.95 with plane (104), (121) give the calcite mineral. Followed by the diffraction peaks 22.08, 23.75, 28.09, 30.31, 33.83, 35.50, 49.84 and 66.33 with the crystalline plane (330), (111), (021), (204), (031), (011), (354) and (112) shows the minerals Sepiolite, Ablite, Kyanite, Zircon, Aragonite, Goethite, Magnetitie and hematitie respectively.

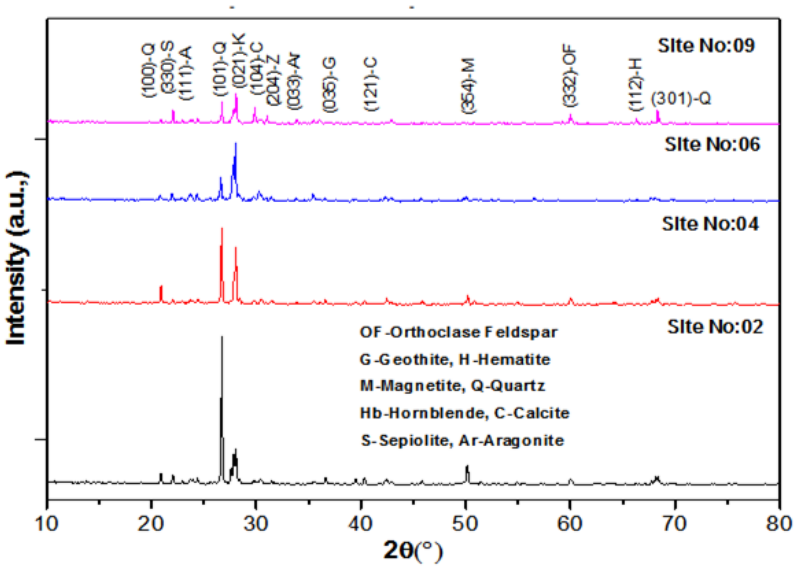

Fig 5. XRD Spectra of Bodamalai hills site numbers (2,4,6 and 9)

EDX with SEM

Energy Dispersive X-ray Spectrometer analysis enables one to identify and confirm microminerals and inspect the distribution of these within the rock samples. The superficial elemental profile, structure and internal morphology could investigated by EDX-SEM [1,9,19]. These individual spectra give information about the additional basic minerals for better comparative scrutiny. In our studies, diverse elemental composition in rock samples like Aluminium (Al), Magnesium (Mg), Iron (Fe), Sodium (Na), Silicon (Si), Potassium (K), Calcium (Ca), Copper (Cu), Carbon (C) and Oxygen (O), and their mass fraction and atomic percentage were measured by EDX. This EDX result has shown that Si and O were significant elements and most dominant in examined samples.

The rock powder samples were dried at 110°C in an oven until no further weight loss was observed. 1: 2 ratios of samples and Boric acid were mixed. The mixture is made as a pellet of 30 mm diameter using a 15 ton hydraulic press. The prepared samples have undergone the study of EDX (Energy Dispersive X ray Spectrometry) at National College, Tiruchirappalli, India. The power specifications of the tube are 4-30 kV; 1 mA – 1 mA. The removable sample charger of the instrument accommodates 12 samples at a time. Selection of filters, tube voltage, filters, current and sample position are fully controlled by computer.

The elliptical beam spot area instrument is 81.7 mm^2. The instrument has features of Multi channel Analyser (MCA) test, standardless determination, and Gain correction. The beam stop is in the reference position the Gain correction could be performed. The beam spot contains a reference sample (an alloy of aluminium and copper). Copper is used for gain correction. Cu along with Al could be used for instrument energy calibration. The standard stream (GBW 7305) sediment was used as reference material for standardizing the instrument and the values are presented in Table 3 of site numbers 2,5.

Concentrations of elements of interest Na, Mg, Al, Si, K, Ca, Fe, Cu, C, O in Bodamalai Hills using EDX are reported in Table 3. From EDX analysis the abundant amount of Si, Al, Mg, Fe, K, Ca and Na was found and it supports the vibrational spectroscopic findings; the presence of Quartz, Iron oxides (Hematite), Alumino silicates and Feldspar. The size of the powdered rock samples size around 60-70 mm is shown in Figure 7.

