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STUDIES OF THE STRUCTURE OF ORGANOCLAYS PREPARED FROM CHINESE BENTONITE AND TETRADECYLTRIMETHYLAMMONIUM BROMIDE

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ABSTRACT

Organoclays were prepared in conditions following: the reaction temperature 500C, the tetradecyltrimethylammonium bromide (TTAB)/bentonite rate of 0.5, the pH of 9.0, the reaction time of 4 hours with the basal spacing d_001=37.897Å, the cation penetration of 22.93%, prepared organoclays have layer structure and high porosity. They have a good red phenol adsorption capacity with the maximum adsorption capacity q=333.333mg/g and Langmuir isotherm b= 0.041.

Keyword: Bentonite; Organoclays; Tetradecyltrimethylammonium Bromide.

1. INTRODUCTION

Bentonite is naturally capable of adsorbing organic substances; however, the adsorption capacity is not high enough to be applied in practice. Hence, it needs to be modified to enhance adsorption capacity, and amine salts have been generally employed as cost-effective reagents. Once hydrocarbon chain is inserted into layers can increase interlayer distance and hydrophobic properties, leading to higher affinity toward organic substances.

Organoclays were synthesized from bentonite and the amount of quaternary ammonium salts (primary, secondary, tertiary, and quaternary structure, with linear chain, branched one, and cyclic one). When a hydrocarbon chain is added to layers of bentonite, will increase layer spacing, and hydrophobic, this leads to an increase in the adsorptive capacity of materials with organic chemicals, especially cyclic organics, chemicals with large molecular weight and bulky structure.

However, a survey of prepared organoclays from Chinese bentonite and tetradecyltrimethylammonium bromide (TTAB) did not fully perform. Therefore, we studied some factors which impact the process of preparing organoclays from Chinese bentonite and TTAB found appropriate conditions to prepare organoclays with d001 and a large cation penetration (%) and initially poked into the sorption capacity of red phenol.

2. EXPERIMENT

2.1. Chemicals

Chemicals: using Chinese bentonite clays with above 90% MMT, less than 10 microns of particle size, 105mdlg/100g of ion exchange capacity, and less than 50% of humidity. Quaternary ammonium salt is used: C17H38BrN (M=336,4g/mol) tetradecyltrimethyl ammonium bromide (TTAB) (Sigma-Aldrich).

2.2. Organoclay synthesis

The survey of some conditions to prepare organoclays was performed as follows: 1 gram of bentonite was added to 100ml of water, stirring for 2 hours so that clays maximum expand, which created 1% of bentonite dispersion. Tetradecyltrimethylammonium bromide solution (TTAB) was stirred in 50ml of heated water at a temperature between 40oC ÷ 50oC with respective weight. After that, gradually pour every TTAB drop into a flask containing 1% bentonite dispersion, and then adjust the pH to the survey value. Continue stirring at constant temperature and time on a magnetic stirrer. After the reaction time, stably place the mixture for 12 hours at room temperature, then filter and wash the precipitate. The product was dried at 800C for 2 days.

The content of organic cation penetration (%) is calculated by the difference between the mass loss rate of the bentonite samples that were prepared with and without TTAB.

3. RESULTS AND DISCUSSION

3.1. Studies on the structure of prepared organoclays

The process of preparing organoclays in a water environment from Chinese bentonite and tetradecyltrimethylammonium bromide was surveyed, the results showed that the optimum conditions for this preparation process were: the reaction temperature 500C, the TTAB/bentonite rate of 0.5, the pH in the solution of 9.0 and the reaction time of 4hours.

3.1.1. Studies using X-Ray diffraction (XRD)

X–Ray diffractograms of bentonite and organoclay were respectively shown in Figure 1 and Figure 2.

