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Characterization of Riverbed Sand from Mullai Periyar, Tamilnadu by FT-IT, XRD and SEM/EDAX

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The riverbed sand collected from mullai periyar in Gudalur, Theni district, Tamilnadu is subjected to mineral analysis. The mineralogical characterization of the riverbed sand were performed by different techniques such as Fourier transform infrared spectroscopy, X-ray powder diffraction, scanning electron microscopy and energy dispersive X-ray spectroscopy. FTIR studies show the presence of quartz and kaolinite mineral matters whereas X-ray powder diffraction confirms the presence of these minerals in the riverbed sand. Scanning electron microscopy analysis showed the platy flakes and spongy structure of silica and alumina. Elemental analysis (EDAX) confirmed the presence of Si (20.82 %) in large quantities than the other oxides such as Al (6.13 %), Fe (0.75 %), Ca (1.02 %), Na (1.42 %), K (3.42 %) and oxygen (66.44 %).

Keywords: Mineralogical characterization, Riverbed sand, Mulli periyar, Silica, Quartz.

INTRODUCTION

Clay minerals are well known and familiar to mankind from the earliest days of civilization because of their low cost, abundance in most continents of the world. High sorption properties and potential for ion exchange, clay materials are strong candidates as adsorbents. Clay materials possess a layered structure and are considered as host materials. They are classified by the differences in their layered structures. There are several classes of clays such as smectites (montmorillonite, saponite), mica (illite), kaolinite, serpentine, pylophyllite (talc), vermiculite and sepiolite and wide application of natural aluminosilicates and clays in the fields of catalysis like water treatment and desulfonation (petrochemistry) is caused by their surface active groups of different nature: acidic, basic and amphoteric, for example, protons, hydroxyls, SiOH, AlOH-groups, coordinately saturated and unsaturated cations. Clay is composed mainly of silica, alumina and water, frequently with appreciable quantities of iron, alkalies and alkali earths[1]. 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 aluminum, 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, $Si_4O_6(OH)_4$[2].

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. Such clay minerals in soil play the role of natural scavenger by removing and accumulating contaminants in water passing through the soil through exchange adsorption mechanism. The high specific area and the ability of holding water in the interlayer sites gave clays excellent adsorbent capacity[3]. Elemental or chemical analysis is an important step of establishing the nature of minerals such as clay minerals for their structural formulae[4,5]. 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[6]. 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[7]. Kramer[8] and Uribe[9] studied the adsorption behaviour of this type of organoclays. SEM/EDS investigations of clay minerals were performed by

Rajkumar and Ramanathan[10]. Thambavani *et al*.[11] studied the mineralogical composition of river bed sand using XRD, FT-IR and SEM/EDAX. In recent years, there has been an increasing interest in utilizing clay minerals such as bentonite, kaolinite and 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 study encompasses the identification of various clay minerals present in the river bed sand through FT-IR, XRD, SEM and EDAX techniques.

EXPERIMENTAL

The Periyar river originate in Periyar Tiger Reserve (Kerala) and flow through the Theni Forest Division. The present intensive study was carried out at Gudalur Range (23 km 2) located (9° 37' N, 77° 16' E) in southern Western Ghats of Theni forest division (723 km^2), Theni district, Tamilnadu, India. The soil samples were collected from mullai periyar river basin near Gudalur, Theni District, Tamilnadu, India.

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[12].

Mineralogical and chemical analysis: The infrared spectra were recorded in the mid IR region 4000-400 cm^{-1} using shimadzu fourier transform infrared spectrometer (IR-Affinity-1). To identify the different mineral phases in the samples, X-ray diffractograms for the shreds in the powdered form recorded using PANI ANALYTICAL XPERT PRO equipped with PW 3050/60 Goniometer by operating at 30 kV and 20 mA with CuK_α radiation at 25 ºC of λ = 1.5405 Å. Mictrostructures 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 scanning electron microscope.

RESULTS AND DISCUSSION

Energy dispersive X-ray spectroscopy analysis: Energy dispersive X-ray spectroscopy spectrum shows the presence of dominant elements of river bed soil and represented in Fig. 1 (Table-1). The result indicated the presence of silica, alumina, calcium, sodium and potassium oxide as major constituents, while other oxide such as iron oxide is present in smaller amount. Therefore the adsorbate species in solution are expected to be removed mainly by SiO_2 and Al_2O_3.