Table 3. The percentage of Minerals in Site No 02 and 05

Element	Site Number 02		Site Number 05	
	Weight%	Atomic%	Weight%	Atomic%
C	26.29	35.64	7.7	11.93
O	50.03	50.91	54.75	63.69
Na	1.87	1.32	2.32	1.88
Mg	0.18	0.12	-	-

Continued on next page

Table 3 continued

Al	4.24	2.56	5.46	3.76
Si	14.59	8.46	25.75	17.06
K	0.45	0.19	0.89	0.43
Ca	1.09	0.44	1.59	0.74
Fe	1.12	0.33	1.54	0.51
Cu	0.14	0.04	-	-
Totals	100		100	

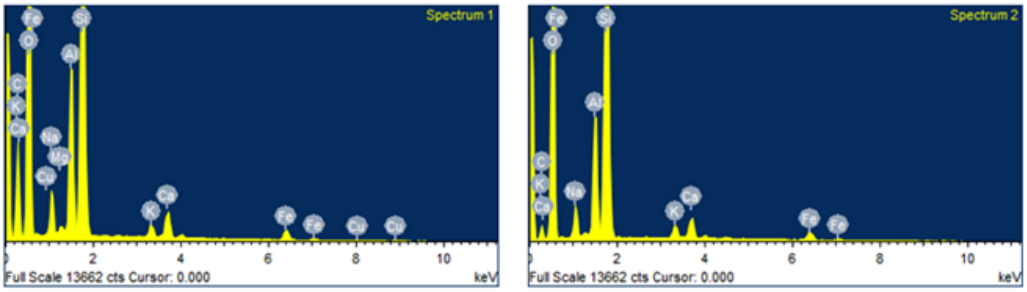

Fig 6. EDX of Site No 02 and 05

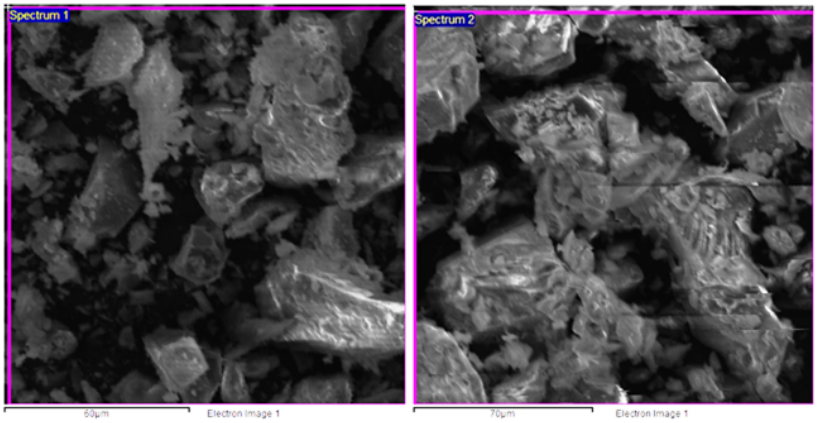

Fig 7. SEM image of Bodahills powered samples – Site No 2, 5

The micrograph from Figure 7 shows clayey particles from irregularly contoured blocks and some aggregate clusters due to other minerals. These characteristics of kaolinite are poorly crystallized [14]. From EDX analysis Aluminium presents 4.2% and 5.46% in site numbers 2 and 5 due availability of Kaolinite and Feldspar. Magnesium (Mg) presented in 0.18%, Copper (Cu) in 0.14% available in site number 02. Silicon (Si) presents a maximum of 25.75% and 14.59% in site numbers 5 and 2 respectively. 1.12% and 1.54% of Iron minerals show their presence in rock samples. Sodium (Na) shows the percentage of weight is 1.87% and 2.32%. Carbon occupies the weight among the samples at 26.29% and 7.7% along with Atomic percentages 35.64% and 11.93% in site numbers 2 and 5 respectively. Among all elements, Oxygen (O) plays a vital role in two site numbers, 50.03% and 54.75% of weight in rock materials due to it being part of almost all the minerals such as Quartz, Calcite, Cristobalite, Gibbsite, Goethite, etc.

