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Characterization Of Clay Samples Separated From Subsurface Alluvial Soils Of Tinsukia And Jorhat Districts, Assam, India

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Abstract: *It has been observed from chemical analysis of the clay samples separated from sub-surface alluvial soils of Tinsukia and Jorhat districts of Assam that the percentage of major constituents like silica, alumina and iron were in the range of 40.50-53.03, 23.41-32.34 and 4.62-14.24 respectively. X-ray diffraction pattern of the clay samples confirmed the presence of different minerals along with the impurities. The major minerals found are kaolinite, halloysite and mixed kaolinite-halloysite minerals. The impurities found in the clay samples are quartz, calcite, goethite, siderite, hematite and pyrite.*

Keywords: *Clay, Kaolinite, Halloysite*

INRODUCTION

The term clay actually designates naturally occurring material composed primarily of fine grained [1] mineral, which is generally plastic at appropriate water contents and will harden when dried or fired. Clay minerals consist of aluminosilicates, which are mostly layered. Clay mineralogy is a multi-disciplinary science and hence clay may signify entirely different things to different disciplines like geologists, engineers, chemists, agronomists, pharmacologists, ceramists etc. A chemist studies clay as catalyst, adsorbent, filler or as hybrid nanocomposite with polymer or other materials[2]. Because clays have layered lattice structures [3], two types of surface area are recognized: internal and external. The external surface area expose for adsorption of molecules of gas or solute and in other hand internal surface area is available for adsorption between two crystal sheets. Clay produced good results for treating hazardous wastes and catalyzing organic reactions [4-7]. It is also used for removal of water pollutant such as fluoride or arsenic in drinking water [8-10].

X-ray Diffraction analysis for Identification of clay minerals:

The layer silicates are arranged in planes with equal spacing which is called d spacing. This plane is given the number 001, on the basis of Miller indices system. Most layer silicates have same length of the edges of unit cell and thus 001 spacing plays a fundamental role in identification of clay minerals [11,12].

The X-ray diffraction is the most widely used technique in the identification of clay. Generally, X-ray with $\lambda = 1.5404$ Å is used with Cu-Kα radiation. The diffraction follows the Bragg's relation

$n\lambda = 2d \sin\theta$

where d = spacing between atomic planes in the crystal
λ = wavelength
θ = glancing angle of diffraction
n = order of diffraction

From X-ray diffraction, the average particle size can be calculated by using Scherrer's formula [13,14]

$$t = k\lambda / B\cos\theta$$

where t is the average size of the particles assuming particles to be spherical, k = 0.9, λ is the wavelength of X- ray radiation, B the full width at half maximum of the diffracted peak and θ_B the angle of diffraction.

Infrared Spectral Analysis:

Infrared spectroscopy is used extensively for qualitative determination of clay constituent during recent years [15,16]. IR spectroscopy is also used to study the intercalation complex formed between clay minerals and other compounds.

The necessary frequency range for IR studies of clay minerals is from 4000 to 400cm^{-1} and it has been found that the IR vibrations are sensitive both to structural and to compositional vibrations in the clay minerals [17,18].

REVIEW OF LITERATURE:

The identification of the principal clay-mineral species is extremely important in both mineralogical and geochemical studies. Yoshiki[19] first identified the *kaolin* mineral in the Shokozan area, Japan. He identified the clay obtained from Hiroshima Prefecture, as *dickite* on the basis of its optical and thermal properties. *Dickite* occurs as lenticular masses associated with nacrite, diaspore, or alunite [20].

Several hydrothermal *halloysite* deposits are known. At the Jashine mine, Gunma Prefecture, *halloysite* occurred with small amounts of opal and tridymite. Other examples of hydrothermal *halloysite* deposits have been reported from the Omura mine, Nagasaki Prefecture[21], the Okuchi mine, Kagoshima Prefecture[22] and the Rangoshi mine, Hokkaido.

Kaolinite is an important product of weathering. In fact, *kaolin* minerals are the most abundant and widely distributed constituents of Japanese soils[23]. Although *halloysite* with spherical or tubular morphology has been reported in soil [24], the form of the *kaolin* mineral in soils is usually platy. Such platy *kaolin* has sometimes been referred to as *meta halloysite* due to its low structural integrity [25].

