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Experimental Studies on Surface Morphology of Nano clay and Mg-Al Layered Double Hydroxide Using Atomic Force Microscopy and EDAX Analysis

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Abstract: Nano-sized particulate materials are finding increase in applications for Engineering and Medical Sciences. In this paper study the two containing Nano- powders, i.e., Nanoclay (Cloisite 30B) and Layered Double Hydro-oxide (LDH) composed of Aluminium-Magnesium was considered for determining their morphological behaviour. Atomic Force Microscopy (AFM) was used to find the roughness of the Nano-particulates. EDAX was used to ascertain the actual composition of the selected Nano-powders. The findings from AFM test were indicated in the result and discussion. It was found that the Nano clay had smooth surface for the same particle size than the LDH. Hence nanoclay can be opted for applications such as powder coatings over metal surface for flame retardant properties.
Keywords : Nano clay (Closite 30B), LDH (Mg-Al), EDAX., AFM (Atomic Force Microscope).

1. Introduction

The group of LDH family was first discovered in 1842 in Sweden. Layered double hydroxides (LDHs), also known as anionic clays, are a family of layered materials which have attracted much attention in recent years. LDH materials are usually classified under nanofillers because of its smaller sized particulates. They are used to remove bacteria and viruses in water. The general formula for the LDH materials is [MII1-x MIIIx (OH)2]x+ (Am-)x/m] •nH2O [1]. LDH are used as the heat stabilizers as well as acid scavengers in most of the halogenated polymers [2]. Due to presence of hydrogen bond, LDH have stable structure. LDH can be prepared by four methods which are co-precipitation, ion exchange, calcination rehydration and hydro thermal methods [3]. In this study the LDH is observed using AFM technique. This technique is a powerful tool in studying the properties at nano level structure. When LDH powder is added to very minute quantity over the surface of metals, it increases the thermal stability of the powder, which enhances its fire retardant property [2]. nanoclay is one of the inexpensive natural mineral used for the preparation of nanocomposite and other applications such as Carbon Fibre Reinforced Plastic (CFRP) preparation because of its good thermal resistivity [4].

1.1. Atomic Force Microscopy

Atomic force microscopy is a technique employing a high resolution Scanning Probe Microscopy (SPM) with resolution demonstrated in order of fractions of nanometers which is 1000 times better than optical diffraction limit. AFM is also called Scanning Force Microscopy (SFM). The use of AFM for layered materials provides a high resolution morphological observation [5]. It is also used to measure the roughness of the sample surface with detail and find its mechanical properties and surface topography features. AFM employs a cantilever beam with sharp tipped probe to scan over the surface of the sample. Contact between probe tip and the sample causes deflections which are detected by a laser beam. A position sensitive photo diode (PSPD) is used to note the deflection [6].

The operation modes of AFM are generally classified into image formation. The image formation is a plotting method as a color mapping. Color mapping is introduced as a function of the Origin represented by the symbol R. This is achieved by changing the X-Y position of the tip by scanning and a constant [7]. This constant is the ratio of a measured variable to the intensity of control signal to each X-Y coordinate. In this respect, the color mapping is a method which corresponds to a measured variable to each coordinate and shows it [8]. It means the image which especially expresses the intensity of a value as a hue. Usually, the relationship between the intensity of a value and a hue is shown as a color scale in the color mapping. The other group includes various aspects such as force spectroscopy and potential mapping [9].

1.2. EDAX Analysis

EDAX analysis is the analysis by which the composition of the powders can be identified and it gives the results in the table form EDAX or EDS is an x-ray spectroscopic method for determining elemental compositions. It can be used with/during imaging in SEM, TEM etc. When done in an SEM instrument, signal can be acquired from a spot, an area, a line profile or a 2D map. It is energy dispersive X-ray system by which the energy absorption sites are found in the specimen [10]. It is an analytical technique by which the elemental analysis and chemical analysis can be done. The results are plotted in the graph [11]. The FESEM (Field Emission Scanning electron Microscope) machine is shown in Fig. 1. The graph has peaks because the electrons in the l-shell return to the k-shell orbit when the excitation caused by the X-Ray is off [12].

Fig 1. Field Emission Scanning Microscope

2. Experimental Procedure

2.1. Pelletization

Pelletization is a process of compacting the materials in powder form into a definite shape as the equipment shown in Fig. 2. The pellets can be used to test metal specimen instead of powder. In this process the

powders are compacted and molded into a shape by the means of mechanical dies and force to create components such as balls used for ball milling [13].

