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Characterization and Optimization of Salicylic Acid Bentonite Clay Nanocomposite

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This work aimed to synthesize and characterize bentonite clay with the assist of Fe(III) ion as salicylic acid chelator. Variation on intercalated concentration and contact time during intercalation process were performed to obtain the best bentonite-salicylic acid nanocomposite. The characterisation of the resulting nanocomposite was done by analyzing the surface area, pore volume, pore diameter and particle size using N_2 gas adsorption analysis method (BET). The magnitude of loading capacity of bentonite to salicylic acid was measured by UV spectrophotometer and the composite topography was analyzed using SEM method. The results of BET method analysis showed that the resulting composite had a surface area of 107.8, 65.6, 109.2 m^2/g, respectively and with a particle size of 55.65, 91.41 and 54.92 nm, respectively. The largest loading capacity is owned by composite produced with 24 h contact time with a capacity of 66.4720 mg/g.The results of topographic analysis with SEM showed that resulting composite has a layered structure with uniform particle shape and heterogeneous particle size.

Keywords: Nanocomposite, Bentonite clay, Salicylic acid.

INTRODUCTION

Bentonite is the name of clay containing more than 80 % montmorillonite minerals. It has enormous benefits in the fields of industry, health and environment [1]. The most prominent benefit of bentonite is due to its high adsorptivity. Therefore, it is best used as an adsorbent of various pollutants, impurities, and other harmful substances in both organic and inorganic compounds [2].

Montmorillonite as a major component of bentonite has a high cation exchange capacity that is easily modified with a variety of other compounds that can improve the performance of bentonite [3]. In addition, bentonite has a high surface area which is the main requirement for the material functioned as an adsorbent. Traditionally, bentonite is widely used as a cleanser and very popular as an additive (ingredient) in a number of detoxification programs [2]. Bentonite is also used as a formula for dermatology. In recent years, montmorillonite has been intercalated into drug compounds hence it has attracted the attention of researchers for producing new materials with excellent chemical and physical properties [4]. Zheng *et al*. [5] investigated the intercalation of ibuprofen into montmorillonite. Fejer *et al*. [6] reported intercalation and release of promethazine chloride and buformin hydrochloride from montmorillonite. Smectite (montmorillonite) may increase *in vitro* solubility rate of non-ionic drugs and drugs that are not soluble in acids [7]. Drug release from the clay surface is facilitated by weak bonds between them and simultaneously the wettability of the drug is supported by the hydrophilic properties of clay [8].

In this research, salicylic acid is interceded into interlayer space of bentonite clay with the purpose of utilizing bentonite as salicylic acid carrier in order to decrease the toxicity effect of salicylic acid. The characterization of resulting composite including parameters of surface area, pore diameter, pore volume and particle size were analyzed by BET method, loading capacity by UV spectrophotometer and topography of salicylic acid-clay bentonite composite by SEM.

EXPERIMENTAL

The chemicals used in this study were all qualified in pro-analysis, HF, $FeCl_3{\cdot}6H_2O$, dry salicylic acid ion (free of water), ethanol (Sigma-Aldrich), Whatman 42 filter paper and bentonite clay used was Indonesian bentonite.

Brunauer-Emmett-Teller (BET) specific surface area (S_{BET}), total pore volumes and average pore diameter of the samples were measured from N_2 adsorption/desorption isotherms at 77 K, using Micrometric Type Tristar II, 3020. The morphology and composition of bentonite and salicylic acid-bentonite clay nanocomposites were observed using a Scanning Electron Microscope SEM EDX.

Synthesis of bentonite clay-salicylic acid composite: Bentonite clay (100 g) was washed with 1 L of 1 % hydrofluoric acid (HF) to remove any impurities present and then dried. After dried, it was crushed, then sieved with a 10^6 µm sieve. A mixture of intercalates *viz*., 0.0176 M Fe(III) and 0.025 M of salicylic acid (600 mL) was added to bentonite clay suspension prepared by dispersing 30 g of clay into 300 mL of dry salicylic ion. The mixture was stirred for 24 h, filtered and washed several times until it become free from chloride ions. The obtained composite was then dried in an oven at 100-120 ºC. A variation of intercalate concentration was performed to obtain composites having the best physico-chemical characteristics.

Characterization of surface area, pore volume, and size particle: Analysis with N_2 gas adsorption method (BET) is a method used to measure the surface area of solid material based on multiple phenomenon takes place at fixed temperature, often called isothermic adsorption [9]. The isotherm adsorption equation used on the surface of solid is given as:

$$\frac{\frac{P}{P_o}}{W\left(1-\frac{P}{P_o}\right)}=\frac{1}{W_m C}+\frac{C-1}{W_m C}\left(\frac{P}{P_o}\right)$$

where: W = weight of adsorbed gas at a relative pressure P/P_o (g); W_m = weight of the adsorbed gas on a single layer (g); C = BET constant; P_o = saturated adsorption vapor pressure (mm Hg) and P = gas pressure.

RESULTS AND DISCUSSION

Several parameters *viz*., particle surface area, pore filtration rate, pore volume and particle size can be obtained using N_2 gas adsorption method (BET). As can be seen from Table-1, nanocomposites with significantly different characteristic properties produced, when different conditions were applied during the process of synthesis. From the three variations of the intercalator concentration, with N_2 gas adsorption method (BET), it was found that nanocomposites **3** synthesized from the greatest intercalated concentrations of salicylic acid produced the composites with the smallest particle size of 54.9225 nm with the largest surface area of 109.2448 m^2/g. If all the three nanocomposites were compared with bentonite, it can be concluded that the intercalation of salicylic acid, which is packed by Fe (III) into interlayer space of bentonite clay causes an increase in the quality of physical properties of bentonite, especially the surface area and particle size.

