Effects of Acid Treatment on the SEM-EDX Characteristics of Kaolin Clay

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Abstract. Raw kaolin was refluxed by sulphuric acid in variable concentrations of 2 M, 4 M, 6 M and 8 M. The morphology and elemental compositions of the acid-leached kaolin were analyse by Field Scanning Electron Microscopy (FESEM) and Energy Dispersive X-ray analysis (EDX) respectively. The disintegration and leaching of Al^{3+} ions of the clay are determined by FESEM studies. The acid treatment increases the silicon content and decreases aluminium content as revealed by EDX analysis. The leaching of Al^{3+} ions increases with gradual increase in concentration of the acid. Therefore, kaolin reflux with acid at lower strength (2 M and 4 M) are more dispersed and more industrially useful than that which is treated at higher acid strength.

Keywords: kaolin; morphology; acid treatment; FESEM, EDX.

INTRODUCTION

Kaolin is an inorganic aluminosilicate clay mineral with natural flaky shape [1].The general chemical formula is articulated as $Al_2Si_2O_5(OH)_4$. The basal (d_{001}) spacing of kaolin is (7 ˚A). Kaolin has a crystal structure composed 1:1 type of a two-layer structure established by an angle sharing [SiO_4] tetrahedral layer and an edge sharing [AlO_6] octahedral layer [2, 3]. The water molecule separates each of these two layers by a monolayer. Kaolin has a versatile range of applications in industrial sectors; predominantly as paper filler, in formulation of medicine, coating pigment, cosmetics, main component of ceramics, extender in water based paints and ink, as a filler for rubber and other polymeric materials [4].

Report from Department of Mineral and Geoscience, Malaysia; proclaimed that substantial reserves of kaolin are discovered throughout Malaysia. Additionally, reports by Joint Meteorological Group (JMG), mentioned that the country possess about 112 million tonnes of kaolin reserves found in the states of Perak, Johore, Kelantan, Selangor, Pahang and Sarawak. Seventeen other active kaolin mines operating during the year mostly located in the Bidor/Tapah district in Perak. Production of kaolin in 2015 was 261,574 tonnes value at RM 24.85 million. For the purpose of the means of obtaining hopeful and attractive venture processes; kaolin can be used as additives in manufacture of polymer nanocomposites [5].

The surface properties of clay determines their industrial application [6]. Numerous techniques have been proposed to enhance the surface properties of clay materials, comprising intercalation [7], chemical activation [8] mechanochemical activation [9, 10] and thermo-chemical treatment [11]. Equally as the chemical activation technique has been established, acid treatment is an effective method to develop the functioning of clay minerals [3]. Normally, acid treatment causes delamination/disaggregation of clay particles, removal of mineral impurities, and disintegration of the external layers. When these happens, clay minerals structure become damaged, initiated an increase in surface area, BET surface area, surface activity and pore volume [6, 12].

Several research works on the acid treatment of kaolin has been carried out. Studies by [13] use meta-kaolin to formulate mesoporous catalysts through acid treatment. The study of the effect of acid and alkaline activation on kaolin was also carried out by [14] and established that 6 M HCl activated under reflux states for 6 h will eliminate roughly 90 % of the octahedral Al^{3+} cations, followed by amorphous silica with greater surface area [14]. Authors [6] deliberates on the effect of treatment of kaolin with sulphuric acid on its physico-chemical characteristic, advocated that acid treatment is a suitable process for manufacturing porous and surface active materials with greater surface area.

In this study, we analytically investigate the effects of acid treatment of kaolin by sulphuric acid on its morphology and elemental composition. The effects on the morphology and elemental composition of kaolin were appraised by means of SEM and EDX analyses. Kaolin crystal structure was partly destroyed, however, serious chemical leaching was improved by acid treatment.