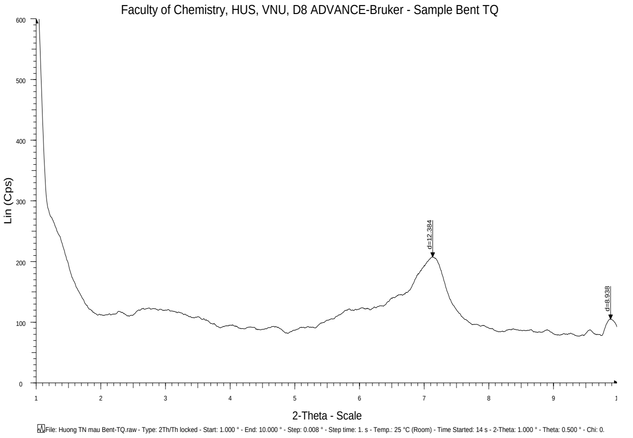


Figure 1: X-ray diffractogram of Chinese bentonite sample

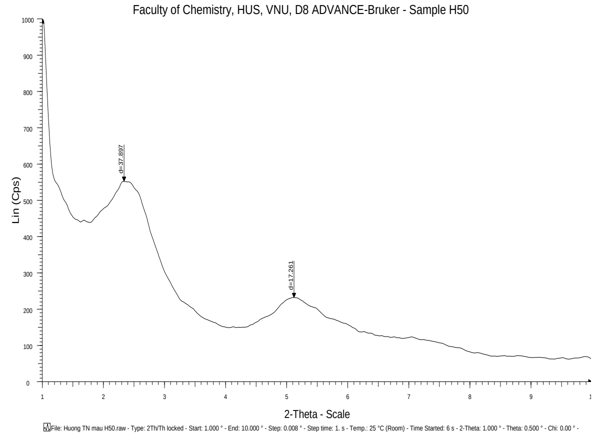


Figure 2: X-ray diffractogram of organoclay

The result indicated that the diffraction angle 2θ dramatically shifted in the range of 60 – 70 and 2.30 – 2.60 for Chinese bentonite and organoclay, respectively. The basal spacing d001 strongly rises from 12.384Å for Chinese bentonite to 37.897Å for organoclay.

3.1.2. Studies using Fourier transform infrared (FT-IR) spectroscopy

The results of studying FT-IR spectroscopy of the Chinese bentonite, TTAB, and organoclay were shown in Figure 3 and Table 1. Results obtained from IR spectra agree with reports of other authors.

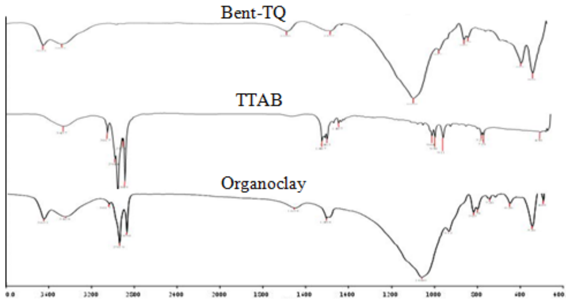


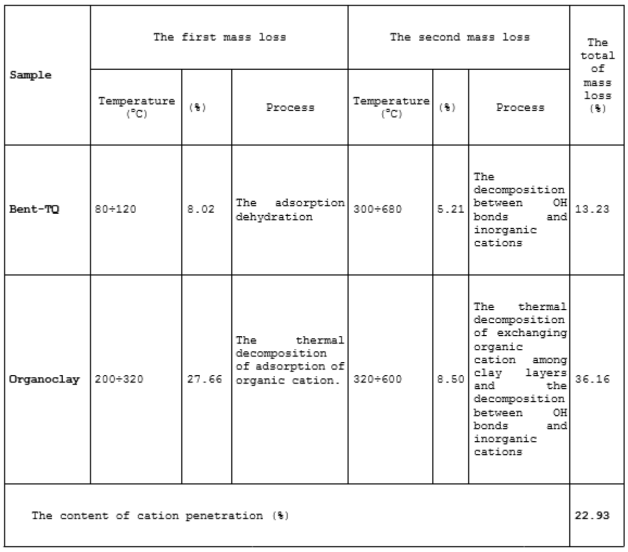
Figure 3: FTIR spectroscopy of the Chinese bentonite, TTAB, and organoclay.

Sample	ν_{OH}	ν_{CH_3,CH_2}	ν_{C-N}	$\nu_{Si-O-Al}$	ν_{Al-O}
Bentonite	3631	-	-	1039	912
TTAB	-	2850, 2917	1487	-	-
Organoclay	3631	2850, 2917	1487	1039	912

Table 1: The wavenumber of characteristic IR adsorptions of bentonite, TTAB, and organoclay (cm-1)

It indicated the appearance of TTAB in prepared organoclay.

