Kochi Chapter

Indian Geotechnical Conference
IGC 2022
15th – 17th December, 2022, Kochi

Mineral and Chemical Characterisation of Geosynthetic Clay Liner Bentonite

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Abstract. Geosynthetic clay liner (GCL) is commonly used in cover system and bottom lining of waste containment facilities due to very low hydraulic conductivity of GCL bentonite. The GCL is consisting of thin layer of bentonite supported by geotexiles and/or geomembranes and held together by needle punched or stitch bonded fibres. For effective functioning of cover system, GCL must be hydrated and retained moisture/water. For retaining moisture within GCL, mineral and chemical composition of bentonite core of GCL needs to be investigated critically. Hence, in the present study, mineralogical and chemical characterization of bentonite core of a commercially available GCL were studied using X-ray diffraction**,** ion chromatography, Fourier transform infrared spectroscopy, scanning electron microscope, cation exchange capacity, specific surface area and swelling of bentonite. The X-ray diffraction of bentonite indicates the presence of primary mineral montmorillonite and minor minerals such as quartz, dolomite, feldspar and cristobalite. The interlayer cations of bentonite obtained from ion chromatography mainly consists of Na^+ and some traces of NH_4^+, K^+, $Ca^{2+}$and Mg^{2+} while Fourier transform infrared spectroscopy provides the detail of molecular interaction corresponds to stretching or bending vibration of particular bands and the positions of peaks. Cation exchange capacity, specific surface area and swelling index of bentonite were found to be 73.42 meq/100g, 674.84 m^2/g and 21 ml/2g respectively.

Keywords: Geosynthetic clay liner, Bentonite, Characterisation, Mineral

1 Introduction

Geosynthetic Clay Liners (GCLs) are commonly used in cover system and bottom lining system of waste containment facilities due to very low hydraulic conductivity of GCL bentonite (<10^{-10} m/s) [1]. The primarily functions of cover system and bottom lining system are to reduce infiltration of rainwater into the landfill, limit biogas escape into the atmosphere and mitigate leachate to ground water table respectively [1, 2-6]. GCLs are commercially manufactured hydraulic barrier consisting of thin layer of sodium/calcium bentonite (5-10 mm thick) sandwiched by geotextiles and /or geomembrames and bonded by needle punched or stitch bounded fibres. GCLs are employed in place of compacted clay liner (0.6-1.5 m thick) due to superior hydraulic performance, easy to install, better self-healing property and sustainable to distortion [1, 7-8]. The GCL of cover system absorbs moisture/water from the foundation soil and from top soil [9-12]. For effective functioning of cover system, the GCL must be hydrated and retained moisture/water within the GCL. For retaining moisture within GCL, mineralogical and chemical composition of bentonite core of GCL plays a primarily role.

Various researchers have studied mineralogical and chemical characterization of GCL bentonite. For example, mineralogical characterization of GCL bentonite had been studied by conventional X-ray diffraction (XRD) [13]. Lange et al. [14] characterized the mineral and chemical composition of GCL bentonite at micro level using micro-analytical techniques like conventional XRD, µXRD, micro X-ray fluorescence (µXRF), synchrotron based µXRD (S- µXRD) to investigate relation between trace elements and clay.

To understand the retained moisture within the GCL, mineralogical and chemical composition of bentonite core of GCL further needs to be investigated. In the present study, the mineral and chemical characterization of bentonite core of a commercially available GCL were investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM)**,** ion chromatography, Fourier transform infrared spectroscopy (FTIR), cation exchange capacity, specific surface area and free swelling index.

2 Materials and Methods

2.1 Geosynthetic clay liner

Geosynthetic Clay Liner (GCL) used in the current study was consists of granular sodium bentonite sandwiched between woven (carrier) and non-woven (cover) geotextiles bonded by needle punched fibres. Mass per unit area of GCL (M_{GCL}) was measured in accordance with [15]. Mass per unit area of bentonite core of GCL ($M_{Bentonite}$) was obtained by subtracting the mass per unit area of geosynthetics from the mass per unit area of GCL. Mass per unit area of geosynthetics was measured according to [16]. The properties of GCL and its bentonite core are tabulated in Table 1.

