

# Synthesis of high-temperature viscosity stabilizer used in drilling fluid

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**Abstract** For a well performance drilling fluid, when it operates in deep wells under high temperature, the most important property required is the thermal stability. The drilling fluid properties under high temperature can be controlled by proper selection of viscosity stabilizer, which can capture oxygen to protect polymer agent in the drilling fluid. In this paper a viscosity stabilizer PB-854 is described, which was synthesized by 4-phenoxybutyl bromide, paraformaldehyde, and phloroglucinol using etherification method and condensation reaction. We studied the effect of catalyst dosage, temperature, time, and stirring rate on the synthetic yield. Under this condition: molar ratio of 2-tert-Butylphenol, paraformaldehyde and phloroglucinol of 2:1:2.5, reacting temperature of 100 °C, stirring rate of 100 r min<sup>-1</sup>, and mass content of catalyst of 15 %, char yield of 5-bromine-3-tert-butyl salicylaldehyde reached 86 %. Under this condition: molar ratio of 5-bromine-3-tert-butyl salicylaldehyde and phloroglucinol of 4, reacting temperature of 60 °C, reacting time of 30 min, volume content of sulphuric acid of 80 %, char yield of the target product viscosity stabilizer PB-854 is 86%. Finally, in this paper, infrared spectroscopy is adopted to analyse the structure of the synthetic product PB-854. The improvement in the stability of drilling fluid was further shown after adding the viscosity stabilizer in the common polymer drilling fluid under high temperature conditions of 120 °C ~ 180 °C. The results show significant change in terms of fluid stability in the presence of this new stabilizer as it provides better stability.

## 1. Introduction

Polymers react by cross-link or free radicals with oxygen under high temperature[1-3], so stabilizer should have spatial structures and capture oxygen free radicals. Therefore, a phenolic compound with a substituted base on one or both sides of a benzene rings was designed and synthesized by 2-tert-butylphenol, paraformaldehyde, and phloroglucinol[4-7]. How to get the highest yield of target product under laboratory conditions? This paper optimized synthetic conditions by orthogonal experiment. The results show that molar ratio of raw material, temperature, reacting time and stirring rate have different effects on the yield of target product. By the yield under different conditions, the optimal react conditions are confirmed. In addition, the structure of synthetic product PB-854 was analysed using infrared spectroscopy[8-10].

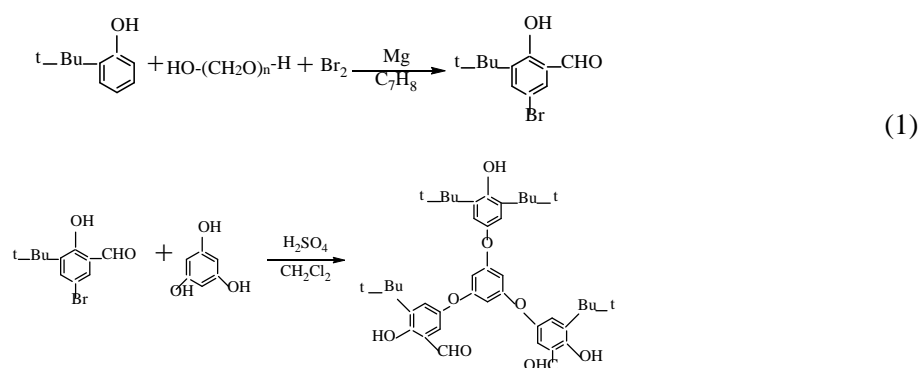
Finally, the new high-temperature stabilizer PB-854 was evaluated in the common polymer drilling fluid system under different temperature by comparing its property and filter loss. PB-854 could protect the polymer from degradation and keep the stability of drilling fluid. In addition, it could be



easily obtained and had good usability in the drilling fluid. Therefore, it has a great application prospect in the oil industry.