Chandrasekhar [26] studied the clay from the village Udiaguda, Koraput district of Orissa state in the south eastern part of India. From X-ray diffraction and thermal-analysis, they found that *kaolinite* is the major mineral, ancillary impurities are quartz and pyrite which are present in varying amounts. The high acidity of the clay and the presence of soluble salts like sulphates and chlorides is found to create problems in processing. Decomposed products of pyrite may be limonite or different types of iron sulphates which are water soluble and poses flocculating effect. The *kaolinite* content increases from 85 to > 95% during size classification as indicated by the quantitative XRD analysis.

Borthakur et al.[27], studied the clay from Deopani, Assam of northeastern region of India. They characterized the clay by FTIR, XRD, DTA and by wet chemical analysis methods. The major impurities in the characterized *kaolinite* clay are *quartz* and *siderite*. They reduced the iron content of the clay by leaching with organic acids as well as by treating with wet high intensity magnetic separation (WHIMS). The non-structured iron may be removed completely at room-temperature by using 0.4M oxalic acid solution.

MATERIALS AND METHODS:

The alluvial subsurface soils (from which clay samples were separated) were collected from the following places of Tinsukia and Jorhat district of Assam, India. The locational index in terms of latitude and longitude is given within the bracket. Depending upon the topography of the sample collection point, the soil was collected from a depth of 0.5 to 2 meter.

1. Digboi (23^{0}23′28″ N, 95^{0}37′09″E).
2. Tinsukia (27^0 29′25″N, 95^0 21′ 29″E).
3. Makum (27^0 29′ 13″N, 96^0 26′ 20″E).
4. Ledo (27^0 17′ 30″N, 95^{0}21′ 29″E).
5. Dumduma (27^0 33′ 53″N, 95^0 33′ 32″E).
6. Dhola (27^0 45′ 04″N, 95^0 35′ 43″E).
7. Margherita (27^0 17′39″N, 95^0 39′ 23″E).
8. Harudhadum (27 40′ 30″, 95^0 34′ 0″E).
9. Rongagora (27^0 30′ 42″N, 95^0 20′ 05″E).
10. Bongaon, Majuli (26^0 50′ 52″N, 94^0 10′ 0″E).
11. Dergaon (26^0 43′ 34″N, 93^0 59′11″E).
12. Bahona, Jorhat (26^0 46′ 54″N, 94 12′ 07″E).

The chemical used are:
Zinc Chloride (dry purified, Merck)
Nickel Chloride (LR, Ranbaxy), purified by recrystallization from alcohol.
Cobalt Chloride (LR, Ranbaxy), purified by recrystallization from alcohol.
Copper Acetate (LR, Ranbaxy), purified by recrystallization from alcohol.
Benzene (LR, Ranbaxy), purified by distillation.
Methyl Alcohol, (GR, Merck)
Ethyl Alcohol, (LR, Bengal Chemicals).
Pyridine (LR, Merck) purified by distillation.
p-Cresol (LR, s.d. fine Chem Ltd)
Castor oil (Avon Herbal India Limited)
Oleic acid (AR, Loba Chemie)
Stearic Acid (AR, Loba Chemie).
Lauric Acid (AR, Loba Chemie).
Sodium Fluoride (AR, Ranbaxy).

Polyvinyl Alcohol (AR, Merck)
Ethylene Glycol (LR, Loba Chemie).

X-Ray Diffraction:

X-ray diffraction pattern of the finely powdered clay samples was recorded on a Philips PW 1700 APD diffractometer at USIC, Gauhati University, Guwahati as well as at Materials Science Division, NEIST, Jorhat, Assam (model no, Ultima IV, Mac Rigaku, Japan using Cu Kα radiation.

IR-Spectrophotometer:

The IR spectra were obtained as KBr pellet by using Perkin Elmer Spestro-photometer, model no. 883 at Dibrugarh University as well as by Perkin Elmer, model no.1600 FTIR in the Department of Chemistry, Gauhati University.

Separation and Purification of Clay[28]:

100 g of the alluvial soil, source of clay was first powdered in a mortar and then sieved through 200 mesh and retained on 400 mesh ASTM sieve. 20 g of this material was dispersed in 1000 ml of distilled water with constant stirring. This was then allowed to stand for ~ 3 ½ h. 50 ml of the suspended solution was drawn out into a beaker. The process was repeated by re-adding 50 ml of water to the cylinder for 4 to 5 times. The clay solution in beaker was evaporated in a water bath to make a slurry. The slurry was then kept in an electric oven at 60^0C. The obtained clay fraction containing a large quantity of organic matter was treated with sufficient quantities of hydrogen peroxide to remove the organic matter. The resulting slurry was kept in the electric oven at 60-70^0C to remove excess hydrogen peroxide. The dried clays were ground and kept in well stoppered bottles.