Natural Radioactivity Measurement

Table 4. Natural Radioactivity of Bodamalai hills from site number (1 – 10)

S. Id	^{40}K (Bq/kg)	^{238}U (Bq/kg)	^{232}Th (Bq/kg)
B 1	243.0±25.9	BDL	BDL
B 2	182.9±26.1	BDL	BDL
B 3	141.3±22.3	BDL	BDL
B 4	216.5±24.7	BDL	BDL
B 5	210.6±27.2	BDL	BDL
B 6	209.1±23.6	BDL	BDL
B 7	58.8±21.1	BDL	BDL
B 8	264.1±26.2	BDL	BDL
B 9	222.1±23.5	BDL	BDL
B 10	198.2±23.8	BDL	BDL

BDL=Below Detection Limit <MDA
MDA=Minimum Detection Activity levels are given below
^{40}K-6.1 Bq/kg; ^{238}U-1.2 Bq/kg; ^{232}Th -1.8 Bq/kg
Studies carried out in IGCAR, Chennai

The Bodamalai hills show the ionizing radiation present only due to ^{40}K. The minimum radiation is 58.8±21.1 Bq kg^{-1} at site number 7 and the maximum is 264.1±26.2 at site number 8. The variation of radioactive radiations randomly formed in these Hills. The ^{232}Th and ^{238}U radiations are below the detection level. It shows their no harmful radiation for living organisms. The radiation levels are tabulated in Table 4.

4 Conclusion

Using FTIR techniques the Rock sediments of Bodamalai hills were carried out, 19 minerals were identified and tabulated with their respective wave number, the crystallinity index and nature of crystallinity were tabulated and graphs were plotted and the Extinction coefficient of Quartz, Feldspar, and Kaolinite are also calculated and tabulated. For confirmation of minerals identification, the samples have undergone the studies of XRD, from this analysis minerals such as Burnswigite, Organic Carbon, Gibbsite, Quartz, Aragonite, Sepiolite, Calcite, Cristobalite, Hectorite, Kaolinite, Montmorillonite, Hallyosite, Nacrite, Goethite, Microcline Feldspar, Megnetite, Hematite and Pyrophyllite are confirmed. From XRD studies "5" additional minerals were identified.

The Graph and Table of contents of XRD have been given. From the above experiment analysis, we inferred that Quartz, Kaolinite, Calcite, and Aragonite are in Crystalline form and other minerals are in a non-crystalline form which is also confirmed in XRD studies. However, the disordered nature of crystals was found using the crystallinity index in rock samples. The Nature of Crystallinity lies between 1.066 to 1.444, with mean value of 1.209. The Nature of crystallinity is not uniformly neither decreased nor increased, the results showed in a random manner from top to bottom of the Bodamalai Hills. Among these minerals, Quartz showed a large number of peaks in diffractograms. This indicates that Quartz is the most abundant mineral in the sediment samples.

Natural Radioactive studies showed the radiation occurs due to ^{40}K ionizing radiation lies between 58.8±21.1 Bq/kg and 264.1±26.2 Bq/kg with mean value 194.6±24.44 Bq/kg. The radioactive elements ^{232}Th and ^{238}U are found below the detection level. So, their no harmful radiation in Bodamalai hills in studied places.

FTIR, XRD and EDX with SEM combined technique gives the information for the minerals and elemental composition in rocks and their formation. Some of the minerals observed through FTIR analysis are not identified in the XRD study which indicates its loss of crystalline nature.

References

1) Hachem R, Meftah N, Bouaziz A. A comparative analysis of the microstructural and physicochemical properties of alluvial and dune sands from northeast Algerian Sahara. *The Journal of Engineering and Exact Sciences* . 2023;09(6):1–14. Available from: https://www.researchgate.net/publication/372693083_A_comparative_analysis_of_the_microstructural_and_physicochemical_properties_of_alluvial_and_dune_sands_from_northeast_Algerian_Sahara.
2) Jozanikohan G, Abarghooei MN. The Fourier transform infrared spectroscopy (FTIR) analysis for the clay mineralogy studies in a clastic reservoir. *Journal of Petroleum Exploration and Production Technology*. 2022;12:2093–2106. Available from: https://link.springer.com/article/10.1007/s13202-021-01449-y.

