ISSN NO. 2320-5407

Journal homepage: http://www.journalijar.com

INTERNATIONAL JOURNAL OF ADVANCED RESEARCH

RESEARCH ARTICLE

MINERALOGICAL CHARACTERIZATION OF RIVER BED SOIL FROM TAMILNADU BY FT-IR, XRD AND SEM/EDAX

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Manuscript Info

Manuscript History:

Received: 14 December 2013
Final Accepted: 25 January 2014
Published Online: February 2014

Key words:
Mineralogical composition, FT-IR, XRD, SEM, EDAX and elemental analysis.

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Abstract

River bed soil collected from Suruli Theertham, Theni District, Tamilnadu, India was characterized using Fourier Transform Infrared Spectroscopy (FT-IR), X-ray powder diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDAX) techniques. FT-IR analysis revealed the presence of various functional groups of Si-O, Al-OH, Al-O, Si-O-Al bands which influence the adsorption process. Identification and estimation of mineralogical composition were carried out by XRD. XRD patterns indicate the presence of silicon dioxide and aluminium oxide in major quantities. SEM analysis showed the platy flakes and spongy structure of silica and alumina. Elemental analysis (EDAX) confirmed the presence of Si (20.66 %) in large quantities than the other oxides such as Al (9.76 %), Fe (6.52 %), Ca (3.05 %), Mg (0.59 %), Mn (0.14 %), Na (3.33 %), K (3.66 %) and phosphorous (0.02 %)..

1.1. Introduction:

Soil is a complex of living, changing and dynamic component of earth crust. A thorough understanding of the mineralogy of soil is very important because of their low cost, abundance in most continents of the world, high sorption properties and potential for ion exchange. Soil materials are strong candidates as adsorbents (Preeti and Singh, 2007). Study of the clay minerals present in soil with varying quantities and types depending on the geological environment and discussion of the relations between the layer covering the surface of earth crust and the underlying layers have been utterly important. Clay is a general name for an important mineral group which is used for the production of great number products, encompasses every part of daily life, such as from soil to ceramics and from fine arts to advanced technological industry (Adams, 1987). Physical and chemical properties of the clays make them right material for different purposes in different fields (Murray, 1999). The past few decades have seen increasing use of clays in a wide range of fields such as production from paper industry to ceramics, bleaching of the vegetable oils, beer, wine and fruit juices, cleaning of the radioactive wastes and waste waters and production of drugs, perfumes, soaps, detergents, rubbers and plastics (Murray, 1991; Breen et al., 1997; Falaras et al., 2000). There are several classes of clays such as smectites (montomorillonite, saponite), mica (illite), kaolinite, serpentine, pylophyllite (talc), vermiculite and sepiolite (Shichi and Takagi, 2000). The adsorption capabilities result from a net negative charge on the structure of minerals present in soil. This negative charge gives soil the capability to adsorb positively charged species. Their sorption properties also come from their high surface area and high porosity (Alkan et al., 2004).

Clay is composed mainly of silica, alumina and water, frequently with appreciable quantities of iron, alkali metals and alkaline earths (Preeti and Singh, 2007). Clay materials possess a layered structure and are classified by the differences in their layered structures. Two structural units are involved in the atomic lattices of most clay minerals. One unit consists of closely packed oxygen and hydroxyl in which aluminium, iron and magnesium atoms are embedded in an octahedral combination so that they are equidistant from six oxygens or hydroxyls. The second unit is built of silica tetrahedrons. The silica tetrahedrons are arranged to form a hexagonal network that is repeated indefinitely to form a sheet of composition, $Si_4O_6(OH)_4$ (Ralph 1968). A large number of researchers determined semi-quantitative clay mineral composition on the basis of area under X-ray diffraction peak duly corrected by appropriate factors accounting for variation of scattering due to variation of angle.

