



**Geotechnique, Construction  
Materials & Environment**  
**VOLUME 5(1)**

**Edited by**

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GEOMATE 2015 OSAKA, JAPAN  
GEOTECHNIQUE, CONSTRUCTION MATERIALS AND ENVIRONMENT

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PROCEEDINGS OF FIFTH INTERNATIONAL CONFERENCE – GEOMATE 2015  
GEOTECHNIQUE, CONSTRUCTION MATERIALS AND ENVIRONMENT OSAKA, JAPAN  
16-18 NOVEMBER, 2015

# Geotechnique, Construction Materials and Environment

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Published by:  
The GEOMATE International Society  
Tsu city, Mie, Japan  
E-mail: [society@geomate.org](mailto:society@geomate.org)  
<http://www.geomate.org/>

ISBN Number: 978-4-9905958-4-5 C3051

# CLAY-SHALE MATERIALS AS LOW-COST LANDFILL LINERS: AN INTEGRATED GEOCHEMICAL AND GEOTECHNICAL ASSESSMENTS

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## ABSTRACT

Increasing rate of wastes generation due to urbanization and industrialization, as well as the need for proper waste disposal management has been the concerns of many low income countries like Nigeria. Hence, this study assessed the geotechnical and chemical characteristics in respect of suitability of selected clay-shale deposits in Nigeria as low cost landfill liners for waste disposal. Twenty four (24) clay-shale samples were subjected to engineering tests, mineralogical XRD and geochemical analyses. Apart from normal kaolinitic clay, the XRD analyses revealed smectitic and mixed layer clays with Liquid Limit (LL) of 58.3 – 116.5 (av. 75.4) and 170.4 – 173.2 (av. 171.8) respectively while the Plasticity Index (PI) ranged from 20.3 – 51.6 (av. 31.8) and 80.9 – 93.3 (av. 87.1) respectively. Methylene blue adsorption index (MBI) ranges from  $\approx 10$  to 18.6 meq/100g for both smectitic and mixed layer clay-shales with corresponding surface area of 0.8–1.5m<sup>2</sup>/g, suggesting the dominance of active clay minerals. In addition, the geochemical analyses show that the clay-shale materials contain significant amount of Al<sub>2</sub>O<sub>3</sub> with average value of 17.0 and 15.9% respectively while Fe<sub>2</sub>O<sub>3</sub> has average value of 8.2 and 6.5% respectively, suggesting Fe-rich smectitic clays. The overall evaluation revealed that the clay-shale materials are chemically and geotechnically suitable for application as landfill liners subject to appropriate beneficiation /amendment such as mixing with cement and other binding materials.

**KEYWORDS:** *Geochemical, Geotechnical Assessment, Landfill, Clay-Shale, Liner Materials*

## INTRODUCTION

Increasing urbanization and industrialization have led to an increase in the flow of goods and services in many low income developing nations, resulting to increase in the volume and varieties of generated wastes. Thus, the environmental implications of the increasing rate of solid waste production and the need for proper waste disposal management has been the concerns of many low income countries like Nigeria. In addition, safe containment of wastes and control of leachate from landfill or waste-dump sites are paramount in the mitigation of groundwater pollution. Hence, proper design and careful selection of lining materials are vital parts of engineered waste disposal system [1].

For economical reasons, natural clays and clay-shales available within a reasonable hauling distance, can be used as landfill liners while compacted natural clays are also widely used as landfill liners due to their low hydraulic conductivity and high contaminant attenuation [2], [3]. In other words, compacted clay liner (CCL) must have a low hydraulic conductivity to control leachates from the wastes and must have sufficient shear strength to accommodate possible settlement [2], [4]. Therefore, strict specifications are usually imposed on the selection of a liner material, design and construction

of the compacted clay liners in order to satisfy the above requirements [1], [2], [5].

Another major constraint to the development of properly engineered landfills is the high cost of imported synthetic liners in local markets of developing countries like Nigeria, which therefore calls for alternative local sources of materials for landfill liner. Consequently, crushed shale / clay-shale deposits appear to be inexpensive alternative that can be utilized to contain leachate in landfill and protect the underlying groundwater resources. This study, therefore, assesses the geotechnical and geochemical characteristics of shale (clay-shale) units from southern part of Nigeria, for suitability as low cost compacted clay liner (CCL) and as components of landfill barrier system in waste disposal systems.