3) Paramasivam R, Shanmugam S, Venkidasamy R, Shanmugam A. Mineralogical Characterization Of Sediments, Kalrayan Hills South India - A FTIR Study. *Journal of Advanced Scientific Research*. 2021;12(1):66–74. Available from: https://www.researchgate.net/publication/367749388_MINERALOGICAL_CHARACTERIZATION_OF_SEDIMENTS_KALRAYAN_HILLS_SOUTH_INDIA_-_A_FTIR_STUDY.
4) Zoungrana JB, Sorgho B, Bakouan C, Sawadogo S, Ouedraogo RD, e Guel B, et al. Mineralogical study of phosphate rocks by quantitative rietveld refinement. *Open Ceramics*. 2024;18. Available from: https://doi.org/10.1016/j.oceram.2024.100567.
5) Nicoli M, Eftekhari N, Vaccaro C, Giraldo HC, Garcês S, Gomes H, et al. A multi-analytical evaluation of the depositional pattern on open-air rock art panels at "Abrigo del Lince. *Environmental Science and Pollution Research*. 2023;30:24344–24360. Available from: https://link.springer.com/article/10.1007/s11356-022-23589-2.
6) Iranfar S, Karbala MM, Shakiba M, Shahsavari MH. Effects of type and distribution of clay minerals on the physico-chemical and geomechanical properties of engineered porous rocks. *Scientific Reports*. 2023;13. Available from: https://www.nature.com/articles/s41598-023-33103-4.
7) Ravisankar R, Rajalakshmi A, Eswaran P, Meenakashisundram V, Gajendiran V, Manikandan E, et al. Gamma- ray spectroscopic and pixe analysis of beach rock samples of south east coast of Tamilnadu, India. *International Journal of PIXE*. 2007;17:193–203. Available from: https://dx.doi.org/10.1142/s0129083507001253.
8) Rahman MS, Metselaar HSC, Razak BBA. Mineralogical and Thermal Analysis of Ancient Ceramic Artifacts Based on Modern Techniques of Ceramic Studies. *Mediterranean Archaeology and Archaeometry*. 2024;2024(3):38–56. Available from: https://www.maajournal.com/index.php/maa/article/view/1131/1081.
9) Seetha D, Velraj G. FT-IR, XRD, SEM-EDS, EDXRF and chemometric analyses of archaeological artifacts recently excavated from Chandravalli in Karnataka State, South India. *Radiation Physics and Chemistry*. 2019;162:114–120. Available from: https://doi.org/10.1016/j.radphyschem.2019.03.017.
10) Papakosta V, Lopez-Costas O, Isaksson S. Multi-method (FTIR, XRD, PXRF) analysis of Ertebølle pottery ceramics from Scania, southern Sweden. *Archaeometry*. 2020;62(4):677–693. Available from: https://doi.org/10.1111/arcm.12554.
11) Ruba S, Abdulraouf M, Hussein AS, Wassef AS, Jürgen P. FTIR and Thermogravimetric Analysis of Ancient Mortar from Al-Amuwaqqar Early Islamic Bathhouse in Jordan for Conservation use. *International Journal Of Conservation Science*. 2023;14(1):3–18. Available from: https://www.researchgate.net/publication/370319381_FTIR_AND_THERMOGRAVIMETRIC_ANALYSIS_OF_ANCIENT_MORTAR_FROM_AL-AMUWAQQAR_EARLY_ISLAMIC_BATHHOUSE_IN_JORDAN_FOR_CONSERVATION_USE.
12) Lorna C, Peter OO, Moses RK. Multi-analytical Approach (FTIR, XRF, XRD, GC-MS) Characterization of Source Rocks from the Anza Basin, Kenya. *Mediterranean Journal of Chemistry*. 2023;13(2):145–155. Available from: https://www.medjchem.com/index.php/medjchem/article/view/1687/941.
13) Plessis PID, Gazley MF, Tay SL, Trunfull EF, Knorsch M, Branch T, et al. Quantification of Kaolinite and Halloysite Using Machine Learning from FTIR, XRF, and Brightness Data. *Minerals*. 2021;11(12). Available from: https://doi.org/10.3390/min11121350.
