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Laboratory Evaluation and Field Application of a Type of Polyhydric Alcohols Fracturing Fluid

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Abstract: Aimed at the characters of poor fracture conductivity and low natural output of Changqing Oilfield An-83 area, a type of Polyhydric Alcohols (PHA) fracturing fluid with low molecular was researched. Then laboratory evaluation was implemented according to SY/T 5107-2005: Water-based fracturing fluid performance evaluation method. It showed that PHA fracturing fluid had better performances on rheological resistance, proppant-carrying capacity and gel-breaking resistance on the condition of PHA-1 concentration was 3%, crosslinking ratio was 100:3. Average output of the test wells was $18.5 \text{ m}^3 \text{ day}^{-1}$, about twice as much as the average output of An-83 area. Viscosity of gel-breaking fluid of PHA fracturing fluid was below $5 \text{ mPa}\cdot\text{s}$, the average backflow ratio was 66.5%. It proved that PHA fracturing fluid had good adaptability for low permeability reservoirs.

Key words: Fracturing fluid, polyhydric alcohols, low-damage, temperature resistance, backflow performance

INTRODUCTION

Low permeability reservoirs are not only mainly characterized by fewer surface porosities, microscopic throats and high displacement pressure but also contain more interstitial materials than effective development areas, so clay swelling can be easily caused by external liquid (Bohlooli and de Pater, 2006; Parris *et al.*, 2008). To solve the above-mentioned problems, firstly, thorough study on gel-breaking technology is necessary, thus the fracturing fluid will gain better backflow performance. Secondly, intramolecular recombination of the thickeners such as the introduction of low molecular functional groups, can make the thickeners hardly remain in the pores of the reservoirs, so that residue content of the fracturing fluid will be reduced dramatically (Khair *et al.*, 2011; Brannon and Ault, 1991).

An-83 area of Changqing Oilfield, which has the typical characteristics of low permeability reservoirs, is very difficult to be exploited effectively. In view of this, a type of PHA thickener-PHA-1-was adopted. High content of hydroxyls, low molecular weight and a certain amount of hydrophobic groups made PHA-1 have some anti-swelling property and less residue content brought less damage for reservoirs. In addition, hydrophobic groups of PHA-1 had some surface activity. Thus, interfacial tension of the fracturing fluid could be reduced

significantly and backflow performance of the system would be improved effectively (Lea *et al.*, 2012; Zuzaniuk and Prins, 2003; Vollmer and Alleman, 2001).

Laboratory evaluation showed PHA fracturing fluid had advantages of good shear resistance, excellent proppant-carrying capacity, lower interfacial tension and residue content. Meanwhile, field applications showed average output of the test wells which were fractured by PHA fracturing fluid in An-83 area was $18.5 \text{ m}^3 \text{ day}^{-1}$, about twice as much as the average output of An-83 area.

MATERIALS AND METHODS

Materials: Organic boron-titanium crosslinker (BTC-1) was synthesized by Key Laboratory of Auxiliary Chemistry and Technology for Chemical Industry. Polyhydric alcohols (PHA-1, PHA-2, PHA-3, PHA-4) and Sodium hydroxide were provided by Tianjin Hongyan Chemical Reagent Factory. Ammonium persulfate (APS) and polyoxyethylene (PEO) were supplied by Qingyang Changqing Downhole Assistant Company.

Preparation of PHA fracturing fluid: The 1000 mL tap water was put into the blender and 30 g PHA-1 was slowly added into the water on the condition of high-speed stir. After the charging was finished, remained stirring for 1-2 h until the solution became uniformity. Then the

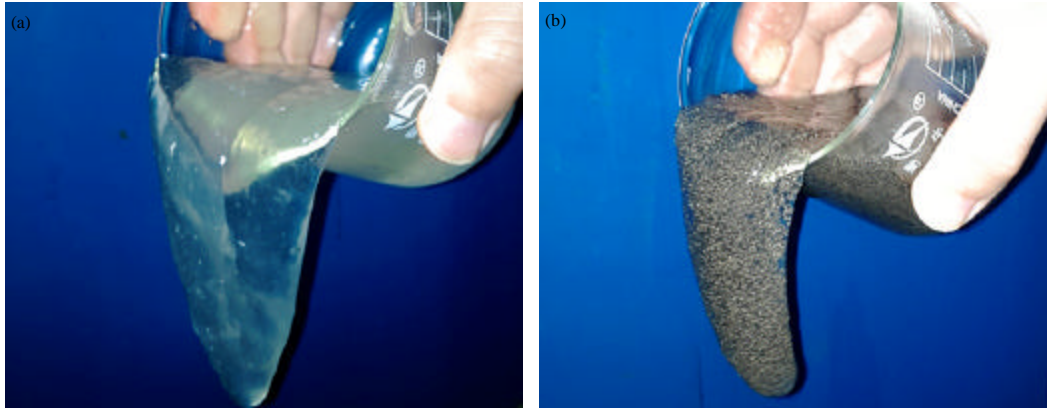


Fig. 1(a-b): (a) Gel shape and (b) proppant-carrying shape of PHA fracturing fluid

solution was immersed into the constant temperature bath (30°C) for 2 h, so that viscosity of the solution would tend to be stabilized.

