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Studies on Characterization and Mechanical Behavior of Natural Clay

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Abstract

This paper analyses the results of an experimental study based on the engineering properties of clay samples collected from both the banks of river Nagavali flowing through Srikakulam, India. 5 samples are collected from each bank at an interval of 200cm and each at a depth of 100cm. The physical and mechanical properties of these samples are determined. The clay is characterized by Scanning Electron Microscopy (SEM) and Energy-Dispersive X-Ray diffraction (XRD). The samples of both the banks are compared with respect to the properties. The detailed test results and observations are presented and conclusions are drawn.

Keywords: *Engineering properties, Physical properties, Mechanical properties, XRD, SEM.*

1. Introduction

In this present study the clay samples are collected from both the banks of river Nagavali flowing through Srikakulam, India. 5 samples are collected from each bank at an interval of 200cm and each at a depth of 100cm. The properties of clay depend significantly on its initial conditions. These samples contain layered silicates.

1.1 Layered silicates

The layered silicates are natural or synthetic minerals consisting of regular stacks of aluminosilicate layers with a high aspect ratio and high surface area. Layered silicates are easily available and are of low cost. Clay is referred to a part of soil fraction with the particle size of less than 2 µm. The clay layers have a thickness of about 1 nm which is in the nanoscale. There are many members of clays with some difference in their formulae, structure and properties including swelling and exfoliation. Those members who are able to be exfoliated by polymer chains or monomers and distributed as individual clay layers within polymer matrix are suitable for the preparation of polymer nanocomposites. The individual clay layers are responsible for the dramatic improvements in polymer properties due to their high aspect ratio and high interfacial interactions with polymer matrix.

1.2 Structure of clays

Clays have layered structure. The layers are built from tetrahedral sheets in which a silicon atom is surrounded by four oxygen atoms and octahedral sheets in which a metal like aluminum or magnesium is surrounded by eight oxygen atoms. The tetrahedral (T) and octahedral (O) sheets are fused together by sharing the oxygen atoms. Unshared oxygen atoms are present in hydroxyl form. Two main arrangements of tetrahedral and octahedral sheets fused together make the one layer structure of clay. One tetrahedral sheet fused to one octahedral (1:1) sheet is known as kaolin group with the general composition of $Al_2Si_2O_5(OH)_5$ forms a layer thickness of ~ 0.7 nm [1]. The crystal lattice consists of one octahedral sheet and wished between two tetrahedral sheets (2:1) with a total thickness of 0.94 nm and is well-known as phyllosilicates.

The 2:1 phyllosilicate layers can be electro statically neutral with no inter layer ion present known as pyrophyllite. Due to non presence of inter layer ions, the layers do not expand in water. When silicon in tetrahedral sheets is substituted by aluminum, the 2:1 structure is called mica. The negative charge induced by this substitution is balanced by the insertion of potassium cat ion between the layers. Due to the equal size of potassium cat ion and the hole created by Si/Al tetrahedral sheets, the presence of potassium cat ion makes no inter layer spacing. Consequently the 2:1 layers are held together strongly and the swelling or exfoliation of layers is not possible. When in neutral pyrophyllite, the aluminum cat ions in the octahedral layers are partially substituted by divalent magnesium or iron cat ion when the smectite clay group is formed. The negative charge

created by this substation is balanced by inter layer counter sodium or calcium ions. The charge created on the layers is not locally constant and varies from layer to layer. An average charge value is considered for the layers which can be determined by cat ion exchange capacity (CEC) measurement. Due to the unequal size, the layers stay near together with a regular gap between them. The gap is called as inter layer or gallery.

The thickness of the repeated units in a regular multilayer structure contained of one layer and one inter layer space is called d-spacing (d001) or basal spacing (figure 1). The basal spacing of clays can be measured or calculated from their X-ray diffraction patterns. The inter layer dimension is also dependent on the nature of clay and swelling or hydration degree of inter layer cat ion. The inter layer distance is about 1 nm for montmorillonite.