## GEOLOGICAL SETTING OF STUDY AREA

The three major rock types - igneous, metamorphic and sedimentary - abound in Nigeria. Igneous and metamorphic rocks constitute the Precambrian crystalline Basement Complex units and underlie the physical foundation of the country, while the Cretaceous-Tertiary sedimentary units, which lie unconformably on the Basement Complex constitute the sedimentary basins as depressions between basement landmass in the south, northeast

and northwest [6], [7]. The Basement Complex and the sedimentary basins are equally dispersed in Nigeria with the basement rocks most extensive in northern-central, south-western parts and along the eastern margin of Nigeria (Fig.1).

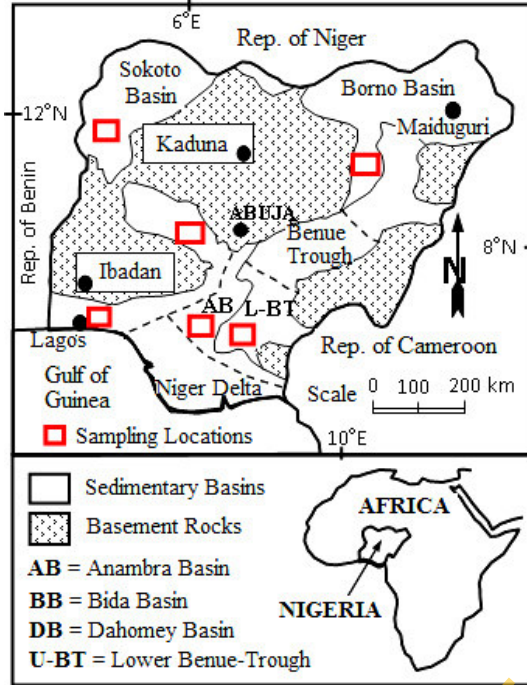


Fig. 1: Geology of Nigeria indicating Sampling Locations (Inset: Map of Africa)

The sedimentary basins occupy the central X-shaped area of the country and underlie the southern part as well as northwestern (Sokoto Basin) and north-eastern areas (Borno Basin). The Precambrian Basement units consist of gneisses, granites, migmatitic and granitic gneisses, quartzites, slightly migmatized to unmigmatized meta-sedimentary schists and dioritic rocks [6]. The Cretaceous and Tertiary sedimentary units consist of alternating sequence of rock units ranging from shales, limestones, mudstone / siltstone intercalated with sandy units in places. The occurrences of extensive clay-shale and mudstones in many of the sedimentary basins (Fig. 1) offer an opportunity of local sourcing of materials for this study.

**MATERIALS AND METHODS**

For the purpose of this study, selected shale / clay-shale samples were collected from different lithologic units within the sedimentary basins in Nigeria (Fig. 1). The samples were obtained from road cut sections and in excavation pits of sand/laterite quarry sites. Efforts were made to collect fresh un-weathered samples for the

laboratory tests. Most of the samples were either in forms of soft rock when wet or in blocky compact and laminated form. Physical appearance indicates that the colour of the sampled clay-shale varied from light grey, to whitish and light brown as well as light reddish when iron-stained. After drying in the laboratory, the samples were disaggregated and sieved to obtain fine-grained materials with a maximum size of about 4.75 mm (sieve number 4).

In this preliminary study, representative samples of the selected clay/shale units were subjected to tests such as Atterberg consistency limits (liquid limit, plastic limit, shrinkage) and hydrometer tests while methylene blue adsorption index (MBI) was estimated following standard method [8]. For the chemical studies, oxides of major elements (SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, CaO, MgO, K<sub>2</sub>O, Na<sub>2</sub>O and TiO<sub>2</sub>) were analysed using a Philips PYE UNICAM SP9 atomic absorption spectrophotometer (AAS) and determination of the loss on ignition (LOI) using Heraeus muffle furnace with sequential heating up to 1,050°C.

The mineralogical analyses for the identification of clay minerals via X-ray diffraction were carried out using a Siemens D500 Diffractometer with Cu-K<sub>alpha</sub> radiation. Powdered as well as textural preparations of samples were measured in three forms; untreated, treated with ethylene glycol and heated to 550°C. The resulting diffractogram charts were analysed using the DIFFRAC-plus Evaluation programme (EVA), version 6.0 rev. 0. The criteria for the mineral identification as in [9] and [10] are presented in Table 1.