TABLE-1
CHEMICAL ANALYSIS OF SOIL

Elemental composition	Atomic (%)	Weight (%)
Si	20.82	28.35
Al	6.13	8.02
K	3.42	6.49
Fe	0.75	2.04
Ca	1.02	1.98
Na	1.42	1.58
O	66.44	51.55

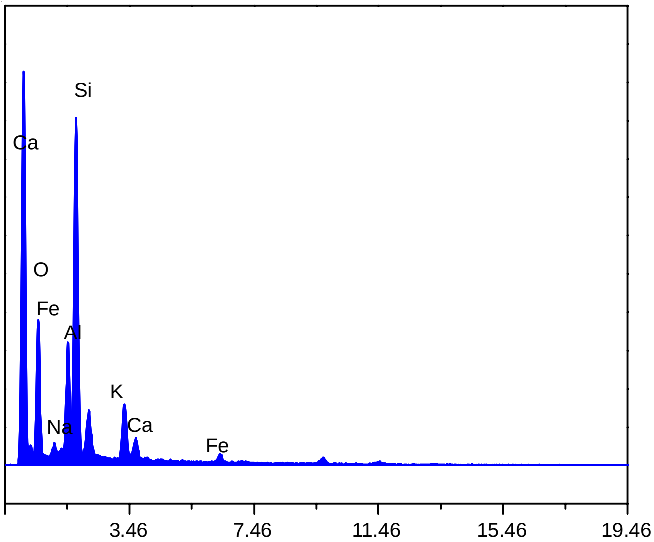

Fig. 1. Energy dispersive X-ray spectroscopy spectrum of riverbed sand

X-Ray diffraction analysis: The presence of above minerals was further tested by XRD studies (Fig. 2). X-Ray diffraction is used to determine the mineralogical composition of the raw material components as well as qualitative and quantitative phase analysis of multiphase mixtures. The occurrences of minerals in clay were identified by comparing 'd' values. No quantitative estimation phases in these adsorbents have been made but their characterization of XRD patterns indicates the presence of quartz (silicon dioxide, SiO_2) and kaolinite (Aluminiumsilicate hydroxide, $Al_2Si_2O_5(OH)_4$) as the major phases. Further the occurrence of the above minerals in the riverbed sand adsorbents was confirmed by FTIR study.

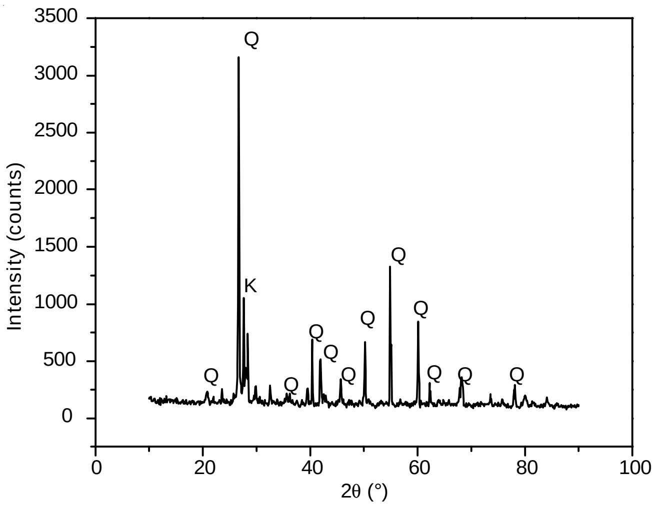

Fig. 2. X-Ray diffraction pattern of riverbed sand

Fourier transform infrared spectroscopy analysis: To elucidate the components of riverbed sand, the FT-IR spectroscopy was used and the spectrum is shown in Fig. 3 and spectral data of soil with standard data for different bonds 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 789.2, 658.9, 543.7 and 464.5 cm^{-1} showing the presence of quartz[13]. The appearance of ν(Si-O-Si) and δ (Si-O) bands also support the presence of

TABLE-2
INFRARED BAND OF SOIL SAMPLES AND ITS ASSIGNED MINERALOGY

Functional group	Standard	Riverbed sand (cm^{-1})	Assigned minerals
O-H (Stretching vibration)	3710-3200	3627	Kaolinite
Si-O (plane bending)	1150-900	1446, 1041.5	Si-O
Al-OH (bending)	900-600	789.2	Si-O, Si-O-Al
Fe-O (bending)	490-460	464.5	Si-O, Quartz
Al-O-Si (skeletal vibration)	550-450	543.7	Quartz

