

Dynamic and static gelation behavior of phenol formaldehyde resin gel system in ampoule bottles and porous media

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Abstract. The dynamic and static gelation process in porous media of the phenol formaldehyde resin gel was studied for successful application in conformance control and water shutoff. Shear rate was the most important factor affecting the dynamic gelation in porous media. Compare to static gelation in ampoule bottles and in porous media, the dynamic gelation time in porous media was extended by 2–6 times. The ability of migration and plugging after dynamic gelation was controlled by system concentration. The porous media permeability was the key factor for dynamic gelling time and strength. The quantitative relationship equation was established between dynamic gelling strength and permeability to choose the appropriate permeability for polymer gel treatment.

1 Introduction

The proven technology of water shutoff and conformance control used polymer gel has been widely introduced to block the high permeable zone, realize in-depth flow diversion and enhance sweep efficiency (James *et al.*, 2003; Mercado *et al.*, 2009; Ojukwu *et al.*, 2007; Wang *et al.*, 2019; Zhou *et al.*, 2020). Several kinds of polymer gels have been used in oilfield, including inorganic metal ions gel system (McCool *et al.*, 2009), Phenol Formaldehyde Resin (PFR) gel system (Banerjee *et al.*, 2006), polyethyleneimine gel system (Al-Muntasheri *et al.*, 2009) and so on. Among these gel systems, due to the controllable gelation time and stable plugging ability, the PFR gel is widely used in conformance control and water plugging operations in oil fields.

There have been many researches on the static gelation of PFR gel in ampoule bottles and porous media. Seright and Martin (1993) and Bryant *et al.* (1997) studied the impact of pH, rock permeability and lithology on the performance of PFR gel. Bryant *et al.* (1998) studied the injectivity of PFR gelant in porous media. Albonico *et al.* (1995) studied the effects of pH, temperature, concentrations of polymer and crosslinker on gelation, injectivity of gelant and thermal stability in bulk and in porous media. Herbas *et al.* (2004) and Banerjee *et al.* (2006) studied the reaction rate of PFR gel and effects of pH and temperature on reaction rate.

All the researches confirmed that PFR gelant cross linked over a wide range of pH and insensitive to lithology, but strongly influenced by the temperature and the types of polymer. Dynamic gelling behavior has been extensively studied in recent years (El-karsani *et al.*, 2013; He *et al.*, 2015; Qin *et al.*, 2017). Yu *et al.* (2015) studied the dynamic gelation of HPAM/Cr(III) under shear in an agitator and porous media, which revealed the gelling behavior of chromium gel under shear condition. Chromium gel could not migrate in porous media because of its short gelation time.

Polymer hydrogels need to have good injectivity, low adsorption of their components in the porous medium and good propagation and programmable gelation time (Tessarolli *et al.*, 2014). Because of long gelation time, PFR gel could be used conveniently in a range of in-depth treatments in matrix as well as in fractured formations. To verify the applicability of hydrogel in conformance control, it is important to evaluate the gelling behaviors, such as: gelation time, gel strength and stability (de Aguiar *et al.*, 2020). The bulk gelation time was an important influence to choose pumping time. And the PFR gel had good thermal stability and plugging ability for the intended lifetime of the treatment under the reservoir conditions.

But it is lack of research on PFR gel performance after dynamic gelation in porous media, such as the relationship between static gelation time in bulk and dynamic gelation time in porous media. Based on system of HPAM/Phenol Formaldehyde Resin (PFR), the purpose of this work was to obtain the relationship of dynamic gelation in porous

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media and static gelation in ampoule bottle and porous media. The influence factors of dynamic gelling in porous media were studied. The method was established to select permeability for dynamic gelation of PFR gelant in porous media.

2 Materials and methods

2.1 Materials

The polymer employed in these researches was classical partially Hydrolyzed PolyAcrylaMide (HPAM), whose molecular weight is 1.2×10^7 and the degree of hydrolysis is 22%. The crosslinker named Phenol Formaldehyde Resin (PFR) is synthesized by phenol and formaldehyde under alkaline condition, whose effective content is 30.53%. The used Synthetic Water (SW) contained 6921 ppm Na^+ , 412 ppm Ca^{2+} , 148 ppm Mg^{2+} and 11 853 ppm Cl^- , which represents the salinity of formation water in Bohai oilfield.