Table 1. Properties of geosynthetic clay liner and bentonite core

Parameter	Value
M_{GCL} (g/m^2)	5283.40
$M_{Bentonite}$ (g/m^2)	4890.40
$M_{Carrier\ GT}$ (g/m^2)	193.40
$M_{Cover\ GT}$ (g/m^2)	199.60
Thickness (mm), at as received water content	6.74
Properties of Bentonite core	
Liquid limit (%)	394.93
Plastic limit (%)	55.39
Specific gravity	2.76
As received water content (%)	12

2.2 Methods

The test methods used to examine mineral and chemical composition of bentonite core of GCL were performed at various laboratories of Indian Institute of Technology Kanpur, Kanpur, India. The details of sample preparation and testing procedures are presented below.

X-ray diffraction

Phase identification of GCL bentonite was obtained utilizing X-ray diffraction (XRD). Fig. 1a shows X-ray diffractometer device (Rigaku MiniFlex 600) used in present study. The dry bentonite powder was placed on a glass slide and few drops of acetone was added to bentonite so that bentonite was glued to glass slide. The glass slide was fixed at sample holder of X-ray diffractometer apparatus. The sample and detector were rotated continuously to obtain the intensity values (counts per second) at various scattered angles (2θ).

Scanning electron microscopy

Surface morphology and elemental analysis of bentonite core of GCL were conducted utilizing scanning electron microscopy (SEM) coupled with energy dispersive X-ray (EDX). Fig. 1b shows the scanning electron microscope device (FEI Quanta 200). The oven-dried bentonite powder was carbon coated and installed in a scanning electron microscope device to record the image and elemental composition at different points.

(a) (b)

Fig. 1. (a) XRD device and (b) Scanning electron microscope device.

Ion chromatography

The concentrations of ionic species, i.e., cations and anions of bentonite core of GCL were measured by ion chromatography. The concentration of ionic species was measured by separating them based on their interaction with a resin. The bentonite extract solution was mixed with buffer solution and loaded in the reservoir of ion chromatography instrument. The buffer solution was passed through a pressurized chromatographic column, where the ions are absorbed by the column constituents. Finally, the elution buffer solution (i.e., elution buffer is a solvent used to washing) was passed through the column, the absorbed ions separated from the column and measured using detector of ion chromatography.

Fourier transform infrared spectroscopy

Chemical bonds and composition of bentonite core of GCL were examined using Fourier transform infrared (FTIR) spectroscopy. The bentonite solution was loaded in the FTIR spectroscopy and an infrared radiation was passed through the solution. A part of infrared radiation was absorbed by the solution while other was transmitted through the solution. The transmitted radiation was recorded by interferometry of spectroscopy device and a spectrum was obtained that represents the molecular fringerprint of bentonite sample.

Cation exchange capacity, specific surface area and free swell index

Cation exchange capacity (CEC) and specific surface area (SSA) of bentonite core of GCL were evaluated utilizing methylene blue adsorption test [17]. For determining CEC value of bentonite, two gram oven dried bentonite passed through 425 µm sieve was added in 100 ml distilled water and mixed uniformly to form bentonite slurry. The methylene blue (MB) solution for titration was prepared by mixing a known amount of MB powder (generally 1 gram) with distilled water (100 ml). The MB solution at a concentration of 10g/l was then poured into a burette. 1 to 2 ml MB solution were added in the bentonite slurry and then stirred. After mixing thoroughly, a drop of bentonite slurry and BM mixture was put on a clear filter paper for visual inspection. A faint blue patch of solids was seen but sometimes, it may be surrounded by a translucent water ring of clear liquid. The process of adding MB solution to the bentonite slurry was continued till the transparent outer ring turns into a diffuse light blue-green glow, radiating outward. At this point, addition of MB solution was stopped and then the drop test on filter paper was repeated for five times. If the glow was observed in all the five cases, then it can be inferred that endpoint was reached; if not, addition of BM solution needs to be continued. The CEC is determined as

$$CEC = \frac{100}{w_M} \times V_{MB} \times N_{MB} \tag{1}$$

where, CEC is cation exchange capacity (meq/100g), w_M is weight of bentonite to make bentonite slurry (g), V_{MB} is volume of MB solution added for titration (ml) and N_{MB} is normality of the MB solution (eq/L), which is expressed as

$$N_{MB} = \frac{M_{MB}}{M \times V_W} \times \frac{100-X}{100} \tag{2}$$

where, M_{MB} is weight of MB used to make a solution (g), V_W is volume of water to make MB solution (ml), X is water content of BM (%) and M is molecular weight of MB (g/mole).