The electrostatic and van der waals forces holding the layers together are relatively weak and inter layer distance varies depending on the charge density on the layers, interlayer cat ion radius and its degree of hydration. Because of the inter layer spacing and weak interlayer forces, the cat ions present between the layer can be hydrated in aqueous solutions well known as clay swelling. The swelling causes to the more increasing of interlayer space. The charge density on the clay layers is different in various clay groups with different clay structures (Mittal, 2009) [2]. Due to the inter layer spacing and weak forces between layers, especially in the hydrated form, other molecules also can be intercalate between layers, leads to the expanding of layered lattice and finally may be caused to the separation of individual layers. The unique intercalation/exfoliation behavior of smectite clay minerals which is responsible to the high aspect ratio of this clay type makes them very important and powerful as reinforcing filler for polymers.

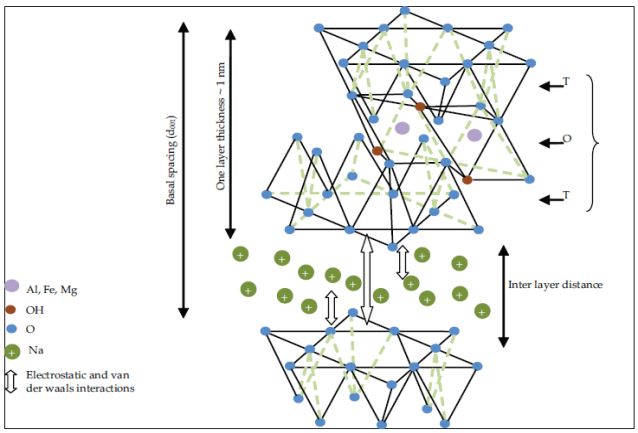

Figure 1: Scheme of the 2: 1 smectite clay structure (Samir M.A.S.A, Alloin F., Sanchez J.Y., Dufresne A., Cellulose nanocrystals reinforced poly (oxyethylene), Polymer, vol.45, 4149-4157, 2004 [3])

The dimension of 2:1 layer structures are about 1 nm in thickness and 30 nm to several microns in lateral depending on the source and preparation method of clay. The clay lateral dimensions and its ability to disperse into separated layers determine the aspect ratio of clay which is strongly important in polymer/clay interfacial interactions. Separation of individual clay layers having about 1 nm thickness display a perfect structure which strongly affects on the improvement of polymer properties.

However due to the higher internal surface in high degrees of dispersed structures, the internal forces for agglomeration of individual layers is also increases. Therefore the layers have tendency to hold together or aggregate. Several layers organized parallel together with the total thickness of about 10 nm make primary clay particles. Primary particles are laterally joined together and make micro aggregates. Aggregates are formed by randomly joining of micro aggregates and primary particles, and finally the staying of aggregates near together makes association in the smectite structures. Clay minerals are belonging to the nanoplatelet structured filler particles in polymer composite technology. Montmorillonite, Hectorite and Saponite are the well known smectites which are most commonly used in the preparation of polymer nanocomposites (table 1) (Pavlidou & Papaspyrides, 2008 [4]). Montmorillonite (MMT) has the widest acceptability for use in polymer nanocomposites because of its ease availability, well known intercalation/exfoliation chemistry, high surface area and high surface reactivity. The MMT layer aspect ratio can be in the range 1000 in well dispersed state without breaking of layer sand surface area of about 750 m2/g. Usually because of the breaking of clay layers in to small plates during preparation process of nanocomposites, the aspect ratio of MMT layers decreases to about 300-500.

Table 1: Chemical Structure of commonly used smectite type clays, m: monovalent cation, x: degree of cation isomorphous substitution in octahedral sheets, (pavlidou & Papaspyrides, 2008 [5]).

Smectites	Chemical formula
Montmorillonite	Mx(Al4-xMgx)Si8O20(OH)4
Hectorite	Mx(Mg6-xLix)Si8O20(OH)4
Saponite	MxMg6(Si8-xAlx)O20(OH)4

2. Experimental Works

In this study, Natural-montmorillonite clay is chosen as layered silicate reinforcement. 10 samples (Figure 2 & 3) are collected from both the banks of river Nagavali flowing through Srikakulam, India. On each bank at an interval of 200cm and at a depth of 100cm 5 samples are collected from Bank-A and 5 samples from Bank-B.

