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Instrumental Characterization of Montmorillonite Clay by FT-IR and XRD from J.K.U.A.T Farm, in the Republic of Kenya

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Abstract

Montmorillonite clay is a phyllosilicate mineral belonging to smectite clay group with a wide range of applications. This study demonstrates the Fourier Transform Infrared Spectroscopy (FT-IR) and X-ray Diffractometer (XRD) characterization of raw montmorillonite clay from J.K.U.A.T farm located in Juja, Kiambu county in the Republic of Kenya. The raw montmorillonite clays from different locations contained impurities and were subjected to mineral acids of different concentrations in order to enhance the analysis. The structural composition was carried out using FT-IR which revealed presence of various functional groups of Si-O, AL-OH and Si-O-AL as the major constituents. Mineralogical composition analysis was carried out using XRD and the patterns confirmed presence of aluminium oxide and silicon dioxide. Peaks of the resultant montmorillonite clay samples were compared with the published literature. This further confirmed the usefulness of spectroscopic technique in determination of crystalline nature of montmorillonite clay from J.K.U.A.T farm, Juja in the Republic of Kenya.

Keywords: Instrumental characterization; FT-IR, XRD.

1. Introduction

Montmorillonite clay is a type of clay mineral consisting of hydrous phyllosilicates with variable amounts of magnesium, iron, alkaline earth metals and alkaline metal cations. Raw formula of montmorillonite clay is $(Ca_{0.12}\ Na_{0.32}\ K_{0.05})\ [AL_{3.01}\ Fe(III)_{0.41}\ Mn_{0.001}\ Mg_{0.54}\ Ti_{0.02}][Si_{7.98}\ AL_{0.02}]O_{20}(OH)_4$ with colors varying from yellow green, yellow-white, gray and white due to presence of other trace elements (Navjeet *et al*., 2012). Montmorillonite clay has two silica-oxygen tetrahedral sheets sandwiching and aluminum or magnesium octahedral sheets. Magnesium or aluminum ions are octahedrally co-ordinated to six oxygen or hydroxyls. The replacement of silicon by aluminium in the tetrahedrallaye makes the layers negatively charged (Briendly and Brown, 1980). This results to cations like Na+, K+ and Ca2+ to get attracted to the layers surface to neutralize the negative charges. According to Sheng *et al*., (2001) montmorillonite clays are widely used due to its wide range of applications because of their high cation exchange, high surface area, swelling capacity and these results to strong adsorption/absorption capacities.

Fourier Transform Infrared Spectroscopy (FTIR) is an analytical tool which mainly compliments X-ray Diffraction (XRD) analytical technique in investigating clays and clay minerals (Adikary and Wanasinghe, 2012). This technique is very common, economical and the clay spectrums are obtained in a short period (Madejova, 2003). It gives information about the mineral structure, isomorphic substituent's nature and distinct molecular water from constitutional hydroxyls.

XRD technique o the other hand is used for mineral analysis (Ravisankar *et al*., 2010). Madejova (2003) stated that the XRD technique distinguishes the different types of clays deriving information about their structure composition. XRD analytical method is thus the best analytical method as it is accurate, inexpensive, reliable, rapid and non-destructive (Oumabady *et al*., 2014).

2. Material & Methods

2.1 Study Area

Clay samples were collected from four sites located in J.K.U.A.T farm. Exact locations of these sites were as follows;

Clay A – Farm area
Clay B- Piggery area
Clay C- Outside swimming pool area
Clay D- J.K.U.A.T main gate area

2.2 Sample Collection &Purification

Sample collection was done at the depth of 20cm from the surface. The collected samples were sieved through 2mm mesh size to remove coarse particles present I the clay samples. The samples were further mechanically milled to smaller clay particles. This was then divided into two parts where one part was further purified using mineral acids at different concentrations. About 100g of each clay samples was dissolved in 10% hydrogen peroxide and was allowed to stand for 3hrs. The clear suspension was decanted and 1L 0.5M NaOH added and was stirred moderately for 2hrs. The resulting solution was allowed to oven dry at 105°C for 24hrs.