The SSA of bentonite core of GCL was determined using methylene blue adsorption test as described aforementioned. The SSA is calculated as

$$SSA = \frac{1}{M} \times \frac{M_{MB}}{V_W} \times V_{MB} \times A_V \times A_{MB} \times \frac{1}{W_M} \quad (3)$$

where, V_{MB} is volume of MB solution added (ml), A_v is Avogadro number (6.02×10^{23}), A_{MB} is area covered by one methylene blue molecule $[(130A^{\circ})^2]$ (m^2), $A°$ is one Angstrom (10^{-10} m). The free swell index of bentonite core of GCL was determined in accordance with [18].

3 Results and Discussion

The XRD spectra of bentonite was analyzed using MDI JADE software and the results are shown in Fig. 2. It indicates the presence of primary minerals like montmorillonite (M), and minor minerals like quartz (Q), dolomite (D), feldspars (F) and Cristobalite (C). The montmorillonite mineral appears at a position (2θ) of 7.12°, 19.84°, 35.7°, 50.28° and 61.7° with distances of 12.4, 4.47, 2.51, 1.81 and 1.50Å respectively. Similarly, quartz mineral appears at 28.54° with 3.12Å, whereas dolomite and feldspar were positioned at 33.25° with 2.69 Å, and 23° with 3.86 Å, respectively.

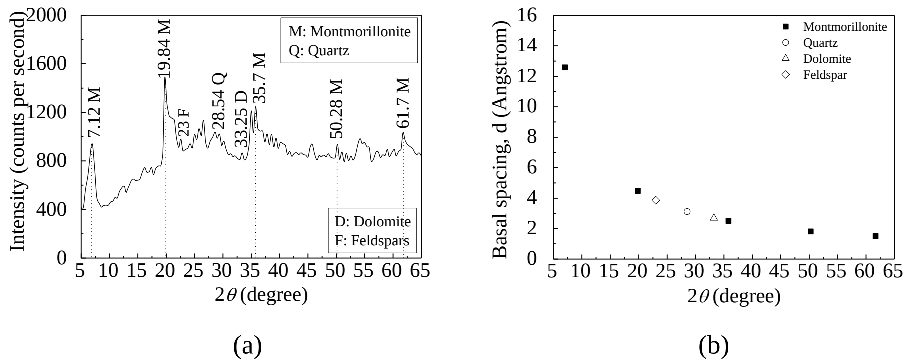
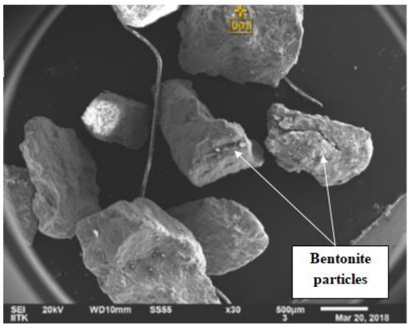
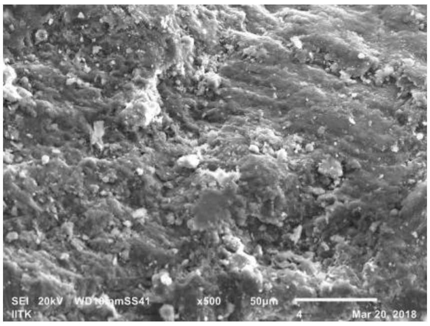


Fig. 2. (a) XRD spectrum and (b) Basal spacing of bentonite core of GCL.

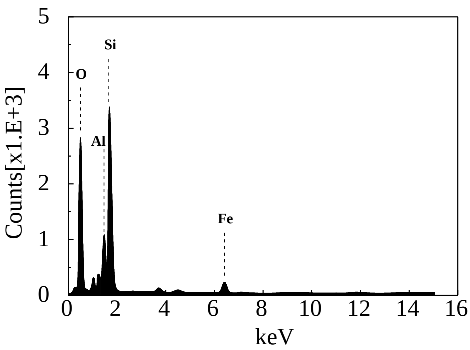
The SEM image of bentonite obtained from scanning electron microscopy are illustrated in Fig. 3a and b. SEM image at 30x magnification indicates that bentonite particles are discrete in nature. The elemental composition of at point 001 (shown in Fig. 3a) is shown in Fig. 3c. It indicates the presence of major elements such as oxygen, Aluminium, Silicon and iron. Similar elemental observation was observed at other points.