14) Patra B, Pal R, Paulraj R, Pradhan SN, Meena R. Mineralogical composition and C/N contents in soil and water among betel vineyards of coastal Odisha. *SN Applied Sciences*. 2020;2. Available from: https://link.springer.com/article/10.1007/s42452-020-2631-5.
15) Madejova J. FTIR techniques in clay mineral studies. *Vibrational Spectroscopy*. 2003;31(1):1–10. Available from: https://doi.org/10.1016/S0924-2031(02)00065-6.
16) Chandrasekaran A, Rajalakshmi A, Ravisankar R, Kalarasai S. Analysis of beach rock samples of Andaman Island, India by spectroscopic techniques. *Egyptian Journal of Basic and Applied Sciences*. 2015;2(1):55–64. Available from: https://dx.doi.org/10.1016/j.ejbas.2014.12.004.
17) Sathya P, Velraj G. FTIR spectroscopic and X-ray diffraction analysis of archaeological grey potteries excavated in Alagankulam, Tamil nadu, India. *Journal of Experimental Sciences*. 2011;2:4–6. Available from: https://updatepublishing.com/journal/index.php/jes/article/view/1838.
18) Ramasamy V, Rajkumar P, Ponnusamy V. Depth wise analysis of recently excavated Vellar river sediments through FTIR and XRD studies. *Indian Journal of Physics*. 2009;83(9):1295–1308. Available from: https://link.springer.com/article/10.1007/s12648-009-0110-3.
19) Cannane NOA, Rajendran M, Selvaraju R. Determination of Binary Mixtures of Chemical Composition Ffor Industrial Area Soils using FT-IR Method. *Asia Pacific Journal of Research*. 2014;1(12):90–105. Available from: https://citeseerx.ist.psu.edu/document?repid=rep1&type=pdf&doi=72210b8f2c1970dc1e21025e91bdd204ab806c88.
20) Cannane NOA, Rajendran M, Selvaraju R. FT-IR spectral studies on polluted soils from industrial area at Karaikal, Puducherry State, South India. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*. 2013;110:46–54. Available from: https://doi.org/10.1016/j.saa.2013.03.040.
21) Sivakumar S, Ravisankar R, Chandrasekaran A, Jebakumar JPP. FT-IR Spectroscopic Studies on Coastal Sediment Samples from Nagapattinum District, Tamilnadu, India. *International Research Journal of Pure and Applied Chemistry*. 2013;3(4):366–376. Available from: https://journalirjpac.com/index.php/IRJPAC/article/view/254.
22) Bayazit M, Kaynak E, Coşkun N. Chemical And Mineralogical Analyses Of The Late Neolithic Ceramics From Şah Valley (Singuber), Turkey. *Journal of Scientific Reports - A*. 2023;p. 1–25. Available from: https://dergipark.org.tr/tr/download/article-file/2780095.
23) Ibrahim MBA, Yao KA, Digbehi ZB. Mineralogical characterization of Upper Cretaceous petroleum source rocks of Termit sedimentary basin (Niger). *Journal of Materials and Environmental Science*. 2020;11(7):1173–1183. Available from: https://www.jmaterenvironsci.com/Document/vol11/vol11_N7/JMES-2020-11103-Ibrahim.pdf.
24) Saikia BJ, Goswami SR, Borah RR. Estimation of heavy metals contamination and silicate mineral distributions in suspended sediments of Subansiri River. *International Journal of the Physical Sciences*. 2014;9(21):475–486. Available from: https://www.researchgate.net/publication/283322816_Estimation_of_heavy_metals_contamination_and_silicate_mineral_distributions_in_suspended_sediments_of_Subansiri_River.
25) Gnanasaravanan S, Rajkumar P. Characterization of minerals in natural and manufactured sand in Cauvery River belt, Tamilnadu, India. *Infrared Physics & Technology*. 2013;58:21–31. Available from: https://dx.doi.org/10.1016/j.infrared.2012.12.042.