Figure 2: Location of Soil Sample Taken

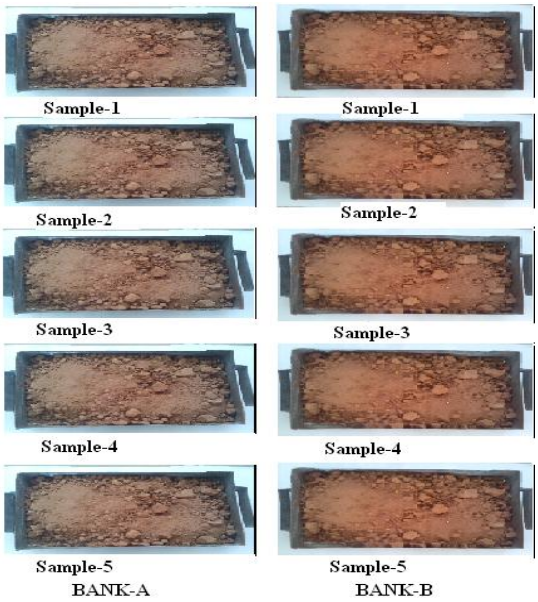

Figure 3: Different Clays from both the bank

The experimental procedures followed to determine their mechanical characterization are:

a) Specific gravity test
b) Hydrometer test
c) Unconfined compression test
d) Scanning and electron microscope (SEM)
e) X-Ray Diffraction (XRD)

2.1 Specific gravity test

This test is done to determine the specific gravity of fine-grained soil by density bottle (50ml capacity) method as per IS: 2720 (Part III/Sec 1) – 1980. Specific gravity is the ratio of the weight in air of a given volume of a material at a standard temperature to the weight in air of an equal volume of distilled water at the same stated temperature.

2.2 Hydrometer test

Hydrometer analysis is a widely used method of obtaining an estimate of the distribution of soil particle sizes from the No. 200 (0.075 mm) sieve to around 0.01 mm. The data are presented on a semi log plot of percent finer vs. particle diameters and may be combined with the data from a sieve analysis of the material retained (+) on the No.200 sieve. The principal value of the hydrometer analysis appears to be to obtain the clay fraction (generally accepted as the percent finer than 0.002 mm). The hydrometer analysis may also have value in identifying particle sizes < 0.02 mm in frost susceptibility checks for pavement sub grades. This test is done when more than 20% pass through No.200 sieve and 90% or more passes the No. 4 (4.75 mm) sieve.

Table 2: Calibrating chart for hydrometer and sedimentation jar

Hydrometer reading (Rh)	H= the distance of hydrometer reading point from the neck of hydrometer bulb measured in cm, R_h	Effective depth He (cm) $He = H + \frac{1}{2}\left[h - \frac{Vh}{A}\right]$ = H + 13.85
-5	12.7	19.62
0	11	17.92
5	9.3	16.22
10	7.5	14.22
15	5.8	12.72
20	4.1	11.02
25	2.4	9.32
30	0.6	7.52

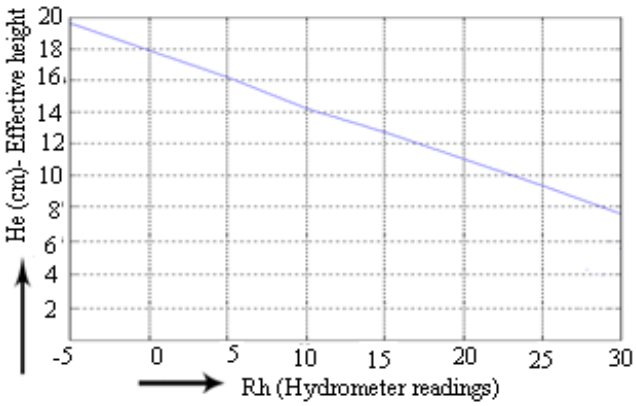

Figure 4: Calibration curve of hydrometer and sedimentation jar

Test Procedure for hydrometer

(a) Dispersing agent solution by mixing 33gm of sodium hexametaphosphate and 7 gm of sodium carbonate in distilled water for one liter of solution is prepared.
(b) The cleaned hydrometer is placed in a 1000 c.c of dispersing agent solution. 50 gm of pretreated fine dry soil passing through 75 micron sieve is added to this solution.
(c) The soil is placed in evaporating dish and covered with 100c.c dispersing solution, and warmed gently

for about 10 minutes. The sample is transferred to the cup of mechanical stirrer using distilled water, until the cup is ¾ the full and operate the mixer for about four minutes.