(a)



(b)



(c)

Fig. 3. SEM image at (a) 30X magnification (b) 500x magnification and (c) EDX spectrum (at point 001) of bentonite core of GCL.

The interlayer cations of bentonite core of GCL botained from ion chromatography mainly consists of Na^+ (49.97 meq/100g) and traces of NH_4^+(0.68 meq/100g), K^+(0.17 meq/100g), Ca^{2+}(0.59 meq/100g) and Mg^{2+}(0.09 meq/100g).

Fourier transform infrared (FTIR) spectroscopy provides information about chemical bonds and composition. It also offers information on how chemical interaction affects the stretching or bending of certain bands of vibration and the locations of their peaks. [17]. Fig. 4 shows FTIR spectra of GCL bentonite. It was observed that structural OH stretching vibrations present from wavelength 3850 cm^{-1} to 2850 cm^{-1}, whereas Si-O stretching vibration and OH bending modes appear from wavelength 1250 cm^{-1} to 400 cm^{-1}. The peak at 3619 cm^{-1} represents Al_2OH stretching while peak at 3442 cm^{-1} refer to OH stretching of H-bonded water of pure bentonite. Peak at 1641 cm^{-1} represents OH deformational mode of water. Si-O-Si and Al-O-Si stretching peaks of tetrahedral layer observed at wavelength of 1033 cm^{-1} and 524 cm^{-1} respectively. OH bending peaks of octahedral layer appear at wavelength of 914 cm^{-1} and 875 cm^{-1}. The obtained observations are in agreement with published results in the literature [19, 20].

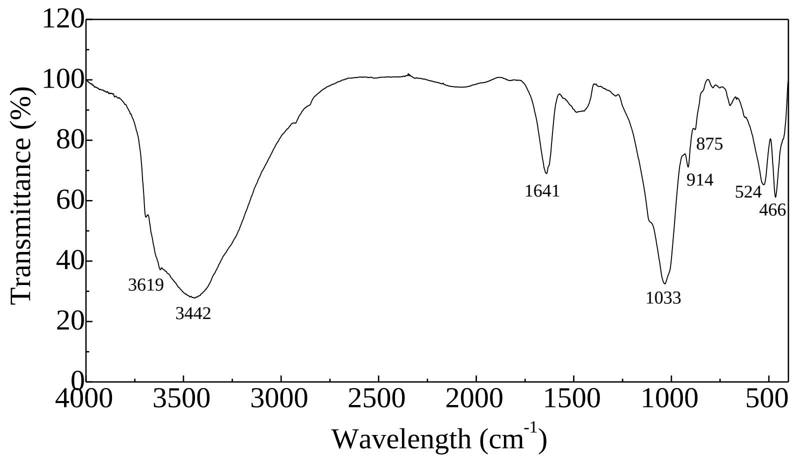


Fig. 4. Variation of tranmittance with wavelength of bentonite core of GCL

The CEC, SSA and free sell index of bentonite core of GCL are shown in Table 2. The CEC, SSA and free swell index of bentonite core were obtained 73.42 meq/100g, 674.84 m^2/g and 21 ml/2g respectively.

Table 2**.** Properties of bentonite core of geosynthetic clay liner

Parameter	Value
CEC (meq/100g)	73.42
SSA (m^2/g)	674.84
Free swell (ml/2g)	21

4 Summary and Conclusions

The mineralogical and chemical composition of bentonite core of GCL were studied using X-Ray diffraction, Ion chromatography, Fourier transform infrared spectroscopy, Scanning electron microscopy, cation exchange capacity, specific surface area, and swelling index. The findings from the study are as follows:

- Bentonite core of GCL consist of major mineral such as montmorillonite and minor mineral like quartz, dolomite, feldspars and Cristobalite.
- GCL bentonite mainly comprise of elements like oxygen, Aluminium, Silicon and iron.
- Interlayer cations of bentonite core of GCL mainly consists of Na^+ and some traces of NH_4^+, K^+, Ca^{2+} and Mg^{2+}.

- FTIR results of GCL bentonite confirmed that structural OH stretching vibrations present from wavelength 3850 cm^{-1} to 2850 cm^{-1}, whereas Si-O stretching vibration and OH bending modes appear from wavelength 1250 cm^{-1} to 400 cm^{-1}.
- CEC, SSA and free swell index of GCL bentonite were obtained 73.42 meq/100g, 674.84 m^2/g and 21 ml/2g respectively.