MATERIALS AND METHODS

Powdered form of kaolin used for this study was obtained from Kaolin (Malaysia) Sdn. Bhd. The raw kaolin was sieved to particle size of 45 µm. Sigma Aldrich supplied the sulphuric acid used; a Colourless viscous liquid with formula weight 98.08 g/mol and purity 99.999 %. Before acid activation, kaolin powder was dried in an oven for 24 hrs at 65 °C and subsequently sieved to 45 micron size. Activation was carried out by adding 10 g of kaolin sample to 100 ml of sulphuric acid in a 250 ml dry beaker [15]. The resulting solution was heated with vigorous magnetic stirring and refluxing on hot plate at temperatures 80 °C. Variable acid concentrations of 2 M, 4 M, 6 M and 8 mol/dm^3 sulphuric acid was used to repeat the activation process. The acid and kaolin were separated from the residual slurry by use of beaker and funnel. The neutral point was achieved with pH indicator after washing the residual kaolin with distilled water. Drying of the kaolin residues was carried out for 4 hrs in an oven at 80 °C [5, 16]. To obtain the initial size of 45 microns; dried samples were grounded and sieved. Samples were designated N-0, N-2, N-4, N-6 and N-8, where the numbers represents the acid concentrations.

RESULTS AND DISCUSSIONS

FESEM Analysis

Figure 1 shows the micrographs of kaolin particles by FESEM and EDX studies. Additive materials are more appropriately analysed on the nanometer scale by FESEM. In order to detect the elements present in the samples, Energy Dispersive X-ray analysis (EDX) was also carried out on the samples. The scanning electron micrographs of kaolin samples are presented in Figure 1.

Figure 1 (a) below shows particles of the layered silicate of untreated kaolin in form of books. This indicates that the particles are not fully dispersed into individual layers. The particles on acid treatment partially appeared as slices of paper with many sizes. In sample N-2, stacks of kaolin particles are quiet establish with slight distribution. N-4 has slight dispersion of the particles into filament, or plate-like fragments. Likewise, both N-6 and N-8 comprise of kaolin particle collected in form of agglomerates. Therefore N-6 and N-8 samples possibly will not be appropriate for dispersion into polymers. This report is in similar to the studies by [4].

EDX Analysis

Figure 1 represents EDX images before and after acid treatment on kaolin and their corresponding spectra of five peaks. The major elements that are detected in kaolin are silicon and aluminium and little magnesium; as demonstrated in Table 1. The EDX peaks of the kaolin samples indicated that kaolin simply contain five elements namely Mg, Al, Si, K, Fe and the contents are 1.06 %, 36.11 %, 50.37 %, 8.49 %, 3.97 % respectively. The EDX analysis result shows dealumination; hence as silicon content continued to increase correspondently with greater acid concentration, the aluminium content decreases. However, aluminium content continues to decline proportionally to acid content; except for sample N-4 which could not correlate with dealumination activity of the kaolin leaching. This report is in agreement with [17]. The summary of EDX result is represented in Table 1.