(d) After stirring, the specimen is washed in a 1000 c.c. jar and enough water is added to bring the level to 1000 c.c mark. The suspension in the jar is mixed thoroughly placing the palm of the hand on the open end of the jar and turning the jar upside down and back.

(e) The jar is placed on the table and the hydrometer is inserted slowly with least disturbance. Stop watch is read simultaneously. The top of meniscus at 0.5 min, 1min, 2 min, and 4min are noted carefully.

(f) After 4 min. the hydrometer, is removed outside and kept in the comparison cylinder, i.e., the 2nd identical jar, containing only the distilled water and dispersing agent solution. The temperature of the suspension is recorded for very precise computations.

(g) The hydrometer readings are noted for soil suspension at suitable times like 8 min, 15 min, 30min, 1 hr., 2hr., 8 hr., 24 hr., after the start of test. For taking each of these readings the hydrometer is inserted just before the reading slowly with as less disturbance as possible.

(h) For determining the composite correction c, record the hydrometer reading at the top of the meniscus in the comparison cylinder. The negative of this reading will indicate the composite correction. This composite correction is found before the start of the test and also at every time interval exceeding 30 minutes.

(i) Similarly, also record the meniscus correction (m) by noting down the difference of reading between the top of the meniscus and bottom of meniscus in the identical jar (comparison cylinder) at the start of experiment, only.

2.3 Unconfined compression test

The primary purpose of this test is to determine the unconfined compressive strength, which is then used to calculate the unconsolidated untrained shear strength of the clay under unconfined conditions. According to the AIM-264 standard, the unconfined compressive strength (q_u) is defined as the compressive stress at which an unconfined cylindrical specimen of soil will fail in a simple compression test.

For soils, the untrained shear strength (S_u) is necessary for the determination of the bearing capacity of foundations, dams, etc. The untrained shear strength (S_u) of clays is commonly determined from an unconfined compression test. The untrained shear strength (S_u) of a cohesive soil is equal to one-half the unconfined compressive strength (q_u) when the soil is under the f = 0 condition (f = the angle of internal friction). The most critical condition for the soil usually occurs immediately after construction, which represents untrained conditions, when the untrained shear strength is basically equal to the cohesion(C). This is expressed as:

$S_u = C = \frac{qu}{2}$

Test Procedure for unconfined compression test

(a) Extrude the soil sample from Shelby tube sampler. Cut a soil specimen so that the ratio (L/d) is approximately between 2 and 2.5. Where L and d are the length and diameter of soil specimen, respectively.

(b) The exact diameters of the top of the specimen at three locations 120° apart is measured and then make the same measurements on the bottom of the specimen. Average the measurements and record the average as the diameter on the data sheet.

(c) The exact lengths of the specimen at three locations 120° apart is measured and then recorded the average measurements average length on the data sheet.

(d) Weigh the sample and record the mass on the data sheet. Calculate the deformation (DL) corresponding to 15% strain (e). Strain (e) = $\frac{\Delta L}{L_0}$
Where L_0 = Original specimen length

(e) Place the specimen in the compression device carefully and center it on the bottom plate. Adjust the device so that the upper plate just makes contact with the specimen and set the load and deformation dials to zero.

(f) Apply the load so that the device produces an axial strain at a rate of 0.5% to 2.0% per minute, and then record the load and deformation dial readings on the data sheet at every 20 to 50 divisions on deformation the dial.

(g) The load applying until (1) the load (load dial) decreases on the specimen significantly, (2) the load holds constant for at least four deformation dial readings, or (3) the deformation is significantly past the 15% strain that was determined in step 4.

(h) Draw a sketch to depict the sample failure. Remove the sample from the compression device and obtain a sample for water content determination.

2.4 Scanning Electron Microscopy (SEM)

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial vari-

-ations in these properties.

Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM (Figure.4) techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations (using EBSD). The design and function of the SEM is very similar to the EPMA and considerable overlap in capabilities exists between the two instruments.

