Original Research Article

PHYSICOCHEMICAL CHARACTERIZATION OF A FEW CLAYS FROM ASSAM, INDIA

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Abstract: A few clay samples are collected from Karbi Anglong and North Cachar districts in Assam (India), were characterized with respect to moisture content, LOI, CEC, SiO_2, Al_2O_3, Fe_2O_3, CaO, K_2O, TiO_2, P_2O_5, Na_2O, MgO, and various trace elements and rare earth elements. The XRD measurement indicated that the predominant clay mineral present in the clays was kaolinite with some amount of quartz impurities. The IR measurements yielded appropriate frequencies expected for the OH and SiO stretching regions, and OH and SiO bending regions in kaolinite, and the data complemented the conclusions drawn from XRD analysis.

Key Words: Kaolin, Characterization, kaolin KGa-1b, montmorillonite SWy-2

INTRODUCTION

In North Eastern region of India, rich clay deposits mostly formed by weathering of Archean and Precambrian rocks[1] are found in Assam and Meghalaya. Alluvial clay deposits are found in large amount at Margherita in the Tinsukia district and Jalukbari in the Kamrup district of Assam. Large deposits of black alluvial clay, found at Mathurapur in the Sibsagar district, is now used in drilling mud in oil exploration. Alluvial clay deposits also occur at Khetri, Dharapur and Ajara areas of the Kamrup district. Good quality white clay deposits exist at Deopani, Sheelvita and Silonijan areas of the Karbi Anglong district. The deposits are estimated at 2 million tones. An estimated deposit of about 7 million tons of white clay also exists at Sohrarim and Cherrapunji areas of the Khasi Hills district in Meghalaya[2]. Deposits of lithomarge have been reported from many areas of the Garo Hills district of Meghalaya. Fire clay deposits occur along the coal belt of Margherita-Lido areas in the Tinsukia district of Assam. Fuller's earth is found at Subankhata and Bhutan Khuti areas of the Nalbari district of Assam.

In the natural environment, clays in soil and river sediments always play the role of a scavenger for the pollutants. The edges and the faces of clay particles can adsorb anions, cations, non-ionic and polar contaminants from natural water. These contaminants accumulate on clay surface and are retained through the processes of ion exchange, co-ordination, or ion-dipole interactions. Sometimes the pollutants can be held through H-bonding, van der Waals interactions or hydrophobic bonding effects. In other cases, the pollutants may be held through weak or strong adsorptive interactions. The strength of such interactions is determined by the particular structural and other features of the clay mineral.

The present work was planned to characterize a few clay samples of Assam in order to investigate their role in natural purification process of eliminating contaminants from water.

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MATERIAL AND METHODS

Collection and Purification of clays

The clay samples were collected from the Karbi Anglong (Silonijan area) and North Cachar districts in Assam (India) in the form of big, bright white lumps. After collection, the samples were washed repeatedly with water to remove stones, sand and other heavy materials. The lumps were crushed, ground and sieved through a 230-mesh sieve for removing the large non-clay fractions for obtaining raw clay. A part of the raw clay was kept suspended in double-distilled water in a 1 L beaker for several hours and the mixture was stirred with the addition of small amounts of 30 % hydrogen peroxide solution to remove organic substances till all effervescence ceased. The mixture was kept overnight, the clear liquid was decanted from the top, more water was added, stirred, and allowed to settle down for 3-4 h. The clear liquid at the top was removed carefully and the process repeated several times to get rid of excess hydrogen peroxide solution. After adding more water, the mixture was agitated vigorously for some time and the suspended clay was kept in wide glass plates in an air oven at around 343 K for slow evaporation to dryness[3] to obtain pure clay. Samples of both raw and pure clay were heated at 770K for 6 h to obtain thermally treated raw clay and pure clay. Samples of clay were also treated with NaOH. For this purpose, 6 g each of raw and pure clay was agitated with 100 mL of 1M NaOH for 4 h in a mechanical shaker, left overnight, filtered and washed with water. The residue was dried at 350K to obtain NaOH-treated raw clay and pure clay. The results were compared with those obtained for kaolin sample (KGa-1b) and montmorillonite sample (SWy-2) obtained from University of Missouri-Columbia, Source Clay Minerals Repository, Columbia, USA.