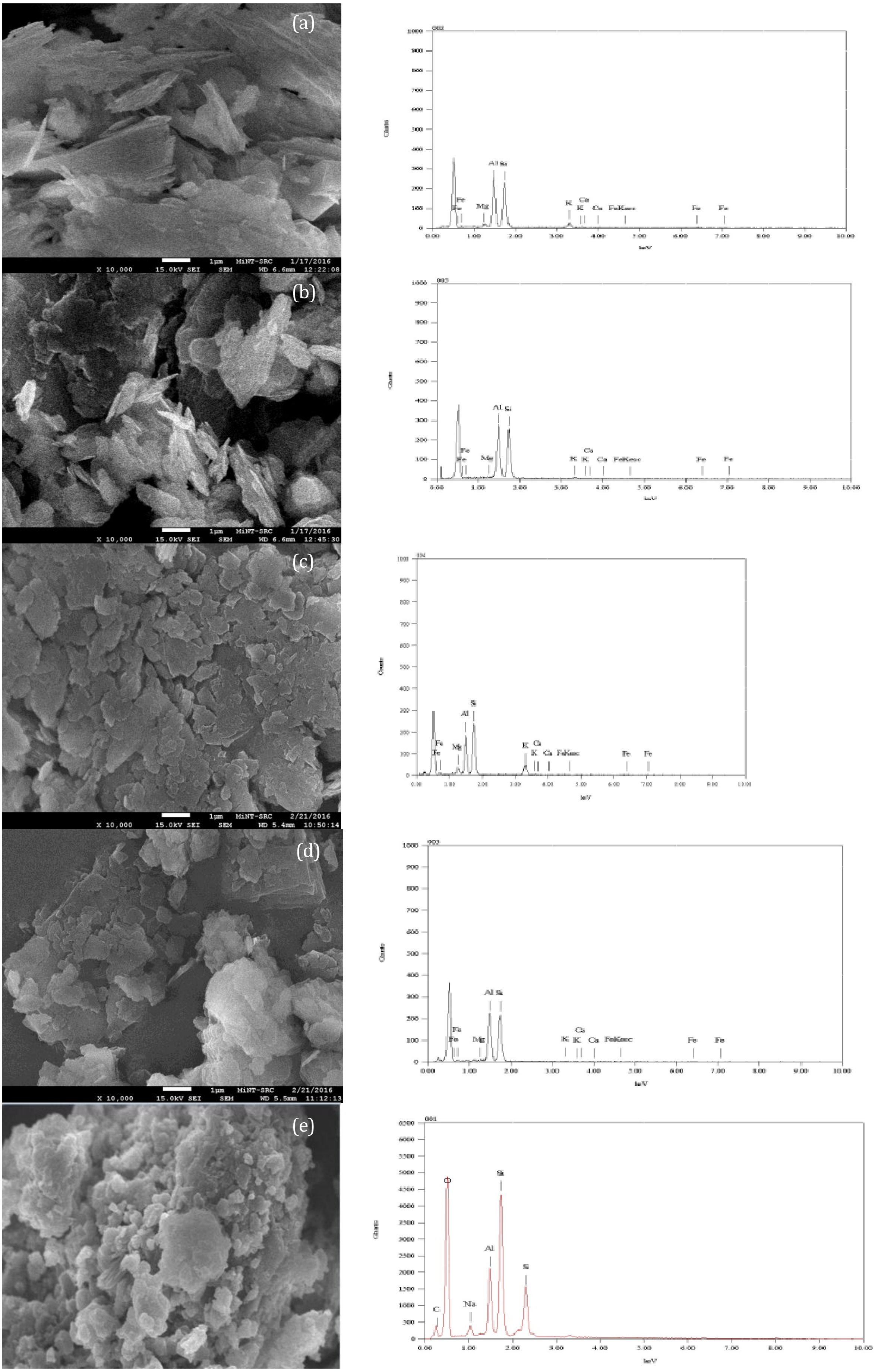

Figure 1 – FESEM and EDX of untreated and treated kaolin at different H_2SO_4 concentrations

Notes: (a) N-0; (b) N-2; (c) N-4; (d) N-6; (e) N-8.

Table 1 – EDX of kaolin samples before and after acid treatment

Sample	Composition (W/%)					
	Mg, K	Al, K	Si, K	K, K	Ca, K	Fe, K
N-0	1.06	40.05	50.37	8.49	-	3.97
N-2	0.17	36.11	57.53	1.51	0.14	1.40
N-4	3.98	39.25	50.52	7.84	-	-
N-6	-	38.51	58.98	1.91	0.52	0.08
N-8	0.13	17.66	59.78	1.36	0.32	2.15

CONCLUSION

In this research, changes in morphological and elemental composition due to effect of acid treatment on kaolin has been determined. The leaching and fragmentation of the kaolin layers through acidification at various acid concentration has been revealed by FESEM studies. Increase in acid strength leads to increase in disintegration of clay sheets. However, higher concentration leads to agglomeration of the kaolin particles. Silicon and Aluminium are the two major elements identified by EDX analysis. EDX also indicated that, as the acid strength increases, silicon content increases progressively while aluminium content decreases.

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REFERENCES

1. Olaremu, G. (2015). Physico-Chemical Characterization of Akoko Mined Kaolin Clay. *Journal of Mineral and Materials Characterization and Engineering, 3*(5), 353–361. doi: 10.4236/jmmce.2015.35038

2. Zhang, B., Pan, L., Zhang, H. -Y., Liu, S. -T., Ye, Y., Xia, M. -S., & Chen, X.-G. (2012). Effects of acid treatment on the physico-chemical and pore characteristics of halloysite. *Colloids Surfaces A Physicochemical and Engineering Aspects, 396*, 182–188. doi: 10.1016/j.colsurfa.2011.12.067

3. Kumar, S., Panda, A. K., & Singh, R. K. (2013). Preparation and Characterization of Acids and Alkali Treated Kaolin Clay. *Bulletin of Chemical Reaction Engineering & Catalysis, 8*(1), 61–69.