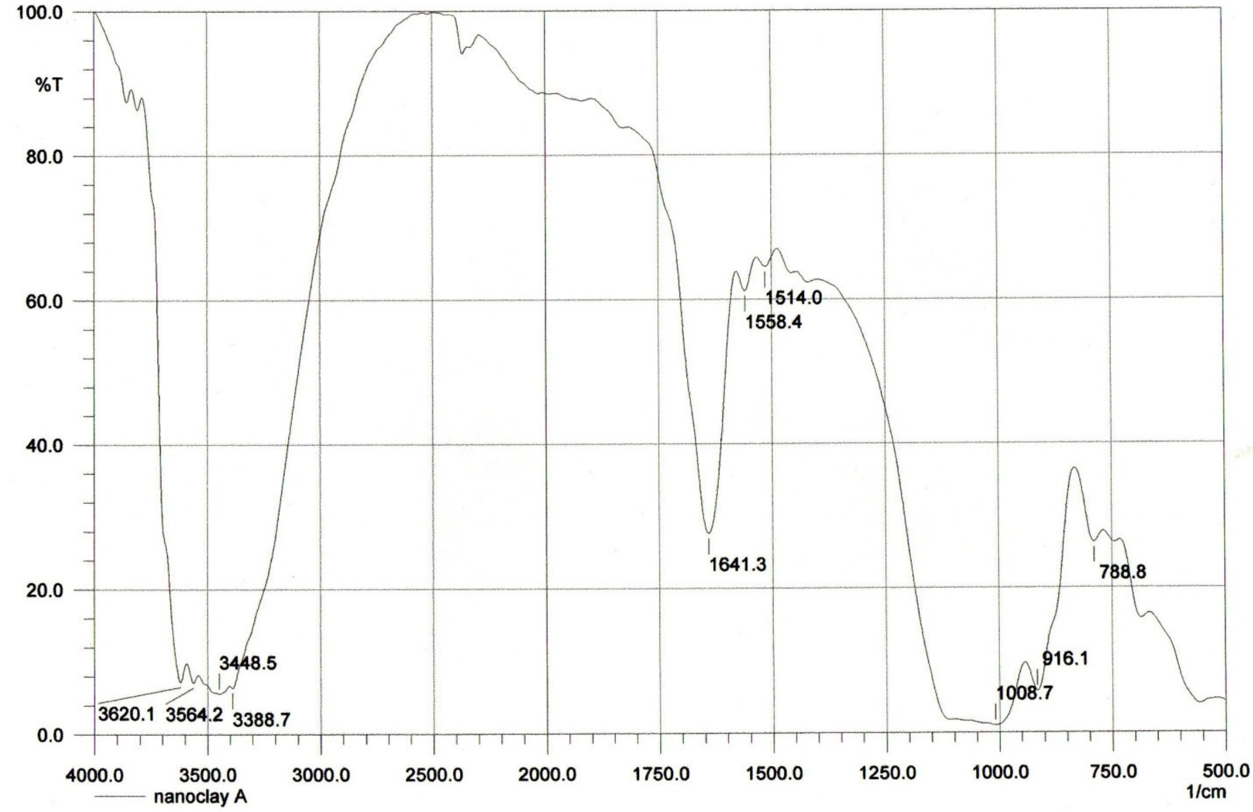
2.3 Isolation of Nano clays

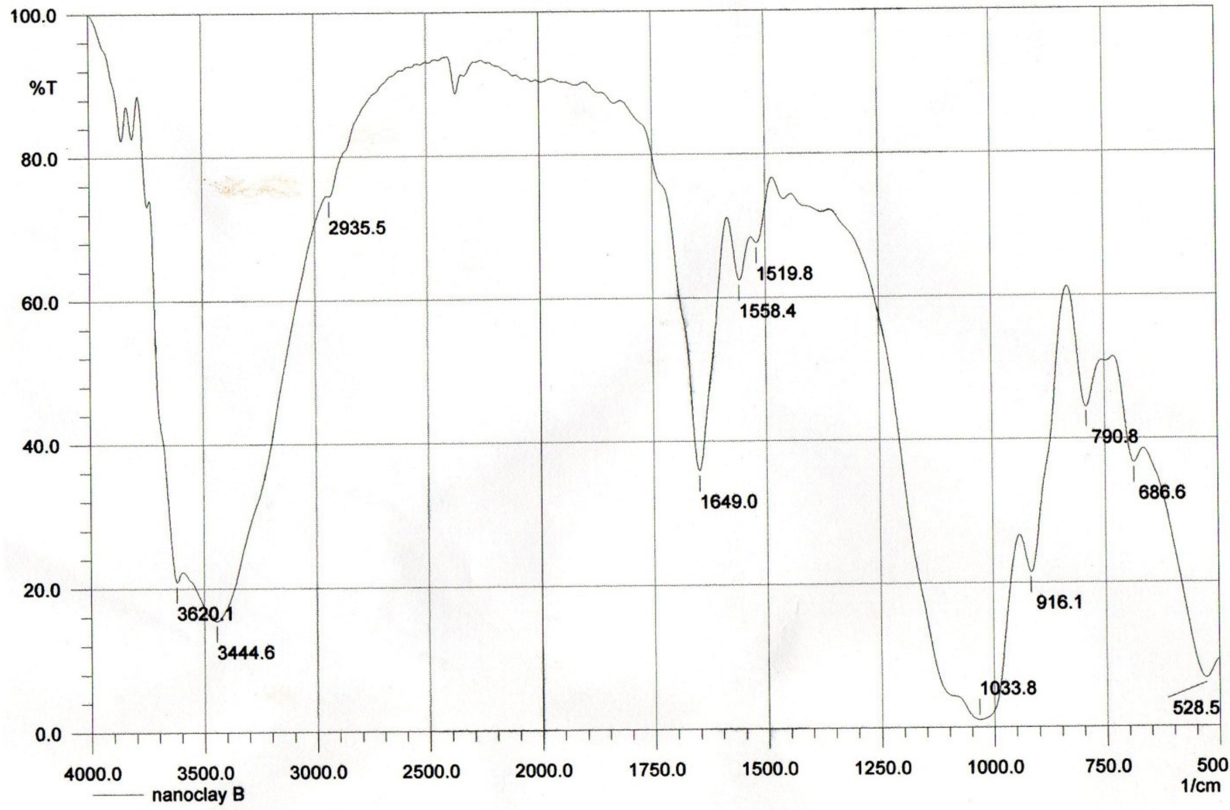
Sedimentation of clay samples followed which involved dissolving 100g of each clay sample in 1000ml distilled water and the solution stirred for 30 minutes. The solution was covered and allowed to stand for 12 hrs where the supernatant liquid was pipette out. The supernatant solution as later centrifuged at 6,000 rpm for 20 minutes and deposited clays obtained using sterilized spatula. The samples were then oven dried at 105oC for 2hrs and crushed into smaller particles using motor and pestle.

2.4 Mineralogical & Chemical Analysis

2.4.1 Characterization using F.T.I.R. Spectroscopy

All the nano sized samples were subjected to FT-IR for functional groups analysis. The spectrum results were similar in all the samples. Range of F.T.I.R was between 400-4000cm^{-1} and the sample was scanned 20 times. 0.01mg of air dried nano clay was mixed with 0.25mg dried potassium bromide (KBr). With gentle circular and back-and forth rubbing motion of the two windows, the mixtures were distributed between the plates. The mixture appeared slightly translucent, with no bubbles after preparation. The sandwiched plates were placed in the spectrometer and the spectrum was obtained.