Experimental Procedure

Moisture content and Loss on ignition: The moisture content represents the amount of water remaining sorbed on the clays. Four samples taken randomly from each location were used for moisture

content determination by measuring the loss in weight of 1g clay sample after it was dried continuously for 6 h in an oven at 378 K. This was repeated till a constant weight was obtained. The moisture content values were expressed in percentage.

The loss on ignition measures the total amount of volatile matter present in the clays along with structural water, which comes out through the process of dehydroxylation at the temperature of ignition. The LOI was determined by the igniting a moisture free clay sample in a muffle furnace at 1273 K for 4 h. The LOI was calculated in percentage from the loss in weight.

Cation Exchange Capacity: The cation exchange capacity (CEC) of the kaolins was estimated by the conventional $BaCl_2$ method.

Chemical Composition

Standard analytical methods[4] were adopted for chemical analysis of the clay samples using analytical grade reagents. The composition of the clays in terms of the contents of SiO_2 and Al_2O_3 was worked out.

The chemical composition of the clay samples with respect to other major oxides was determined by XRF measurements in the University Science Instrumentation Centre, Gauhati University using Phillip's PW 1480 XRF spectrometer provided with Au-Cr dual anode system. The trace element composition of the pure clay samples was determined by ICP-AES technique at the National Geophysical Research Institute, Hyderabad.

Clay mineral identification

XRD measurements were done to identify the clay mineral present in the clay sample at the USIC, Gauhati University using Phillip's X-ray Powder Diffractometer (PW1710 Based) using CuK_α radiation at 40 kV, 20 mA and Ni filter. The scanning range was from 5° to 30° (2θ). The identification of clay minerals was done by using standard techniques [3, 5-7].

IR measurements were done using Perkin-Elmer 1600 series FTIR spectrophotometer (range 4000 -400 cm^{-1}) by the KBr pallet technique. The clay content[8] in the KBr pallets was kept at between 0.15-0.25 %.

RESULTS AND DISCUSSION
Moisture content and Loss on ignition

The results of moisture content and L.O.I. determination are given in Table 1. The raw clays contain appreciable amount of moisture content. The Silonijan clay from the Karbi Anglong district had slightly lower average moisture content at 1.88 %

compared to the North Cachar clay, which has moisture content of 2.01 %. The different clay samples from the same area have very similar moisture content, which can be attributed to identical environmental conditions. The L.O.I. values varied from 11.75 to 13.10 % for the Karbi Anglong clay and from12.94 to 13.60% for the North Cachar clay. The North Cachar clay had thus slightly higher content of volatile matter compared to the Karbi Anglong clay.

Table 1: Moisture content and LOI data

Sample		Moisture content (%)		Loss on Ignition (%)	
		Value	Mean	Value	Mean
Karbi-Anglong Clay	1	1.85		12.50	
	2	1.60	1.88	11.75	12.55
	3	1.95		13.10	
	4	2.10		12.85	
North Cachar Clay	1	2.14		13.60	
	2	2.05	2.01	12.94	13.29
	3	1.90		13.25	
	4	1.96		13.36	

Cation Exchange Capacity

The cation exchange capacity (CEC) of some of the clay samples was estimated to be 2.62 (Raw Silonijan clay), 2.85 (Pure Silonijan clay), 1.94 (Thermally treated Raw Silonijan clay), 3.08 (Thermally treated Pure Silonijan clay), 2.17 (NaOH treated Raw Silonijan clay) and 2.42 (NaOH treated Pure Silonijan clay) meq/100g. The pure clay and its thermally treated sample had higher CEC compared to the other samples. Thermal treatment improved the CEC of the pure clay while that of the raw clay decreased. The treatment with NaOH decreased the CEC of the raw and pure clay.

Chemical Composition

SiO_2 and Al_2O_3 ratio: The chemical composition of the clay samples with respect to the main components SiO_2 and Al_2O_3 was determined for the following samples by wet chemical analysis :

C1 :	Raw Karbi Anglong clay
C2 :	Pure Karbi Anglong clay
C3 :	Thermally treated raw Karbi Anglong clay
C4 :	Thermally treated Pure Karbi Anglong clay
C5 :	NaOH-treated raw Karbi Anglong clay
C6 :	NaOH-treated Pure Karbi Anglong clay
C7 :	Raw North Cachar clay
C8 :	Pure North Cachar clay
C9 :	Thermally treated raw North Cachar clay
C10 :	Thermally treated pure North Cachar clay
C11 :	Kaolin sample (K Ga-1b)