Then, BTC-1 was added according to the crosslinking ratio 100:3. PHA fracturing fluid was prepared after stirring well, as shown in Fig. 1.

Characterization: Performance evaluation of PHA fracturing fluid was according to SY/T 5107-2005: Water-based fracturing fluid performance evaluation method. An NDJ28S rotary viscometer (Shanghai Analytical Instrument Factory, China) was used to measure viscosity of the fracturing fluid. An AR2000ex coaxial cylinder rotary rheometer (TA Instruments Company, U.S.) was applied to rheological behavior analysis. The measuring conditions were as follows: prepared fracturing fluid sample was put into specimen cup of AR2000ex coaxial cylinder rotary rheometer, started experimenting from 30°C, with shear rate 170 sec^{-1} , controlled heating rate of $3 \pm 0.2^\circ\text{C min}^{-1}$, remained shearing the fracturing fluid sample until the gel viscosity or the shear time reached a specified value.

Static suspended sand method was used to analyze proppant-carrying capacity: Prepared fracturing fluid sample was put into a 250 mL graduated cylinder, then the system was heated to allotted temperatures. Added proppant in different sand ratios, then elapsed time that proppant subsided 20 cm at different temperatures were recorded, thus sedimentation rate could be calculated.

Viscosity of the gel-breaking fluid was measured by NDJ28S rotary viscosity, prepared fracturing fluid sample was placed in Waring blender (Fann Instrument Company, U.S.), then the system was heated to the reservoir temperature, viscosity of the supernatant was measured

at this temperature. A BZY-1 automatic surface tension meter (Shanghai Hengping Instrument Factory, China) was used for measuring surface tension of the gel-breaking fluid. Interface tension analysis of the gel-breaking fluid was performed in a TEXAS-500 rotary drop interfacial tension meter (Temco Instrument Company, U.S.). An 80-2 centrifuge (Shanghai Medical Instruments Ltd., Corp, China) was employed for residue content test. The experimental procedure was as follows: 50 g prepared gel-breaking fluid sample was put into centrifuge tube of 80-2 centrifuge, after centrifuging for 30 min at the rate of 3000 r min^{-1} , the supernatant was removed. Then the centrifuge tube was put into the oven, dried to constant weight. The residue content was calculated by the equation:

$$\text{Residue content} = m/V \times 100\%$$

The dried residue was weighed and expressed with m and prepared gel-breaking fluid sample was measured and expressed with V .

RESEARCH ON FORMULA OF PHA FRACTURING FLUID

Screening of thickeners: Basic principle of the screening was devoted to decreasing molecular weight and controlling alcoholysis degree of the thickeners.

Lower molecular weight and shorter chain length can reduce the gel strength, so as to achieve the purposes of reducing initial viscosity of the gel and decreasing damage to the equipments (Colinet *et al.*, 2009). Meanwhile, water-solubility of the thickeners can be improved by controlling the alcoholysis degree, which will help raise the labor efficiency.

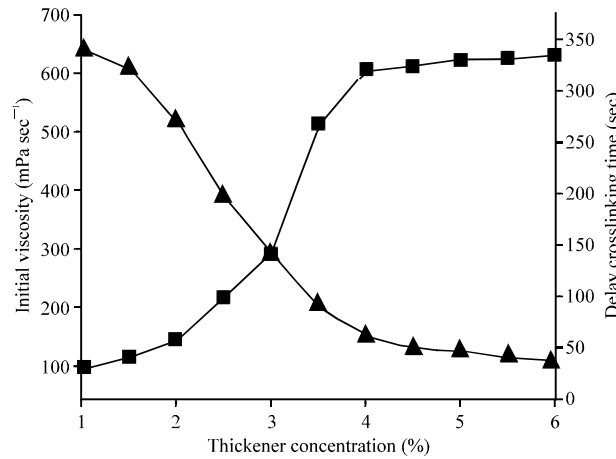


Fig. 2: Effects of PHA-1 concentration on delay crosslinking time and initial viscosity