3.1.3. Studies using thermal analysis



Sample	The first mass loss			The second mass loss			The total of mass loss (%)
	Temperature (°C)	(%)	Process	Temperature (°C)	(%)	Process	
Bent-TQ	80÷120	8.02	The adsorption dehydration	300÷680	5.21	The decomposition between OH bonds and inorganic cations	13.23
Organoclay	200÷320	27.66	The thermal decomposition of adsorption of organic cation.	320÷600	8.50	The thermal decomposition of exchanging organic cation among clay layers and the decomposition between OH bonds and inorganic cations	36.16
The content of cation penetration (%)							22.93

Table 2: Results of analyzing thermal diagrams of Chinese bentonite and organoclay

The thermal analysis result indicated that the optimum condition for preparing organoclay is 22.93% of the content of cation penetration.

3.1.4. Studies using the scanning electron microscope (SEM)

SEM images of Chinese bentonite and organoclays prepared in the optimum condition are shown in Figure 4. The result indicated a great difference from prepared organoclays with layer structure and high porosity.

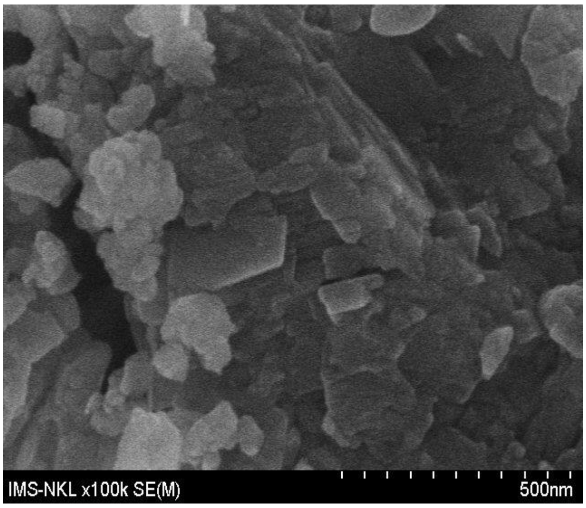


Figure 4(a): SEM image of Chinese bentonite

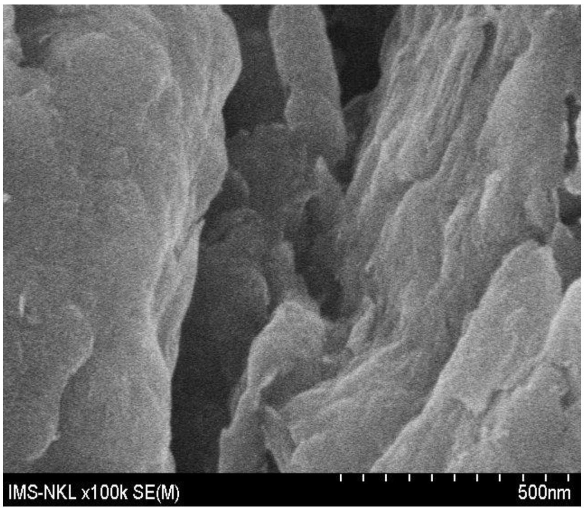


Figure 4(b): SEM image of organoclays

3.2. Studies on red phenol adsorptive capability

Having researched some factors that affected the red phenol adsorptive capability of organoclays in comparison with Chinese bentonite. The finding showed that in surveying conditions: the adsorption equilibrium time of Chinese bentonite and organoclay were 60 minutes and 45 minutes respectively. If the weight of the adsorbent material is 0.05 grams, the adsorption capacity is the largest, and adsorptive performance is constant.

Having described the adsorption process of Chinese bentonite and organoclay based on the Langmuir adsorption isotherm, determined that the maximum red phenol adsorption capacity of organoclay is 333.333mg/g and Langmuir isotherm b = 0.041; the one of Chinese bentonite is lower q = 30.581mg/g.

4. CONCLUSION

Organoclays were prepared in such conditions: a reaction temperature 50oC, a TTAB/bentonite rate of 0.5, a pH of 9.0, reaction time of 4 hours, using some methods such as XRD, IR, thermal analysis, and SEM to study their structure. The finding indicated that organoclays have d001 = 37.897A, the content of organic cation penetration of 22.93%, a layer structure and high porosity.

Prepared organoclays have a good phenol adsorption capacity with the maximum adsorption capacity q = 333.333mg/g and Langmuir isotherm b = 0.041.

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