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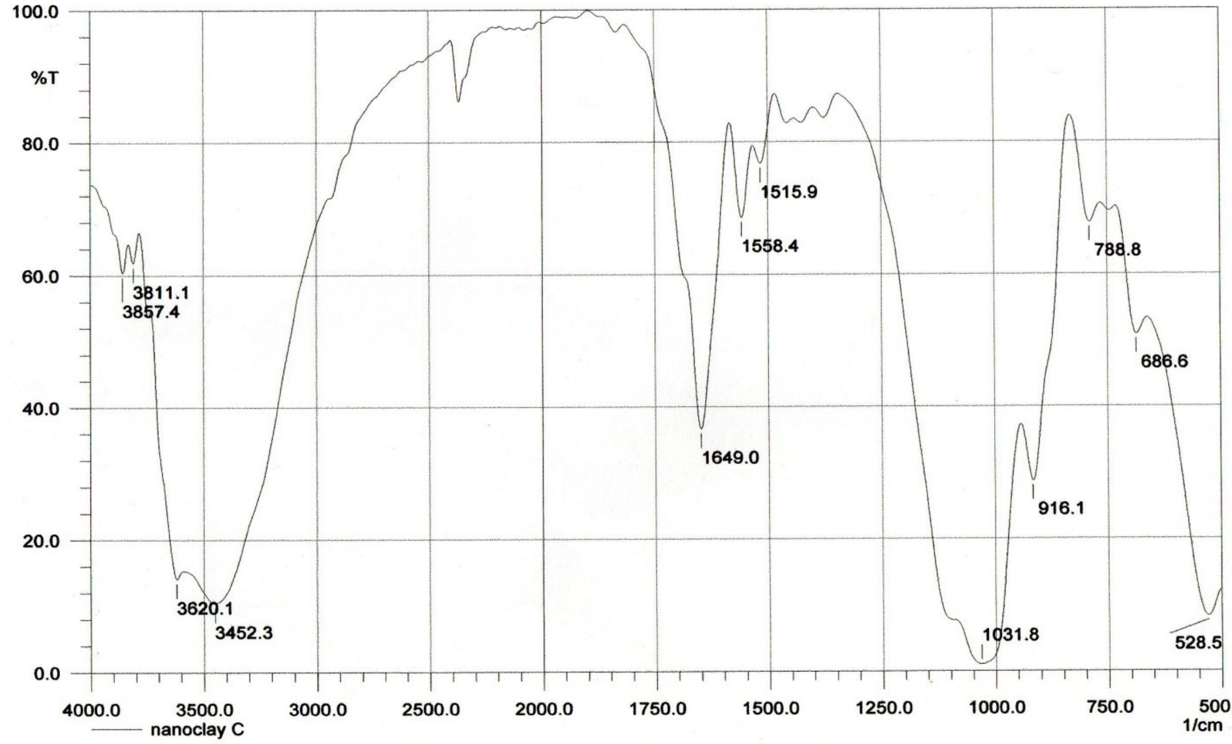