Table 1: Effects of Mw and alcoholysis degree on the gel strength

Thickeners	Mw	Alcoholysis degree (%)	Gel strength
PHA-1	8000~12000	80~90	low
PHA-2	27000~32000	90~97	low
PHA-3	70000~80000	91~98	medium
PHA-4	110000~120000	98~99	high

Table 2: Formula of PHA fracturing fluid

Reagents	Components	Mass fraction (%)
Thickener	PHA-1	3.0
Crosslinker	BTC-1	3.0
Drag reducers	PEO	0.01
pH modifier	40% NaOH solution	0.8
Gel breaker	APS	1.0~1.5

Table 1 showed the relationships among molecular weight, alcoholysis degree and gel strength of the four thickeners PHA-1, PHA-2, PHA-3 and PHA-4. As presented in Table 1, the gel strength reduced but the solution rate increased with the decrease of molecular weight and alcoholysis degree. PHA-1 was eventually identified as the thickener of PHA fracturing fluid.

Effects of PHA-1 concentration on delay crosslinking time and initial viscosity have been studied on the condition of pH value 8~9, crosslinking ratio 100:2~4, the experimental results shown in Fig. 2. Seen from Fig. 2, when the crosslinking ratio and pH value remained unchanged, with increasing concentration of PHA-1, initial viscosity of the gel increased slowly but its delay crosslinking time decreased slowly. When the concentration of PHA-1 was 4~5%, the two performances tended to be stabilized.

When the concentration of PHA-1 was 3%, the gel had suitable delay crosslinking time and initial viscosity, therefore PHA-1 concentration was determined as 3%.

Screening of crosslinkers: BTC-1 was chosen as the crosslinker for PHA fracturing fluid. Bond energy of the coordination bond that formed by boron ion and hydroxyl is smaller, thus the organic boron crosslinker has better shear resistance but weak temperature resistance.

Instead, bond energy of the coordination bond that formed by titanium ion and hydroxyl is larger, thus the organic titanium crosslinker has better temperature resistance but weak shear resistance (Hou *et al.*, 2013).

BTC-1 was based on crosslinking mechanism of the two crosslinkers, made the boron particles combine more closely. Figure 3 showed the effects of crosslinking ratio on delay crosslinking time and initial viscosity.

As shown in Fig. 3, when PHA-1 concentration and pH value remained unchanged, with increasing crosslinking ratio, initial viscosity of the gel increased slowly until the crosslinking ratio achieved 4~5%, then initial viscosity of the gel began to decrease rapidly. The main reason for this phenomenon was that excess crosslinker made the system occur excessive crosslinking behavior (Dawson *et al.*, 1998). In this case, initial viscosity of the gel would be reduced; even the gel would be broken. Delay crosslinking time was gradually shortened with increasing crosslinking ratios. When the crosslinking ratio was between 2~4%, the gel had suitable delay crosslinking time and initial viscosity. After a systematic consideration, crosslinking ratio was determined as 100:3. It was found that PHA fracturing fluid had good performances on delay crosslinking time and initial viscosity when PHA-1 concentration was 3%, crosslinking ratio was 100:3. Formula of PHA fracturing fluid was appeared in Table 2 (main components).

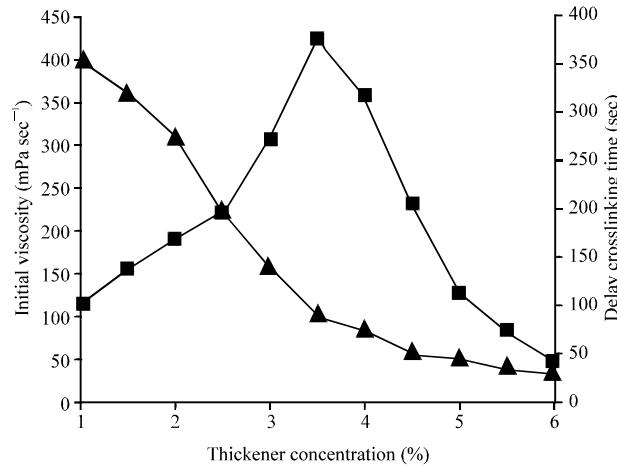


Fig. 3: Effect of crosslinking ratio on delay crosslinking time and initial viscosity

LABORATORY EVALUATION OF PHA FRACTURING FLUID

Temperature resistance: Prepared fracturing fluid sample was put into specimen cup of AR2000ex coaxial cylinder rotary rheometer, started experimenting from 30°C, with shear rate 170 sec⁻¹, controlled heating rate of 3±0.2°C min⁻¹, remained shearing the fracturing fluid sample until the gel viscosity dived to 50 mPa sec⁻¹. The corresponding temperature could characterize temperature resistance of PHA fracturing fluid (Wei *et al.*, 2011).

