Journal of Radiation and Nuclear Applications
An International Journal

http://dx.doi.org/10.18576/jrna/080103

The Use of Spectroscopy Techniques to Determine the Mineral Content of Soils in the Tiruvannamalai District of Tamil Nadu, India

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Received: 9 Oct. 2022, Revised: 3 Nov. 2022, Accepted: 11 Dec. 2022

Abstract: This study used infrared spectroscopy to determine the mineral content of soils. The soil samples were collected from the top layer of the soil at a depth of 5 cm from 63 locations in Tiruvannamalai District, Tamilnadu, using a grid technique. The top layer was so chosen in order to assess the anthropogenic influence on the nature of the soil. Charnockite, Granite gneiss, Epidote Hornblende gneiss, Amphibolite, Pyroxenite, Dunite, migmatites, Banded Magnetite Quartz, Shale, and clay are among the rocks found in the district. In the infrared spectrum, the results show that soils are composed of different minerals, such as quartz, kaolinite and montmorillonite, aragonite, hematite, feldspar and calcite. For quartz, the crystallinity index was calculated in order to determine its crystalline nature; there is disordered crystalline quartz present in the soils. Accor0000000000000g to the findings of this study, kaolinite and quartz minerals were the most abundant minerals, while montmorillonite and illite were found in moderate amounts, and organic carbon was identified in trace amounts. The FT-IR study revealed that quartz, calcite, and kaolinite were the most prevalent minerals in the soils. From the XRD analysis, confirmation of the mineral compositions of soil samples was obtained. The results of the mineral analysis using the FT-IR method are in agreement with the findings of the XRD studies. The proposed FTIR technique was shown to be useful in determining soil mineralogy because it is quick and easy to use.

Keywords: Soil, FT-IR, XRD, Mineral Analysis.

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1 Introduction

Soil is described as a heterogeneous system with complicated and difficult-to-understand systems and processes. Numerous traditional approaches are used to characterize the relationship between various soil qualities, including physical, chemical, mineralogical, and major soil components. As a result, both field and laboratory soil testing processes must be straightforward and accurate. Infrared absorption spectroscopy (IAS) is a non-destructive systematic technique for characterizing and identifying soil mineralogy. This technique is frequently employed in a variety of areas, including agriculture, food, polymers, pharmaceuticals, and petrochemicals, as an incredible tool for evaluating these products. Numerous publications asserted that the infrared absorption technique is a viable tool for determining the mineralogy of soils [1,2].

Soil is a complicated mixture of minerals, rocks, and organic materials. The majority of soils are made up of weathered rocks and minerals. Quartz, feldspars, micas, hornblende, calcite, and gypsum are examples of these minerals. The most often used methods for characterisation of soil minerals are X-ray diffraction, infrared, and optical studies. These techniques enable the identification and quantification of distinctive mineral properties that are critical for forecasting their impact on overall soil behavior. Among the techniques, infrared spectroscopy as an analytical tool has a number of advantages since it is precise, affordable, and dependable, the sample quantity required is in the sub milligram range, and the sampling procedure is straightforward. A significant advantage of infrared spectroscopy is that it enables the detection of changes in both crystalline and amorphous forms. Infrared

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spectroscopy is less frequently used in quantitative mineral analysis.

The purpose of this research is to use infrared absorption spectroscopy to evaluate the mineral content of soil samples obtained in Tiruvannamalai District, Tamilnadu.

2 Materials and Methods

2.1. Study area and Sample Collection

The district of Tiruvannamalai is located between 11.55° and 13.15° North Latitude and 78.20° and 79.50° East Longitude. It covers an area of 6188 km^2. Charnockite, Granite gneiss, Epidote Hornblende gneiss, Amphibolite, Pyroxenite, Dunite, migmatites, Banded Magnetite Quartz, Shale, and clay are among the rocks found in the Study area. Soil samples were collected using the grid method [3]. Fig. 1 depicts the study area's location map and sampling strategy. At each grid point, samples were obtained from depths ranging from 0 to 5 cm. In Each location the samples are collected from the corners of the square and one sampled at the central point [4]. A 1 kilogram bulk sample was taken and air-dried, while bigger stone shards or shells were hand-picked. The materials were finely pulverized using an agate motor and stored in desiccators until analysis.

