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# An experiment about Morphological Structure of Mg-Al Layered Double Hydroxide Using Field Emission Scanning Electron Microscopy with EDAX Analysis

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**Abstract:** In this article we are going to see about morphological structure and elements of Mg-Al Layered Double Hydroxides (LDHs). LDHs are nothing but nanofillers. It is better than conventional hydroxides, especially known for its flammability, thermal and good mechanical properties. Mg-Al LDHs powder was undergone field emission scanning electron microscopy (FESEM) with EDAX analysis and the obtained results show us the clear idea about morphological structure and elements of Mg-Al LDHs powder respectively.
**Keywords:** Magnesium-Aluminium (Mg-Al) LDHs, FESEM, EDAX.

## 1. Introduction

In 1842 hydrotalcite, a mineral of LDH family was discovered in Sweden. In recent days LDHs are used in many fields they have been studied in many aspects like structure, synthesis to know about their use in catalyst, ion-exchange, Adsorption, pharmaceutics and other fields. The composition of layered double hydroxide is $[M^{II}_{1-x}M'^{III}_{x}(OH)2](A^{n-})x_{/n}\cdot mH2O$ (m = 0·33–0·50). LDHs structure is same as brucite (Mg(OH)2) a type of octahedral layers. It can be reinforced with cation like (e.g. Mg, Al, Cr, Mn, Fe, Co) [1-2].

LDH have stable structure due to presence of hydrogen bond in it. There are four methods to prepare LDH they are co-precipitation, ion-exchange, calcination-rehydration and hydrothermal methods [3]. LDHs powder is observed under a fine technique called Field Emission Scanning Microscope (FESEM) to understand about their morphological structure with EDAX. The reason why we are going for FESEM is because it is more accurate than SEM.

### 1.1. Field Emission Scanning Electron Microscope (FESEM)

In early days Hitachi developed electron microscopes which can produce magnified image with beam of electrons. After a long time electron microscope was produced in the year of 1942 [4]. Ultrahigh resolution imaging FESEM was product developed by physicist Albert Crewe with support of Hitachi in the year of 1972.

Scanning electron microscope is used to inspect the surface of solid to know about its morphological character. Field Emission Scanning Electron Microscope (FESEM) is well known method for structural analysis and it is better than conventional method of analysis of optical microscope [5]. Here, the emitted electrons are used to analyze the surface and the best known source of electron is field emission source and it was developed in the year of 1930's [6]. In FESEM a wire of tungsten with sharp point is the Field Emission Gun (FEG) and the electrons produced by FEG is focused onto the sample by three types of lens, they are condenser lens, objective lens, electro-magnetic lens. Here the test is carried out in the FESEM Zeiss supra 55vp [7].

## 2. Experimental Procedure

### 2.1. FESEM Observation

Emitted electron from field emission gun is allowed to focus on the solid surface to create the surface image. FESEM should be carried out in vacuum chamber for that pre-vacuum pump and turbo vacuum pump are employed. Vacuuming the chamber may take an hour. After when the chamber gets evacuated N2 gas is allow to flow into the closed chamber. Following that, the emitted electron is focused on sample as the result electrons are scattered when it hits the surface of the sample. The scattered electrons are detected by x-ray detector and that emitted electrons helps in forming image in the monitor.

The morphological structure of sample was examined under FESEM with an accelerating voltage of 10 to 20 KV [7]. Observation can be done up to 8m under different magnification to find its exact morphological structure. Here the sample is observed at 5 k x, 10 k x, 25 k x, 50 k x and 100 k x. When we go for higher magnification we will come to know more about the surface structure. The 100 k x magnification clearly showed us the surface of Mg-Al LDH was in polygonal shape.

**Fig.1. FESEM Machine ( Zeiss supra 55vp)**

## 3. Result and Discussion

### 3.1. FESEM Analysis

FESEM Analysis is conducted to find the morphological structure of the sample, here the sample Mg-Al LDH is tested for morphological structure and it is found that it is in polygonal shape especially it is hexagonal in shape at the magnification of 100.00 k x. The polygonal shape is shown in the Fig.6.

In Fig.2. Clearly show the penetration of electron from its source at a velocity of EHT = 5.0 KV with respect to that magnification index is set up to 5 x and viewed in order to obtain the former result. Here the first three tests are conducted with constant velocity and varying magnification.