Table 1: Criteria for Identification of Major Clay Minerals

Minerals Basal d-spacing, Å (Untreated)	Glycolation Effect	Heating Effect
Kaolinite (d <sub>1</sub> = 7.1, d <sub>2</sub> =3.5, d <sub>3</sub> = 10.4)	No change / reaction	Degraded@ 550° C
Illite (d <sub>1</sub> = 10.0, d <sub>2</sub> = 5.0)	No change	No reaction
Palygor. (d <sub>1</sub> =10.5, d <sub>2</sub> = 4.5, d <sub>3</sub> = 3.2)	No change	Degraded @ 600° C
Smectite (d ≈ 11.9–15.3)	Peak change d ≈16.6–17.2	Change to d ≈ 9.5–10.0
Vermiculite (d ≈ 13.9 – 14.3)	No change	Change to d ≈ 9.8–10.0
Chlorite (d <sub>1</sub> =14.3, d <sub>2</sub> = 7.1)	No change	Change to d ≈ 13.8

Palygor = Palygorskite

All the geotechnical, chemical and mineralogical tests, following standard laboratory procedures, as well as follow-up data evaluation and interpretations were carried out at the Department of Geology, University of Trier, Germany.

**RESULTS AND DISCUSSIONS**

**Grain-size Distribution and Index Properties**

The grain-size distribution curves for the representative samples of the clay-shales used in this study are presented in Fig. 2. The curves revealed that the crushed clay-shales are generally clayey with percentage fines (silt and clay) ranges from 90–98% for most of the samples. However, the proportion of clay fractions (<0.002mm) are in the range of 60–75% for the more plastic (smectitic) samples, 50–55% for mixed layer clay-shale materials and 15–30% for silty (kaolinitic) samples. The values are more than minimum of 20% recommended for liner materials, especially for the smectitic and mixed layer clay-shale materials [11].

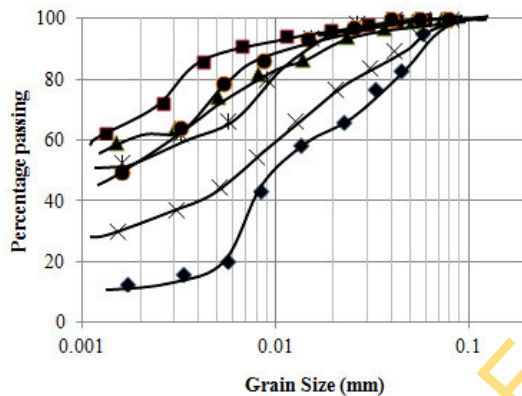


Fig. 2: Representative Grain size Distribution Curves for the clay-Shale Samples.

The NRA (1992) define suitable materials as those clays with a liquid of  $30 > (LL) < 90\%$  and plasticity index of  $20 > (PI) < 65\%$ . For this study, the results of the Atterberg limits as presented in Table 2 revealed Liquid Limit (LL) of 58.3 – 116.5 (av. 76.2) and 170.4 – 173.2 (av. 171.8) for smectitic and mixed layer clay materials respectively, while the Plasticity Index (PI) ranged from 20.3 – 51.6 (av. 31.8) and 80.9 – 93.3 (av. 87.1) respectively. However, the kaolinitic clay materials revealed LL of 38.8 – 75 (av. 54.6) and PI of 10.5 – 29.6 (av. 20.7), suggesting little expansive properties desirable of landfill liner materials. The implication is that the relatively low plasticity index for the kaolinitic clay will enhanced leachate attack from the landfill as a result of its potential high permeability.

As shown in the Casagrande Plasticity Chart (Fig. 3), nearly all the clay-shale samples plot below the "A" line. In other words, the clay-shale materials can be classified as inorganic silts of medium plasticity and intermediate compressibility (ML) for the kaolinitic clay-shales while the smectitic and

mixed layer clay-shale materials exhibited high plasticity as inorganic clays (CL-OH) of higher compressibility.

Table 2: Geotechnical characteristics of the clay-shale materials (in %)

Parameters	Min.	Max.	Mean	Std. Dev.
<b>Smectitic Clays (N=10)</b>				
LL	58.3	116.5	75.4	19.9
PL	32.2	64.9	43.4	10.4
PI	20.3	51.6	32.0	10.4
SL	25.6	52.0	35.4	8.4
<b>Mixed layer Clays (N=4)</b>				
LL	170.4	173.2	171.8	2.0
PL	79.9	89.6	84.7	6.8
PI	80.9	93.3	87.1	8.8
SL	56.6	69.3	63.0	9.0
<b>Kaolinitic Clays (N=10)</b>				
LL	38.8	75.0	52.3	10.9
PL	21.9	48.6	32.2	8.0
PI	10.5	29.6	20.2	6.1
SL	16.3	42.1	27.2	7.8

Therefore, with possible minimal beneficiation, these characteristics will be suitable for capping and bottom liner materials or as components of such, in landfill system.

