



Characterization of Sand Fractionated from Bijoypur Soil, Bangladesh and its Application as an Adsorbent

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Abstract

Sand was obtained from the fractionation of Bijoypur soil of Bangladesh. Different advanced techniques such as SEM (Scanning electron microscope), LIBS (Laser induced breakdown spectroscopy), FT-IR (Fourier transform infra-red spectroscopy), XRD (X-ray diffraction) were used to characterize the physical and chemical nature of sand. The adsorptive property of sand toward methylene blue (MB) was also investigated in batch process. The SEM micrographs in different magnifications show that the surface of sand is heterogeneous in nature and the particle's sizes are almost uniform (particle size $\geq 140 \mu\text{m}$). LIBS analysis confirmed the presence of Fe, Si, Ti, Cu, F, I, Tc, Ni, Pu and Na in sand. Sand fraction thus obtained was mainly quartz, which was supported by XRD analysis. FT-IR spectral analysis showed the presence of Si-O bond in sand. The adsorptive property of sand toward MB showed that about 24% ($\approx 0.28 \text{ mg/g}$) of MB adsorbed on sand within 90 min. of contact time at neutral pH of solution and ambient temperature.

Keywords: Sand, Characterization, SEM, LIBS, FT-IR, XRD, Adsorption, Methylene blue.

Introduction

Soil can be fractionated to different categories¹⁻³. Based on particle size, three different fractions as sand, silt and clay can be obtained from soil. Sand is a naturally occurring granular material composed of finely divided rock and mineral particles. The composition of sand is highly variable, depending on the local rock sources and conditions, but the most common constituent of sand in inland continental settings and non-tropical coastal settings is silica (silicon dioxide, or SiO_2), usually in the form of quartz⁴. Sand is transported by wind and water and deposited in the form of beaches, dunes, sand spits, sand bars and related features. Sand may be obtained in different grain size in environments such as gravel-bed rivers and glacial moraines⁵. Sand is used for many purposes in practical life e.g. sandy soils are ideal for crops such as watermelons, peaches and peanuts, and their excellent drainage characteristics make them suitable for intensive dairy farming, Sand is principal raw materials of glass etc. Sand's many uses require a significant dredging industry; raising environmental concerns over fish depletion, landslides, and flooding⁶. Sand is generally non-toxic but sand-using activities such as sandblasting require precautions. Bags of silica sand used for sandblasting now carry labels warning the user to wear respiratory protection to avoid breathing the resulting fine silica dust⁷. Material safety data sheets (MSDS) for silica sand state that "excessive inhalation of crystalline silica is a serious health concern"⁸. Soil and its fractions can be characterized by several analytical techniques e.g. elemental analysis, surface charge/surface pH, phase and molecular analysis etc. Bijoypur soil was dried at 120°C and calcined at 500°C, and characterized by FT-IR, TGA/DTA and XRD⁹. In our previous studies, SEM, LIBS,

FT-IR, XRD were used to characterize Clay and Silt obtained from Bijoypur soil¹⁰⁻¹¹.

The objective of present investigation was to characterize sand fraction of Bijoypur soil by different physical methods. Surface morphology and particle size was determined by SEM, elemental analysis done by LIBS, bond pattern and vibrational modes were analyzed by FT-IR, and phase analysis done by XRD. Typical adsorption experiment was performed to determine sand's adsorption capacity as adsorbent for MB.

Material and Methods

Preparation of sand: Sand was fractionated from Bijoypur (Netrokona) soil following Hydrometer method¹.

SEM analysis: A small portion of fractionated sand was separately taken in a SEM sample holder and made it platinum coated using a Pt-coated auto system (JFC-1600, JEOL, Japan). Platinum coated sand was placed in the SEM sample chamber and SEM picture was taken at 20 kV under 1,000 and 2,000 magnification.