Fig. 2. Pelletizer machine.

2.2. AFM Observation

The material which was pelletized was measured using Atomic Force Microscopy. It was found that the maximum size of the pellets were in the range of 10nm to 20nm.

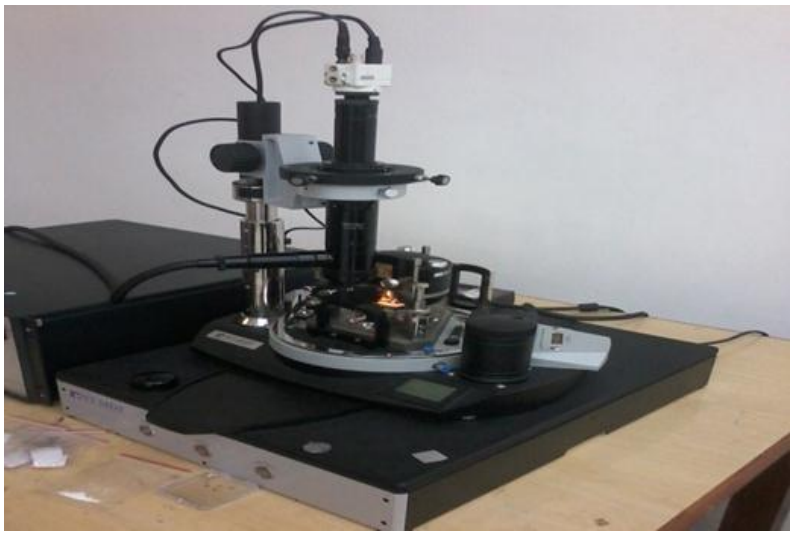

Fig 3 Atomic force Microscope

3. Results and Discussion

3.1. AFM Analysis of LDH

The AFM topographic images of the layered double hydroxide are represented in Fig. 4a, 4b, at 10 µm magnification and the corresponding roughness parameters, Ra, derived from the AFM scans are shown in Table 1.

