

Research Article

DEFORMATION AND ADSORPTION CAPACITY OF KAOLIN THAT IS INFLUENCED BY TEMPERATURE VARIATION ON CALCINATION

YOGA WINDHU WARDHANA, ALIYA NUR HASANAH, PRISKA PRIMANDINI

Faculty of Pharmacy, University Padjadjaran, Jl. Raya Bandung - Sumedang Km. 21 Jatinangor 45363, Sumedang, Indonesia.

Email: yoga.ww@unpad.ac.id or ayodhna@gmail.com

Received: 05 Feb 2014 Revised and Accepted: 11 Apr 2014

ABSTRACT

Kaolin is a mineral composite derived from clay soil. It has been used for long time in medicine as an antidiarrhea. Antidiarrheal function of kaolin comes from its work locally at intestine as a strong adsorbent. Some contaminants which cause diarrhea in intestine will be adsorbed by kaolin and wasted with faeces. Adsorption capacity of adsorbent agent is influenced by surface properties and its porosity. Improving surface adsorption characteristics of the adsorbent agent will increase its adsorption capacity. Several efforts to increase adsorption capacity of adsorbent have been carried out such as structure modification of matter by chemical or physical ways. Chemical modification uses a complicated method than physical ways. Physical modification can be done by reducing particle matter or with high temperature heating like calcination. Reducing particle needs more time and energy, but with calcination structure modification is formed spontaneously and rapidly with a simple way. Calcination is an endothermic process with high temperature heating, but still under its melting point. With calcinations, the structure will thermally deformed with changes in mineral composite formation. In this research, the effect of calcination temperature variations on kaolin's deformation and adsorption capacity has been studied. Deformation of mineral composite was studied by X-Ray diffractogram from Powder X-Ray Diffractometer, and the adsorption capacity was analyzed by measuring number of Pb being adsorbed by using Atomic Absorption Spectroscopy. The result showed that at the 400°C deformation did not occur yet, but at 600° and 800°C temperature the deformation began. The adsorption capacity of Pb at 400°C raised by 0.377%, but on the contrary, at 600° and 800°C decreased significantly by 7.198% and 20.761%, respectively. Conclusion from those data indicated kaolin has raised adsorption capacity at 400°C, getting more heated the deformation of kaolin occurred with lower adsorption capacity.

**Keywords:** Kaolin, Calcination, Deformation, Adsorption capacity

INTRODUCTION

Kaolin is a natural aluminum silicate hydrate, which is free of most contaminants. Minerals are included in the kaolin is kaolinite, nakrit, decree, and hallosyite ( $Al_2(OH)_4SiO_5$ ). As an antidiarrheal agent, kaolin layers the intestinal wall, exactly as adsorbent that adsorbs toxins and bacteria in the digestive tract, acts to protect the gastrointestinal mucosa exile[1].

Adsorption phenomenon is an accumulation of a number of molecules, ions, or atoms that occur at the boundaries of the two phases. The amount of adsorbate accumulates on the adsorbent is affected by particle size and surface area of the adsorbent and the adsorbate. Condensed molecule is called adsorbate, while the substrate surface (solid or liquid) is called adsorbent[2].

Adsorption capacity of the adsorbent can be enhanced with increasing surface area, pore volumes and percent microporosity. One method to increase surface activity for adsorption that is modifying crystal structure or crystal habit of the adsorbent material through heating. Heating material process under its melting point has been commonly used in the field of metallurgy to purify the metal material. This process is called calcination. Calcination usually is used to bring decomposition thermal, phase transition and to remove volatile fractions such as  $CO_2$  and  $H_2O$  [3].

This work aims at enhancing adsorption capacity of kaolin by calcination and find the deformation of kaolin by variation of calcination temperature.

MATERIALS AND METHODS

Pharmaceutical grade powder of kaolin (ex. BDH, England),  $PbSO_4 \cdot 7H_2O$ , Nitric Acid concentrated (ex. Merck, USA).

Calcination Process

Kaolin calcinated in graphite crucible at electric furnace (Nabertherm). The graphite crucible has content of Kaolin heated each in different temperature at 400°, 600° and 800°C during 6 hours.

Porosity Evaluation

Porosity is evaluated using porosimeter device *Fisher Sub-Sieve Sizer*. Placed the sample test on test tube then closed with whatman paper filter and porous stopper. Press the stopper till all paper filters got into sample test, run device and watched the mano-meter indicator.

Density Evaluation

The true density of material is tested using pyc-nometer method, where in this test using methanol with 0.788 g/ml as test fluid.

Crystal Characterization

Characterization of kaolin deformation was conducted using Powder X-Ray Diffractometer (PXRD) (Rigaku, Geiger Flex).

Adsorption Capacity

Adsorption capacity of the test material was done by measuring the contents of  $Pb^{2+}$  that adsorbed by the test material. The levels of  $Pb^{2+}$  were determined using the Atomic Absorption Spectroscopy (Shimadzu, AA-6501S).

RESULTS AND DISCUSSION

Reported from previous studies that Kaolin when subjected to the heating process will go through two phases of weight loss. In the low temperature phase (25-650°C), kaolin will be facing loss of water molecules and at the high temperature phase (655-1365°C), kaolin began to decompose [4].

After the calcination process, the tested material has changed the nature differently. The porosity and the true density of kaolin as adsorption surface activity parameters showed changes after different temperature heating. After heating at 400°C, the surface activity was increased by increasing of porosity and true density value, but when the temperature of heating was raised, a decline was noted.