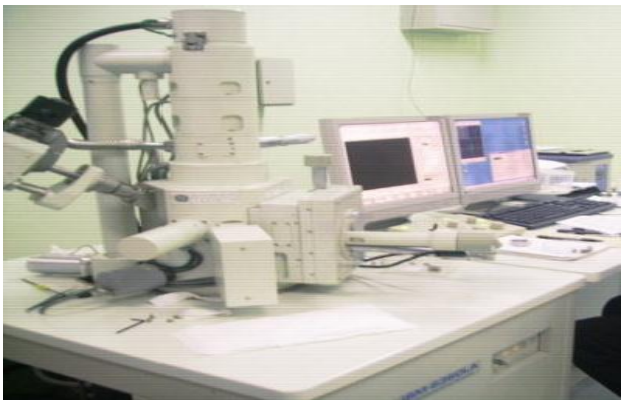

Figure 5: Photos of SEM & EDX

2.5 X- Ray diffraction test (XRD)

X-ray diffraction is a technique that provides detailed information about the atomic structure of crystalline substances. It is a powerful tool in the identification of minerals in rocks and soils. The bulk of the clay fraction of many soils is crystalline, but clay particles are too small for optical crystallographic methods to be applied. Therefore, XRD has long been a mainstay in the identification of clay-sized minerals in soils. However, its usefulness extends to coarser soil fractions as well. X-ray diffraction analysis can be conducted on single crystals or powders. This technique is applicable to soil mineralogy.

3. Results and Discussions

From Specific gravity test, Hydrometer test, unconfined compression test, Scanning and electron microscope (SEM), X-Ray Diffraction (XRD) tests the following results are noted:

3.1 Soil consistency

A system called Atterberg Limits is used to describe the Liquid Limits (LL), Plastics Limit (PL), and Shrinkage Limit (SL) of a soil. According to this system as water is added to a dry soil, the soil changes from solid to semi-solid to plastic to liquid. (Das, B.M., 2002).

The moisture content in the soil at the threshold between semi-solid and plastic is called the plastic limit. The moisture content in the soil at the threshold between plastic and liquid is called the liquid limit. Liquid limit is determined by forming a groove in a dish of soil and impacting the dish until the groove closes. The test is conducted as per the ASTM. D-4318. The plastic limit is determined by rolling a thread of soil on a glass plate until the 1/8-inch-diameter thread begins to crumble. A large liquid limit indicates high compressibility and high shrinks swell tendencies. Subtracting the plastic limit from the liquid limit yields the plasticity index. A large plasticity index indicates low shear strength. (Das, B.M., 2002).

Liquid limit (LL) - 35%
Plastic limit (PL) - 28%
Plasticity Index (PI) - 12%

3.2 Specific gravity test

Specific gravity of Nagavali River clay is determined by density bottle method. The specific gravity of the Bank-A, soil particles is found to be 2.65, and the specific gravity of the Bank-B, soil particles is found to be 2.72.

3.3 Hydrometer test

Table 3: Hydrometer Readings from Bank-A

Bank –A			
Sample No.	Clay %	Silt %	Sand %
1	17.49	57.3	25.3
2	20.8	48	31.2
3	25.6	46.4	28
4	32	46.4	24.8
5	33.5	41.7	24.8

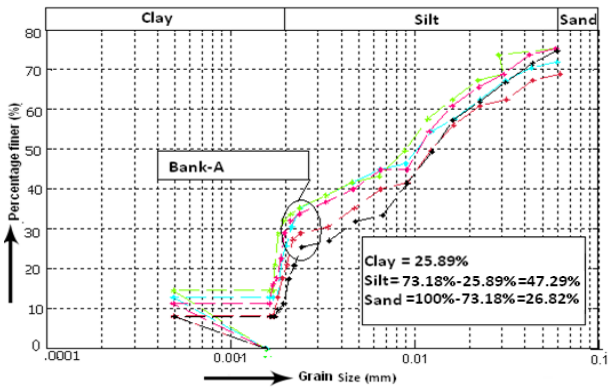

Figure 6: Grain size distribution curve from Bank-A (on

The size distribution of primary mineral particles, called soil texture, has a strong influence on the properties of a soil. Particles larger than 2 mm in diameter are considered inert. Little attention is paid to them unless

they are boulders that interfere with manipulation of the surface soil. Particles smaller than 2 mm in diameter are divided into three broad categories based on size. Particles of 2 to 0.05 mm diameter are called sand; those of 0.05 to 0.002 mm diameter are silt; and the <0.002 mm particles are clay.

The Grain size distribution curve (Figure-6) is drawn for 5 samples from Bank-A. These samples contain 26.86 % of sand, 47.29% of silt and 25.89% of clay.

