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Characterization of pillared clay by SEM-EDX

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***Abstract*—A Algerian bentonite is successfully pillared with solutions of Al, Fe and Al-Fe polyhydroxocations or polymeric under moderate conditions of synthesis in order to obtain insertion at 800°C. The characterization by SEM EDX confirms that the preparation procedure of pillaring method is efficient for obtaining pillar at high temperature.**

Keywords— Bentonite; polyhydroxocations, Fe, Al; pillared clay; SEM-EDX

I. INTRODUCTION

Clays are the most important of our industrial minerals, millions of tons are utilized annually in a large variety of applications. Some of the most important include ceramics, paper, paint, plastics, drilling fluids, chemical carriers, liquid barriers, adsorbent, decolorization, and catalysis [1, 2, 3] Pillared clays (PILCs) constitute one of the most widely studied series among the microporous materials. They are prepared by exchanging the charge-compensating cations between the clay layers with larger inorganic hydroxyl cations, which are polymeric or oligomeric hydroxyl metal cations formed by hydrolysis of metal salts. Upon calcinations, dehydration and dehydroxylation occurred from these metal hydroxyl cations and the structural OH groups, thus formed stable metal oxide pillars (e.g. Al_2O_3, Fe_2O_3 etc.) to keep the clay layers apart and create interlayer and interpillar spacing's of molecular dimension [4, 5]. Generally, the catalytic activity of pillared clays is studied after the calcination at 500°C, of these solids [6, 7, 8]. In the present work we report the preparation and characterisation by SEM-EDX of Al, Fe and Fe/Al-PILC calcined at 800°C.

II. EXPERIMENTAL

A. Preparation of Purified Bentonite

The clay used in this work is a natural bentonite from Hammam Boughrara (Maghnia West of Algeria), used in purified form. The sodium montmorillonite are obtained by two steps: sedimentation and the chemical treatment as described by [7, 8]. Carbonates are removed by sodium acetate/acetic acid buffer, iron oxides by dithionite/sodium citrate, and organic materials by hydrogen peroxide (30 v%). To ensure complete transformation into the sodium from, all samples are washed several times 1M NaCl, Finally, the all samples are centrifuged and washed with distilled and deionised water until free from chloride ion as tested by AgNO3 solution and calcined at 100°C. The exchange capacity or CEC of Na-montmorillonite is 0,91 meq /g (by methylene bleu exchange) and has a BET surface area of 100,2 m^2/g.

B. Pillaring methods

Pillaring solutions containing either Al or Fe polymeric species are prepared following an established procedure [7] by slowly adding a 0,207M and 0.1M solution of sodium hydroxide to a 0,207M and 0,1M solution of $FeCl_3$, $AlCl_3$ respectively to obtain the molar ratio of OH^- /M^{+3} = 2,5 even for the combination of Fe and Al (metallic ratio= 0,5). The aluminum pillaring solution, the iron pillaring solution and the Fe/Al solution are aged for one week at room temperature. After the ageing process the sample is added directly into the pillaring solution (with ratio mass M^{3+} /clay = 6,25%) under vigorous stirring and allowed to age overnight. The products were then collected for centrifugation and washed with deionised water until free of chloride (as judged by the silver nitrate test) then calcined for 2 h at 800 °C.

III. CHARACTERIZATION METHOD

Scanning electron microscopy (SEM)

The morphology, and Chemical composition of the samples are investigated using JEOL5510 Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy (EDX), operating at acceleration voltages = 20 kV. These methods are used in phase analysis (SEM) and determining major element composition of PILC bentonite. The samples are prepared in the form of pastilles; the microstructural imaging, particle size and elemental composition analysis at different spots (selected grains) are made using SEM and INCO software linked to the SEM machine.

IV. RESULTATS AND DISCUSSION

- To further understand the morphology of pillared clay by insertion of pillaring solutions process and visualize the surface. The samples used are characterized by electron microscopy in conjunction with EDX. Fig. 1 shows the SEM images of the samples in the range 100 μm revealing a smoothed coated porous and rough surface. The three samples are appeared to have an abundance of crystals on the external surface of the supports (Fig. 1) as well as inside the pores. The introduction of Fe and Al cations or mixed Fe/Al into clay layers results in the appearance of the crystals are circle in the Fig. 1. As expected for amorphous materials, they are irregular in shape. EDX (Table. 1, 2, 3) elemental analysis over a number of selected crystals showed that they are composed of Si, Al, O, Ca, Mg and Fe the biggest

constituent composition it is oxygen average of 56% by weight. Si is a component of the clays, which is supposed to stay in the clay, the EDX (Fig. 2, 3, 4) analysis of the three samples exhibited a nearly uniform distribution of iron throughout the external surface area. This indicates that the preparation procedure of pillaring method is efficient for obtaining pillar.