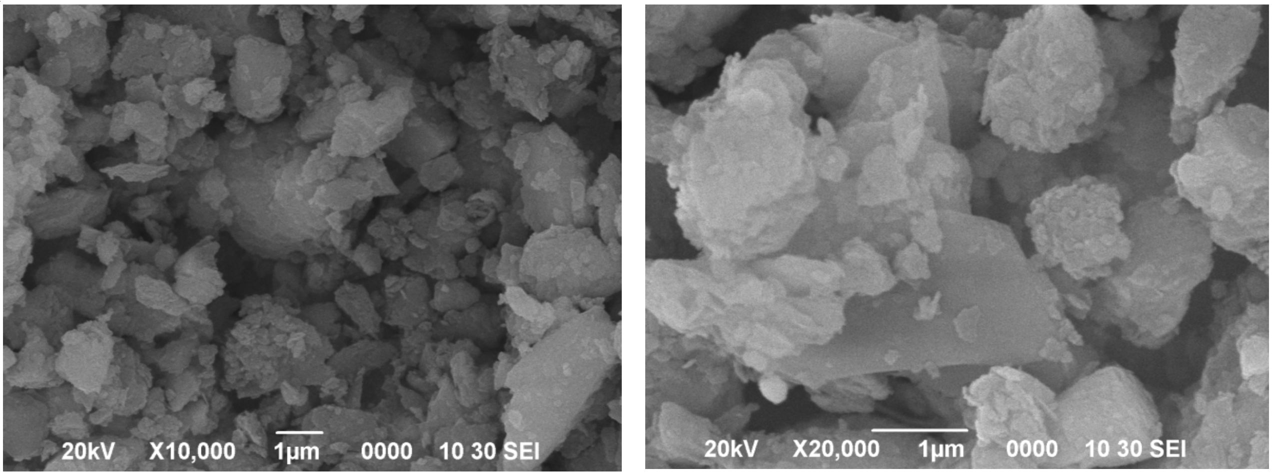

Fig. 4. Scanning electron microscope image of riverbed sand with low and high magnification

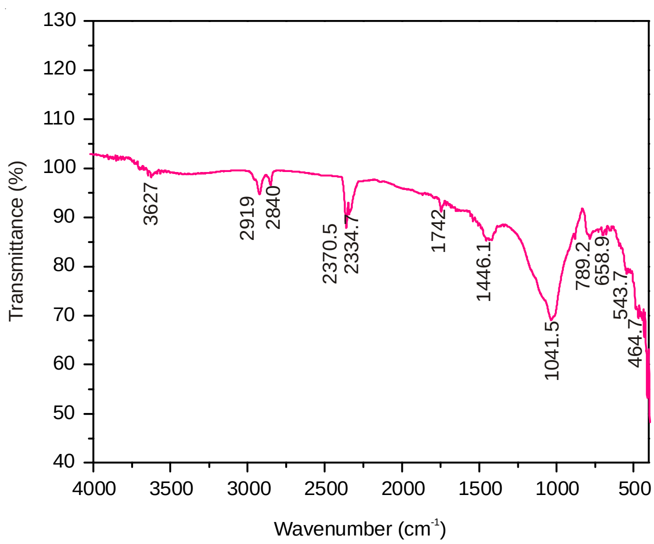

Fig. 3. FT-IR spectrum of riverbed sand

quartz. A strong band at 3627 cm^{-1} indicates the possibility of the hydroxyl linkage. However, a broad band at 3627 cm^{-1} in the spectrum of soil suggests the possibility of water of hydration in the adsorbent. Most of the bands such as 3627, 1446, 1041.5, 789.2, 658.9, 543.7 and 464.5 cm^{-1} shows the presence of kaolinite[14].

Scanning electron microscope analysis: Scanning electron microscope picture of soil was taken at 20 kV with 10000 and 20000 magnifications and presented in Fig. 4. It depicts the platy flakes and spongy structure of the soil.

Conclusion

The chemical analysis, EDAX, XRD and FTIR show that soil is mainly constituted of alumina and silica. The FTIR was used over 4000-400 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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