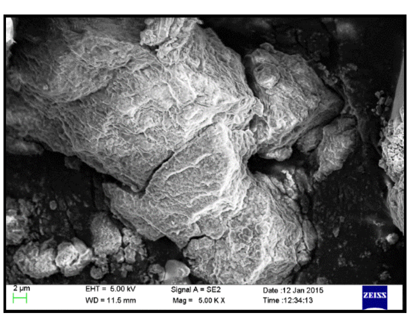

**Fig.2. Shows FESEM image of sample at 5.00 k x**

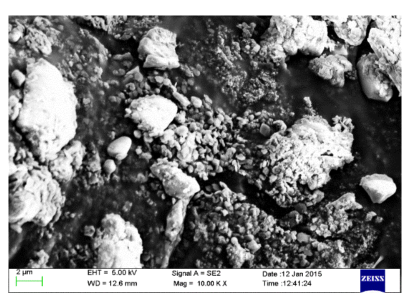

**Fig.3. Shows FESEM image of sample at 10.00 k x**

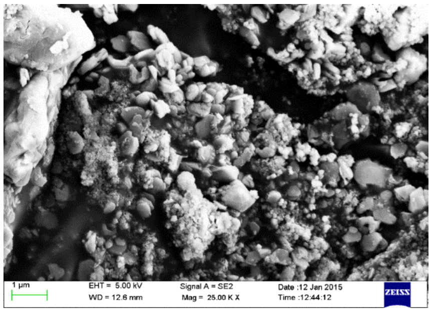

**Fig.4. Shows FESEM image of sample at 25.00 k x**

Similarly, further test is carried out by having different values of velocity and magnification. As shown in Fig.5 & 6, the velocity remains constant and magnification changed like 50.00 k x and 100.00 k x. The result of changing magnification is shown below.

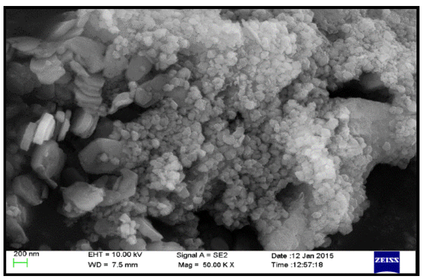

**Fig.5. FESEM image at magnification of 50.00 k x**

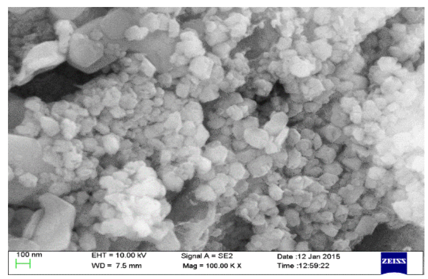

**Fig.6. Shows FESEM image of sample at 100.00 k x**

**3.2. EDAX Analysis**

Energy Dispersive X-Ray Analysis (EDX), is also known as EDAX or EDS, is a technique of using X-ray to find the elements present in sample. This technique is both qualitative and quantitative. The composition of sample is shown in Fig.8. Each peak shows as the presence of different elements in Fig. 7(a). In this test sample is not undergoes any physical or chemical changes. This analysis needs almost no sample preparation.