The experiments of core flooding were operated by the circulating device with sand packs with 2.50 cm ID and 10.00 cm length or 100.00 cm length, whose were fitted by clean micro glass beads with different mesh counts and saturated by SW. The mesh counts include three specifications of 80–120 mesh, 140–180 mesh and less than 200 mesh. The circulating device is comprised of two piston containers at room temperature and a sand pack in 75 °C calorstat. And all of the gelation experiments were performed under 75 °C.

2.2 Methods

2.2.1 Static gelation in ampoule bottles

HPAM solutions of different concentrations were prepared with SW as gelants, and then the crosslinkers of different concentrations were added. 15 mL gelant was placed into an ampoule bottle made of thermal resistant glass. The bottle has a screw cap, and it is 10 cm long with ID of 2 cm. The air head space above the solution in the ampoule was purged with nitrogen to remove any oxygen that could cause gel degradation. A series of samples of each formula were prepared for the following static gelation experiments.

2.2.2 Static gelation in porous media

Prepare a series of sand packs with 2.50 cm ID and 10.00 cm length. After being saturated by SW, calculated Porous Volumes (PV) and permeabilities (K). The sand pack was injected in one PV gelant with 1 mL/min. Then sealed sand packs were placed at 75 °C calorstat. At regular intervals the pressure gradient breakthrough of one sand pack was measured by SW flooding at the speed of 1 mL/min.

2.2.3 Dynamic gelation in porous media

After 30 days at room temperature, the viscosity of gelant included 0.2 wt% HPAM and 0.6% PFR was 10.9 mPa s, whose initial viscosity was 8.4 mPa s. So dynamic gelation of HPAM/PFR gel in porous media could be estimated

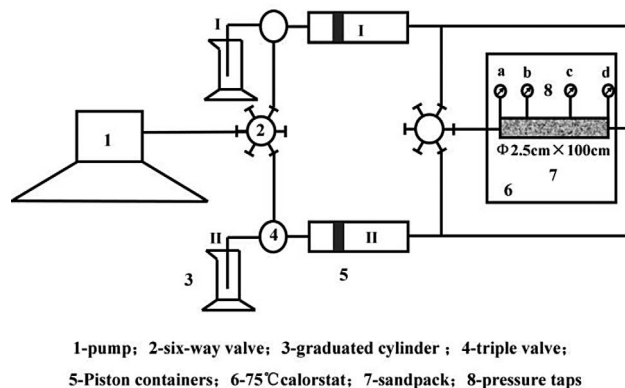


Fig. 1. Schematic of dynamic gelling process.

with the circulating device, which was comprised of two piston containers at room temperature and a sand pack in 75 °C calorstat. First, one PV gelant in container I was injected into the sand pack. Then connect the outlet of sand pack with container II and adjust temperature to 75 °C. One PV gelant in container II was injected into sand pack and initial one PV gelant in sand pack was displaced into container I again. By adjusting the valves, the one PV gelant in container I was injected into sand pack, and the one PV gelant in sand pack was displaced into container II again. The two PV gelants were alternately injected into the sand pack with 2.50 cm ID and 100.00 cm length. There are two internal pressure taps 30 and 70 cm from the inlet, shown in Figure 1. In the whole process, record the change of injected pressure drops with time. The parameters for flow experiments of gelant in porous media were listed in Table 1.

3 Experimental results

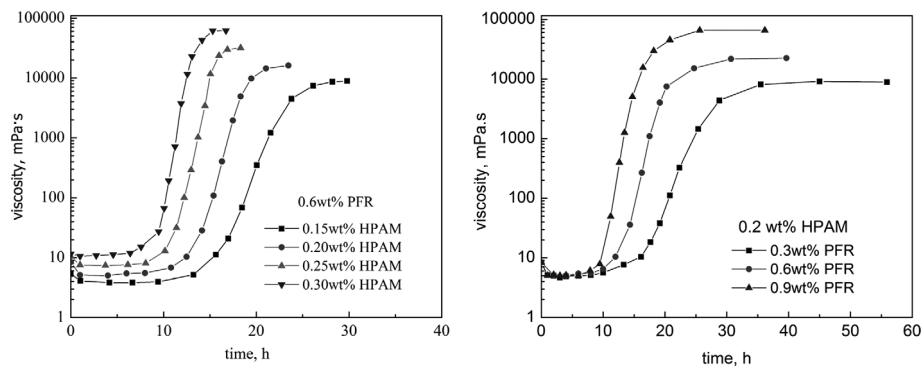
3.1 Static gelation in ampoule bottles and sand pack

Gelation time is one of the most important performances. The method of measuring viscosity versus time was used to determine the gelation time, which was divided into Initial Gelation Time (IGT) and Final Gelation Time (FGT). IGT is the time of sudden increase in viscosity. FGT is the time of nearly finish reaction (Mokhtari and Ozbayoglu, 2010). The results of gelation time of different concentrations were shown in Figure 2. The process of gelation in ampoule bottles was divided into slow induction, sudden increase and stability. The data of gelation time in ampoule bottles was shown in Table 2. Gelation time was shortened with increasing concentrations.