**Table 1: Evaluation of Kaolin Porosity**

| Treatment                      | Porosity |
|--------------------------------|----------|
| Kaolin before calcination      | 0.61     |
| Kaolin after 400°C calcination | 0.75     |
| Kaolin after 600°C calcination | 0.42     |
| Kaolin after 800°C calcination | 0.36     |

**Table 2: Evaluation of Kaolin True density**

| Calcination Treatment          | True Density (g/ml) |
|--------------------------------|---------------------|
| Kaolin before calcination      | 2.6199              |
| Kaolin after 400°C calcination | 2.6275              |
| Kaolin after 600°C calcination | 2.6097              |
| Kaolin after 800°C calcination | 2.6001              |

**Table 3: Contents of Pb<sup>2+</sup> and Adsorption Capacity**

| Calcination Treatment     | No. | contents of Pb <sup>2+</sup> (ppm) | Adsorption Capacity (%) |
|---------------------------|-----|------------------------------------|-------------------------|
| Kaolin before Calcination | 1   | 0.2936                             | 99.3516                 |
|                           | 2   | 0.2877                             | 99.3647                 |
|                           | 3   | 0.2948                             | 99.3490                 |
| Kaolin after 400°C        | 1   | 0.1149                             | 99.7463                 |
|                           | 2   | 0.1246                             | 99.7248                 |
|                           | 3   | 0.1239                             | 99.7264                 |
| Kaolin after 600°C        | 1   | 3.5519                             | 92.1563                 |
|                           | 2   | 3.5493                             | 92.1620                 |
|                           | 3   | 3.5537                             | 92.1523                 |
| Kaolin after 800°C        | 1   | 9.6802                             | 78.6231                 |
|                           | 2   | 9.6735                             | 78.5723                 |
|                           | 3   | 9.6967                             | 78.5867                 |

Changes during calcination cast for the adsorption capacity of the levels of Pb<sup>2+</sup>. The results of the determination of Pb<sup>2+</sup> contents adsorption at the test material seen in mutual accord with the increase and decrease in surface activity parameters (Table 3).

From the data in Table 3, it was found that at calcination temperature 400°C adsorption capacity higher than without calcination, but higher temperature has lowered the adsorption capacity.

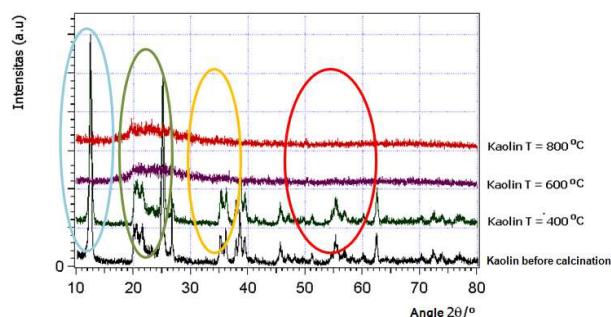
**Table 4: Comparison of Adsorption Capacity between before and after Calcination**

| Calcination | %                 |
|-------------|-------------------|
| 400°C       | Raise 0.3774      |
| 600°C       | Decrease 7.198233 |
| 800°C       | Decrease 20.76107 |

For getting some answer what happen with different nature after calcination treatment. We analyzed the sample by powder X-ray diffractometer then we found picture below.

Figure 1 shows that after 600°C, crystallinity of kaolin was broken. Raise more heat further damaged the deformation of kaolin structure to becoming more amorphous. There has connectivity of kaolin structure and adsorption capability. At 400°C, the kaolin crystallinity

looked more increase cause of removing some volatile compound in its pores then making percentage of microporosity increase.

**Fig. 1: Powder X-Ray Diffractogram (PXRD) of kaolin before and after calcination**

## CONCLUSION

Calcination at 400°C made crystallinity kaolin higher that is due to elimination of some volatile compound and raise its percent microporosity then the adsorption capability increased. Calcination temperature higher than 600°C destroyed the kaolin structure making loss of its adsorbability nature. There has relationship between adsorption capability with crystalline form of kaolin. The crystalline form has better adhesivity force, conversely in amorphous form has reduced adhesivity force.

## ACKNOWLEDGEMENTS

The authors conveyed their sincere thanks to National Nuclear Energy Agency of Indonesia (BATAN) at Serpong, Banten Province Indonesia for their retribution on PXRD evaluation. Gratefully thanks to Pharmacy Faculty at Universitas Padjadjaran for their meritorious service on all laboratory services.

## REFERENCES

- Kosasih, K. 2009. *Comparison of Heavy Metal Adsorptions by Thai Kaolin and Ballclay*. (Jurnal) Fakultas MIPA UGM. Yogyakarta
- Prawira. 2008. *Biosorption of Heavy Metals*. *Research Journal Of Chemistry And Environment*. p. 71-78.
- Mac Kenzie, R., 1957. *The Differential Thermal Investigation of Clays*. *Mineralogy Society London*. 141-148.
- Endang, Tri Wahyuni. 1993. *Using X-ray Diffraction Methode for Detection of Mineral Clay Crystal Deformation Cause by Activation and Thermal*, *Jurnal. Laboratorium Kimia Analitik, FMIPA UGM*. Yogyakarta. 37-44.
- Reynolds JEF. *Martindale: The Extra Pharmacopoea*,. 29 ed. London: The Pharmaceutical Press, 1989 pp. 1092.
- British Pharmacopoea. London: Pharmaceutical Press, 1985 pp. A143.
- British Pharmacopoea, vol. 2. London: Pharmaceutical Press, 1980 pp. 747
- Mujiyanti, D.R. 2007. *Adsorption of Some Metal Ag(I), Pb(II), Cr(II), and Ni(II) On Merkpto-silica Hybride from Rice Plant Husk Ashes*. (Thesis) *Fakultas MIPA UGM*. Yogyakarta : p 29
- Ilić, Biljana R. et al., 2010. *Thermal Treatment Of Kaolin Clay To Obtain Metakaolin*. *Hem. ind.* 64 (4) 351–356.