4. Panda, K., Mishra, B. G., Mishra, D. K., & Singh, R. K. (2010). Effect of sulphuric acid treatment on the physico-chemical characteristics of kaolin clay. *Colloids Surfaces A Physicochemical and Engineering Aspects, 363*(1–3), 98–104. doi: 10.1016/j.colsurfa.2010.04.022

5. Trabelsi, W., & Tlili, A. (2017). Phosphoric acid purification through different raw and activated clay materials (Southern Tunisia). *Journal of African Earth Sciences, 129*, 647–658. doi: 10.1016/j.jafrearsci.2017.02.008

6. Yang, N., Zhang, Z.-C., Ma, N., Liu, H.-L., Zhan, X.-Q., Li, B., … Shi, D. (2017). Effect of surface modified kaolin on properties of polypropylene grafted maleic anhydride. *Results in Physics, 7*, 969–974. doi: 10.1016/j.rinp.2017.02.030

7. Valášková, M., Rieder, M., Matějka, V., Čapková, P., & Slíva, A. (2007). Exfoliation/delamination of kaolinite by low-temperature washing of kaolinite-urea intercalates. *Applied Clay Science, 35*(1–2), 108–118. doi: 10.1016/j.clay.2006.07.001

8. Valášková, M., Martynková, G. M., & Matìjka, M. (2007). Chemically activated kaolinites after de-intercalation of formamide. *Ceramics, 51*(1), 24–29.

9. Horváth, E., Frost, R. L., Makó, É. , Kristóf, J. , & Cseh, T. (2003). Thermal treatment of mechanochemically activated kaolinite. *Thermochimica Acta, 404*(1–2), 227–234. doi: 10.1016/S0040-6031(03)00184-9

10. Jikan, S. S., Badarulzaman, N. A., Yahaya, S., & Adamu, A. D. (2017). Delamination of Kaolinite by Intercalation of Urea Using Milling. *Materials Science Forum, 888*, 136–140. doi: 10.4028/www.scientific.net/msf.888.136

11. Zykova, J., Kalendova, A., Matejka, V., Zadrapa, P., & Malac, J. (2010). Influence of kaolinite modification on the PVC composites properties. In *Advance in sensors, signals and materials,* November , 3–5 (pp. 30–34). Faro: n. d.
12. Tang, W., Song, L., Zhang, S., Li, H., Sun, J., & Gu, X. (2016). Preparation of thiourea-intercalated kaolinite and its influence on thermostability and flammability of polypropylene composite. *Journal of Materials Science, 52*(1), 208–217. doi: 10.1007/s10853-016-0323-8
13. Lenarda, M., Storaro, L., Talon, A., Moretti, E., & Riello, P. (2007). Solid acid catalysts from clays: Preparation of mesoporous catalysts by chemical activation of metakaolin under acid conditions. *Journal of Colloid and Interface Science, 311*(2), 537–543. doi: 10.1016/j.jcis.2007.03.015
14. Belver, C., Bañares Muñoz, M. A., & Vicente, M. A. (2002). Chemical Activation of a Kaolinite under Acid and Alkaline Conditions. *Chemistry of Materials, 14*(5), 2033–2043. doi: 10.1021/cm0111736
15. Woumfo, D., Kamga, R., Figueras, F., & Njopwouo, D. (2007). Acid activation and bleaching capacity of some Cameroonian smectite soil clays. *Applied Clay Science, 37*(1–2), 149–156. doi: 10.1016/j.clay.2006.12.008
16. Srasra, E., & Trabelsi-Ayedi, M. (2000). Textural properties of acid activated glauconite. *Applied Clay Science, 17*(1–2), 71–84. doi: 10.1016/s0169-1317(00)00008-9
17. Sengupta, P., Saikia, P., & Borthakur, P. C. (2008). SEM-EDX characterization of an iron-rich kaolinite clay. *Journal of Scientific & Industrial Research, 67*, 812–818.