V. CONCLUSION

The method (SEM EDX) is very useful in the investigations of pillared clays. The EDX result shows the insertion clearly the oligomeric hydroxyl metal cations (Al, Fe /Fe/Al) in the clay. This indicates that the preparation procedure of pillaring method is efficient for obtaining pillar interlayer at 800°C.

A. Figures and Tables

TABLE I. ELEMENT COMPOSER OF BENTONITE-AL CALCINED AT 800 °C BY EDX

Element	Bentonite-Al	
	Weight%	Atomic %
OK	55.32	71.46
NaK	1.15	1.03
MgK	2.10	1.78
AlK	5.51	4.22
SiK	23.94	17.61
KK	1.03	0.54
CaK	0.12	0.06
FeK	1.35	0.50
CuK	4.88	1.59
BrL	4.61	1.19
Tolals	100	

TABLE II. ELEMENT COMPOSER OF BENTONITE-FE CALCINED AT 800 °C BY EDX

Element	Bentonite-Fe	
	Weight%	Atomic%
OK	55.19	69.57
NaK	0.72	0.63
MgK	2.10	2.33
AlK	9.04	6.75
SiK	25.59	18.38
KK	0.37	0.19
FeK	5.09	1.84
BrL	1.18	0.30
Tolals	100	

TABLE III. ELEMENT COMPOSER OF BENTONITE-AL/FE CALCINED AT 800 °C BY EDX

Element	Bentonite-Al/Fe	
	Weight%	Atomic %
OK	57.98	70.73
NaK	3.16	2.68
MgK	2.47	1.98
AlK	9.69	7.01
SiK	23.70	16.47
KK	0.75	0.37
CaK	0.00	0.00
FeK	2.13	0.75
PtM	0.12	0.01
Tolals	100	

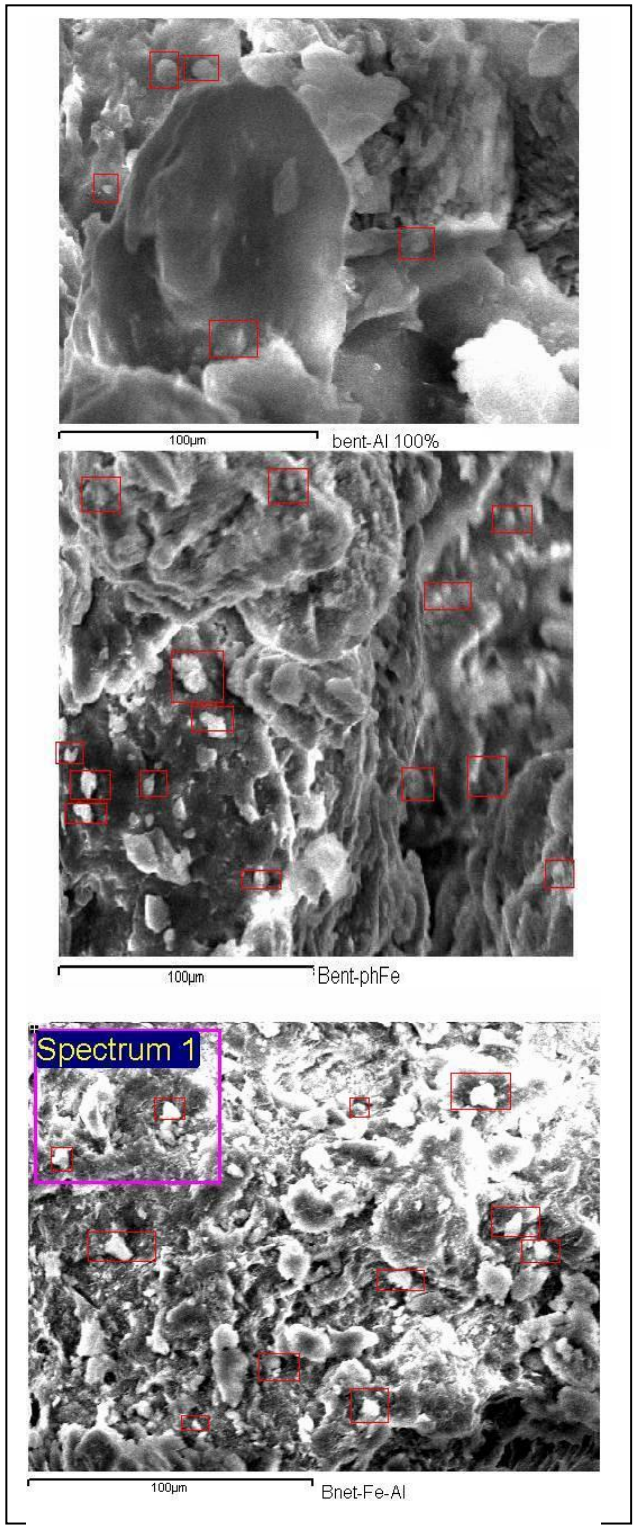

Fig. 1. *SEM of bentonie modified by Al, Fe and mixed Al/Fe calcined at 800°C, with magnification of × 300.*