2.2. Sample Preparation

A sample of two mg was mixed with 40 mg of spectroscopic KBr in a ratio of 1:20 taken into a mortar and pestle for grinding to make poweder form. Prior to mixing, the appropriate amount of KBr powder was dried in an oven at 120°C for 6 hours. Otherwise, the broad spectral peak created by the free OH ion will significantly affect the interpretation of the bound hydroxyls associated with any of the minerals. A pellet with a thickness of 1 mm and a diameter of 13 mm was prepared. The pellet was made and place in a moisture-free glass container before it was placed in a sample holder and placed in the infrared beam for analysis.

The Perkin Elmer FT-IR spectrometer was used to record the FT-IR spectra of the samples at room temperature in this investigation. Mineral analysis was performed using the KBr pellet technique (1:20).To accurately characterize a mineral using infrared spectroscopy, the spectrum should be recorded in the 4000-400 cm^{-1} region. This range coverage ensure that the majority of useful infrared vibrations were included. The instrument scans the spectrum 16 times per minute with a resolution of 5cm^{-1}. Fig. 2 shows a typical FT-IR spectrum.

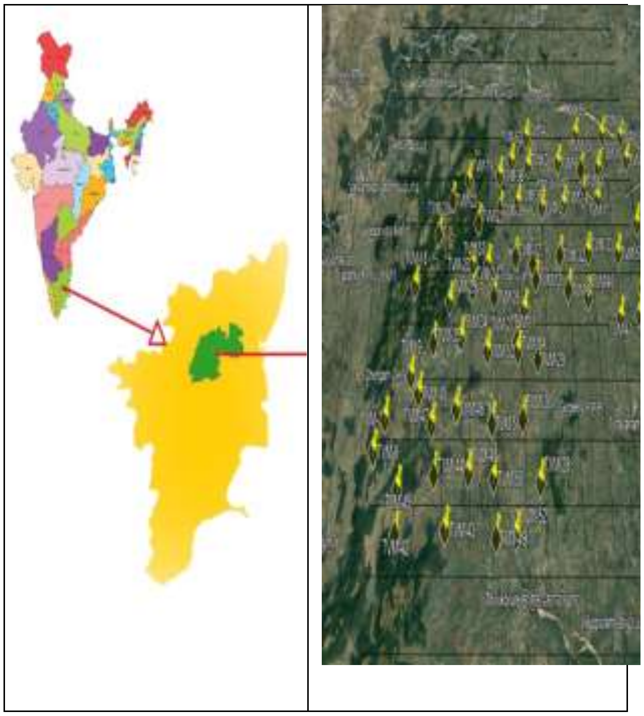

Figure1: Study area of Tiruvannamalai District and Sampling sites.

2.3. FT-IR Analysis

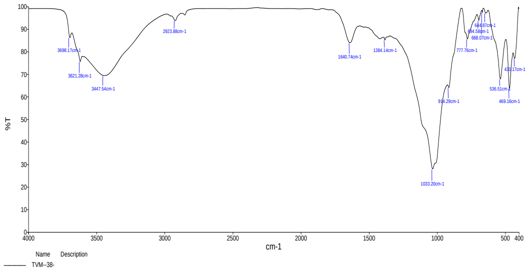

Figure 2: A typical FT-IR spectrum of the soil sample collected at TVM-38

2.4. XRD Analysis

The X-ray patterns of soil samples were recorded at room temperature using an X-ray diffractometer (D500, Siemens) equipped with a curved graphite crystal diffracted monochromator, a CuK radiation source, and a NaI(Tl) scintillation detector. The diffraction patterns were obtained in the range of 10 º to 80 º degrees for the 2θ value. The estimated lattice parameter is in the order of ±0.01. The mineralogy of the soil samples is qualitatively determined using standard XRD interpretation methodologies. Fig.3 shows a typical XRD spectrum of soil samples.