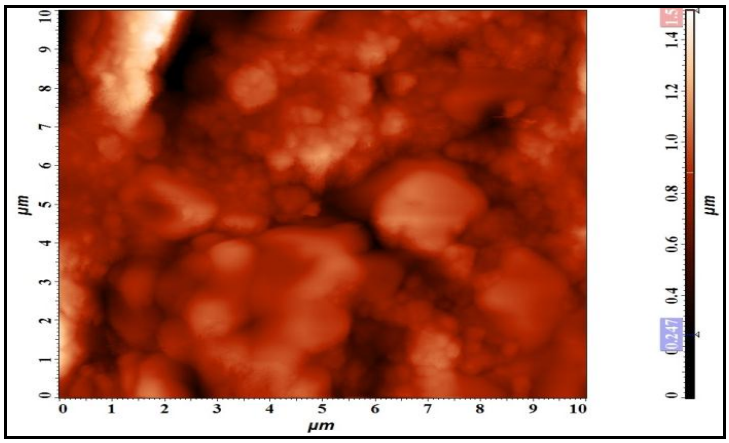

Fig 4a. AFM image of LDH sample at 10µm

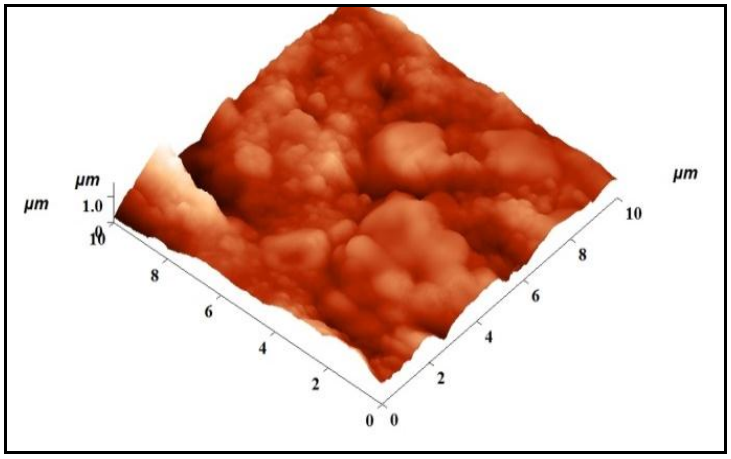

Fig 4b. 3D Image of LDH in AFM in 10µm

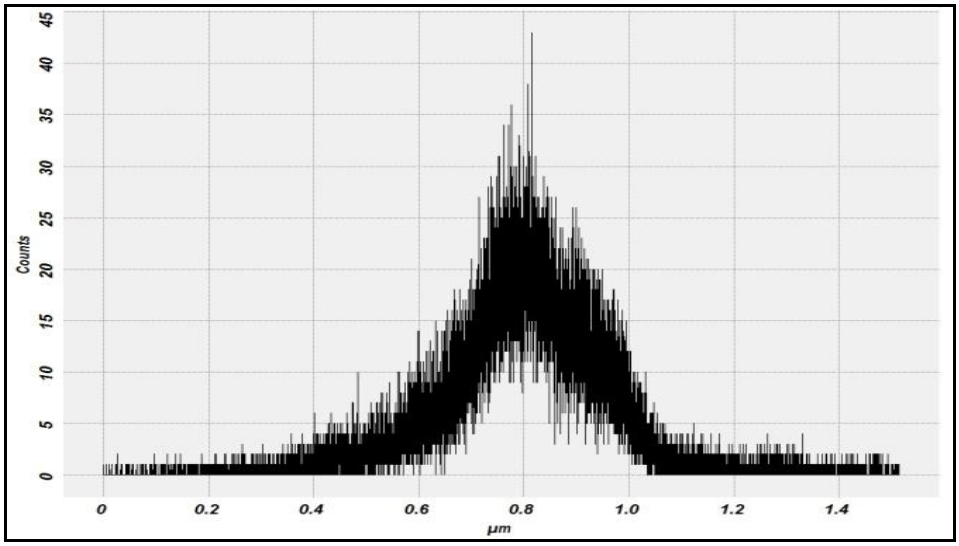

Fig 5. Histogram analysis of LDH at 10µm.

Fig. 5. shows histogram in which the Y-axis has roughness measurement in counts and X-axis has size of the particulates in µm. The highest peak in the graph is 43 counts for a particulate size of 0.82 µm.

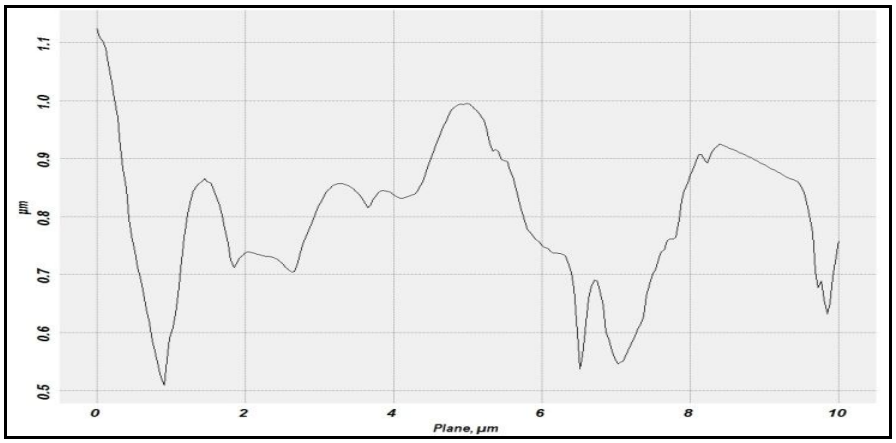

Fig 6. Line profile of LDH at 10µm.

The line profile of LDH has shown in the Fig. 6 and it is seen that at 1µm the roughness is so high and the values drops and due to roughness the line values varies.

Table 1 Roughness analysis of Layered double Hydroxide

S.No	Details	Parameters
1	Number of sampling	65536 no
2	Max	1513.84 nm
3	Min	0 nm
4	Peak-to-peak, Sy	1513.84 nm
5	Ten point height, Sz	761.419 nm
6	Average	800.254 nm
7	Average Roughness, Sa	117.855 nm
8	Root Mean Square, Sq	160.574 nm
9	Second moment	666191
10	Surface skewness, Ssk	-0.0916793
11	Coefficient of kurtosis, Ska	2.21324
12	Entropy	12.516
13	Redundance	-0.18489

3.2. EDAX Analysis of LDH

The SEM image of LDH powders are shown in Fig 7. The EDAX of LDH and its corresponding composition is shown in Fig 8 and Table 2 respectively. It can be found that coarse grains were present at the surface of the specimen. The EDAX analysis revealed the composition of the specimen.