The SiO_2 and Al_2O_3 percentage, SiO_2/Al_2O_3 ratio and the total amount of SiO_2 and Al_2O_3 in all the above clay samples are given in Table 2.The results showed that the raw clay contained large amount of SiO_2 compared to Al_2O_3. This may be due to presence of quartz, sand and other heavy particles. The SiO_2/Al_2O_3 ratio for C1 decreased on thermal treatment and on

treatment with NaOH. The contents of both SiO_2 and Al_2O_3 increased on thermal treatment. However, NaOH treatment did not much alter SiO_2 percentage while Al_2O_3 percentage showed improvement. The raw clay from the North Cachar district (C7) had chemical composition with an identical trend. There was a marked decrease in the SiO_2/Al_2O_3 ratio, when the clay samples were purified. This is reflected in the total amount of SiO_2 and Al_2O_3 present in the clay samples. It was interesting to note that the kaolin KGa-1b had a similar SiO_2 / Al_2O_3 ratio, but the total amount of SiO_2 and Al_2O_3 measured up to 98.98%.

Table 2: Chemical composition of the clay samples

Sample	Clay	SiO_2 (%)	Al_2O_3 (%)	Total	SiO_2/Al_2O_3
C1	Raw Karbi Anglong	47.26	8.22	55.48	5.70
C2	Pure Karbi Anglong	54.87	40.09	94.96	1.36
C3	C1 after thermal treatment	59.20	11.76	70.96	5.00
C4	C2 after thermal treatment	55.27	42.23	97.50	1.31
C5	NaOH treated C1	47.68	10.96	58.64	4.40
C6	NaOH treated C2	55.75	42.37	98.12	1.32
C7	Raw North Cachar	41.83	3.89	45.72	10.80
C8	Pure North Cachar	43.71	33.32	77.03	1.31
C9	C7 after thermal treatment	58.22	8.37	66.59	7.00
C10	C8 after thermal treatment	44.37	32.37	76.74	1.37
C11	K Ga-1b	55.99	42.99	98.98	1.30

The chemical analysis throws some light on the alteration of chemical composition brought about by treatment of the clays with NaOH. Only the Karbi Anglong clay was tested for this purpose. The raw clay (C1) on being treated with NaOH showed a decline in SiO_2/Al_2O_3 ratio, which was basically due to improvement in Al_2O_3 percentage. The total amount of the SiO_2 and Al_2O_3 also increased. In the case of pure Karbi Anglong clay (C2), the SiO_2/Al_2O_3 ratio remained more or less constant on treatment with NaOH. However, there was some improvement in the contents of both SiO_2 and Al_2O_3 with a resultant enhancement of the total amount of SiO_2 and Al_2O_3.

Pure Kaolin should have ideally a SiO_2/Al_2O_3 ratio of 1.0. The values obtained in the present work even for the purified clay samples were never below 1.3. This perhaps indicates that the clay samples even after purification contained some free SiO_2. The ratio of 1.3 could not be improved with further purification. However, the SiO_2/Al_2O_3 ratio of the clay samples used in this work compared well with that of the standard kaolin KGa-1b.

Other Oxides
The presence of a few major oxides namely Fe_2O_3, MnO_2, MgO, CaO, Na_2O, K_2O, TiO_2 and P_2O_5 were estimated by XRF measurement for the clay samples C2, C4, C6, C8, C10 and C11. The data are presented in Table 3.

Table 3: Major oxides other than SiO_2 and Al_2O_3 in the clay samples

Sample	C2	C4	C6	C8	C10	C11
Fe_2O_3 (%)	0.73	0.78	0.77	0.88	0.91	0.51
MnO_2 (%)	B.D.L.	B.D.L.	B.D.L.	B.D.L.	B.D.L.	B.D.L.
MgO (%)	<0.15	<0.15	<0.15	5.12	5.85	<0.15
CaO (%)	0.22	0.22	0.17	10.20	9.11	0.11
Na_2O (%)	B.D.L.	B.D.L.	B.D.L.	6.08	6.66	B.D.L.
K_2O (%)	0.23	0.25	0.29	0.11	0.11	0.02
TiO_2 (%)	0.17	0.17	0.17	0.19	0.21	2.08
P_2O_5 (%)	0.044	0.057	0.056	0.39	0.42	0.008

B.D.L= Below Detection Limit.