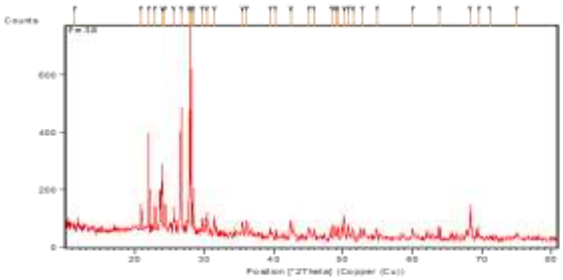

Figure 3: A typical XRD spectrum of the soil sample collected at TVM-38

4.1Results and Discussion

The absorption frequencies of the peaks in the spectra of each site in wave number units (cm^{-1}) are reported in Table 1 together with the identified minerals. The minerals were identified as quartz, albite, microcline, orthoclase, kaolinite, montmorillonite, illite, and organic carbon [5-15], in the soil samples. From the i.r. absorption peaks, quartz and feldspars are the major constituents and kaolinite is the minor constituents in the study area. The mineral wise discussion is as follows.

The absorption frequencies of the peaks in the spectra of each site (wave number units (cm^{-1}), along with the identified minerals are listed in Table 1.Quartz, albite, microcline, orthoclase, kaolinite, montmorillonite, illite, and organic carbon were identified in soil samples [5-15], From the number of i.r. absorption peaks it is noticed that quartz and feldspars are the primary minerals in the study area while kaolinite is a minor constituent. The following is a discussion of minerals.

4.1.1. Quartz

Quartz is a mineral composed of silicates with the chemical formula SiO_2. Table 1 list quartz mineral indicated by the presence of infrared absorption bands at 693-695 cm^{-1}, 775-780 cm^{-1}, 790-795 cm^{-1}, and 1874-1879cm^{-1} [5-10]. Quartz can be identified by the existence of Si–O bonding. Silicate minerals, in particular, have a stronger Si–O bond. In the range 900–1100 cm$^{-1,}$ the Si–O bond is owing to stretching mode, while in the range 400–800 cm^{-1}, it is due to bending mode. A minimum of two to a maximum of five peaks were found for each sample.

The absorption pattern in quartz may be explained by assigning the bands in the 695 cm^{-1} region to Si-O symmetrical bending vibrations, the bands in the 775 cm^{-1} region to Si-O symmetrical stretching vibrations, and the bands in the 795 cm^{-1} region to Si-O symmetrical stretching vibrations. 795-800cm^{-1} and 695-700cm^{-1} are the most often used diagnostic peaks for quartz [11]. The presence of quartz was established in the majority of samples using Si–O bonding from the typical peak around 777 cm^{-1}. At 695 cm^{-1}, the symmetrical bending vibrations of the Si-O group are more effective for determining whether the material is crystalline or amorphous. The 695 cm^{-1} peak will be absent in the amorphous condition [18]. Due to the fact that the majority of the samples' spectra exhibit the 695 cm^{-1} it may be assumed that the quartz mineral in these samples is crystalline.

4.1.2. Feldspar Minerals

Feldspar is the most prevalent mineral group on the Earth. The feldspar minerals are distinguished by their composition of aluminum silicates combined with additional metals such as potassium, calcium, sodium, and occasionally barium. The orthoclase, microcline and plagioclase feldspars are included in this group. According to table-1., the detected i.r.frequencies are 430-435, 465-470, 535-540, 575-580, 580-585, 585-590, 640-645, and 645-650 cm-1, which were all assigned to feldspars in all samples [6-11],[18,22,23].The presence of microcline is indicated by the occurrence of peaks at 585-590 and 640-645cm^{-1} in the samples. The ir absorption peaks at 425-430 cm^{-1} suggest the existence of feldspar in the microcline form. The peaks at 465–470, 535–540, 580–585, and 645–650cm^{-1} show the presence of orthoclase in the samples. The absence of a peak at 635–640 cm^{-1} in all samples shows that sanidine is not present, which could be attributed to sanidine crystallizing at high temperatures.