CHARACTERIZATION OF CLAY:

Chemical Analysis:

To determine the percentage of chemical constituent of clays the following methods were adopted.

The quantitative analysis of silica, sesquioxide, MgO and CaO were done by using the same solution of clay sample as follows:

1 g of a clay sample was digested with 10 ml of conc. HCl (AR, BDH), 2 ml of conc. HNO_3 (LR, BDH) and small amount of saturated bromine water (GPR, BDH) in a 100 ml beaker. The clay samples with acids were evaporated to dryness in a water bath. This was repeated for 2-3 times by treatment with the acids viz. conc. HCl and conc. HNO_3.

Determination of Silica:

To the clay sample described above 10 ml of distilled water was added and then boiled and filtered through Whatman filter paper no. 42. The filtrate was preserved for finding the percentage of other constituents. The filter paper containing residue was ignited at 1000^0C in a previously weighed platinum crucible till getting constant weight. The difference in weight between crucible and the crucible containing silica gave the amount of silica. The exact amount of SiO_2 was determined by adding 1 ml of water, two drops of conc. H_2SO_4 and 5 ml of HF acid. The crucible was heated on a hot plate slowly. The loss in weight represents the exact weight of silica, SiO_2.

Determination of Sesquioxide[29]:

The filtrate after filtering silica was taken in a 250 ml beaker. To this beaker 3 to 4 gms of solid NH_4Cl (AR, BDH) was added and boiled. The hydrated sesquioxide was precipitated in boiling solution by adding 1:2 ammonia (AR, BDH) solution. After filtration, the precipitate was washed with hot 2 % NH_4NO_3 (LR, BDH) solution. The precipitate was burnt in a previously weighed silica crucible at 800^0C till attaining constant weight. The weight difference gave the amount of sesquioxide.

Determination of CaO and MgO[29]:

The filtrate obtained after filtration of sesquioxide was preserved for Ca and sesquioxide was preserved for Ca and Mg estimation in a 250 ml volumetric flask and made the volume with distilled water.

25 ml of the solution was transferred to a conical flask and to it 25 ml of distilled water, 4 ml of 8 M KOH solution and 0.02 g of HHSNNA indicator were added. The mixture was titrated with standard EDTA solution until colour changes from wine red to pure blue.

A blank titration was carried out by replacing the sample solution with distilled water. The difference of sample titre value and blank titre value gave the actual volume of EDTA and the amount of CaO was calculated this way.

To determine the amount of combined Mg^{2+} and Ca^{2+}, 25 ml of the solution from the same volumetric flask was transferred to a conical flask. The solution in the conical flask was diluted with 50 ml distilled water and to it 5 ml buffer solution of pH range 10-12, and 2 drops of EBT indicator were added. This was then titrated with standard EDTA solution to a pure blue end point. A blank titration was done with 25 ml distilled water in the same way. The difference between the sample titre value and blank value gave the actual volume of EDTA and the amount of combined Ca^{2+} and Mg^{2+} was calculated.

The amount of Mg^{2+} or MgO was calculated from the difference of combined amount of Ca^{2+} and Mg^{2+} and the amount of $Ca^{2+.}$

Determination of Total Iron:

1 g of the clay sample was digested with 10 ml of conc. HCl. After digesting the sample 30-40 ml of distilled water was added and heated to boiling. A few drops of stannous chloride solution were added with constant stirring till the solution become colourless. The solution was cooled to room temperature and then 10 ml saturated mercuric chloride solution and 10 ml of dilute (1:1) phosphoric acid were added. The solution was then titrated adding two drops of diphenylamine indicator with standard potassium dichromate solution with bluish violet coloured end point. The amount of iron oxide was calculated from the volume of potassium dichromate.

Determination of Sodium and Potassium [30]:

A standard curve was prepared by using NaCl (AR) of least six different concentrations. Then the clay sample was introduced in the flame photometer and reading was recorded. From the standard curve the concentration of sodium in each of the clay sample was determined.

For determination of potassium a standard curve was prepared for KCl (AR) of different concentrations. 5 ml of the filtrate obtained from sesquioxide determination was taken in a 50 ml volumetric flask and thus diluted by a factor of 10 times. The solution was then introduced in the flame photometer, and reading was recorded. From the standard curve concentration of potassium in the clay sample was determined.

Determination of loss on ignition (H_2O^+):

1 g of the sample was taken in a previously weighed crucible and burnt in an electric muffle furnace at 850 ^{0} C to constant weight. The loss in weight gave the loss on ignition.