All the samples examined contained an appreciable amount of Fe_2O_3, but they did not contain any MnO_2. The North Cachar samples, C8 and C10, contained a large amount of MgO, CaO, and Na_2O. The oxides (Fe_2O_3, K_2O, P_2O_5 and TiO_2) showed certain differences between the Assam clays and the standard kaolin KGa-1b. With respect to Na_2O, MgO, CaO content, the Silonijan clay samples were very similar to KGa-1b, but the North Cachar clay sample had some differences.

Trace Element Composition
30 trace elements and rare earths were determined in 5 samples of clay after purification. Two samples (C2 and C2$^{()}$) were from Silonijan area and three samples (CD1, CD2, CD3) from Deopani area, both in the Karbi Anglong district of Assam. The results along with the ranges are given in Table 4.

No particular distribution pattern for the trace elements can be distinguished. Some of the trace elements occur at appreciable concentration. The presence of such a large number of trace elements in the clay samples cannot have a simple interpretation. It is suggested that many of the trace elements and rare earths have identical geochemical behaviour[9] and therefore they occur together. Further, weathering processes have less impact on those trace elements whose compounds have low solubility[10] and such elements are not removed by the normal weathering process. It is an established fact that the trace element composition of clays varies widely from place to place [11] and therefore the results obtained in the present work are no exception. The present results are in agreement with the earlier results obtained for similar clay samples of Assam and Meghalaya [8].

Clay mineral identification
XRD Measurements. *XRD measurements were done for 2θ values of 5° to 30° at step size of 0.05^o per 0.5 sec for the clay samples C2, C4, C6, C8, C10, C11 (KGa-1b) and C12 (standard Montmorillonite SWy-2).*

The XRD data for all the seven samples are given in Table 5. The XRD measurements show a number of prominent peaks. The purified Silonijan clay

gave prominent peaks at 2θ values of 12.43° (*d* = 7.13 Å) and 24.94° (*d* = 3.58 Å) along with three medium intensity peaks at 19.92°, 20.38° and 21.28° with *d*-values of 4.46 Å, 4.36 Å and 4.18 Å respectively. The XRD spectra of the thermally treated pure Silonijan clay showed no prominent peaks excepting a high background level. This indicates that the crystallinity of the sample was totally destroyed by treatment at 873 K for 6 h. The pure Silonijan clay after treatment with 1 M NaOH gave an XRD spectrum identical to that of the pure untreated clay. The treatment with NaOH did not alter the structure of the clay nor did it destroy the crystallinity.

Table 4: Trace and Rare Earth elements in the Clay samples (μg/g)

SN	Elements	C2	C2/	CD1	CD2	CD3	Range
1	Se	7.08	10.24	7.89	3.52	9.66	3.52 −10.24
2	Zn	57.01	48.50	59.53	23.39	41.97	23.39 − 59.53
3	Nb	31.98	22.82	29.84	1.43	30.67	1.43 −31.98
4	V	14.34	29.35	12.62	19.56	16.70	12.62 − 29.35
5	Ga	40.01	33.29	44.78	2.68	39.89	2.68 − 44.78
6	In	BDL	BDL	BDL	BDL	BDL	BDL
7	Cr	32.52	19.01	33.79	20.63	8.73	8.73 −33.79
8	Rb	1.22	1.27	1.59	5.75	11.14	1.22 −11.14
9	Ba	29.52	19.96	40.21	41.90	80.28	19.96 − 80.28
10	Co	2.55	3.23	2.81	5.57	1.61	1.61 − 5.57
11	Sr	10.15	10.05	10.73	418.87	33.58	10.05 − 418.87
12	Ta	3.36	4.11	3.69	0.16	3.22	0.16 − 4.11
13	Ni	12.47	10.79	24.29	51.44	5.08	5.08 − 51.44
14	Y	4.29	3.72	4.52	26.28	101.70	3.72 − 101.70
15	Cu	14.84	9.83	14.42	5.35	19.45	5.35 − 19.45
16	Zr	114.58	114.38	108.59	8.94	60.00	8.94 − 114.58
17	La	30.26	35.26	31.99	15.70	216.34	15.70 − 216.34
18	Hf	3.28	3.98	4.06	0.16	1.97	0.16 − 4.06
19	Ce	44.33	63.09	52.00	42.85	411.80	42.85 − 411.80
20	Pr	4.13	5.53	4.64	4.09	53.66	4.09 − 53.66
21	Nd	9.98	13.70	8.80	18.99	162.58	8.80 − 162.58
22	Sm	2.00	1.62	1.29	4.01	28.49	1.29 − 28.49
23	Eu	0.29	0.20	0.32	0.95	5.46	0.20 − 5.46
24	Gd	1.95	1.55	1.46	4.65	30.72	1.46 − 30.72
25	Dy	0.80	1.06	0.78	3.42	20.08	0.80 − 20.08
26	Er	0.77	0.48	0.75	1.95	11.53	0.48 − 11.53
27	Yb	0.80	1.13	0.86	1.62	13.25	0.86 − 13.25
28	Lu	0.16	0.20	0.16	0.25	1.50	0.16 − 1.50
29	Th	10.60	23.76	11.33	2.52	24.00	2.52 − 24
30	U	2.74	1.75	1.12	2.37	5.36	1.12 − 5.36