The FTIR study of water adsorption was carried out by Stevens and Anderson (1996a). Similarly, the orientations of trimethyl phenylammonium (TMPA) in montmorillonite, as well as its implications in the adsorption of aromatic compounds have been studied by Stevens and Anderson (1996b). Kramer (2000) and Uribe (2000) studied the adsorption behaviour of this type of organoclays. SEM/EDS investigations of clay minerals were performed by Rajkumar and Ramanathan (2012). In recent years, there has been an increasing interest in utilizing clay minerals such as bentonite, kaolinite, diatomite and fullers earth for their capacity to adsorb inorganic materials. They showed that this naturally occurring material could act as a substitute for activated carbon as an adsorbent due to its availability and low cost, and its good sorption properties.

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

1.2. Materials and Methods:

1.2.1. Study area:

Suruli falls is a famous tourist attraction located in Theni District in Tamil Nadu. It is 56 kms from Theni and 10 kms from Cumbum. Madurai is almost 123 kms away from the falls and it lies on the way to Periyar National Park. Suruli falls is located in between 9°39′52″ north latitudes and 77°18′12″ east longitude. Suruli River, the mouth of the falls originates from the Meghamalai Mountain range. The soil samples were collected from Suruli river named as Suruli Theertham, Theni District, Tamilnadu, India was shown in Figure 1.

1.2.2. Soil collection and preparation:

The soil samples were initially sun dried for 7 days followed by drying in hot air oven at 383±1 K for 2 days. The dried soil was crushed and sieved and then stored in sterile, closed glass bottles till further investigation (Das and Mondal, 2011).

1.2.3. Mineralogical and chemical analysis:

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 technique 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 different mineral phases in the samples, X-ray diffractograms for the shreds in the powdered form recorded using PAN ANALYTICAL XPERT PRO equipped with PW 3050/60 Goniometer by operating at 30 kV and 20 mA with CuKα radiation at 25ºC of λ=1.5405 A°. Microstructures were examined by Scanning Electron Microscope (SEM) with JM6701F-6701 model. The chemical composition was determined by an Energy Dispersive X-ray Spectroscopy (EDAX) attached to SEM.

1.3. Results and discussion

1.3.1. Characterization of Soil particle

For understanding the nature of adsorption on soil, EDAX, XRD, FTIR and SEM studies were performed, which are described in the following sections.

1.3.2. EDAX characterization

It was performed to know the chemical compositions of the minerals that are present in the soil. The data given in table 1 shows that the alumina and silica are present in major quantities while other minerals are present in trace amounts. This confirms the chemical analysis of the soil.

EDAX spectrum shows the presence of dominant elements of river bed soil and represented in Figure 2. The result indicated the presence of Silica, Alumina, Iron, calcium, Sodium and Potassium oxide as major constituents, while other oxides such as Magnesium oxide, Manganese oxide and Phosphorus pentoxide are present in smaller amount. Therefore the adsorbate species in solution are expected to be removed mainly by SiO_2 and Al_2O_3.

1.3.3. XRD characterization

X-ray powder diffraction (XRD) method was used to confirm the presence of clay minerals such as kaolinite, illite, quartz and chlorite. The d-values of XRD pattern of soil were estimated compared with standard values of clay minerals supplied by Crystallographic and Crystallochemical Database for Minerals and their Structural Analogues. Comparing the d-values, it can be concluded that the soil is mainly quartz (Silicon dioxide SiO_2) containing small amount of Kaolinite type of mineral (Aluminiumsilicate hydroxide, $Al_2Si_2O_5(OH)_4$), trace amount of Chlorite (Sodium Aluminium Silicate Hydroxide Hydrate, $Na_{0.5}Al_6(Si, Al)_8O_{20}(OH)_{10}.H_2O$) and illite (Potassium Aluminium Silicate Hydroxide, $(K, H_3O)Al_2Si_3AlO_{10}(OH)_2$). XRD line and d-values indicate the soil fraction resembles more to quartz and kaolinite. Fig. 3 shows the XRD pattern of the studied soil.