XRD Analysis:

The oriented samples were air dried, glycolated and heated at 500 ^{0}C. A 2% clay suspension was spread homogeneously on a clean microscopic slide. The slide was allowed to dry undisturbed in a desiccator for 24 h. Another slide was placed in a desiccator which contained ethylene glycol and was saturated with glycol vapour in an electric oven at 60^{0}C for 24 h. Another slide was heated was heated at 500^{0}C in a muffle furnace for 4-5 h.

The XRD spectra of the different samples were also taken as powdered sample.

Particle Size Measurement from XRD:

The average particle size of different clay samples was calculated from X-ray diffraction spectrum by measuring the full width at half maximum of the diffracted peak and the angle of diffraction. These values were putting in Scherrer's formula to calculate the average particle size. The Scherrer's formula[13,14] is

$t = k\,\lambda/\,B\mathrm{Cos}\theta$

where t is the average size of the particles assuming particles to be spherical, k = 0.9, λ is the wavelength of X- ray radiation, B the full width at half maximum of the diffracted peak and θ_B the angle of diffraction.

RESULT AND DISCUSSION:

Chemical Analysis:

The chemical constituents of clay samples collected from various locations were determined by both usual analytical and instrumental techniques and are listed in Table 1. The variation in composition of clay samples may be due to the presence of different amount of clay minerals present in different locations. The percentage of major constituents like silica, alumina and iron were in the range of 40.50-53.03, 23.41-32.34 and 4.62-14.24 respectively. This may also reflect the depositional history and weathering pattern prevalent in this region.

Table 1: Chemical Analysis of clay samples

Sample No.	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %	K_2O %	Na_2O %	Loss on ignition %	Total
1, Digboi	42.86	23.60	14.24	0.86	2.22	1.83	0.33	12.01	97.95
2, Tinsukia	50.36	24.32	5.26	0.72	5.12	1.24	0.23	11.20	98.45
3, Makum	43.24	25.30	10.43	2.11	4.01	0.91	0.74	12.02	98.76

4, Ledo	53.03	23.41	9.62	1.01	0.31	0.78	0.22	10.98	99.36
5, Dumduma	46.62	27.10	5.00	0.83	6.23	0.99	0.41	12.80	99.93
10, Bongaon	43.11	32.34	5.15	0.92	5.34	0.15	0.35	12.33	99.69
11, Dergaon	40.50	29.00	6.03	0.15	7.54	1.58	0.75	12.80	98.35
12, Bahona	41.44	31.00	7.13	1.11	0.94	1.20	0.47	10.80	94.09

IR Spectral Analysis:

The IR spectra of sample 1 and 10 are shown in the Figs.1 and 2 respectively. The IR spectrum of clay sample no. 1 shows the presence of Al-Al-OH stretching vibration band at 3698 cm^{-1} and 3621 cm^{-1} respectively. A sharp band at ~915 cm^{-1} is indicative of *kaolinite*[16]. The bands at ~1402 cm^{-1} and doublet near 798 cm^{-1} are due to the presence of *quartz* impurity[31].

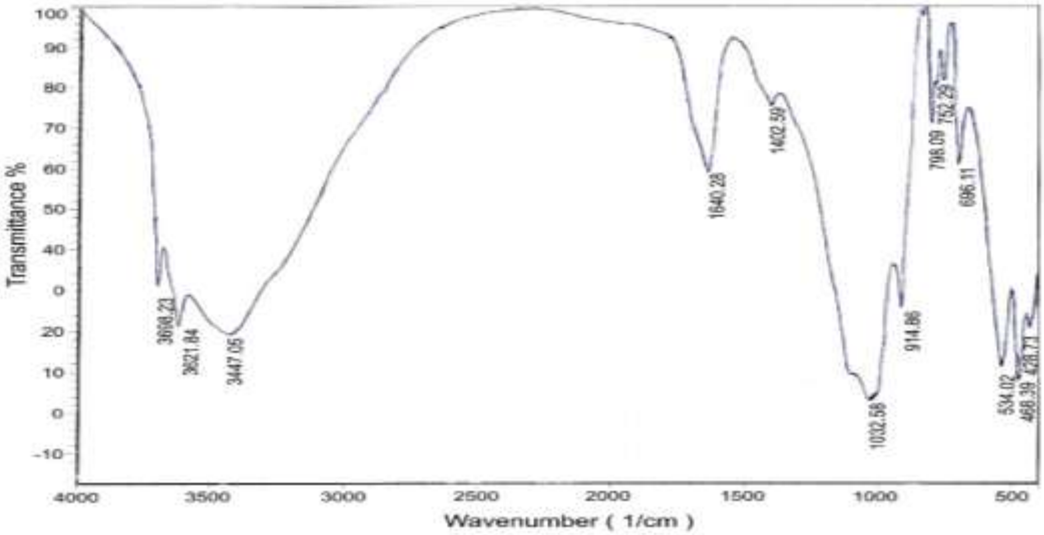

Fig.1: IR spectrum of clay sample no.1, Digboi,($23^0 23'28''$ N, $95^0 37'09''$E)