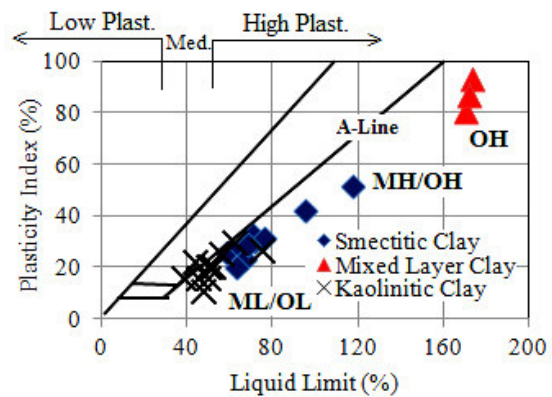


Fig. 3: Casagrande chart classification of the clay-shales

Although a moderately high plasticity index is considered important in the selection of a suitable liner material. However, under extremely high plasticity ( $PI > 65\%$ ) the liner material becomes sticky and difficult to work with when wet and forms hard lumps when dry and at the same time more susceptible to desiccation [1], [3], [5]. In this

study, the PI values of 10-30% for kaolinitic clay-shales and 20-52% for smectitic clay-shales are within the recommended range of 20-65% for most of the samples, especially for smectitic clay-shale materials. However, the values of 80-93% for the mixed-layer clay-shales are extreme enough as to warrant beneficiation with other materials, before application as liner materials.

**Chemical Characterization**

The summary of the results of geochemical analyses are presented in Table 3a-c. The results revealed that the smectitic clay-shales have relatively lower SiO<sub>2</sub> of 38.2 – 63.1wt.% (±8.4%) compared to values of 47.8 – 68.1 wt.% for the mixed layer and kaolinitic clay materials.

Table 3a: Geochemical Characteristics of the smectitic Clays (N=10)

Parameters (%)	Min	Max.	Mean	Std. Dev.
SiO <sub>2</sub>	38.2	63.1	53.1	8.44
TiO <sub>2</sub>	0.87	1.52	1.16	0.21
Al <sub>2</sub> O <sub>3</sub>	11.5	23.2	17.0	4.31
Fe <sub>2</sub> O <sub>3</sub>	6.37	10.9	8.19	1.40
MnO	0.02	0.17	0.08	0.05
CaO	0.01	17.2	4.50	6.04
MgO	0.81	2.82	1.52	0.58
Na <sub>2</sub> O	0.04	0.32	0.12	0.10
K <sub>2</sub> O	0.21	1.90	0.80	0.58
LOI	10.1	21.5	13.4	4.11

Table 3b: Geochemical Characteristics of the mixed layer Clays (N=4)

Parameters (%)	Min	Max.	Mean	Std. Dev.
SiO <sub>2</sub>	48.84	68.0	59.6	9.09
TiO <sub>2</sub>	1.00	1.26	1.11	0.11
Al <sub>2</sub> O <sub>3</sub>	12.5	18.4	15.91	2.78
Fe <sub>2</sub> O <sub>3</sub>	5.07	8.21	6.54	1.39
MnO	0.04	0.08	0.06	0.02
CaO	0.57	3.61	1.48	1.43
MgO	0.62	4.25	2.37	1.93
Na <sub>2</sub> O	0.04	0.44	0.16	0.19
K <sub>2</sub> O	0.17	2.07	1.05	0.78
LOI	7.78	15.5	12.1	3.92

The loss on ignition (LOI) values range between 10.1 and 21.5 wt.% (av. 13.4%) for smectitic clay while the mixed layer and kaolinitic clays exhibited values of 7.8 – 15.5wt.% (av. 12.0%). Furthermore,

the analyzed clay-shales contain significant amount of Al<sub>2</sub>O<sub>3</sub>, with respective average values of 17.0% 15.9% and 24.5% for smectitic, mixed layer and kaolinitic clay materials.