Table 2 shows the EDAX Results for LDH

Element	Wt%	At%
CK	7.25	10.28
NK	6.87	8.36
OK	56.36	60.01
NaK	22.52	16.68
MgK	3.55	2.49
AlK	3.46	2.18
Total	100	100

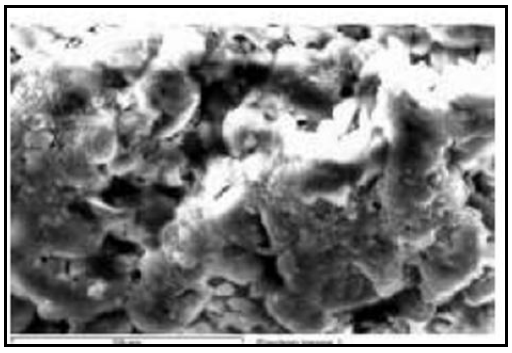

Fig 7. SEM image of Mg-Al LDH

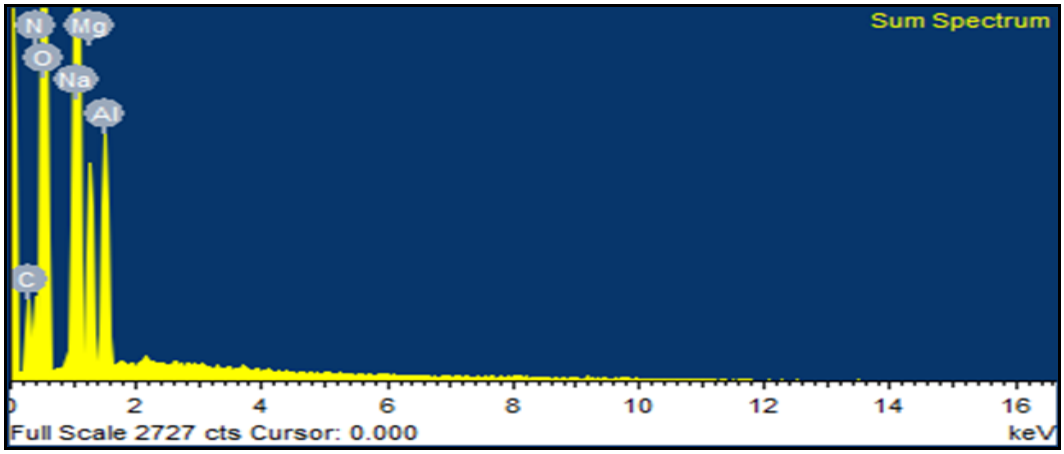

Fig 8. EDAX graph for LDH

3.3. AFM Analysis of NC

The AFM topographic images of the nanoclay are represented in Fig. 9a, 9b, and the corresponding roughness parameters, Ra, derived from the AFM scans are shown in Table 3.

Table 3 Roughness analysis of Nanoclay

S.No	Details	Parameters
1	Number of sampling	65536 no
2	Max	556.136 nm
3	Min	0 nm
4	Peak-to-peak, Sy	556.136 nm
5	Ten point height, Sz	286.122 nm
6	Average	307.039 nm
7	Average Roughness, Sa	56.5725 nm
8	Root Mean Square, Sq	73.606 nm
9	Second moment	99690.6
10	Surface skewness, Ssk	0.0395785
11	Coefficient of kurtosis, Ska	0.752605
12	Entropy	11.54
13	Redundance	-0.265808

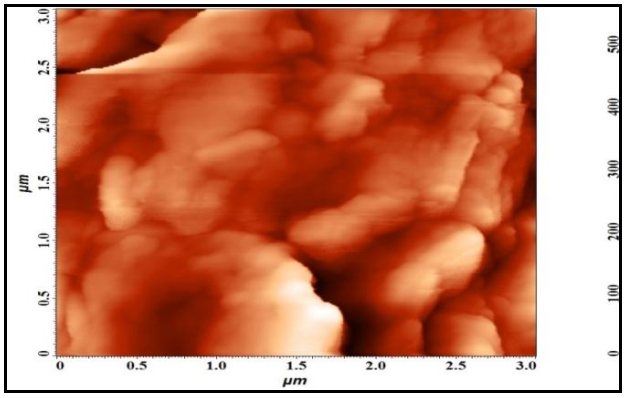

Fig. 9a. AFM image of Nanoclay at 3µm.