The static gelation time in sand pack was determined with the method of change of Residual Resistance Factor (RRF) with time (Vasquez et al., 2005). The increase of following water flooding pressure mainly resulted from plugging by crosslinked gelant. And the RRF increased with the degree of crosslinking. When the RRF was constant, the crosslinking reaction was completed. The IGT was the time of water flooding pressure sudden increase. And the FGT was the first time when the RRF remained

Table 1. Parameters for flow experiments.

Code	HPAM, wt%	PFR, wt%	Permeability, μm^2	Injected speed, mL/min
1	0.2	0.3	7.07	0.5
2	0.2	0.9	8.48	0.5
3	0.15	0.6	7.22	0.5
4	0.2	0.6	8.08	0.5
5	0.25	0.6	8.28	0.5
6	0.3	0.6	8.99	0.5
7	0.2	0.6	13.58	0.5
8	0.2	0.6	8.08	0.5
9	0.2	0.6	2.13	0.5
10	0.2	0.6	0.55	0.5

**Fig. 2.** The change of viscosity with time under static gelling in ampoule bottles at 75 °C.**Table 2.** Static gelation time in ampoule bottles and porous media under 75 °C.

Code	HPAM, wt%	PFR, wt%	Static gelation in ampoule bottles		Static gelation in porous media	
			IGT, h	FGT, h	IGT, h	FGT, h
1	0.15	0.6	14	27	25	45
2	0.2	0.6	12	21	17	40
3	0.25	0.6	9	16.5	10	29
4	0.3	0.6	7	14.4	8	23
5	0.2	0.3	14.3	30	20	45
6	0.2	0.9	9.5	18	10	35

unchanged. The static gelation time in sand packs was listed in Figure 3 and Table 2. The process of static gelation in sand packs was similar to that in ampoule bottles. And with concentration of polymer and crosslinker increasing, the gelation time decreased.

3.2 Dynamic gelling behavior in porous media

The results of dynamic gelation under different concentrations were listed in Figure 4. The process of dynamic gelling behavior in porous media was similar to that of static

gelation in porous media. In the step of slow induction, it needed a certain pressure difference due to viscoelasticity of polymer molecules when polymer deformed distinctly and passed through the pore throat, whose size was smaller than polymer molecules (Delshad *et al.*, 2008). And independent aggregates, whose apparent viscosity was low, were formed by polymer molecules crosslinked with PFR (Chauveteau *et al.*, 1999). So the pressure difference remained unchanged. There existed two acting forces in the sudden increase step, crosslinking reaction and shear degradation. When cohesion among aggregates was beyond

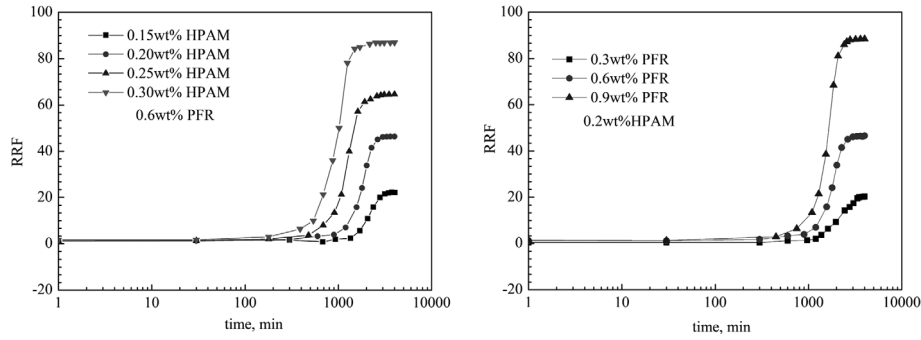


Fig. 3. The change of RRF with time in the process of static gelling in sandpacks.