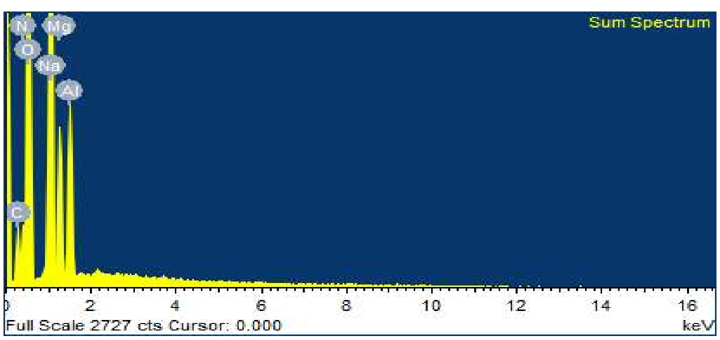


**Fig.7.a. EDAX image represents presence of different elements**

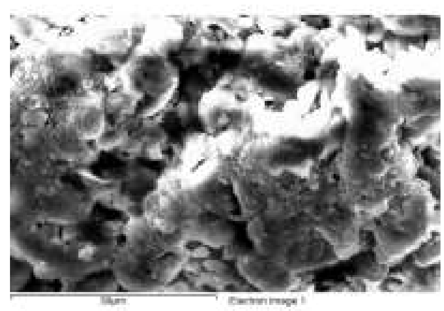

**Fig.7.b. EDAX image of morphological structure of Mg-Al LDH**

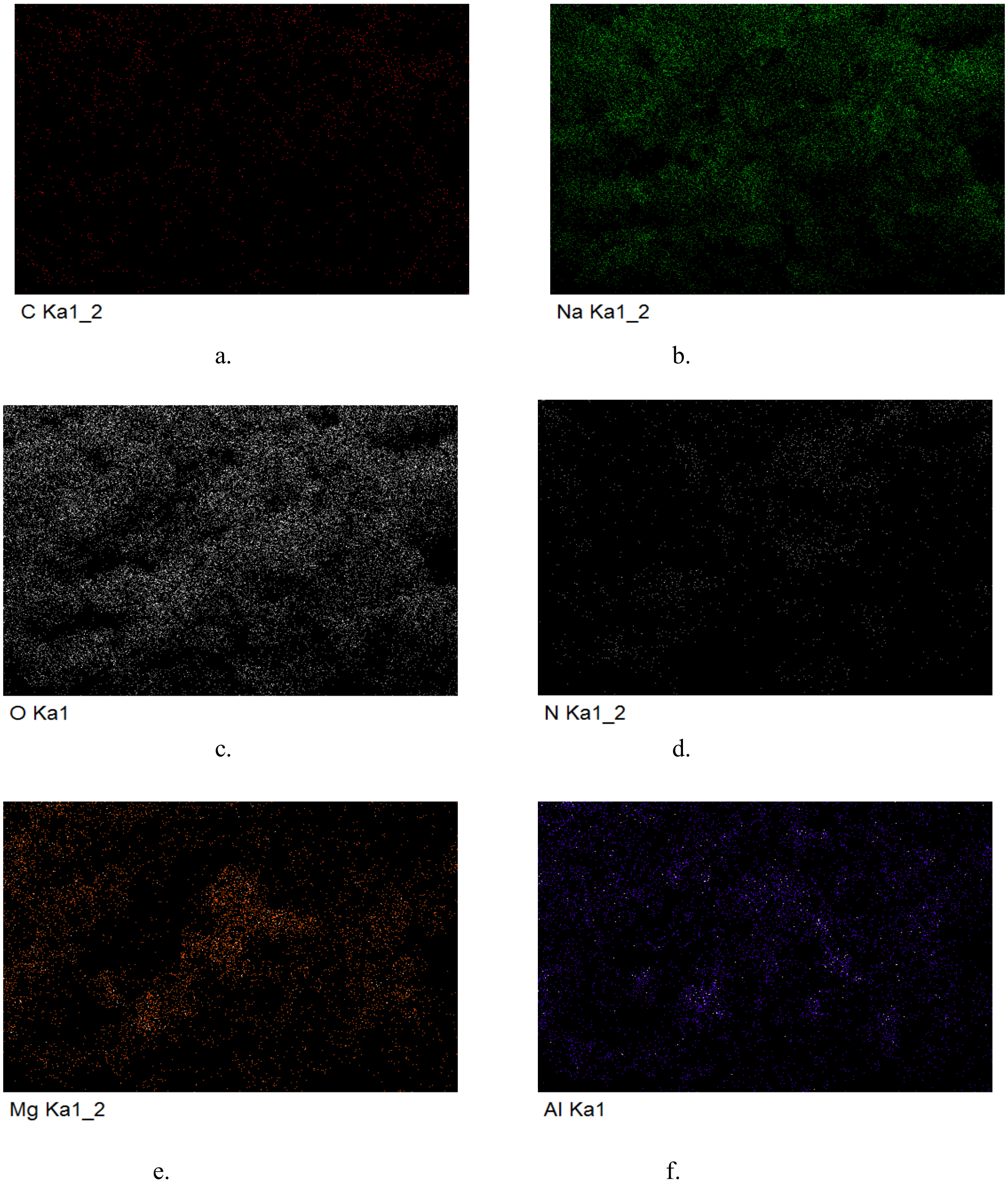


**Fig.8. EDAX image which shows the elements of sample**

## 4. Result and Discussion

LDH sample of EDAX was analyzed and the results shows us the composition and amount of the element in sample. Test is taken at least for 5 iteration. Here the weight % and atomic % of Mg is 3.55 and 2.49 respectively, in case of Al it is 3.46 and 2.18 respectively.

**Table 1 Standards**

| | |
|---|---|
| C | CaCO3 |
| N | Not defined |
| O | SiO2 |
| Na | Albite |
| Mg | MgO |
| Al | Al2O3 |

The EDAX analysis result was shown below; table 2 shows us the clear information about composition and amount of presence of the elements. The value for each element with respect to weight % and atomic % and the result is tabulated as below.

**Table 2 Amount of Mg, Al and other elements.**

| Element | Weight % | Atomic % |
|---|---|---|
| C K | 7.25 | 10.28 |
| N K | 6.87 | 8.36 |
| O K | 56.36 | 60.01 |
| Na K | 22.52 | 16.68 |
| Mg K | 3.55 | 2.49 |
| Al K | 3.46 | 2.18 |
| Totals | 100.00 | |

## 5. Conclusion

From this paper we can clearly conclude with the sum of results which was obtained by performing two unique test over Mg-Al LDH as

- Performing FESEM test to the given Mg-Al LDH sample with constant and varying velocity at corresponding magnification factor provides different morphological structures as per analysis for the test specimen.
- Performing EDAX analysis on same sample for up to 5 iterations and respective table was plotted for weight % and atomic %, which provides clear information over the composition and amount of elements present in sample.

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