## 2. Experimental

In this experiment, 2-tert-Butylphenol, paraformaldehyde and phloroglucinol were taken as raw materials, and the synthetic experiment was completed in two steps. Firstly, 5-bromine-3-tert-butyl salicylaldehyde is synthesized by 2-tert-Butylphenol, paraformaldehyde and Bromine liquid. Secondly, high-temperature viscosity stabilizer PB-854 was synthesized by 5-bromine-3-tert-butyl salicylaldehyde and phloroglucinol[11-13]. The chemical equation is shown below.



## 3. Results and discussion

### 3.1. Synthesis of 5-bromine-3-tert-butyl salicylaldehyde

In the experiment, 9 groups of experiments, 4 factors, and 3 levels of orthogonal experiment for each factor (as shown in Table 1) were adopted.[14] In order to optimize the experiment conditions, the sum of yield was calculated for each factor under three levels, the result is shown in table 2.

**Table 1.** Etherification of orthogonal factor levels.

Level Factor	A molar ratio of raw materials	B temperature/°C	C time/min	D stirring rate/r min <sup>-1</sup>
1	2 : 1 : 2	90	120	200
2	2 : 1 : 2.5	100	190	150
3	1 : 1.5 : 2	120	250	100

**Table 2.** Optimum synthesis conditions of etherification

Molar ratio of raw material	Temperature/°C	Time/min	Stirring rate/r min <sup>-1</sup>
2 : 1 : 2.5	100	190	100

### 3.2. Substitution reaction

In the experiment, 9 groups of experiments, 4 factors, and 3 levels of orthogonal experiment for each factor (as shown in Table 3) were adopted. In order to optimize the etherification synthetic process, [15] the productivity sum of synthetic product was calculated for each factor under three levels. The optimal process combination is presented in Table 4.

**Table 3.** Orthogonal factors of condensation.

Element	A Material ratio	B Reaction temperature /°C	C Reaction time/min	D Sulphuric acid volume fraction/%
1	2 : 1	30	30	98
2	3 : 1	60	60	80
3	4 : 1	90	120	60

Record: material ratio is equal to molar ratio of 4-phenoxybutyl bromide and Phloroglucinol

**Table 4.** Optimum synthesis conditions of etherification reaction.

Material ratio	Reaction temperature/°C	Reaction time/min	Sulphuric acid volume/%
4 : 1	60	30	80

### 3.3. Structural analysis

Sample preparation of dried PB-854 with KBr was tested by America's Nicolet 70SX infrared spectrometer. The spectrogram is shown in Figure 1.

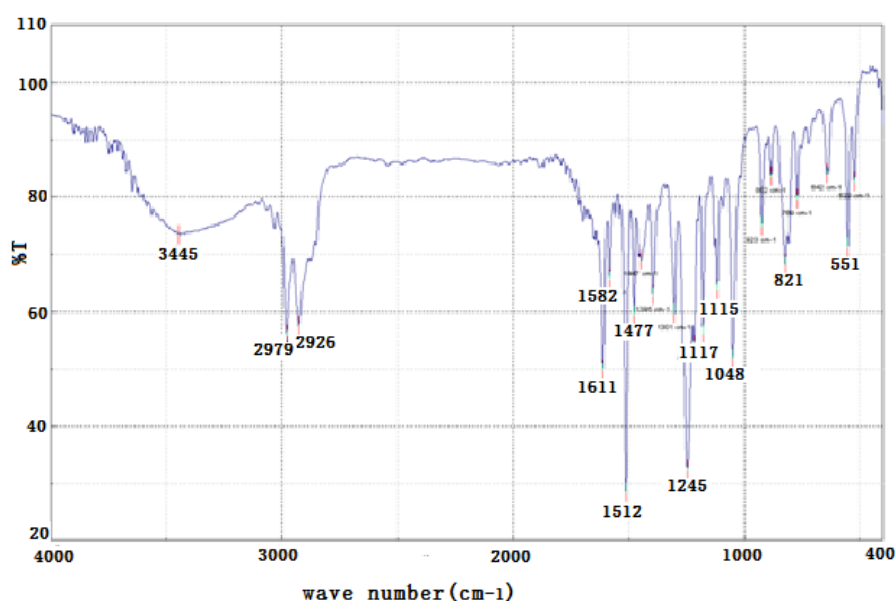


Figure 1 FTIR of PB-854

According to Figure 1, the main absorption peak of PB-854 is as follows:  $1117\text{ cm}^{-1}$  and  $1048\text{ cm}^{-1}$  represent telescopic vibration and anti-telescopic vibration peaks of Ph-O-Ph.