4.1.3. Clay minerals

The clay minerals such as kaolinite, montmorillonite and illite are the present in the soil. These minerals are distinguished by their absorption spectroscopy bands, with the strongest 1.4 µm band in kaolinite and a weaker 1.9 µm band in montmorillonite referring to hydroxide ions (OH$^{-)}$,while the stronger 1.9 µm band in montmorillonite is affected by bound water molecules in this hydrous clay. Kaolinite is invariably found in all of the samples analyzed, as demonstrated in Table 1. Kaolinite observed from the absorption peaks at 3690, 3620, and 1030 cm^{-1}. It is observed that each sample contains at least one peak containing trace levels of kaolinite.

The O-H stretching band between 3690 and 3620cm^{-1} is a distinctive feature of kaolinte that can be used to differentiate it from other clay minerals. This was noticed most of the samples in the study area. The absorption bands at 3690 and 3620cm^{-1} are attributed to inner surface OHs and inner OH stretching vibrations, respectively [19]. At 1030 cm$^{-1,}$ the broad absorption band corresponds to the Si–O stretching of kaolinite (clay material) [18,10] The absorption band at 3440-3445 cm^{-1} in Table 1 indicates the montmorillonite. This band, which is typically centered around 3400cm^{-1}, is caused by the H-O-H stretching of water molecules found in montmorilinite's interlayer region [20].The absorption lines found at 915 cm^{-1} in the samples' spectra indicate the

Table 1: The observed wave numbers and their minerals by FT-IR of spectra of soil samples of Tiruvannamalai Dist, Tamil Nadu, India.

S.No	Mineral	Soil sample locations ID	Observed wave number (cm^{-1})	References
1	Quartz	TVM-1 - TVM-63	693-695	[16] [17] [8] [9] [11] [10]
		TVM-1 - TVM-4, TVM-6, TVM-8 – TVM-9, TVM-11 – TVM-63	775 -780	
		TVM-5, TVM-7, TVM-10,	790-795	
		TVM-17-TVM-19, TVM-26,TVM-45,TVM-46	1874-1879	
2	Microcline Feldspar	TVM-1 - TVM-63	431-435	
3	Orthoclase Feldspar	TVM-1 - TVM-63	465-469	
		TVM-1 – TVM-63	533 to 538	
		TVM-18	585	
		TVM-1 to TVM-4,TVM-12, TVM-22, TVM-26, TVM-28, TVM-29, TVM-32, TVM-33,TVM-35 to TVM-38,TVM-40,TVM-41 TO TVM-42,TVM-44 TO TVM -46,TVM-48,TVM-49,TVM-53,TVM-54,TVM-57,TVM-60,TVM-61,TVM-62,TVM-63	641-649	
4	Calcite	TVM-13, TVM-35,TVM-37,TVM-39, TVM-40, TVM-41,TVM-43,TVM-61 and TVM-63	875-876	
5	Kaolinite	TVM-1, TVM-2, TVM-4, TVM-5, TVM- TVM-6, TVM-8,- TVM-10, TVM-23-28, TVM-32-34, TVM-36, TVM-38, TVM-40,TVM-49,TVM-52,TVM-57,TVM-63	1007 to 1010	
		TVM-3, TVM-12	1017-1018	
		TVM-1 to TVM-63	1031-1034	
		TVM-1 to TVM-14 TVM-16 To TVM-40	3619-3623	
		TVM-1 to TVM-14 TVM-16 to TVM-40 TVM-42,TVM-43,TVM-46 TVM-48 to TVM-63	3693-3694	
6	Montmorillanite	TVM-1, TVM-3 to TVM-27 TVM-29 to TVM-37, TVM-39 to TVM-53,TVM-55,TVM-57,TVM-58,TVM-60 to TVM-63	3440 to 3442	
7	Illite	TVM-1- to TVM-14, TVM-16 to TVM-17, TVM-19 to TVM-43,TVM-46 TVM-48 to TVM-63	913-915	
		TVM-1,TVM- 2, TVM-5 to TVM-8, TVM-10, TVM-12,TVM-13, TVM-15,TVM-16,TVM-25,TVM-26, TVM-28 To TVM-32, TVM-35, TVM-37, TVM-39,TVM-54,TVM-57.	1635-1640	
8	Organic Carbon	TVM-1 to TVM-6,TVM-7 to TVM-40 TVM-42 to TVM-46 TVM-48 to TVM-63	2922 - 2925	