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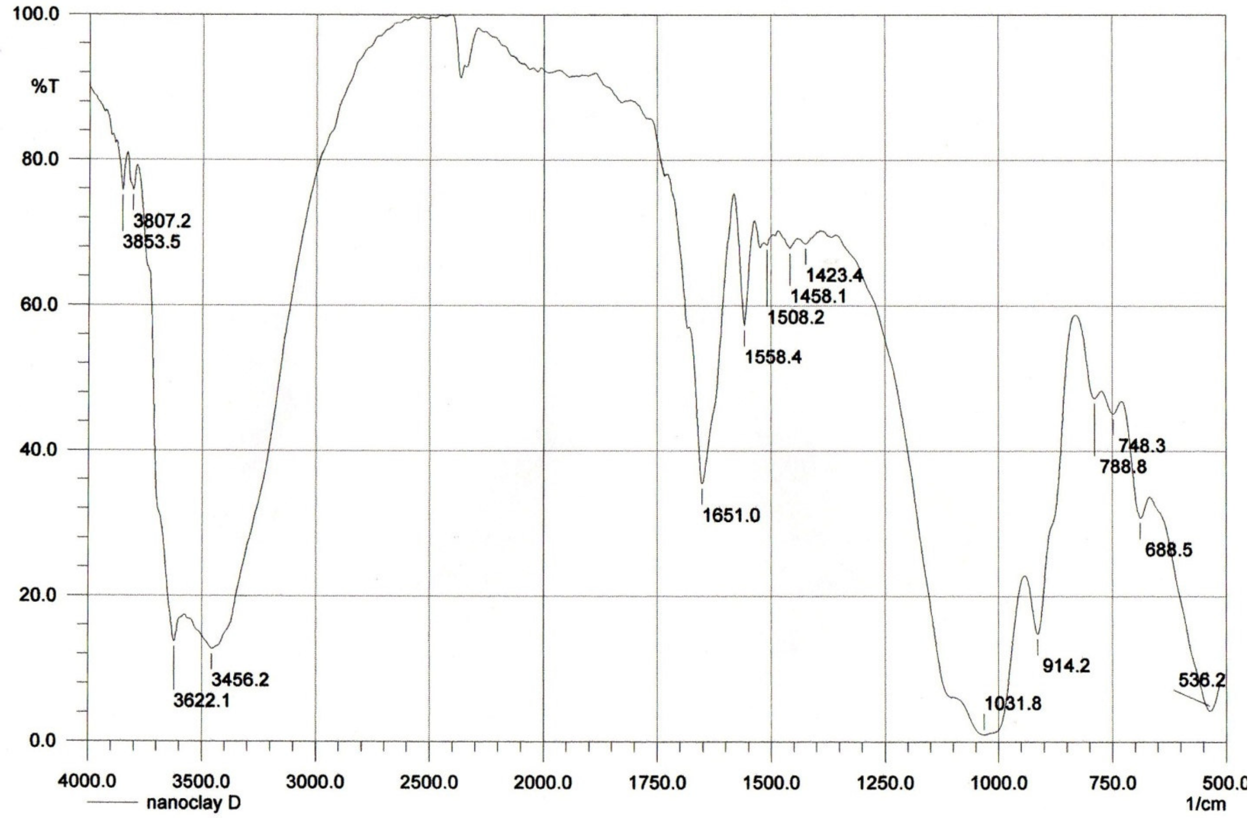
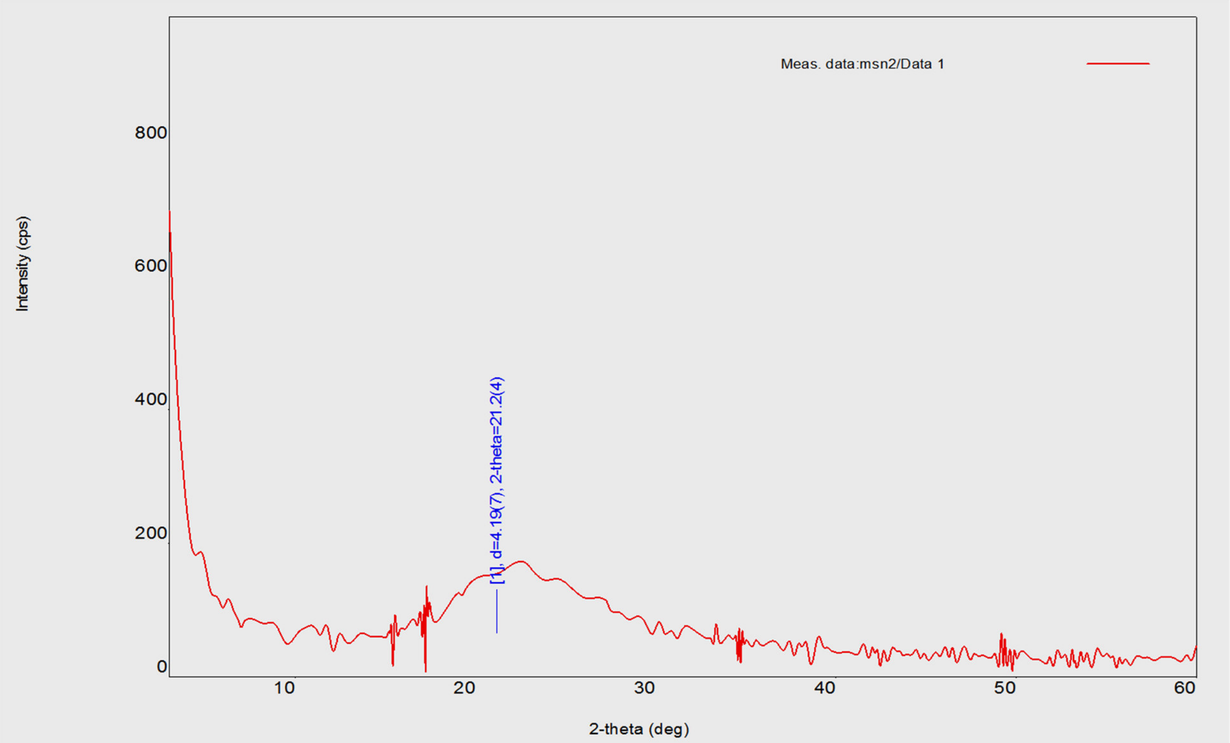