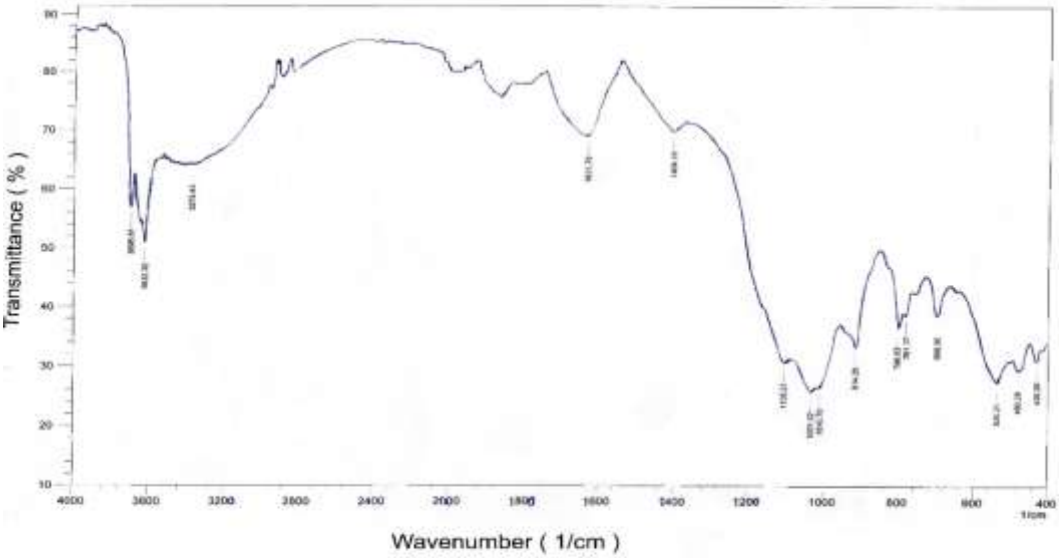

Fig. 2.: IR spectrum of clay sample no.10, Bongaon, Majuli,
(26^0 50′ 52″N, 94^0 10′0″E).

The doublet at 938 cm^{-1} and 915 cm^{-1} in the IR spectrum of clay sample no.11 is also characteristics of *kaolinite*.

A common IR band seen at 1630±5 cm^{-1} is due to deformation vibration of adsorbed water [33]. The symmetric and asymmetric stretching vibrations of adsorbed water give rise to a broad band centered near 3400 cm^{-1}. The clay minerals, the layered silicates contain the planar hexagonal silicon-oxygen (Si-O) network and the Si-O stretching vibration in this study is located [35] in the region 700 to 1200 cm^{-1}. Most of the IR-spectra of the clay samples shows a medium band near 915±5 cm $^{-1}$ as it is a common band for both *kaolinite* and *halloysite*. The band at 938 cm $^{-1}$ which is seen in the IR spectrum of clay sample no. 11 is not seen in other clay minerals. Though this band can strongly identify the presence of *kaolinite* minerals, is not seen in mixed clay minerals or when the percentage of *kaolinite* is below 50.

X-ray Diffraction Analysis:

The clay samples are essentially comprised of minerals and their composition has been verified by X-ray diffraction patterns which are depicted in Figs. 1, 2, 3, 4, 5 and 6. The d- values of the clay samples showed that they are mixed clay minerals such as *kaolinite* along with *halloysite* and other impurities or *halloysite* with muscovite and other impurities. Some of the samples are also predominantly *kaolinite* having other impurities. The impurities found in these samples are *quartz, calcite, goethite, siderite, hematite and pyrite*. The presence of *kaolinite* clay mineral is characterized by the prominent basal reflection at 7.13 Å and 3.56 Å which disappeared [32] upon heating at 500^{0}C and unaffected on glycolation.