References

1. Bouazza, A.: Geosynthetic clay liners. Geotextiles and Geomembranes, 20(1), 3-17 (2002).
2. Rajesh, S., Khan, V.: Characterization of water sorption and retention behavior of partially saturated GCLs using vapor equilibrium technique and filter paper methods. Applied Clay Science 157, 177-188 (2018).
3. Rajesh, S., Khan, V.: An apparatus to measure gas diffusion and gas permeability of unsaturated cap barriers subjected to distortion. Geotechnical Testing Journal 44(2), 462-482 (2021).
4. Rajesh, S., Naik, A. A., Khan, V.: Performance of compacted soil barriers under advection gas flow condition. In: Dey, A., Reddy, K. R., Yesiller, N., Zekkos, D., Farid, A. (eds.) Geo-Chicago 2016: Sustainable Geoenvironmental Systems, pp. 212-221, American Society of Civil Engineers, USA (2016).
5. Khan, V., Rajesh, S.: Wetting path water retention curve of geosynthetic clay liner. In: 6^{th} Asian Regional Conference on Geosynthetics: Geosynthetics for Infrastructure Development 2016, pp. 667-675.
6. Rajesh, S, Roy, S., Khan, V.: Modeling WRC and volume change behavior of GCLs considering net stress and temperature. Geosynthetics International 28(2), 174-185 (2021).
7. LaGatta, M. D., Boardman, B. T., Cooley, B. H., Daniel, D. E.: Geosynthetic clay liners subjected to differential settlement. Journal of Geotechnical and Geoenvironmental Engineering 123(5), 402–410 (1997).
8. Bouazza, A., Vangpaisal, T.: An apparatus to measure gas permeability of geosynthetic clay liners. Geotextiles and Geomembranes 21(2), 85–101 (2003).
9. Anderson, R., Rayhani, M. T., Rowe, R. K.: Laboratory investigation of GCL hydration from clayey sand subsoil. Geotextiles and Geomembranes 31, 31-38 (2012).
10. Bouazza, A, Ali, M. A., Gates, W. P., Rowe, R. K.: New insight on geosynthetic clay liner hydration: The key role of subsoils mineralogy. Geosynthetics International 24(2), 139-150 (2017).
11. Rayhani, M. T., Rowe, R. K., R. W. I., Take, W. A., Siemens, G.: Factors affecting GCL hydration under isothermal conditions. Geotextiles and Geomembranes 29(6), 525-533 (2011).
12. Sarabian, T., Rayhani, M. T.: Hydration of geosynthetic clay liners from clay subsoil under simulated field conditions. Waste Management 31(1), 67-73 (2013).
13. Rowe, R. K., Lake, C. B., Petrov, R. J.: Apparatus and procedures for assessing inorganic diffusion coefficients for geosynthetic clay liners. Geotechnical Testing Journal 23 (2), 206–214 (2000).
14. Lange, K., Rowe, R. K., Jamieson, H., Flemming, R. L., Lanzirotti, A.: Characterisation of of geosynthetic clay liner bentonite using micro-analytical methods. Applied Geochemistry. 25(7), 1056-1069 (2010).
15. ASTM D5993-18. Standard Test Methods for Measuring Mass per Unit Area of Geosynthetic Clay Liners, ASTM International, PA, USA, 2018.
16. ASTM D5261-18. Standard Test Method for Measuring Mass per Unit Area of Geotextiles, ASTM International, PA, USA, 2018.
17. Cokca, E., Birand, A.: Determination of cation exchange capacity of clayey soils by the methylene blue test. Geotechnical Testing Journal 16(4), 1-7 (1993).

18. ASTM D5890-19. Standard Test Method for Swell Index of Clay Mineral Component of Geosynthetic Clay Liners, ASTM International, PA, USA, 2019.
19. Tabak, A., Yilmaz, N., Eren, E., Caglar, B., Afsin, B., Sarihan, A.: (2011). Structural analysis of naproxen-intercalated bentonite (Unye). Chemical Engineering Journal 174(1), 281-288 (2011).
20. Jain, A.: Performance of polymer amended bentonite based geosynthetic clay liners. M. S. Thesis, Indian Institute of Technology, India (2017).