

# Synthesis and Performance Evaluation of Organic Bentonite Modifier Dimethyldistearylammonium Bromide (DODMAB)

Qingwang Liu, Ao Sun, Zhenzhong Fan, Jiguang Wang, Tong Zhang, Dong Cheng

Northeast Petroleum University, Daqing, China

Email: [liuqingwang@163.com](mailto:liuqingwang@163.com), [sunsunaoao@126.com](mailto:sunsunaoao@126.com), [fanzhengzhong@163.com](mailto:fanzhengzhong@163.com), [wangjigang9999@163.com](mailto:wangjigang9999@163.com), [0648553814@qq.com](mailto:0648553814@qq.com), [277134741@qq.com](mailto:277134741@qq.com)

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## Abstract

Dimethyldistearylammonium Bromide (DODMAB) is a bentonite modifier with good performance. In this experiment, 1-Bromo-octadecane is the raw material, and completes tertiary amination and quaternization successively by changing the condition of experiment. The result is that the productivity can achieve 78.94% in the synthesis of DODMAB. It has been used in the experiment of bentonite modification and achieves good results; compared with using trimethylstearylammonium bromide as the organic modifier, the synthetic organic bentonite has better performance than the common organic modifier when used in the oil base drilling fluid of oilfield operation.

## Keywords

Organic Modifier, Stepwise Synthesis, Performance Evaluation

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## 1. Introduction

There are many kinds of bentonite modification agent, and various types of hyamine are the most common used. DODMAB is a surfactant of quaternary ammonium with double long carbon chains, it not only can be used for bentonite modification but also as fabric softener and fungicide [1]. The length and amount of carbon chains of hyamine have a significant impact on the structure and space of organobentonite [2] [3]. So the DODMAB has perfect performance as the organic modifier and the product has the better performance when used in the oilfield operation. This experiment will synthesize DODMAB with method of fractional step and use the products as

bentonite modification agent and evaluate the performance of the bentonite.

## 2. Experimental

### 2.1. Experiment Material

1-Bromooctadecane, Dimethylamine aqueous solution, Na-bentonite clay, Bentonite, rimethylstearylammonium Bromide.

### 2.2. Experiment Method

**The synthesis of DODMAB.** Add dimethylamine aqueous solution to three-neck flask, while add granular sodium hydroxide and CTMAB as phase transfer catalyst. Heat the flask slowly to dissolve the sodium hydroxide with back flow and agitation, then add half of 1-bromooctadecane gradually which has been heated to the flask. Heat to 120°C - 140°C for 3 hours and then cool down the solution to 80°C - 90°C and add the other 1-bromooctadecane into the flask and go on heating for 3 hours. After the action has completed, pour out the liquid to the beaker while it is still hot and the liquid will appear laminate. Then take the white crystalline solid of the bottom layer into anhydrous methanol and heat until the solid dissolve. Then wait the solution cooling down, the DODMAB will educe from the solution, recrystallize the crystalline solid for four to five times and dry out it with vacuum drying oven that is the production [4].

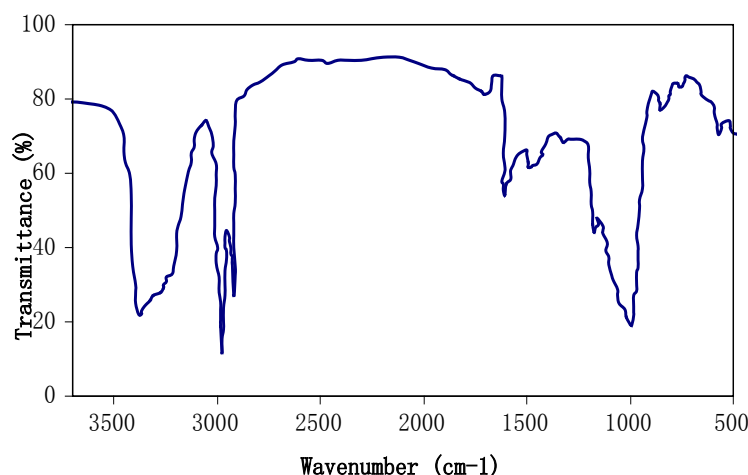
**The synthesis of organic bentonite.** Put 5 g sodium bentonite into 20 mL distilled water and stir constantly until pasted and regulate pH to 7 - 8, then add 40 mL solution which has dissolved 5.5mmol hyamine surfactant. Then heat to 80°C for 2 hours, after the chemism is finished, centrifugalize the solution when it has cooled down. Remove the liquid and wash the solid with distilled water for 4 times, and desiccate under 80°C - 100°C, grind in 300 mesh then get the organic bentonite sample.

**The valuation of performance.** Examine the DODMAB sample with Avatar360FT-IR infrared spectrometer. Compare the FT-IR with standard FT-IR and ascertain the product structure of the sample.