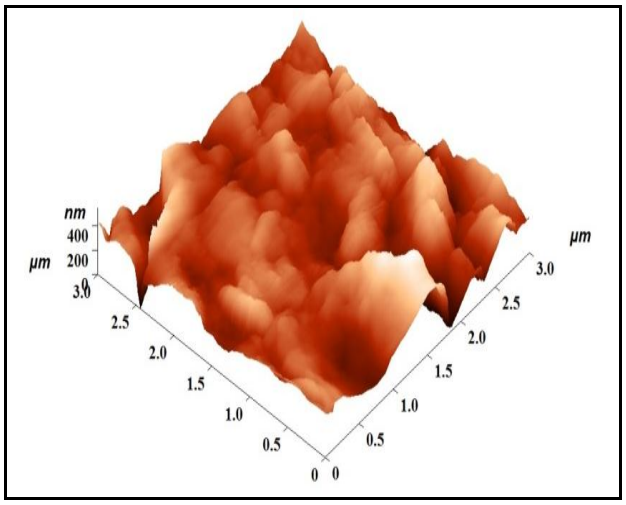

Fig. 9b. 3D image of Nanoclay in AFM in 3µm.

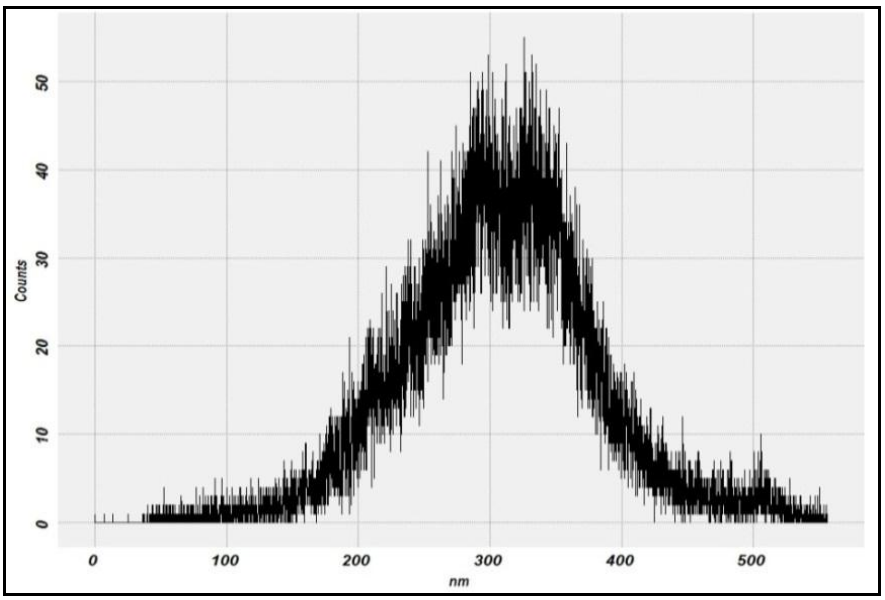

Fig 10. Histogram analysis of Nanoclay at 3µm.

Fig 10 shows the histogram graph of NC and the peak has a region in which the highest value is about 57 counts for the particulate size of 340 nm. Another peak closer to the highest peak was noted at 54 counts for the particulate size of 300 nm.

Fig 11 gives the line profile of NC and the graph varies from 330 and falls at the end and the highest peak occur twice and the peak value is above 400. The line profile reveals that the maximum roughness of the powder was 405 at 205 nm.