The pure North Cachar clay gave four prominent XRD peaks at 2θ values of 12.43°, 24.94°, 26.63°, and 29.41° corresponding to the *d*-values of 7.12 Å, 3.57 Å, 3.34 Å, and 3.03 Å respectively. After thermal treatment, the pure North Cachar clay still showed the peak at 29.45° (*d* = 3.03 Å) very prominently along with a relatively small peak at 26.76° (*d* = 3.33 Å), but the other peaks appearing at 12.43° and 24.94° for the pure clay disappeared. The results showed that the North Cachar clay behaved differently from the Silonijan clay with respect to thermal treatment.

Table 5: XRD data for the clay samples

Peak	2θ (°)	d (Å)	Peak Width (Å)	Peak Intensity (Counts)	Relative Intensity (%)
Pure Silonijan Clay (C2)					
1	6.71	13.19	0.60	86	9.2
2	8.89	9.96	0.40	17	1.8
3	12.43	7.13	0.20	936	100.0
4	17.88	4.97	0.25	14	1.5
5	19.92	4.46	0.20	137	14.6
6	20.38	4.36	0.20	180	19.2
7	21.28	4.18	0.25	142	15.1
8	23.18	3.84	0.20	69	7.4
9	24.94	3.58	0.25	595	63.6
10	26.45	3.38	0.50	46	4.9
11	28.78	3.11	0.60	11	1.2
12	32.48	2.76	0.40	10	1.1
Thermally treated pure Silonijan Clay (C4)					
1	6.61	13.40	1.00	81	100.0
2	17.70	5.02	0.30	13	16.0
3	21.11	4.22	0.30	27	33.4
4	26.77	3.34	0.30	31	38.7
NaOH-treated pure Silonijan Clay (C6)					
1	6.94	12.77	0.80	64	4.8
2	12.43	7.14	0.20	1325	100.0
3	19.91	4.47	0.20	132	10.0
4	20.39	4.36	0.15	149	11.2
5	21.32	4.17	0.20	132	10.0
6	23.09	3.86	0.40	61	4.6
7	24.93	3.58	0.30	818	61.7
8	26.62	3.35	0.20	62	4.7
9	28.60	3.13	0.50	14	1.0
10	32.51	2.76	0.20	16	1.2
Pure North Cachar Clay (C8)					
1	6.91	12.82	1.00	58	8.6
2	12.43	7.13	0.20	428	63.9
3	20.93	4.25	0.20	50	7.5
4	23.09	3.86	0.20	69	10.3
5	24.94	3.58	0.20	262	39.1
6	26.63	3.35	0.20	151	22.6
7	29.41	3.04	0.20	671	100.0
8	31.51	2.84	0.30	10	1.4
Thermally treated pure North Cachar Clay (C10)					
1	6.60	13.38	0.60	92	14.9
2	23.13	3.84	0.20	40	6.4
3	26.76	3.33	0.15	142	22.8
4	29.45	3.03	0.25	620	100.0
5	31.48	2.84	0.30	14	2.3
Standard Kaolin KGa-1b (C11)					
1	6.73	13.16	0.30	86	8.0
2	9.47	9.35	0.20	37	3.5
3	12.50	7.10	0.25	1076	100.0
4	20.01	4.44	0.20	169	15.7
5	20.44	4.35	0.20	174	16.2
6	21.32	4.17	0.10	121	11.2
7	25.00	3.57	0.25	548	50.9
8	26.45	3.38	0.30	17	1.6
9	28.69	3.12	0.40	8	0.7
10	32.73	2.74	1.00	7	0.6
Standard Montmorillonite SWy-2 (C12)					
1	6.77	13.09	0.30	216	86.6
2	13.81	6.43	1.00	13	5.2
3	19.87	4.48	0.30	146	58.6
4	20.87	4.26	0.25	69	27.6
5	26.70	3.34	0.20	250	100.0
6	28.74	3.11	0.80	55	21.9
7	29.62	3.02	0.20	19	7.8