**Figure 1** is the result of FT-IR of DODMAB, from the figure, y-axis represents the transmissibility of IR, the x-axis represents the reciprocal of wavelength. The absorption peak in 2992  $\text{cm}^{-1}$  is absorption peak of  $\nu_{\text{as}}(\text{C}-\text{CH}_2-\text{C})$ ; the  $\nu_{\text{s}}(\text{C}-\text{CH}_2-\text{C})$  absorption peak is in 2853  $\text{cm}^{-1}$ ; 1329  $\text{cm}^{-1}$  absorption peak is absorption of  $\nu(\text{C}-\text{N})$  chemical bond stretching vibration. As a result, the FT-IR result verify the exist of DODMAB.

**Measurement of viscosity of organic bentonite.** Add 36mL dimethylbenzene to 3.6 g organic bentonite sample, agitate for 5 min and add 1.5 mL carbinol, add more 18mL dimethylbenzene. Agitate for 2 min at 1200 rpm, transfer the gel to measuring container, measure viscosity with NDJ-8s Rotatory viscometer.

**Critical volume and swelling volume of organic bentonite.** Weigh 1.0000 g organic bentonite and put in graduate, add mixture of solvation accelerant and medium slowly and agitate with glass rot at the same time. When the gel just start slow flowing the volume of the mixture add into is the critical volume. Then go on adding



**Figure 1.** Infrared spectra of sample.

20 - 30 mL mixture liquid and agitate it until it is steady, set it statically for 24 hours and measure the volume that it has sediment is the swelling volume  $V_E$ . These two parameters reflect the swelling performance and gel performance of organic bentonite which have oleophilic and hydrophobic performance.

#### 4. Results and Discussions of the Synthesis of 1-Bromooctadecane

It can be perceived from **Table 1** that the best ratio of reactant is  $n(\text{C}_2\text{H}_7\text{N}):n(\text{C}_{18}\text{H}_{37}\text{Br}) = 1:3$ , because 1-bromooctadecane can stimulate synthesis of hyamine, on the other hand, the more 1-bromooctadecane is added the cost of the synthesis is more and the purification of hyamine will also be more difficult. The temperature of the first stage of reaction should be  $130^\circ\text{C}$  because the lower temperature detrimental the synthesis process, while the higher temperature would increase the quantity of byproduct which would decrease the productivity; then as the same reason, the second stage of the reaction temperature should be  $85^\circ\text{C}$ .

#### 5. Results and Discussions of the Synthesis of Organic Bentonite

From the **Table 2**, the longer carbon chain of organic modifier has the viscosity of organic bentonite  $\eta$  is higher, it shows that the increase of organic matter content. The critical volume  $V_C$  shows the gel performance of the hyamine organic bentonite in the organic medium, it also increases with the carbon chain of carbon chain lengthening; the swelling volume  $V_E$  shows that the swelling performance of organic bentonite in the organic medium and the performance increases with the growth of the carbon chain of organic modifier. In this article, when the organic bentonite is modified by DODMAB has the best performance.

#### 6. Summary

1) Because the process of the synthesis of DODMAB has two different stages, stepwise synthesis is better than the simple directly synthesized, and the productivity and efficiency of stepwise synthesis are both higher.

2) The 1-bromooctadecane and dimethylbenzene mole ratio is 1:3, and the temperature of tertiary amination process should be  $130^\circ\text{C}$  and the temperature of quaternization process should be  $85^\circ\text{C}$ , the synthesis productivity of DODMAB can achieve 78.94%. When the organic modifier is hyamine, wet method synthesize organic bentonite is best, the organic exchange rate can achieve 92%.

3) Comparing organic bentonite modified by resultant DODMAB with organic bentonite which is modified

**Table 1.** Orthogonal optimization of design and productivity result of DODMAB.

$n(\text{C}_2\text{H}_7\text{N}):n(\text{C}_{18}\text{H}_{37}\text{Br})$	Temperature 1 <sup>st</sup> ( $^\circ\text{C}$ )	Temperature 2 <sup>nd</sup> ( $^\circ\text{C}$ )	Productivity (%)
1:2	130	70	48.54
1:2	120	85	42.65
1:2	140	100	41.36
1:2.5	140	70	55.08
1:2.5	120	85	58.61
1:2.5	130	100	60.32
1:3	140	70	71.24
1:3	130	85	78.94
1:3	120	100	74.11

**Table 2.** The capability of organic bentonite.

Sample	Viscosity (mPa·S)	Critical volume (mL)	Swelling volume (mL)
DODMAB	198	3.62	17.12
STAB	147	2.58	11.96

by trimethylstearylammmonium bromide, the organic bentonite modified by DODMAB has better performance that the viscosity is 198 mPa·S, the critical volume is 3.62 mL and swelling volume can achieve 17.12 mL. The organic bentonite has reached the requirements of the oil base drilling fluid in increasing viscosity and fluid loss.

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