1.3.4. FTIR characterization

The FTIR spectrum was used to determine the nature of functional groups which could possibly influence the adsorption of the soil. The FTIR spectrum of the soil is shown in Fig. 4 and spectral data of soil with standard data for different bands with different vibrational modes as shown in table 2. In the FTIR studies of the soil, the Si-O stretching vibrations were observed at were observed at 779.2 cm^{-1}, 694.4 cm^{-1}, 540.1 cm^{-1} and 447.5 cm^{-1} showing the presence of quartz (Marel and Bentelspacher, 1976). The appearance of υ (Si-O-Si) and δ (Si-O) bands also support the presence of quartz. A strong band at 3425.5 cm^{-1} and 3699.5 cm^{-1} indicate the possibility of the hydroxyl linkage. However, a broad band at 3425.5 cm^{-1} in the spectrum of soil suggests the possibility of water of hydration in the adsorbent. Most of the bands such as 3699.5 cm^{-1}, 3425.5 cm^{-1}, 1033.8 cm^{-1}, 779.2 cm^{-1}, 694.4 cm^{-1}, 540.1 cm^{-1} and 447.5 cm^{-1} shows the presence of kaolinite (Tuddenham and Lyon, 1960).

The vibration observed at 694.4 cm^{-1} shows the possibility of the presence of calcite (Gadsen, 1975). The presence of bands at 3699.5 cm^{-1}, 3425.5 cm^{-1}, 2368.5 cm^{-1}, 1033.8 cm^{-1}, 779.2 cm^{-1} indicate the possibility of the presence of illite (Wolf, 1963). The presence of chlorite was confirmed by a band at 3425.5 cm^{-1} and the band at 1643.5 cm^{-1} indicates the presence of iron content.

The results showed the mixing of various overtones and complexity of the spectrum of soil incading the presence of Kaolinite, Chlorite, illite, Si-O, Si-O-Al and Quartz.

1.3.5. SEM characterization

SEM picture of soil was taken at 20 kV with 750 and 2400 magnification and presented in Fig. 5. It depicts the platy flakes and spongy structure of the soil.

Table 1 Chemical analysis of soil

Chemical composition	Weight (%)
SiO_2	**20.66**
Al_2O_3	**09.76**
Fe_2O_3	**06.52**
CaO	**03.05**
MgO	**00.59**
MnO	**00.14**
Na_2O	**03.33**

K_2O	**03.06**
P_2O_5	**00.02**

Table 2. Infrared band of soil samples and its assigned mineralogy

Infrared bands cm^{-1}	Assigned minerals
3699.5	Kaolinite
3425.5	Chlorite
779.2	Illite, Si-O
694, 640	Si-O, Si-O-Al
640,570,540	Si-O, Quartz
447	Quartz

1.4. Conclusion

The chemical analysis, EDAX, XRD and FTIR show that soil is mainly constituted of alumina and silica. The FTIR was used over 400-4000 cm^{-1} region to study the vibrational spectrum of the river bed soil mineral structure. The various vibrational modes were attributed to the –OH, Si-O and Al-O bonds. XRD study shows the presence of quartz, kaolinite, chlorite and illite as major phases. The morphology of the soil observed by SEM showed platy flakes and spongy structure.