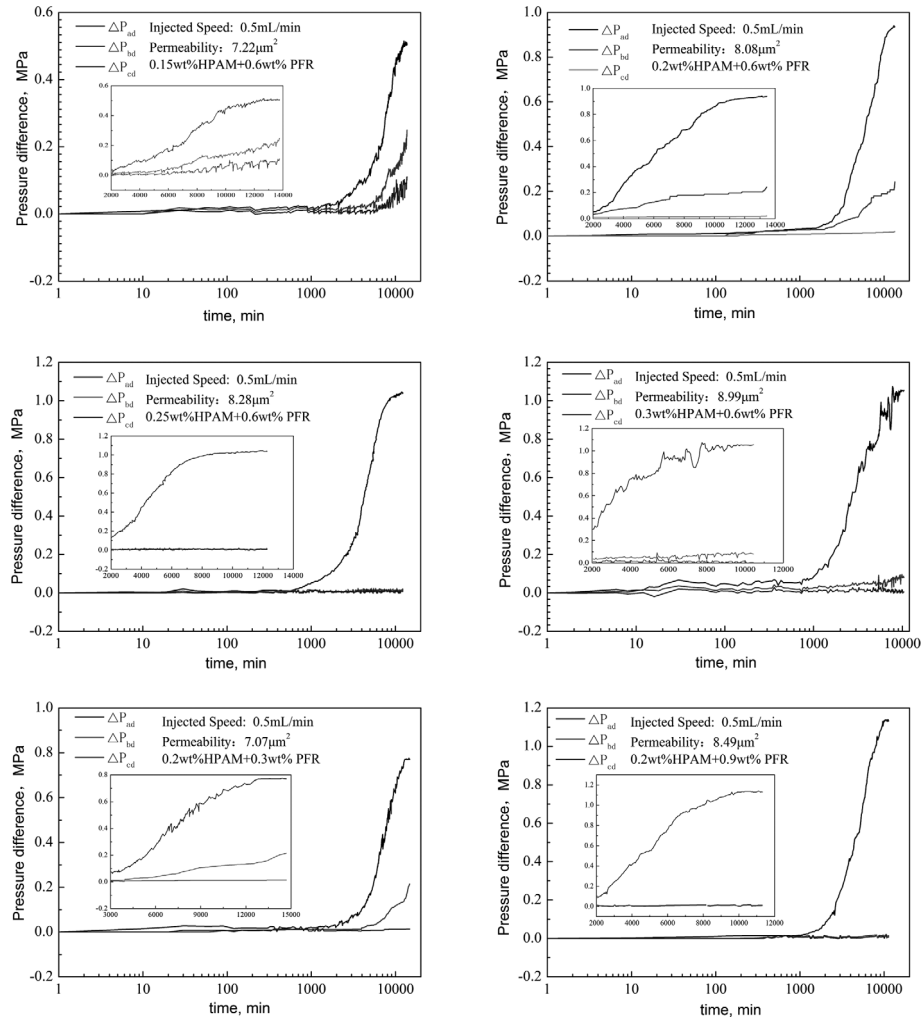


Fig. 4. The change of pressure difference with time in the process of dynamic gelling under different concentrations.

shear stress, the size of polymer gel was limited (McCool *et al.*, 1991). Therefore, the dispersed gel particles formed in the process of dynamic gelation in porous media (Huang *et al.*, 1986; Marty *et al.*, 1991), not the bulk gel. And it was confirmed by the production after dynamic gelation, shown in Table 3. The viscosity of production under different concentrations was lower than that of initial gelant. The

formed dispersed gel particles were held up in porous media by adsorption or bridging. The water bounded by three dimensional network structures became free and flowed from sand pack. The dynamic gelation times were listed in Table 4. The gelation time decreased with concentrations of polymer and crosslinker increasing, as well as the stable pressure difference ΔP_{ad} . When the permeabilities were in

Table 3. Viscosity of production in the processes of dynamic gelation in porous media under different concentrations and permeabilities.

Code	HPAM, wt%	PFR, wt%	Permeability, μm^2	Injected speed, mL/min	Viscosity of production*, mPa s
1	0.2	0.3	7.07	0.5	5.7
2	0.2	0.9	8.48	0.5	1.6
3	0.15	0.6	7.22	0.5	2.1
4	0.2	0.6	8.08	0.5	3.7
5	0.25	0.6	8.28	0.5	6.3
6	0.3	0.6	8.99	0.5	4.2
7	0.2	0.6	13.58	0.5	3.7
8	0.2	0.6	8.08	0.5	3.7
9	0.2	0.6	2.13	0.5	3.2
10	0.2	0.6	0.55	0.5	3.9

* Viscosity of production measured by viscometer was represented of production flowed from sandpack.