- (2)  $2979\text{ cm}^{-1}$  and  $2926\text{ cm}^{-1}$  are the stretching vibration absorption peaks of  $-\text{CH}_3$ ;
- (3)  $1611\text{ cm}^{-1}$  and  $1512\text{ cm}^{-1}$  are skeletal vibration absorption peaks of benzene ring;
- (4)  $1245\text{ cm}^{-1}$  is the stretching vibration absorption peak of aromatic aldehyde.

#### 3.3.1. Temperature resistance evaluation

For evaluating the high temperature stability of PB-854 used in the drilling fluid, the common polymer drilling fluid system was chosen as performance evaluation of the high temperature rheology and filter loss before and after adding the PB-854. The results of the experiments are listed in Table 5.

**Table 5.** General performance of the polymer drilling fluid

(a. No adding PB-854)

Temperature/°C	Condition (16 h)	AV (mPa s)	PV (mPa s)	YP (Pa)	G''/ G' (Pa/Pa)	API <sub>FL</sub> (ml)	pH
25	before heating	32	23.1	8.9	1.4/3.1	4.5	9
120	after heating	22.4	14.4	8	0.5/2.5	6	9
150	after heating	18.9	13.4	5.5	0.8/1.5	8.1	9
180	after heating	10.2	7.8	2.4	0.5/1.1	11.5	9

(b. Adding 2 %PB-854)

Temperature/°C	Condition (16 h)	AV (mPa s)	PV (mPa s)	YP (Pa)	G''/ G' (Pa/Pa)	API <sub>FL</sub> (ml)	pH
25	before heating	39.4	29.2	10.2	1.5/3.4	4.4	9
120	after heating	35	24.8	10.2	1.4/2.6	4.8	9
150	after heating	30.8	24.4	6.4	1.8/2.0	5.4	9
180	after heating	31.5	26.4	5.1	1.1/1.8	5.1	9

From the Table 5, it can be concluded that high-temperature stability of polymer drilling fluid is poor. When temperature increased from 25 °C to 180 °C, the apparent viscosity went down from 32 mPa s to 10.2 mPa s, and its filter loss increased from 4.5 ml to 11.5 ml. However, after adding 2 % PB-854 into the drilling fluid, the apparent viscosity of drilling fluid basically remained around 31 mPa s and its filter loss just increased a little. Therefore, PB-854 has good rheological property at high temperature. It can protect the polymer in the drilling fluid system from degradation, preserving the high-temperature stability of drilling fluid in that way.

#### 4. Conclusions

By the relation between molecular structure and high-temperature stability, in this paper designed and synthesized high-temperature viscosity stabilizer PB-854, and the following conclusion was obtained based on the experiment:

The etherification and condensation method have been used to synthesize high-temperature viscosity stabilizer PB-854, and carry out optimal experiment for the synthetic method to realize the maximum productivity of PB-854.

The structure of PB-854 was evaluated by infrared spectrum, where we can find the characteristic peaks of the main functional groups in accordance with the designed target product.

By comparing the change of the high-temperature rheological property and filter loss of the polymer drilling fluid before and after adding PB-854, the results show that PB-854 has good high-temperature stability and could effectively protect the polymer at the high-temperature.

List of symbols and abbreviations

AV – apparent viscosity

PV – plastic viscosity

YP – yield value

G'' – gel strength of the stationary after 10 s

G' – gel strength of the stationary after 10 min

API<sub>FL</sub> – medium pressure filtration quantity in accordance with API standard

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