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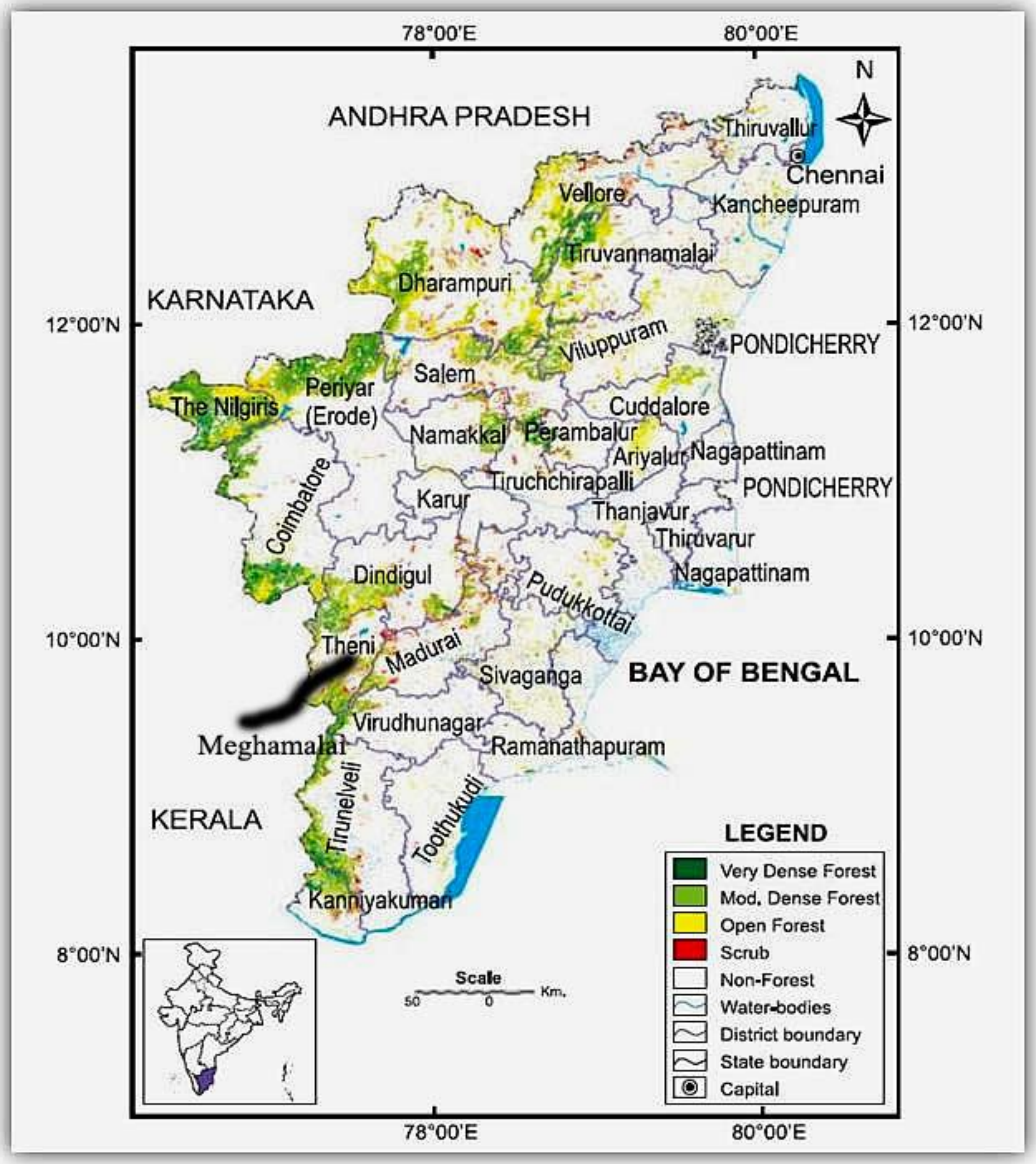