LIBS analysis: The prepared sand was air dried for several days. Then these were crushed and grounded for making powder. The powder samples were stored in plastic jar. For LIBS Analysis, about 0.5-1.0 g sand was mixed with 1-2 drops of glue and small pellet was made. This was air dried before use. LIBS spectroscopy can be produced from high intensity laser pulse (Nd-YAG laser) interacting with the sample producing a plasma plume that evolves with time from the point of impact of the incident laser pulse. The laser pulse usually lasts for 5 to 20 ns.

LIBS spectra were taken using different spectral ranges for two gratings, One of them was 2400 ruling/mm grating blazed at 240 nm using spectral range 200-350 nm and the other was a grating with 600 ruling/mm blazed at 500 nm using spectral range 350-900 nm.

FT-IR analysis: FT-IR spectra of sand were taken from the Analytical research laboratory, Department of Chemistry, University of Dhaka. This was carried out by Shimadzu (IR Prestige 21) FT-IR spectrometer equipment using potassium bromide pellet. The pellet was prepared by mixing 1.0 mg of finely ground dry sample and 200.0 mg of spectroscopic grade dry KBr. The mixture was grounded thoroughly in an agate mortar and pressed between a pair of special dies for 5 minutes under a pressure of 8-9 tons using hydraulic press connected with vacuum pump for removal of CO₂. The spectra were recorded between 4000-500 cm⁻¹ with 2 cm⁻¹ resolution.

XRD analysis: X-ray powder pattern of sand sample was recorded with a Philips PW-1380 X-ray generator operating at 40 kV and 30 mA and an XDC-700 Guinier- Hagg focusing camera using CuK_{α1} radiation. An exposed time of 1 hour was used and the films were processed using commercially available developer and fixer. The d-values of the diffraction lines in the films were calculated manually.

Adsorption capacity: The adsorption experiment was carried out at pH 7.0 on MB (Janssen chemicals, Belgium) of concentration 5.0×10⁻⁵ M at 30°C. 0.3125 g of sand was taken in each of 6 bottles containing 40 mL MB solution. The bottles were shaken in a thermostatic mechanical shaker (SWB-20, HAAKE, Fisons Ltd, Germany). After a definite interval of time

each bottle was withdrawn from the shaker. The supernatant of the bottle was transferred and centrifuged repeatedly until a clear liquid was obtained. The absorbance of the clear solution was measured spectrophotometrically at λ_{max} 663.0 nm by UV-Visible recording spectrophotometer (UV-1650 PC Shimadzu, Japan). In all cases, the pH of the solution was adjusted before and after the adsorption by using acid or alkali without affecting the volume of the solution.

Results and Discussion

Surface morphology: The surface of sand was analyzed by Scanning Electron Microscopy (SEM). Figure 1 shows the SEM micrograph of sand by two different magnifications at 1000 and 2000. There were some pores or cavities in surface which dictate the surface of sand is heterogeneous and can act as an adsorbent like clay of Erzurum, Turkey¹² or clay of Bijoypur, Bangladesh¹³ or silt of Bijoypur, Bangladesh¹⁴. SEM micrographs of clay¹⁰ and silt¹¹ fractions of Bijoypur soil showed heterogeneous surface and effective adsorbents. Particle size of sand is higher than 100 μm which is higher than the particle size of clay and silt, suggesting that sand's adsorptive properties may be less than clay or silt due to less surface area.

Elemental analysis: LIBS were used to analyze the presence of elements in the sand. Table 1 shows the list of elements present in the sand sample according to NIST's Atomic Spectra Ref. Data¹⁵. The LIBS spectra of sand when compared with that of clay¹⁰ and silt¹¹ fractions of Bijoypur soil, many emission lines for identification of elements were detected and additional lines for F, I, Tc, Pu and Ni were also observed which were not found in clay¹⁰ or silt¹¹.