Table 4: Hydrometer Readings from Bank-B

Bank –B			
Sample No.	Clay %	Silt %	Sand %
1	44.24	42.66	13.1
2	46.11	47.7	6.19
3	47.4	48.9	3.62
4	49	44.2	6.78
5	50	46.38	3.62

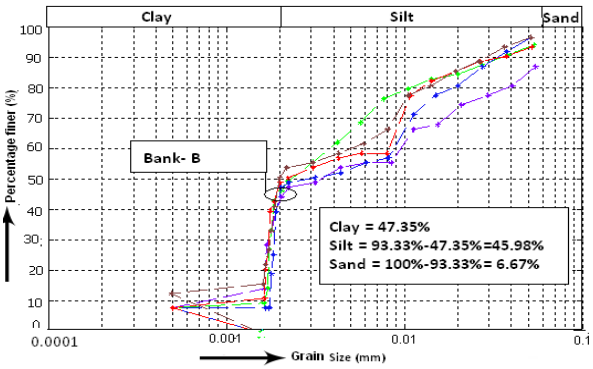

Figure 7: Grain size distribution curve from Bank-B (on semi log paper)

The Grain size distribution curve (Figure-7) is drawn for 5 samples from Bank-B. These samples contain 6.67 % of sand, 45.98% of silt and 47.85% of clay.

3.4 Unconfined compression test

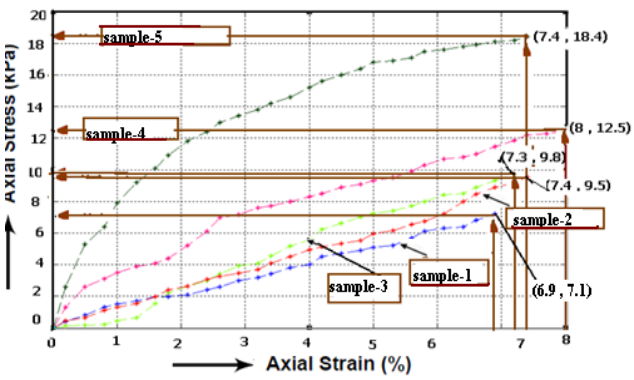

Figure 8: Stress- Strain curve from Bank-A

The Stress- Strain curve (Figure-8) is drawn for 5 samples from Bank-A. These samples contain unconfined compression strength is 13.75 KPS and the Cohesion (C) is 6.87 KPS**.**

Table 5: UCT Readings from Bank-A

Bank –A		
Sample No.	Unconfined compression strength-qu (KPS)	Cohesion-C (KPS)
1	7.1	3.55
2	9.5	4.75
3	9.8	4.9
4	12.5	6.25
5	18.4	9.2
Mean	11.46	5.73

Table 6: UCT Readings from Bank-B

Bank -B		
Sample No.	Unconfined compression strength-qu (KPS)	Cohesion-C (KPS)
1	7.4	3.7
2	8	4
3	14.2	7.1
4	15.79	7.89
5	18.4	9.2
Mean	12.75	7.4

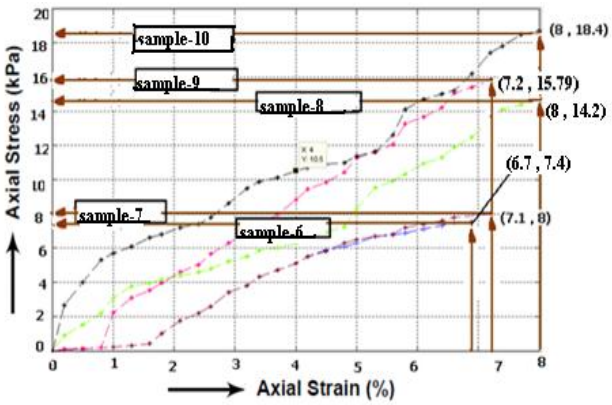

Figure 9: Stress- Strain curve from Bank-B

The Stress- Strain curve (Figure-9) is drawn for 5 samples from Bank-B. These samples contain unconfined compression strength is 15.30 KPS and the Cohesion (C) is 7.85 KPS**.**

3.5 Scanning Electron Microscopy (SEM) & X-Ray Diffraction (XRD) SEM and XRD for Bank-A

The results of chemical characteristics of soil type in this study using SEM and XRD photos (figure 10) indicate that this soil contains chemical elements:

Oxygen (O) = 23.21%, Silica (SiO_2) = 32.98%, Aluminum (Al) = 28.75%, Iron (Fe) = 14.63%, Titanium (Ti) = 1.70%, Calcium (K) = 0.73% and Carbon (C) = 7.10%.