The kaolin sample KGa-1b produced an XRD pattern characterized by two most prominent peaks at 2θ values of 12.50° and 25.00° corresponding to *d*-values of 7.10 Å and 3.57 Å respectively. On the other hand, the montmorillonite sample SWy-2 showed a most prominent peak at 26.70° (*d* = 3.34 Å) and three other medium intensity peaks at 6.77°, 19.87°, and 20.87° with *d*-values of 13.09 Å, 4.48 Å and 4.26 Å respectively. The two XRD patterns showed the distinctive features of kaolinite and montmorillonite.

The XRD patterns for all the clay samples showed that the clay samples from the Karbi Anglong and North Cachar districts were almost pure kaolinite. The identical positions of prominent XRD peaks with those of standard kaolin KGa-1a support this. The principal XRD characteristics of the clay samples used in this work and the standard samples are summarized in Table 6.

Table 6: XRD characteristics of the clays

Clay sample		*d*-values, (Å)
North Cachar Clay	Pure	
	Thermally treated pure	
Silonijan Clay	Pure	
	Thermally treated pure	
	NaOH-treated pure	
Standard Samples	Kaolin KGa-1b	7.98(s), 4.43(mb), 4.24(mb), 4.16(mb), 3.56(s)
	Montmorillonite SWy-2	4.46(m), 4.25(w), 3.34(s), 3.10(mb)

IR Measurements

The principal IR frequencies of the clay samples C2 (pure Silonijan clay), C4 (Thermally treated pure Silonijan), C6 (NaOH treated pure Silonijan clay), C8 (pure North Cachar clay), C10 (Thermally treated pure North Cachar clay), C11 (kaolin KGa-1a), C12 (montmorillonite SWy-2) are given in Table 7.

OH-stretching region

All the clays gave distinct peaks in the OH stretching region. C2 gave three peaks at 3618, 3652, and 3691 cm⁻¹, which were in conformity with those obtained earlier[8]. For a highly crystalline kaolinite, four peaks are obtained at 3619, 3653, 3669, and 3695 cm⁻¹, which are recognized as the fingerprint of kaolinite[12]. The clay sample, C4, showed four peaks, pure Silonijan clay after treatment with NaOH (sample C6) gave three peaks, pure North Cachar clay (C8) and the thermally treated pure North Cachar clay (C10) showed identical behavior to the Silonijan samples C2 and C4. The Kaolinite sample KGa-1b (C11) also gave only three peaks in the OH-stretching region. The montmorillonite sample SWy-2 (C12) gave peaks in this region at 3619, 3646, and 3673 cm⁻¹. These results indicate that the clay samples collected from Karbi Anglong and North Cachar in Assam contained kaolinite as the dominant clay mineral.