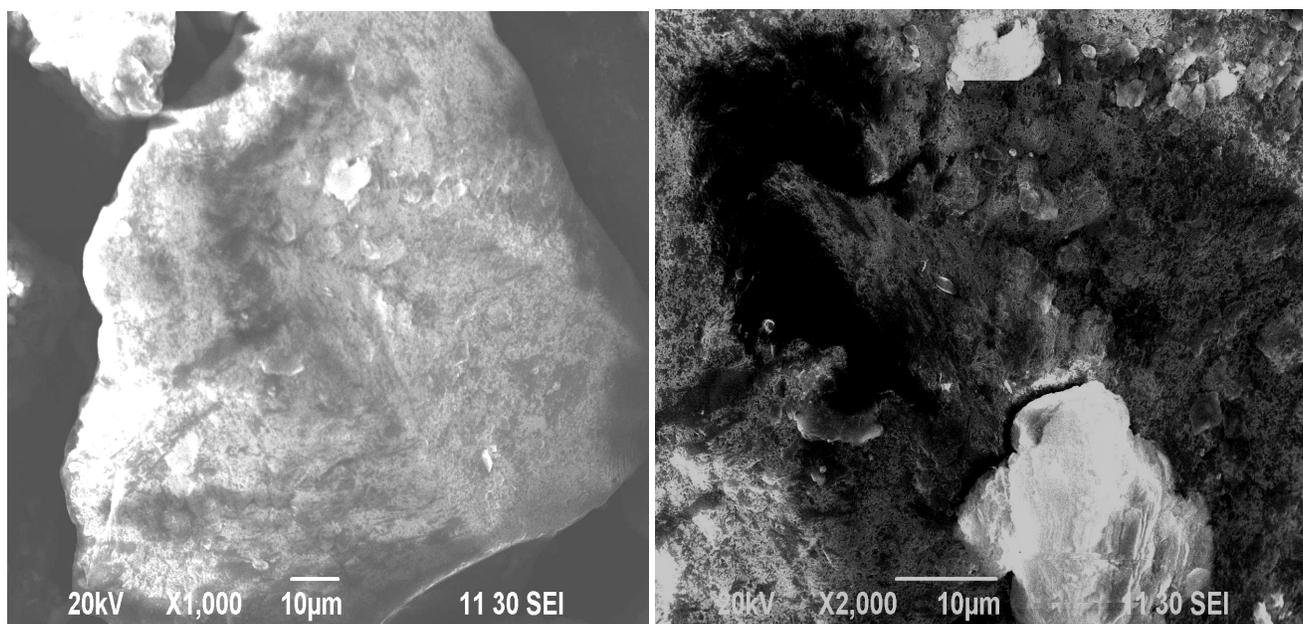


Figure-1
SEM micrograph of sand with 1000 and 2000 magnifications

Bond patterns and vibrational mode analysis: Different vibrational modes of bonding of sand were investigated using FTIR spectroscopy. Figure 2 shows the FT-IR spectrum of fractionated sand. Respective vibrational modes for different functional groups were analyzed with standard FT-IR data¹⁶⁻¹⁷ presented in table 2. FTIR analysis of sand, clay¹⁰ and silt¹¹ of Bijoypur soil when compared, sand spectra were found to contain only peaks for the Si-O plane bonding vibrational mode. So we can infer that sand fraction of Bijoypur soil is quartz.

Phase analysis of sand by XRD: X-Ray powder diffraction (XRD) method was also used to compare the sand with several

clay minerals such as kaolinite, illite, quartz and chlorite. The *d*-values of XRD pattern of sand were estimated and compared with standard *d*-values of different clay minerals supplied by JCPDS (Joint Committee on Powder Diffraction Standards)¹⁸. The values are shown in table 3. It can be concluded from this table that the sand is mainly quartz, (Silicon oxide, SiO₂) containing very poor amount of Kaolinite type of mineral (Aluminum silicate hydroxide, Al₂Si₂O₅(OH)₄), trace amount of Chlorite, (Sodium Aluminum Silicate Hydroxide Hydrate, Na_{0.5}Al₆(Si, Al)₈O₂₀(OH)₁₀·H₂O) and Illite, (Potassium Aluminum Silicate Hydroxide, (K, H₃O)Al₂Si₃AlO₁₀(OH)₂. *d*-values of sand closer to that of silt¹¹ than clay¹⁰.