presence of illite and are Al-O-H stretching vibrations [10]. Illite and kaolinite are the two primary clay minerals found in soils and natural aerosols. Almost all samples contain clay minerals such as kaolinite and montmorillonite. Kaolinite and montmorillonite appear to form as a result of the weathering of a variety of different types of rocks under a variety of different climatic circumstances. Thus, the presence of clay minerals in the samples may imply that they are the predominant minerals of the research area. Table-2 lists the band assignments for the various minerals found in the study area.

Table 2: Band assignments for different minerals of soil samples of Tiruvannamalai district.

Minerals	Frequency (cm^{-1})	Tentative assignments	Reference
Quartz	459	Si - O asymmetrical bending vibration	[6]
	695	Si - O symmetrical bending vibration	[8]
	780	Si - O symmetrical stretching vibration	[9]
	797	Si - O symmetrical stretching vibration	[18]
Feldpar	535	Si - O asymmetrical bending vibration	[10]
	642	Al - O - Co-ordination vibration	[21]
Kaolinte	1030	Si-O Stretching	[22]
	935	O-H deformation	
Montmorlinite	3440	O-H Stretching of absorbed water molecule	[23]
Calcite	875	Fe^{3+} (Al-OH)	[11]

4.1.4. Carbonate Mineral

Carbonates are minerals that contain the anion $(CO3)^{2-}$, such as calcite and aragonite (both born calcium carbonates), dolomite (magnesium/calcium carbonate), and siderite (iron carbonate). As shown in Table 1, the i.r. absorption peaks at,875-880 cm^{-1} are allocated to calcite [11-13],[21-24].

4.1.5. Soil organic carbon

Soil organic carbon (SOC) is a key indicator of soil quality since it has an effect on the variety of organic components and physical qualities of soils [25]. The spectra of all samples reveal the presence of very weak to weak absorption bands between 2920 and 2925 cm^{-1},indicating the presence of organic carbon[10,14,13].The C-H absorption contaminants in the samples are the reason of these bands.

4.2. XRD analysis

The X-ray diffraction technique is applied in this work to determine the soil mineralogy. The XRD pattern of selected representative samples is recorded, and the qualitative mineralogy is determined using the standard XRD interpretation procedures. The observed XRD patterns show that quartz, feldspars, and kaolinite are the predominant constituents of the studied area, while other minerals are minor constituents. This revealed that these were quartz and feldspars are a mineral that is found in abundance in soil samples. The FT-IR investigation confirms the existence of minerals in soil samples, which is strongly supported by XRD analysis.

5 Conclusions

FT-IR and XRD methods were used to qualitatively identify minerals in soil samples. The Fourier transform infrared spectroscopy was used to determine the constituent minerals in soil samples. Quartz, microcline feldspar, orthoclase feldspar, kaolinite, montmorillonite, calcite, and organic carbon were detected in soil samples using FTIR analysis. Quartz, feldspar, and kaolinite are the principal minerals investigated, whereas illite and calcite is trace, based on their intensities of the peaks in the samples. The presence of quartz and feldspar are two minerals are found to occur from the number peaks in the FT-IR spectrum. XRD analysis corroborates the FT-IR study's findings. The bulk mineralogy of soil samples determined by IR and XRD analysis indicates that quartz and feldspar are the dominating minerals, with kaolinte being a trace mineral. The analyses conducted offered valuable information regarding the mineralogy of the soil samples.

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