Table 7: Principal FTIR signals (cm⁻¹) of the clay samples

C2	C4	C6	C8	C10	C11	C12
468 (s)		473 (s)	469 (s)	461 (w), 484 (w, b)	472 (s)	460 (w), 476 (w)
542 (s), 568 (w)	588 (w)	551 (s)	546 (s)	572 (w)	538 (s), 554 (w), 572 (w, b)	512 (s), 552 (s), 587 (w)
646 (b), 686 (s)	682 (w, b)	652 (w, b), 684 (s)	649 (w, b)	614 (w, b), 647 (w, b), 666 (w, b)	650 (w, b), 685 (s, b)	
753 (s), 789 (s)		753 (s), 790 (s)	705 (s, b), 753 (w, b), 791 (w, b)	711 (s)	753 (s), 788 (s)	724 (s), 773 (w)
	805 (w, b)		873 (s)	871 (s)		801 (w), 875 (w, b), 896 (w)
915 (b)		914 (s)	914 (s), 935 (w)	986 (s, b)	914 (s)	976 (s)
1009 (w, b), 1034 (w), 1089 (w)	1067 (s, b)	1004 (w), 1056 (w), 1093 (w)	1004 (w), 1034 (w), 1090 (w)	1019 (w), 1065 (s, b)	1007 (s, b), 1033 (w, b), 1047 (w, b), 1081 (w, b)	1019 (w), 1052 (s)
1115 (w)		1114 (w)	1116 (w, b)	1165 (s, b)	1112 (w, b)	1164 (s)
						3526 (w), 3566 (w), 3590 (w)
3618 (s), 3652 (w, b), 3691 (s, b)	3602 (s), 3647 (w), 3667 (w), 3706 (w)	3619 (s), 3651 (w, b), 3692 (s, b)	3618 (s), 3649 (w, b), 3690 (w)	3607 (w), 3641 (w, b), 3672 (s, b), 3743 (s)	3619 (s), 3650 (w, b), 3689 (s, b)	3619 (w, b), 3646 (s), 3673 (w)

s: strong, w: weak, b : broad

The following regions were given special emphasis in IR data analysis:
(i)   OH stretching region : 3620 – 3700 cm⁻¹
(ii) SiO stretching region : 1000 – 1120 cm⁻¹
(iii) OH bending region : 910 – 940 cm⁻¹
(iv) SiO bending region : 400 – 550 cm⁻¹

SiO stretching region

In this region, the pure Silonijan clay (C2) gave four peaks, the thermally treated clay (C4) gave only one peak, and after treatment with NaOH, the pure Silonijan clay (C6) again gave four peaks in slightly shifted positions. A well crystalline kaolinite sample usually shows four SiO stretching frequencies at 1011, 1034, 1105 and 1117 cm⁻¹ according to Farmer[12]. The frequencies recorded in the present work in the SiO stretching region agree quite well with Farmer's results, excepting the thermally treated samples, where the deviations are not unexpected.

The pure North Cachar clay (C8) showed four IR peaks, while the thermally treated form (C10) gave two peaks for the SiO stretching region. The standard samples kaolin KGa-1a and montmorillonite SWy-2 [C11 and C12] yielded four and three peaks respectively.

The good resolution of the SiO stretching frequencies obtained in the present work and their agreement with well-known published work show that

the kaolinite samples from Karbi Anglong and North Cachar were of reasonably pure kaolinite. The good resolution indicates the clay particles to be of appropriate uniform size and shape.

OH bending region

Farmer[12] from his exhaustive work on clay minerals showed that the OH bending frequencies appear in two ranges i.e. 912 – 915 cm^{-1} and 935 – 940 cm^{-1}. Lyon [13] has found that the 935 cm^{-1} band is diagnostic of the kaolinite clay. In the present work, the pure Silonijan clay did not give a band at 935 cm^{-1}, but it gave a strong, broad band at 915 cm^{-1}, which might have masked the other band. As in the other cases, thermal treatment of this clay (C4) resulted in alteration of the clay surface and therefore, no OH bending frequencies could be detected. This is also true for the thermally treated North Cachar clay (C10). The pure North Cachar clay (C8) gave one strong band at 914 cm^{-1} and a weak band at 935cm^{-1} in conformity with the literature values. The pure Silonijan clay after treatment with NaOH (C6) continued to show a strong band at 914 cm^{-1} corresponding to OH bending frequencies and indicating that treatment with NaOH does not alter the clay surface structure. The KGa-1b kaolin sample (C11) showed only one OH bending frequency at 914cm^{-1} whereas the SWy-2 montmorillonite sample (C12) did not show any frequency in the OH bending region.

CONCLUSION

The following conclusions could be drawn from the above results:

i.   The clays from the districts of Karbi Anglong and North Cachar are definitely identified as kaolinite.

ii. The process of thermal treatment was destructive to the clay surface. Both XRD and FTIR measurements supported this.

iii. The treatment of the clay surface with NaOH did not change the structural features to any large extent and the characteristic XRD peaks and IR bands continued to appear almost in the same positions as they appeared for the pure samples.

iv. Comparison of the results with the standard, well crystalline kaolin sample (KGa-1b) indicated that the clays used in the present work had comparable purity, crystallinity and uniformity in size and shape of the particles.

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