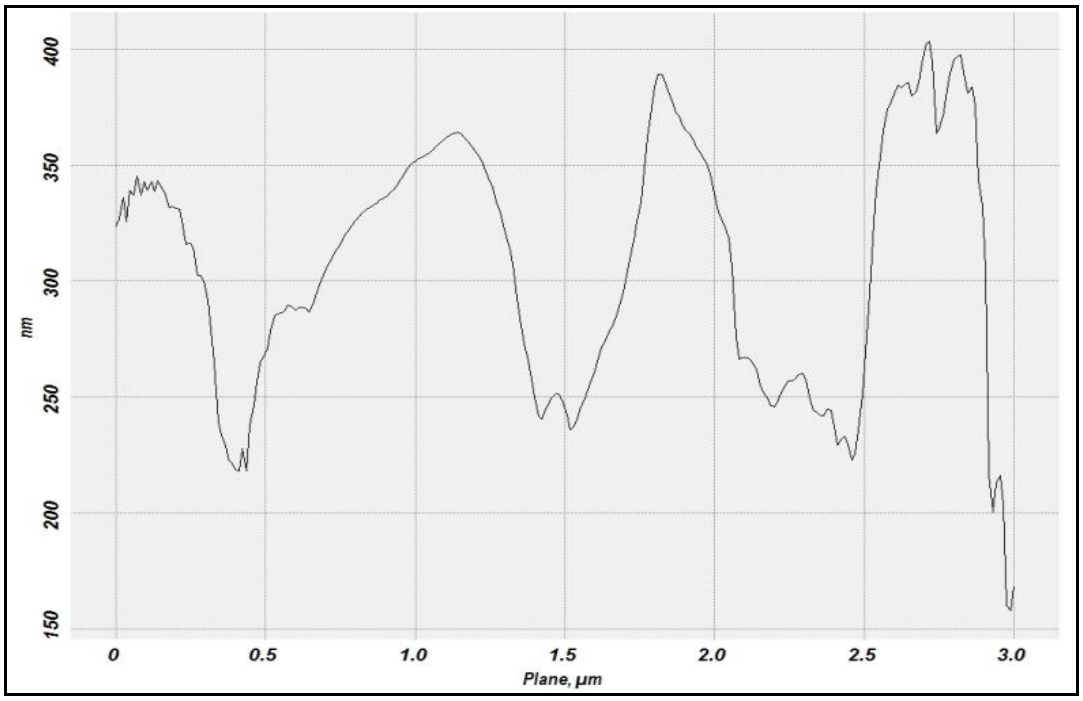

Fig 11. Line profile of Nanoclay at 3µm.

3.4. EDAX Analysis of Nanoclay

The EDAX of NC and its corresponding composition is shown in Fig 12 and Table 4 respectively. The EDAX analysis revealed the composition of the specimen.

Table 4. EDAX Results for Nanoclay

Element	Wt%	At%
CK	26.09	38.42
OK	32.86	36.32
MgK	1.01	0.74
AlK	10.45	6.85
SiK	26.55	16.72
FeK	3.04	0.96
Matrix	Correction	ZAF

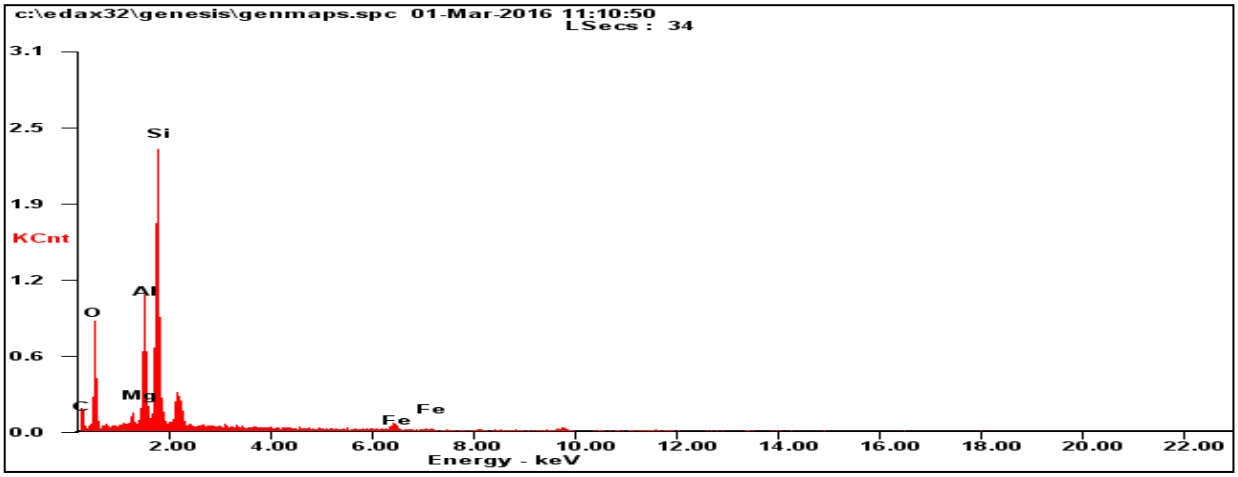

Fig 12. EDAX graph for NC

4. Conclusion

From this paper we can clearly understand the following

- AFM of LDH and Nano clay showed that the size of LDH was larger than Nano clay.
- The LDH powders were coarser in comparison with Nano clay.
- The histogram revealed that LDH roughness of powders Spiked at a single point at 0.82 µm particle size.
- However for Nano clay the histogram revealed that the coarseness of particles ranged over a few sizes of particles.
- The line diagram of LDH and Nano particulates revealed that Nano clay was comparatively fine sized and hence can be adopted as fire resistance coating for metal surfaces which needs good surface finish.

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