Table 3c: Geochemical Characteristics of the kaolinitic Clays (N=10)

Parameters (%)	Min	Max.	Mean	Std. Dev.
SiO <sub>2</sub>	47.8	62.8	56.7	4.52
TiO <sub>2</sub>	1.01	3.17	1.89	0.64
Al <sub>2</sub> O <sub>3</sub>	17.0	30.9	24.5	5.04
Fe <sub>2</sub> O <sub>3</sub>	1.62	7.79	3.56	2.03
MnO	0.01	0.04	0.02	0.01
CaO	0.01	0.36	0.09	0.12
MgO	0.07	2.08	0.51	0.63
Na <sub>2</sub> O	0.02	1.02	0.22	0.32
K <sub>2</sub> O	0.15	2.16	0.91	0.64
LOI	8.18	15.5	12.0	2.72

LOI=Loss on Ignition

However, Fe<sub>2</sub>O<sub>3</sub> have average value of 8.2%, 6.5%, and 3.6% respectively, suggesting Fe-rich assemblages for smectitic, mixed layer and kaolinitic clay-shale materials. Also average values of 4.5% and 1.5% CaO for smectitic and mixed layer clays respectively compared to 0.1% CaO for kaolinitic clay-shales suggest the dominance of Ca-montmorillonite as also supported by the XRD results.

**Mineralogical Characterization**

The X-ray diffraction (XRD) analyses of the clay-shale samples revealed the general presence of quartz (usually at d = 3.34Å) as the principal non-clay mineral in all the analyzed samples while calcite (CaCO<sub>3</sub>) also occur in a couple of the samples. The representative X-ray patterns presented in Fig. 4 revealed the presence of kaolinite in all samples by its basal spacings at 7.1 and 3.5Å. In addition, the oriented mounts of the samples confirm the presence of divalent-cation saturated smectite (Fig. 4b and c) for the mixed-layer and smectitic clay-shale materials. The basal spacing, d<sub>001</sub> of the smectite mineral ranges from 14.45–15.31Å, suggesting the dominance of Ca-montmorillonite.

In both cases, such basal spacing (d<sub>001</sub>) are expanded to about 17.0 Å under glycolation (Fig. 4b and c), and clearly distinguished the observed Ca-montmorillonite from chlorite and vermiculite groups. The X-ray pattern for the mixed-layer clay-shale materials also exhibit two weak bands at d = 10.5 and 4.5Å (Fig. 4b), indicating the presence of palygorskite while a weak peak at d = ≈5.0Å (Fig. 4c) also suggest the presence of illite in the smectitic



clay-shale materials. Moreover, these results correlated well with the estimated activities of the clay-shales and are also clearly consistent with the results of XRD analyses presented earlier.

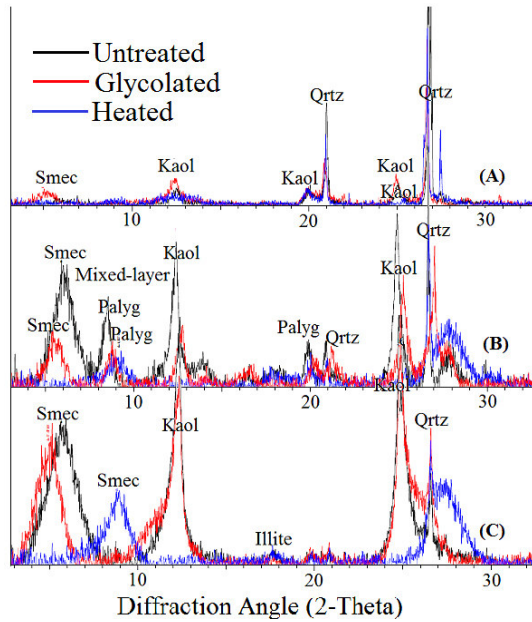


Fig. 4: Representative XRD patterns of the clay-shale samples under different treatments (A= Kaolinitic; B= Mixed layer; C= Smectitic)

### Methylene Blue Index and Activity

In the field of clay chemistry, the adsorption of methylene blue to the edges, external surfaces, and accessible inter-layer regions of clay minerals dispersed in an aqueous solution is often used to measure CEC and specific surface area of clay minerals [12], [13]. In this study, the results of Methylene blue adsorption index, MBI (an estimate of CEC) and surface area of the clay-shale materials are presented in Table 4 alongside the activity.

As shown in Table 4, MBI (an estimate of CEC) range from  $\approx 10$  to 18.6 meq/100g for both smectitic and mixed layer clay-shales with corresponding surface area of 0.8–1.5m<sup>2</sup>/g suggesting the dominance of swelling clay minerals as also revealed by the estimated montmorillonite content of 31.5 to 60.1%. However, the MBI value of 1.8–5.8 meq/100g and surface area of 0.14–0.45m<sup>2</sup>/g for kaolinitic clay-shale materials is a confirmation of the dominance of inactive clay minerals as reflected by the low values (15.1 - 18.1%) of estimated montmorillonite content.