Fig.1 Study area location

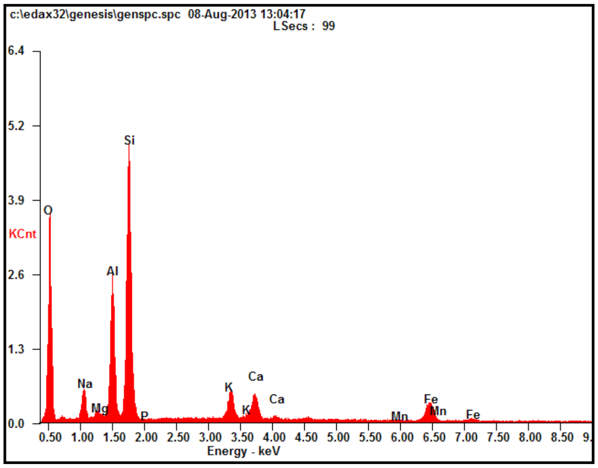

Fig. 2 EDAX spectrum of Soil

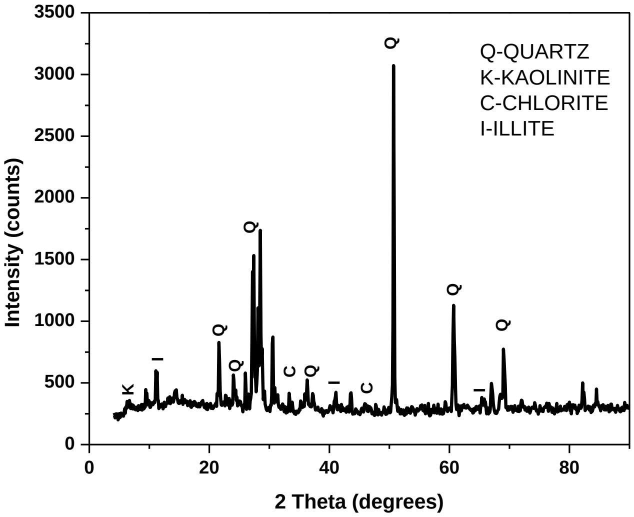

Fig. 3 XRD pattern of Soil

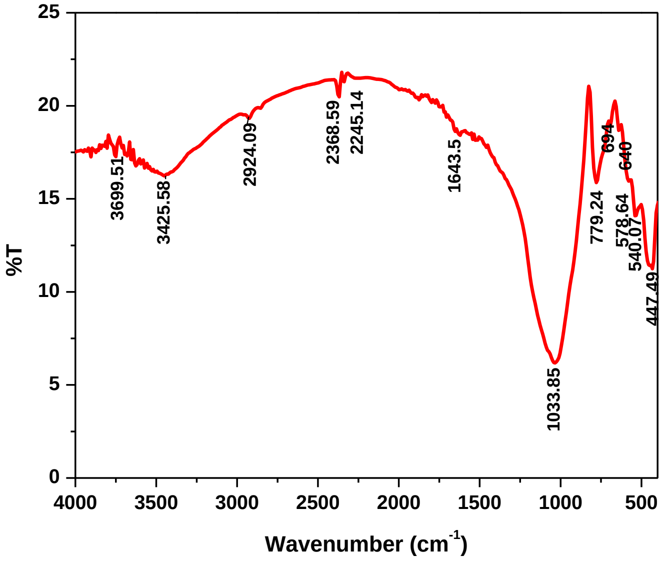

Fig. 4 FTIR spectrum of Soil

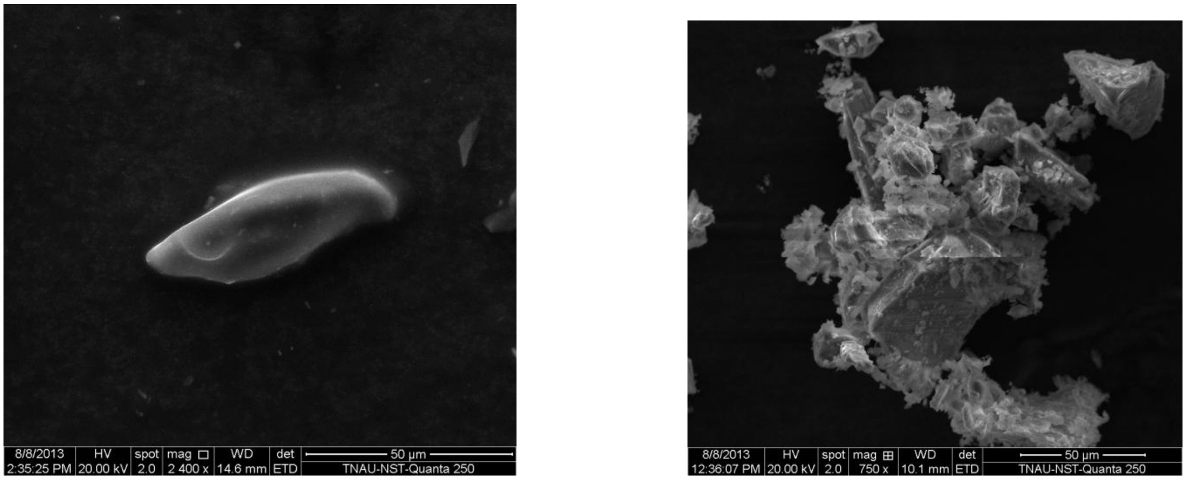

Fig. 5 SEM images of soil at high and low magnification