The *halloysite* clay mineral is identified by basal[22] spacing at 9.97 Å -10.1 Å, 7.09 Å, 4.47 Å, 3.34 Å and 2.56 Å respectively.

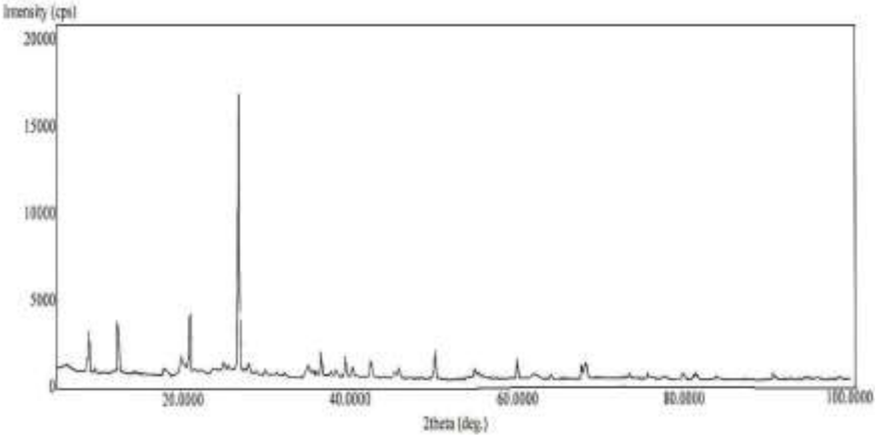

Fig.1: XRD of Dumduma Clay

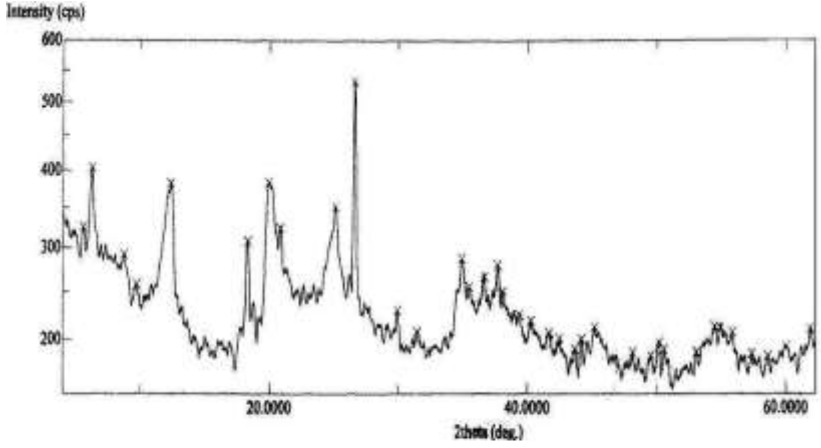

Fig.2: X-ray Diffraction Pattern of Dhola Clay

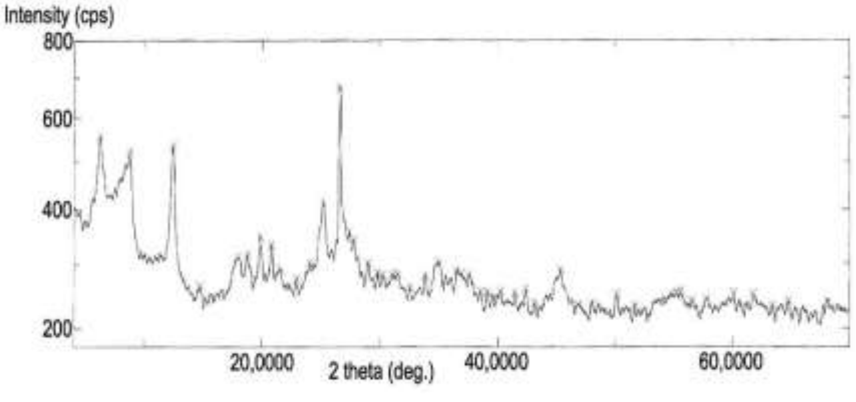

Fig.3: X-ray Diffraction pattern of Margherita Clay

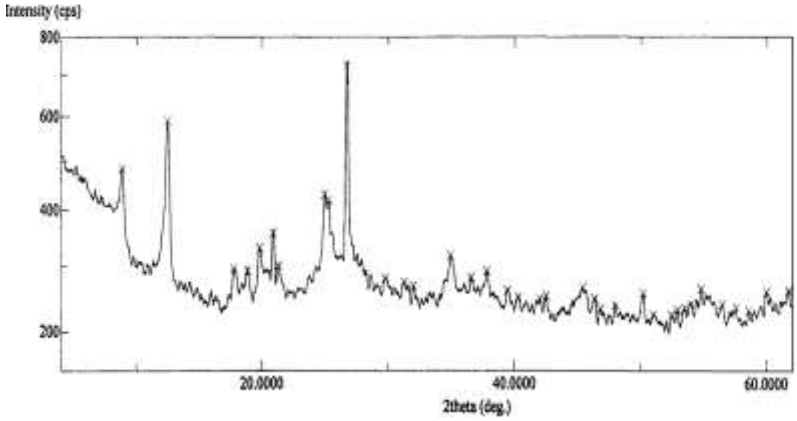

Fig.4: X-ray diffraction Pattern of Harudhadum Clay

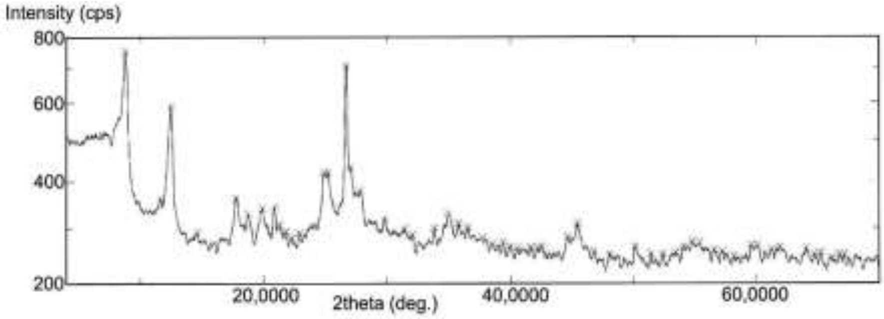

Fig.5: X-Ray Diffraction Pattern of Rongagara Clay

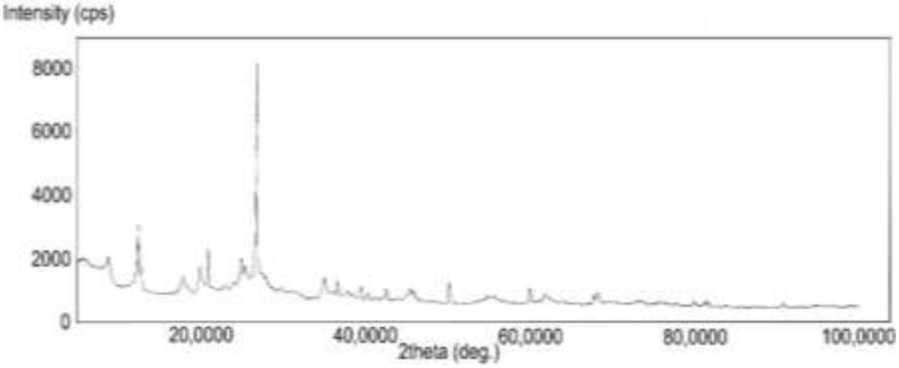

Fig.6: X-Ray Diffraction Pattern of Bahona Clay

Majority of clay contains high amount of *quartz* and *calcite* and also contains both *feldspar* and *muscovite*. Traces of pyrite and *siderite* are also present.

The d values at 4.26 Å, 3.34 Å, 2.45 Å are due to the presence of *quartz*. The presence of calcite is identified by the basal spacing at 3.53 Å, 2.99 Å and 2.79 Å. The peaks at 2.23 Å is also indicative of pyrite along with the peaks at 2.43 Å, 2.02 Å, 1.89 Å and 1.62 Å. *Feldspar* is identified by d values at 3.99 Å, 3.20 Å and 1.81 Å respectively. The peak at the d value 1.81 Å is also indicative of *quartz*. *Geothite* peaks are identified by basal reflections at 4.16 Å, 2.45 Å and 1.72 Å respectively. The occurrence of the peaks at 3.53 Å, 2.84 Å and 1.72 Å indicate the presence of *siderite* in sample 6, Dhola clay. *Siderite* is also present in sample 9. The presence of hematite is also seen in sample 6 and 9. The characteristic peaks for *hematite* are at 2.52 Å, 1.83 Å and 1.68 Å.

The electronic spectrum of clay minerals may not give much information however; the electronic spectrum of clay sample no. 11 shows the weak band around 20400 cm^{-1} which may be due to d-d electron transfer between neighboring Fe^{2+} and Fe^{3+} ions (i.e. $Fe^{2+} \rightarrow Fe^{3+}$). Charge transfers [34] is facilitated along the chains of linked tetrahedra and octahedra.