Clay minerals with high plasticity also generally have higher activity and corresponding high CEC [14]. These tend to plot in the upper right hand

region of an expanded plasticity chart (high liquid limit and high PI) as shown earlier in Fig. 3.

Table 4: Geochemical Characteristics of the kaolinitic Clays (N=10)

Parameter	Acti- vity	MBI (meq/100g)	SA (m <sup>2</sup> /g)	% Mont.
<b>Smectitic Clay (N=10)</b>				
Min.	1.32	10.0	0.78	32.3
Max.	1.92	18.6	1.46	60.1
Mean	1.71	14.7	1.15	47.5
Std. Dev.	0.23	2.63	0.21	8.48
<b>Mixed layer Clay (N=4)</b>				
Min.	0.96	9.75	0.76	31.50
Max.	1.81	17.4	1.36	56.05
Mean	1.49	13.5	1.05	43.45
Std. Dev.	0.38	3.19	0.25	10.31
<b>Kaolinitic Clay (N=10)</b>				
Min.	0.51	1.77	0.14	5.71
Max.	0.72	5.80	0.45	18.7
Mean	0.60	3.59	0.28	11.6
Std. Dev.	0.10	1.92	0.15	6.19

MBI = Methylene blue adsorption index

Mont. = % Montmorillonite

SA = Surface Area; %

Activity provides an indirect indication of the type of clay minerals present, e.g. Allophanes, Illite, Kaolinite, and Halloysite, do exhibit an activity of 0.5, while Illites and Smectites are characterized by an activity of 1 [15]. In this study, based on the grain size distribution and Atterberg limits, the estimated activity as presented in Table 4 range from 0.51–0.72 for the kaolinitic clays; suggesting the dominance of inactive clays with low CEC and little or no swelling potential. However, values of 0.96–1.92 for smectitic and mixed layer clay-shales indicate normal to predominantly active clay materials with relatively higher swell susceptibility and higher CEC. These results correlated well with the estimated activities of the clay-shales and are also clearly consistent with the results of XRD analyses, index properties and Atterberg limits (LL, PL and PI) presented earlier; thus it can be inferred that the study clay-shales are suitable as liner materials.

### SUMMARY AND CONCLUSIONS

The results of the geotechnical tests conducted on the clay-shale samples revealed that smectitic and mixed layer clays have Liquid Limit (LL) of 58.3 – 116.5 (av. 75.4) and 170.4 – 173.2 (av. 171.8) respectively while the plasticity index (PI) ranged from 20.3 – 51.6 (av. 31.8) and 80.9 – 93.3 (av. 87.1) respectively. With percentage fines of more than 85%, percentage clay fraction of 60–75% (smectitic) and 50–55% (mixed-layer), most of the samples satisfy the basic requirements of clay liners

according to the standard specifications (Daniel, 1993). The mineralogical analyses revealed the presence of kaolinite in all samples by its basal spacings at 7.1 and 3.5A°, however, the oriented mounts of the samples confirm the presence of divalent-cation saturated smectite for the mixed-layer and smectitic clay-shale materials. These are consistent with the geochemical data with SiO<sub>2</sub> of 38.2 – 63.1wt.% (±8.4%) and average Al<sub>2</sub>O<sub>3</sub> of 17.0% for smectitic clay-shales and SiO<sub>2</sub> of 47.8 – 68.1 wt.% and Al<sub>2</sub>O<sub>3</sub> of 5.9% and 24.5% respectively for mixed layer and kaolinitic clay materials. Nonetheless, average value of 8.2%, 6.5%, and 3.6% respectively, suggests Fe-rich assemblages.

In summary, based on the results of the geotechnical, mineralogical and chemical properties of the study clay-shales were generally within the range suitable for use as liners or component materials. However, higher plasticity index of 80-93% for the mixed-layer clay-shales are extreme enough as to warrant amendment or blending with other materials in order to obtain suitable plasticity property and shrinkage susceptibility, before application as liner materials. Further studies in respect of permeability, compaction behaviour and sorption characteristics of the clay-shales are recommended.

#### ACKNOWLEDGMENTS

The first author acknowledged, with many thanks, the assistance and supports of Dr. Oscar Baeza-Urrea and Mrs. Jutta Moschansky-Koster of the Fachbereich Geographie/Geowissenschaften (FB-VI), Universität Trier, Germany, during the laboratory phase of this study.

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