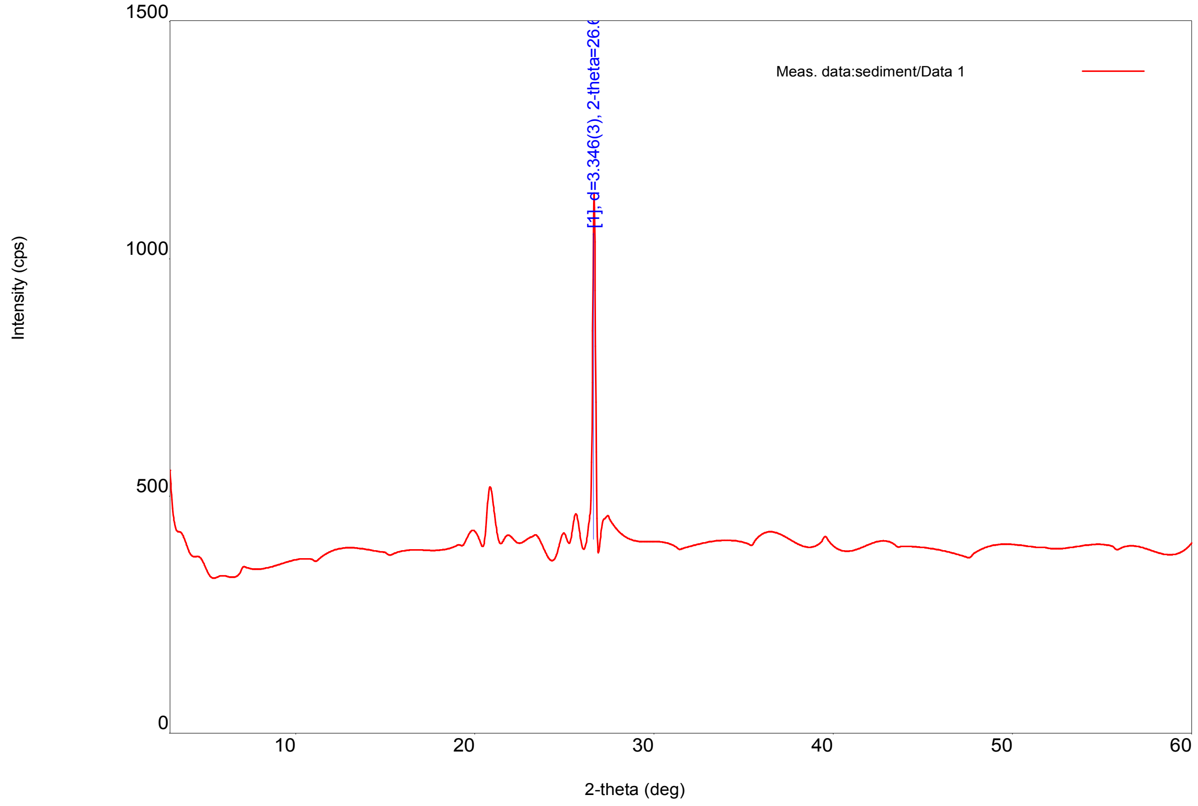
2.4.2 Characterization using XRD

The X-ray diffractometer measurements were done with Cu-Kα radiation of wavelength 1.541A^{O} at 40Kv and 30mA. The scan range was through 10^{o} through 100^{o} at a scanning rate of 1 degree per minute with a step size of 0.04^{o}. Diffraction peaks of the raw nano clay sample were compared with those of standard compounds.

Nano clay A XRD spectrum



Nano clay B XRD spectrum



3.0 Results & Discussions

3.1 Sample collection

The samples were collected from J.K.U.A.T farm as follows.

Sample	Sample collection area	Color
A	Farm area	Light brown
B	Piggery area	Dark brown
C	Outside swimming pool area	Dark grey
D	J.K.U.A.T Main gate area	Light grey

3.2 Sample preparation

3.2.1 Dry purification method

All the samples contained stones, pebbles and leaves and were thus allowed to pass through 2mm mesh size. After drying and milling, the samples were again allowed to pass through 420µm mesh size for further purification and the yield was as follows;

Sample	Yield
A	70%
B	79%
C	71%
D	69%

3.2.2 Wet purification method

On application of wet clay purification method there was high rate of effervescence, emission of heat and gasses and this method proofed to be more effective in the removal of inorganic matter. After wet purification and oven drying, the yield increase in grams due to increased absorption of the water into the clay gallery spaces and consequently increased interlayer space. Sample C and D proofed to have higher gallery space for water absorption as both had the highest and increased by 141.02gms and 138.03gms respectively.

Sample	Sample before purification	Sample after purification & drying
A	100gms	169.98gms
B	100gms	176.76gms
C	100gms	241.02gms
D	100gms	238.03gms

3.3 Isolation of nano sized samples

After sedimentation and centrifugation processes, nano sized (< 2µm sample size) was obtained. Nano clay yield obtained from 2.5 liters of the supernatant liquid was small as observed in the result.