Figure 4 was viscosity-temperature curve of PHA fracturing fluid. As shown in Fig. 4, viscosity of PHA fracturing fluid was 100 mPa sec⁻¹ at 100°C. It proved that temperature resistance of PHA fracture fluid exceed 100°C.

Shear resistance: Prepared fracturing fluid sample was put into specimen cup of AR2000ex coaxial cylinder rotary rheometer, then started experimenting from 30°C, with shear rate 170 sec⁻¹, controlled heating rate of 3±0.2°C min⁻¹. After the temperature reached 100°C, remained temperature and shear rate unchanged, continued shearing the gel within allotted time. Figure 5 was viscosity-time curve of PHA fracturing fluid. Seen from Fig. 5, after continuous shearing for 3600 sec, fracturing fluid viscosity was about 100 mPa sec⁻¹. It explained PHA fracturing fluid had a good shear resistance.

Proppant-carrying capacity: Good proppant-carrying capacity can improve sand ratio of the fracturing fluid, avoid sand plug, sand jamming and other accidents. Sedimentation rate of the proppant inside the fracturing

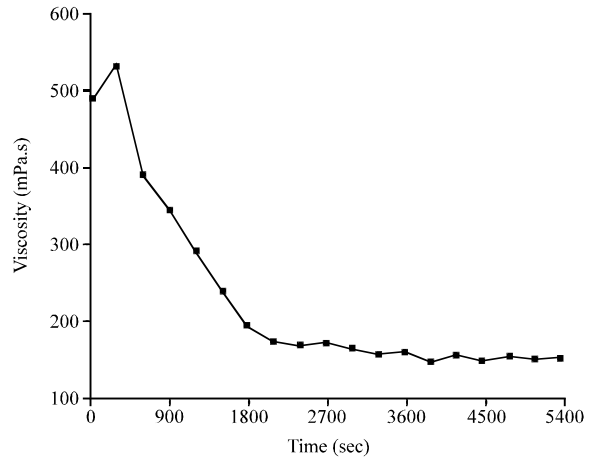


Fig. 4: Viscosity-temperature curve of PHA fracturing fluid

Table 3: Proppant-carrying capacity of PHA fracturing fluid

Sand ratio (%)	Sedimentation rate (mm s ⁻¹)		
	60°C	70°C	80°C
20	0.5043	0.5315	0.5524
30	0.5316	0.6013	0.6661
40	0.5518	0.7012	0.7825

fluid was used for characterizing proppant-carrying capacity of the fracturing fluid (Weaver *et al.*, 2002).

Table 3 showed that when the sand ratio was 40%, sedimentation rate of the proppant at 60, 70 and 80°C were 0.5518, 0.7012 and 0.7825 mm•sec⁻¹, which could commendably satisfy the operation requests.

Gel-breaking performance: In order to improve the gel-breaking performance and reduce damage to the reservoirs, fracturing fluid should achieve quick and thorough gel-breaking (Peles *et al.*, 2002). APS was used as gel breaker for PHA fracturing fluid, gel-breaking

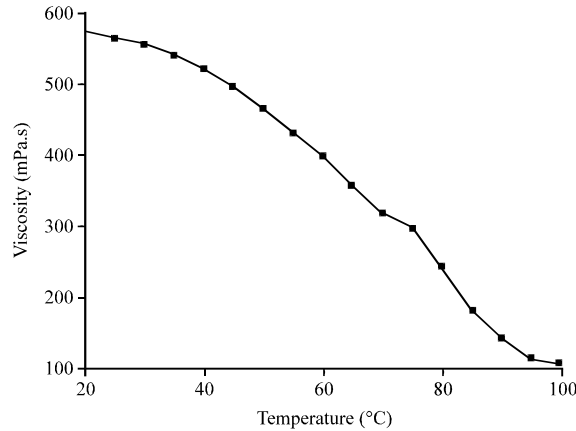


Fig. 5: Viscosity-time curve of PHA fracturing fluid

Table 4: Gel-breaking performance of PHA fracturing fluid

Temperature (°C)	Gel breaker dosage (%)	Gel-breaking time (h)	Gel-breaking liquid viscosity (mPa sec ⁻¹)
60	0.8	120	5.4
	1.0	60	5.2
	1.2	40	5.0
	1.5	20	4.8
70	0.8	100	5.0
	1.0	80	4.6
	1.2	45	4.4
	1.5	20	4.2
80	0.8	75	5.0
	1.0	65	4.8
	1.2	30	4.8
	1.5	18	4.8

Table 5: Performance compared between guar gum and PHA fracturing fluid

Thickeners	Surface tension (mN m ⁻¹)	Interface tension (mN m ⁻¹)	Apparent viscosity (mPa sec ⁻¹)	Residue content (mg L ⁻¹)
Guar gum	34.82	1.28	<8.5	165
PHA-1	22.52	0.83	<5.5	15

experiments were based on the conditions of onsite construction at 60, 70 and 80°C, the experimental results shown in Table 4.