Salicylic acid loading capacity in interlayer space of bentonite clay: The intercalation of salicylic acid into interlayer space of clay begins with the salicylic acid adsorption process by the surface of clay particles. Adsorption occurring on the solid surface will provide various form of isotherms. For example, Langmuir isotherm with one-layer closure (monolayer) or only a few typical molecular layers in micropore solids, or adsorption isotherm occurring when contact frequency between the adsorbate and the high adsorbent in which this adsorption generally occurs in solids with a larger pore diameter than the micropore. Loading capacity is calculated using the following equation:

$$W=\frac{C_1-C_2}{1000}\times V\times\frac{1}{W_{ads}}$$

where W = loading capacity of salicylic acid (mg/g), C_1 = initial salicylic acid concentration (ppm); C_2 = salicylic acid concentration after intercalation (ppm), V = volume of solution (L) W_{ads} = weight of clay used (g).

It is concluded that 24 h of contact time (adsorption) gave the largest loading effect with a loading capacity of 66.4720 mg/g (Table-2).

TABLE-2
LOADING CAPACITY OF SALICYLIC ACID ON BENTONITE CLAY

Adsorption time (h)	[salicylic acid]$_i$ (ppm)	[salicylic acid]$_f$ (ppm)	[salicylic acid]$_{ads}$ (ppm)	Loading capacity (mg/g)
6	3453,000	268,646	3166,354	63,3271
12	3453,000	232,899	3220,101	64,4020
24	3453,000	129,401	3323,599	66,4720
48	3453,000	141,749	3311,251	66,2250
96	3453,000	173,698	3279,302	65,5860

SEM analysis: The SEM images (Fig. 1) show that the salicylic acid bentonite clay nanocomposite particles have an almost identical form of spherical dispersed with non-uniform sizes. With 5000 times magnification it can be seen that the layered nanocomposite structure corresponds to the bentonite structure as its host.

Conclusion

In this research, bentonite-salicylic acid nanocomposite synthezised with the greatest intercalated concentration (0.0176 M Fe(III) + 0.1 M salicylic acid) yielded the best characteristics of nanocomposite with the smallest particle size of 54.9225 nm and the largest surface area of 109.2448 m^2/g. It also gave the largest loading capacity of salicylic acid into interlayer space

TABLE-1
CHARACTERISTIC OF SALICYLIC ACID-BENTONITE NANOCOMPOSITES OBTAINED BY BET METHOD

Sample	Concentration	Surface area (m^2/g)	Pore diameter (nm)	Pore volume (cm^3/g)	Particle size (nm)
Bentonite	–	90.0586	27.0606	32.41110	50.0165
Nanocomposite 1	0.0176 M Fe(III) + 0.025 M salicylic acid	107.8145	2.0815	0.022535	55.6512
Nanocomposite 2	0.0176 M Fe(III) + 0.050 M salicylic acid	65.6373	2.0841	0.017570	91.4114
Nanocomposite 3	0.0176 M Fe(III) + 0.100 M salicylic acid	109.2448	2.0795	0.022033	54.9225

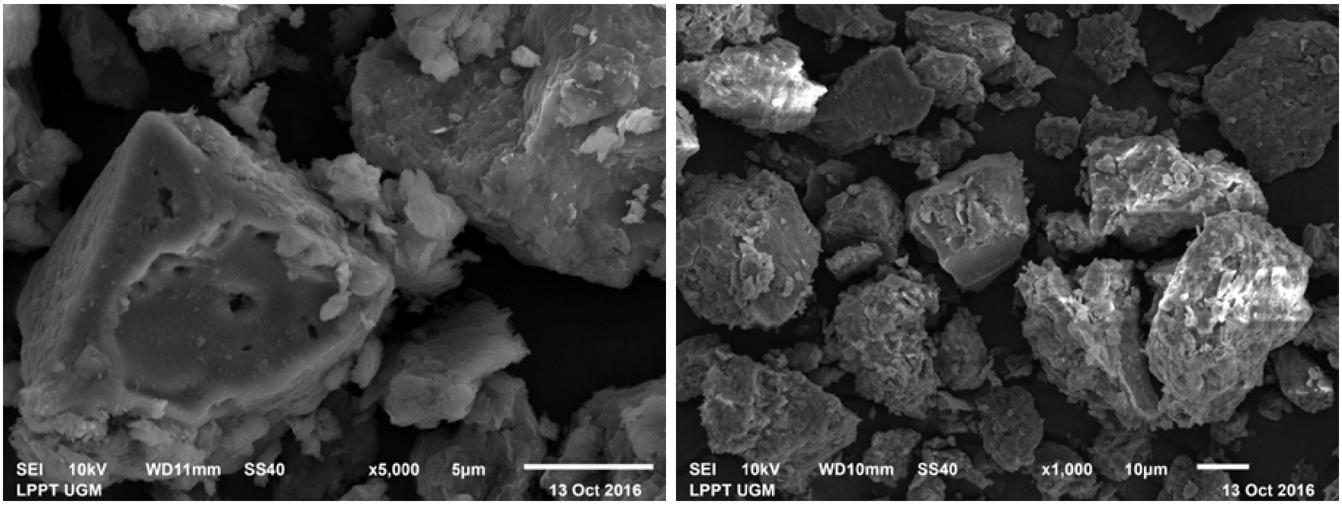

Fig. 1. SEM image of bentonite-salicylic acid clay nanocomposite

of bentonite clay which was 66.4720 mg/g. Based on SEM images, it can be ascertained that bentonite-salicylic acid nanocomposite had the same particle shape with a heterogeneous particle size distribution.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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