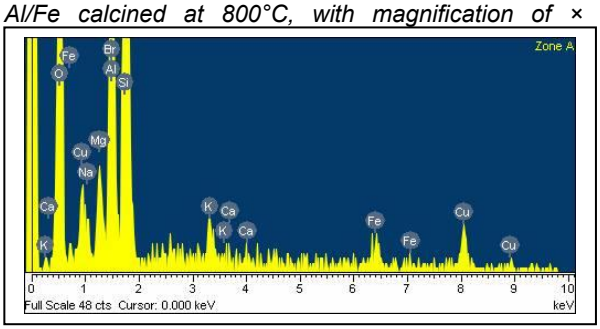

Fig. 2. EDX elemental analyses of bentonite-Al in Fig 1.

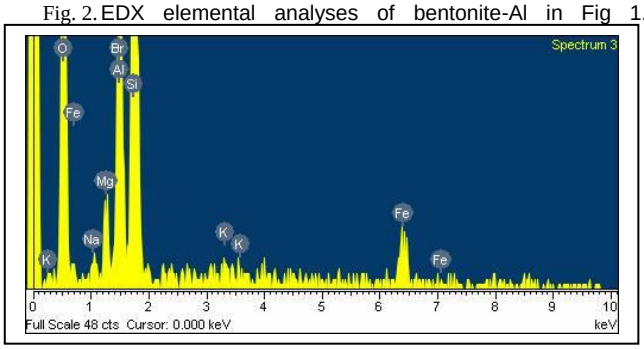

Fig. 3. EDX elemental analyses of bentonite-Fe in Fig 1.

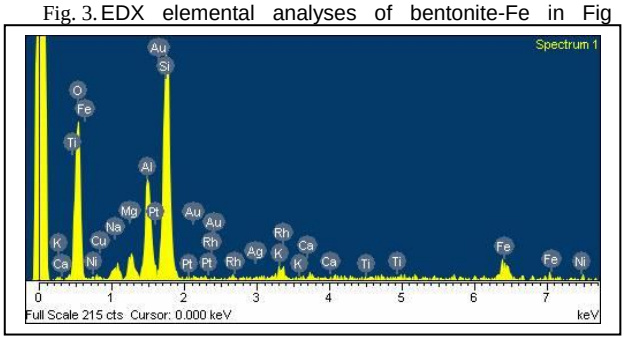

Fig. 4. EDX elemental analyses of bentonite-Al/Fe in Fig 1

REFERENCES

[1] H. H. Murray, “Applied clay mineralogy today and tomorrow”, Clay. Mineral. Vol34, pp. 39-49, Octobre 1999.

[2] W. A. Carvalho, “Cyclohexane oxidation in gas phase using iron and chromium pillared clays as catalysis”, Eclética Quimica, Vol 27, pp. 353-365.

[3] M. Naswir, S. Arita, Marsi and Salni, “Characterization of bentonite by XDR and SEM-EDS and use to increase pH and color removal, Fe and organic substance in peat water”, Jour. Chem. Engr. Technol. Vol4, pp, 313-317, October 2013.

[4] J.T. Kloprogge, “Synthesis of smectites and porous pillared clay catalysts” a review, J. Pourous Mater. Vol5, pp, 5-41, 1998.

[5] N, D, Hutson, M. J. Hoekstra, R. T. Yang, « Control of micro-porosity of Al_2O_3-pillared clays : effect of pH, calcinationt temperature and clay cation exchange capacity” Microporous Mesoporous Mater. Vol28, pp, 447-459, 1999.

[6] M. Akçay, “The surface acidity and characterization of Fe-montmorillonite probed by in situ FT-IR spectroscopy of adsorbed pyridine” App. Cata. A. General Vol294, pp, 156-160, July 2005.

[7] Z. Meçabih , J. Rose, D. Borschneck , “Urban wastewater treatment by adsorption of organic matters on modified bentonite by (iron-aluminum)”. J. Enscapulation Adsor. Sci. Vol4 pp,71-79, September 2014.

[8] Z E, Bojemuelle, A. Nennemann, G. Lagaly, “Enhanced pesticide adsorption by thermally modified bentonites”, Appl. Clay Scien. Vol5, pp, 277-204, May 2001.