Seen from Table 4, viscosity and gel-breaking time reduced with increasing temperatures and APS dosages. Maximum viscosity of gel-breaking fluid was 5.4 mPa sec⁻¹, which was far less than gel-breaking fluid of guar gum fracturing fluid (8.5 mPa sec⁻¹).

Residue content: Fracturing fluid residue mainly includes solid impurities, water insoluble of thickener and water insoluble formed by material inside the reservoir and fracturing fluid. The residue will not only block pores of the reservoirs but also reduce fracture conductivity, so as to reduce outputs of oilfields greatly (Walters *et al.*, 2009). Table 5 compared gel-breaking performance between guar gum and PHA fracturing fluid. As shown in Table 5, surface tension and interface tension of gel-breaking fluid

of PHA fracturing fluid were 22.52 and 0.83 mN m⁻¹, which were far less than gel-breaking fluid of guar gum fracturing fluid. It meant that PHA fracturing fluid had better backflow performance. Meanwhile, gel-breaking fluid of PHA fracturing fluid didn't contain water insoluble and its residue content was very little (15 mg L⁻¹), so that pores of the reservoir won't be blocked easily.

Field application: So far, PHA fracturing fluid has been used in 12 test wells of An-83 area. Overall, the construction effect was excellent, the application results shown in Tab. 6. As shown in Tab. 6, average output of the test wells was 18.5 m³ day⁻¹, which was about twice as much as the average output of An-83 area. After back flowing for 40 min, viscosity of gel-breaking fluid of PHA fracturing fluid was below 5mPa•s, the average backflow ratio was 66.5%.

Table 6: Outputs of PHA fracturing fluid

No. of Well	Reservoirs	Effective thickness (m)	Outputs (m ³ /d)	Backflow rate (%)
1	Chang 7 ₂	17.4	12.7	56.5
2	Chang 7 ₂	15.2	23.7	60.7
3	Chang 7 ₂	21.1	8.9	90.4
4	Chang 7 ₂	22.5	11.0	47.2
5	Chang 7 ₂	16.9	3.1	59.5
6	Chang 7 ₂	19.7	7.9	60.1
7	Chang 7 ₂	8.5	19.9	53.8
8	Chang 7 ₂	19.2	20.6	66.1
9	Chang 7 ₂	17.6	15.8	61.6
10	Chang 7 ₂	21.0	19.5	54.1
11	Chang 7 ₂	8.9	20.6	58.0
12	Chang 7 ₂	17.2	16.5	94.0
Average of test wells(12)		16.2	18.1	66.5
Average of contrast wells(23)		12.8	9.0	57.5
Average of the area(103)		20.2	12.2	62.3

CONCLUSION

It was found that PHA fracturing fluid had good performances on delay crosslinking time and initial viscosity on the condition of PHA-1 concentration was 3%, crosslinking ratio was 100:3. Then formula of PHA fracturing fluid was determined.

Laboratory Evaluation showed that PHA fracturing fluid had better performances on shear resistance and proppant-carrying capacity. Temperature resistance of PHA fracture fluid was over 100°C. Maximum viscosity of gel-breaking fluid was 5.4 mPa sec⁻¹, which was far less than viscosity of gel-breaking fluid of guar gum (8.5 mPa s⁻¹). Surface tension and interface tension of gel-breaking fluid of PHA fracturing fluid were 22.52 and 0.83 mN m⁻¹, which were also far less than gel-breaking fluid of guar gum fracturing fluid. Residue content of PHA fracturing fluid was 15 mg L⁻¹, so that the system had low damage to the reservoirs.

Average output of the test wells was 18.5 m³ day⁻¹, which was about twice as much as the average output of An-83 area. After back flowing for 40 min, viscosity of gel-breaking fluid of PHA fracturing fluid was below 5 mPa s⁻¹, the average backflow ratio was 66.5%. It proved that PHA fracturing fluid could satisfy construction requirements on the condition of different well depths and temperatures.

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