Table-1
Characteristic emission lines of elements present in sand

No	Element	Charge state*	Characteristic emission lines (wavelength, nm) for identification of the elements
1	Si	I	212.375, 221.629, 243.472, 251.424, 251.910, 288.156, 298.795, 742.201
		II	505.622
2	Ti	I	322.620, 323.412, 334.816
		II	322.620, 323.412, 334.816
3	Fe	I	251.610, 252.843, 302.016, 358.038
		II	233.263, 234.324, 238.159, 259.942, 263.126, 261.172, 273.936
4	Cu	I	766.332
5	Na	I	588.888, 589.492, 285.223
6	F	I	567.892, 824.092
7	I	I	395.994, 769.746, 746.687
8	Tc	I	280.274, 715.677
9	Ni	I	220.760, 300.199, 334.816
10	Pu	I	309.261, 795.060

* I and II imply neutral and singly ionized states of the atoms respectively.

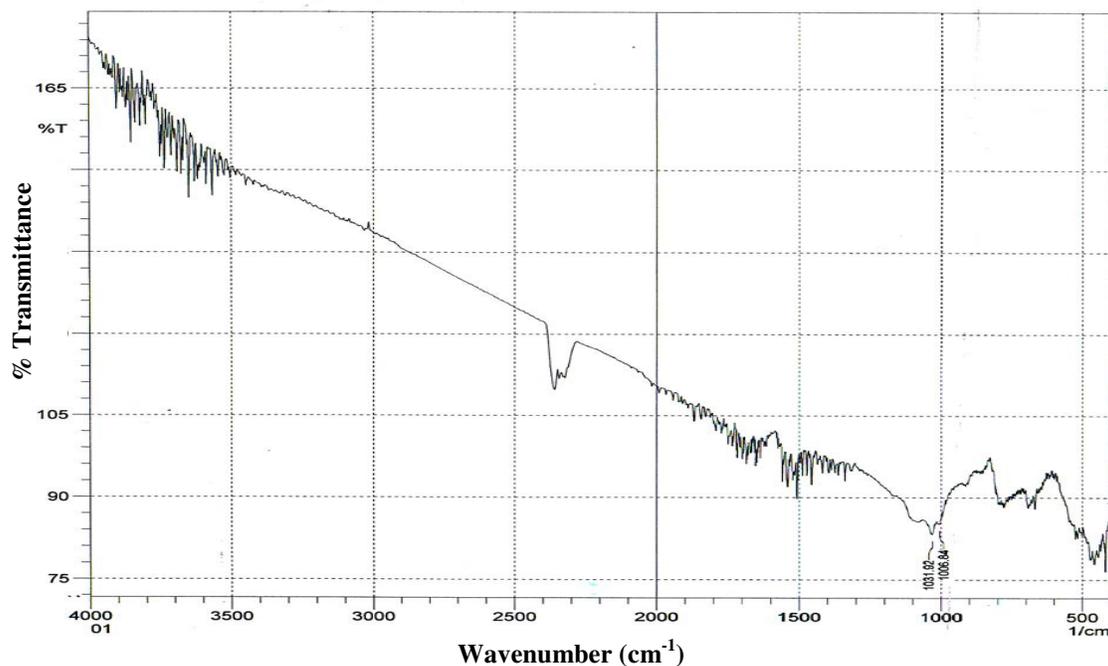


Figure-2
FTIR spectrum of sand fraction from Bijoypur soil

Sand as adsorbent: The adsorption property of sand for methylene blue (MB) was investigated. Figure 3 presents the result of typical adsorption experiment on MB using 0.3125 g of sand at 30°C and pH 7.0. Amount of MB adsorbed on sand surface is increasing with time. But this process is not rapid as considered with clay¹³ (equilibrium time 60 minutes) or silt¹⁴ (equilibrium time 90 minutes). After 90 minutes of adsorption

time, 24% of MB was adsorbed on sand whereas at same conditions 65% and 99% of adsorption was found on silt¹⁴ and clay¹³, respectively. About 0.28 mg of MB was adsorbed on per g of sand which is very low in comparing with clay¹³ (6.93 mg/g) or silt¹⁴ (1.34 mg/g) adsorption capacity. But sand's adsorption efficiency is lower than clay¹²⁻¹³ or silt¹⁴.