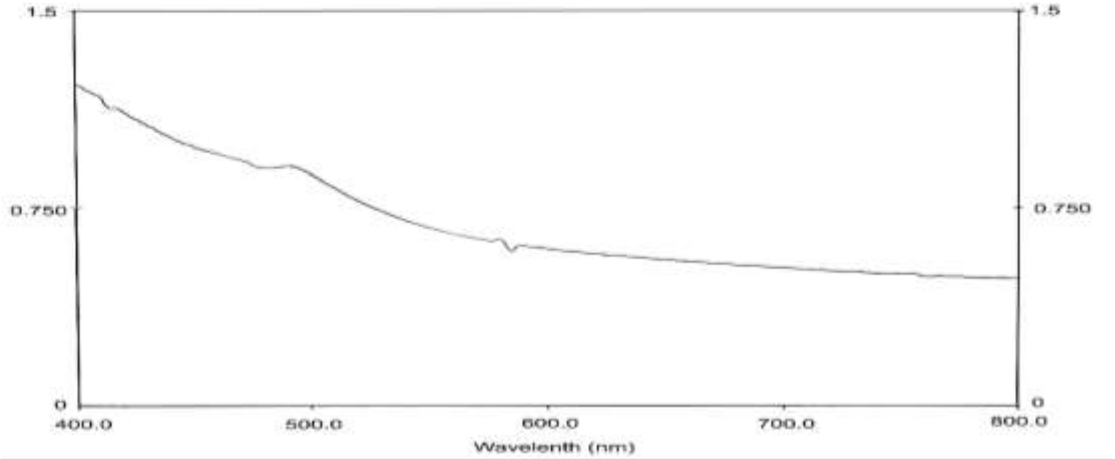

Fig. 7: Reflectance spectrum of sample 11, Dergaon
(26^0 43′ 34″N, 93^0 59′11″E).

Table 2: XRD results of different clay samples

Sample No.	**Minerals present**
1. (Digboi)	Predominantly *kaolinite* with maximum *quartz* impurities.
2. (Tinsukia)	*Kaolinite – halloysite* mixed minerals along with *muscovite*, *quartz* and *feldspar*.
3. (Makum)	Predominantly *halloysite* containing *quartz* and *muscovite impurities*
5. (Dumduma))	*Kaolinite-halloysite* mixed minerals with maximum *quartz* impurities. Traces of *pyrite* and *calcite* impurities are also present.
6. (Dhola)	Mainly contains *kaolinite* along with *halloysite*. It also contains high amount of *quartz* and *calcite*. Other minerals present are *muscovite*, *hematite* and traces of *pyrite* and *siderite*.
7. (Margherita)	It contains high amount of *quartz* and *calcite*. The sample also contains both *feldspar* and *muscovite*. Traces of *pyrite* is also present.
8. (Harudhadum)	It is *halloysite* but it contains high amount of *quartz* and *calcite*. *Muscovite* and *goethite* are also present. Other trace minerals are *siderite* and *pyrite*.
9. (Rongagora)	Predominantly *halloysite* with considerable amount of *quartz*. The presence of feldspar was observed. It also contains a traces of *hematite*, *siderite*, *pyrite* and *goethite*.
10. (Bongaon, Majuli)	Predominantly *kaolinite* with considerable amount of *quartz* impurity.
11. (Dergaon)	Mainly *kaolinite* with *quartz* and *calcite* impurities.
12. (Bahona,Jorhat)	*Kaolinite –halloysite* mixed clay with high amount of *quartz* impurity.

CONCLUSION

It has been observed from chemical analysis of the clay samples separated from sub-surface alluvial soils of Tinsukia and Jorhat districts of Assam that the percentage of major constituents like silica, alumina and iron were in the range of 40.50-53.03, 23.41-32.34 and 4.62-14.24 respectively.

From IR spectral studies it is seen that most of the IR spectra of the clay samples showed a medium band near 915 cm^{-1} as it is a common band for both *kaolinite* and *halloysite*. As the IR spectrum of the clay sample no.11 showed 938 cm $^{-1}$ band, it indicates that the clay sample no. 11 contains more than 50% *kaolinite*.

X-ray diffraction pattern of the clay samples confirmed the presence of different minerals along with the impurities. The major minerals found are *kaolinite, halloysite* and mixed *kaolinite-halloysite* minerals. The impurities found in the clay samples are *quartz, calcite, goethite, siderite, hematite* and *pyrite*.

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