Sample	Yield
A	1.76%
B	1.40%
C	1.88%
D	1.92%

3.4 Characterization using F.T.I.R Spectroscopy

Wave numbers (cm^{-1})						
Absorption band position	Theoritical Montmorillnonite	Nano clay A	Nano clay B	Nano clay C	Nano clay D	Assignments
1	3630	3620.1	3620.1	3620.1	3622.1	AL/Mg--O-H stretching (Inter-octahedral)
2	3427	3448.5	3444.6	3452.3	3456.2	H-O-H stretching of structural hydroxyl groups & water
3	1636	1641.3	1649.0	1649.0	1651.0	OH deformation of water
4	_	1558.4	1558.4	1558.4	1558.4	C-N stretching
5	_	1514.0	1519.8	1515.9	1508.2	Aromatic nitrate
6	1065					Si-O stretching of quartz in plane vibration
7	1049	1008.7	1033.8	1031.8	1031.8	Si-O stretching
8	918	916.1	916.1	916.1	914.2	AL-AL-OH bending/ deformation
9	_	788.8	790.8	788.8	788.8	Si-O quartz
10	695	_	686.6	686.6	688.5	ALALOH bending
11	524	_	_	528.5		AL-O-Si deformation

The observed bands in the range of 4000-500cm^{-1} were assigned to various constituents. In this FTIR study of the soil, the Si-O and AL-OH main functional groups were observed in the 1000cm^{-1} and 500cm^{-1} range. Intensive peaks at 528.5cm^{-1} was due to the bending vibrations of AL-O-Si bond (Zuzana *et al.*, 2007). Bands at 914.2cm^{-1} and 916.1cm^{-1} corresponded to the AL-OH bending vibrations while doublet at 780-798cm^{-1} was due to Si-O-Si inter tetrahedral bridging bonds in SiO_2 and OH deformation bond of gibbsite (Bhaskar & Gopalakrishnarao, 2010). Si-O stretching vibrations were observed at 788.8cm^{-1}, 748.3cm^{-1}, 790.8cm^{-1}, 688.5cm^{-1} and 686.6cm^{-1} showing presence of quartz (Sarala and Kavitha, 2014).

A strong band at 3622.1cm^{-1} and 3620.1cm^{-1} showed presence of hydroxyl linkage while broad band at 3456.2cm^{-1}, 3444.6cm^{-1}, 3448.5cm^{-1} and also bands at 1651.0cm^{-1}, 1649.0cm^{-1} and 1641.3cm^{-1} in the clay spectrum indicated possibility of water of hydration or H-O-H bending of water in the adsorbent (Preeti and Singh, 2007). The structural –OH groups occurring in the clay mineral contributes to the shoulders and broadness of the –OH band. However, the chemical composition of this clay mineral is clearly indicated by the position of the maximum of the band (Patel *et al.*, 2006).

The results of FTIR thus were very helpful in identification of various forms of minerals present in clay. The bands at 3622.1cm^{-1} and 1651.0cm^{-1} of clay showed the hydrous nature of this clay material.

According to Adikary and Wanasinge 2012, the important bands that are needed for identification of montmorillonite clay are at absorption bands at position 1, 2, 7, 8 and 11.

3.5 Characterization using XRD

The XRD results analysis of the four samples were done and the values compared with the published literature. The identification of the clay minerals was accomplished by careful consideration of peak position and intensities. The XRD results showed presence of impurities like quartz and kaolinite. Presence of kaolinite was indicated by d-spacing peaks of 100% intensities at 26.28, 26.62, 26.63 and 35.062 in samples B, C and D which proved to be dominant. Also traces of kaolinite were indicated by d-spacing peak at 20.04 in sample C. The presence of quartz was further proofed as earlier confirmed by the absorption characteristic bands obtained in F.T.I.R Spectroscopy which gave the characteristic band at 778.8cm^{-1} in sample A and B and also characteristic absorption band at 686.6cm^{-1} in samples B and C and 688.5cm^{-1} in samples D.

4.0 Conclusion

The four samples of montmorillonite clay were collected from different parts of J.K.U.A.T farm, Juja in Republic of Kenya and after thorough examination using F.T.I.R Spectroscopy and XRD characterization techniques confirmed the properties of montmorillonite clay. F.T.I.R Spectroscopy confirmed presence of montmorillonite clay in the position of absorption bands as compared with the published literature. However, XRD analysis indicated traces of quarts and kaolinite impurities.

5.0 Acknowledgements

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