Table-2
FTIR analysis for sand fraction from Bijoypur soil

Functional group	Standard	Sand	Comment
O-H (stretching vibration)	3710-3200	-	O-H not present
Zn=O (plane bending)	1650-1350	-	Zn=O not present
Si-O (plane bending)	1150-900	1031, 1006	Si-O present
Al-OH (bending)	900-600	-	Al-OH not present
Fe-O (bending)	460-490	-	Fe-O not present
Al-O-Si (skeletal vibration)	550-450	-	Al-O-Si not present

Table-3

X-ray data of sand along with the data from JCPDS for kaolinite, quartz, chlorite, Illite, clay (Bijoypur) and silt (Bijoypur)

d-values of sand (Bijoypur) A°	d-values of silt ¹¹ (Bijoypur) A°	d-values of clay ¹⁰ (Bijoypur) A°	d-values of Kaolinite A°	d-values of Quartz A°	d-values of Chlorite A°	d-values of Illite A°
4.30	4.25	7.08	7.10	4.32	7.70	10.0
3.34	3.35	4.25	4.41	3.38	4.78	5.02
2.48	2.47	3.35	3.56	2.50	4.44	4.48
2.27	2.28	2.58	2.55	2.30	3.50	4.44
2.22	2.23	2.43	2.49	2.16	2.56	3.46
1.99	2.13	2.28	2.43	2.01	2.50	3.34
1.84	1.98	2.11	2.38	1.84	2.34	3.20
1.69	1.82	1.82	2.33	1.70	1.98	2.99
1.54	1.68		2.20	1.57	1.66	2.56
1.43	1.55		1.98			2.00
1.38	1.45		1.79			1.49
1.27	1.39		1.67			
	1.38		1.66			
	1.29		1.54			
	1.26		1.49			

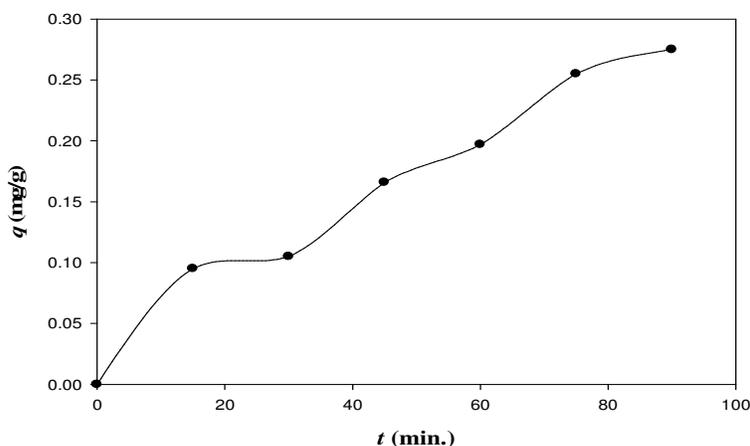


Figure-3
Adsorption of MB on sand fraction from Bijoypur soil at pH 7.0

Conclusion

Sand obtained from fractionation of Bijoypur soil contains many elements. SEM micrographs of sand clearly indicated the heterogeneity of surface and the particle size of silt is higher than 100 μm . LIBS spectra show the presence of different elements like Fe, Si, Ti, Cu, F, I, Tc, Pu, Ni and Na. Si-O bond observed in FT-IR spectra, suggests that sand of Bijoypur is quartz. XRD lines and d -values suggest that sand fraction is mainly quartz and small proportion of kaolinite. Adsorption experiment suggests that sand is poor adsorbent for MB than clay (Bijoypur) and silt (Bijoypur). Adsorption capacity of sand was 0.28 mg/g.

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