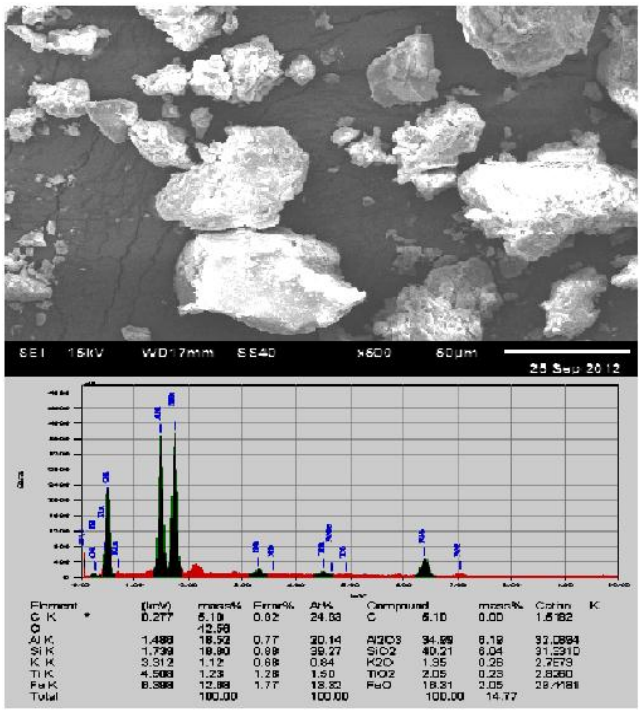

Figure 10: SEM and XRD Photos from Bank –A, Soil

SEM and XRD for Bank-B

The results of chemical characteristics of soil type in this study using SEM and XRD photos (figure 11) indicate that this soil contains chemical elements:

Oxygen (O) = 42.56%, Silica (SiO2) = 38.80%, Aluminum (Al) =32.52%, Iron (Fe) = 22.68%, Titanium (Ti) = 2.23%, Calcium (K) = 1.12% and Carbon (C) = 9.10%.

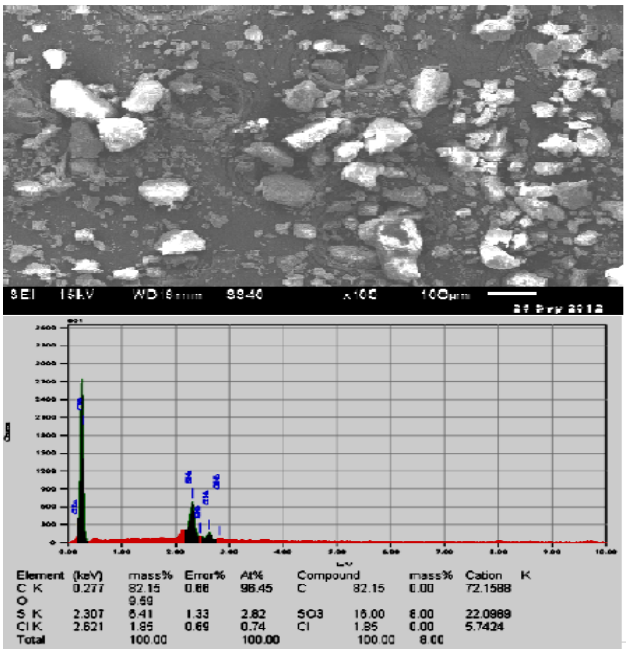

Figure 11: SEM and XRD Photos from Bank-B, Soil

4. Conclusions

In the present work the clay collected from both the banks of the river are reviewed. A laboratory study has been carried out to determine the engineering properties and characteristics of clay sample by different methods and the following conclusions are drawn.

(a) It is observed that the specific gravity of clay from Bank-B is more than that of Bank-A.
(b) The clay percentage varies from sample to sample and between the Banks.
(c) The strength of the clay increases as depth of sample goes on increasing and this is valid for both the banks.
(d) The chemical minerals of the clay from Bank-B is better than the Bank-A.
(e) The best results are obtained using Bank-B clays which exhibits more strength with more clay percentage.

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