Table 4. Dynamic gelation times in the processes of dynamic gelling in porous media under different concentrations and permeabilities

Code	HPAM, wt%	PFR, wt%	Permeability, μm^2	Injected speed, mL/min	Dynamic gelation time, h	
					IGT	FGT
1	0.2	0.3	7.07	0.5	40	106
2	0.2	0.9	8.48	0.5	25	80
3	0.15	0.6	7.22	0.5	20	105
4	0.2	0.6	8.08	0.5	14	95
5	0.25	0.6	8.28	0.5	35	80
6	0.3	0.6	8.99	0.5	20	46
7	0.2	0.6	13.58	0.5	25	94
8	0.2	0.6	8.08	0.5	23	95
9	0.2	0.6	2.13	0.5	21	93
10	0.2	0.6	0.55	0.5	19	95

the range of 7 ~ 9 μm^2 , the change of pressure was influenced by concentrations of polymer and crosslinker. The pressure difference ΔP_{bd} had no change when the polymer concentration was beyond 0.25 wt% and crosslinker concentration was bigger than 0.6 wt%. The increase of pressure difference ΔP_{bd} shown that polymer gel could be migrated into in-depth of sand pack. So the ability of in-depth diversion of polymer gel was limited by the concentration of themselves at a certain permeability.

3.3 Dynamic gelling behavior under different reservoir permeabilities

The results of dynamic gelling behavior of 0.2 wt% HPAM and 0.6 wt% PFR in porous media under different permeabilities were listed in Figure 5. In the process of dynamic gelation of HPAM/PFR, two PV gelants were injected into sand pack alternately. When one PV gelant was injected into sand pack at 75 °C, the other stayed in container at

room temperature. So dynamic gelation time was half of the time of whole flow experiment. And the dynamic gelation times of different permeabilities were shown in Table 4. The permeabilities of sand packs varied from 0.55 to 13.58 μm^2 . With permeability increasing, the size of pore throat was enlarged and the resistance on growth of polymer gel aggregates decreased. The dynamic initial gelation time prolonged slightly with increasing permeability while dynamic final gelation time of HPAM/PFR varied inconspicuously. In the process of dynamic gelation, the stable pressure difference ΔP_{ad} increased with decreasing permeability.

3.4 Water flooding after dynamic gelation in porous media

After dynamic gelation, the following water flooding was conducted to evaluate the plugging of polymer gel, shown in Figure 6. With Pore Volume (PV) increasing,

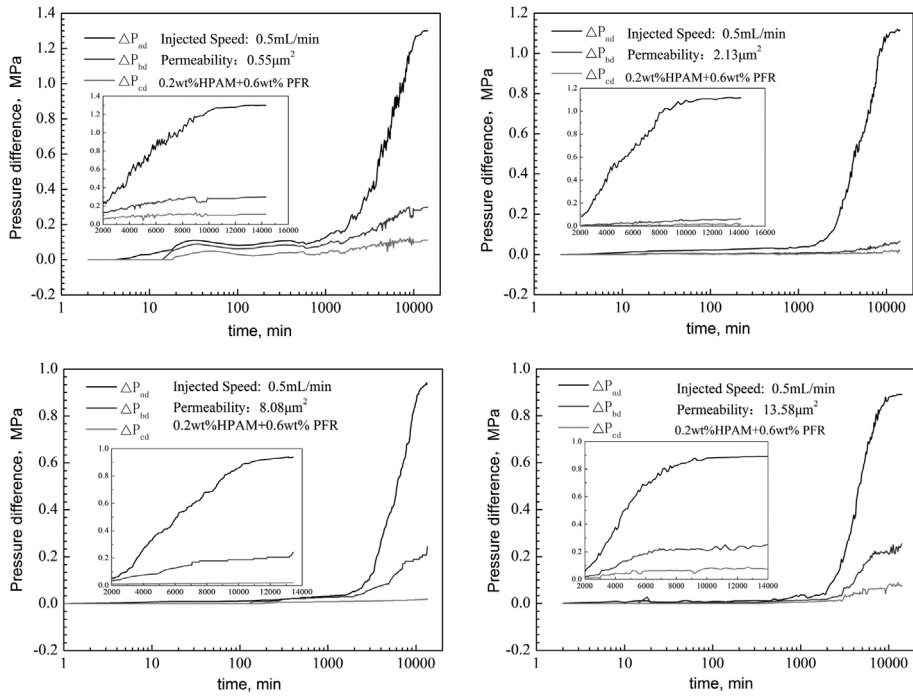


Fig. 5. The change of pressure difference with time in the process of dynamic gelling under different permeabilities.

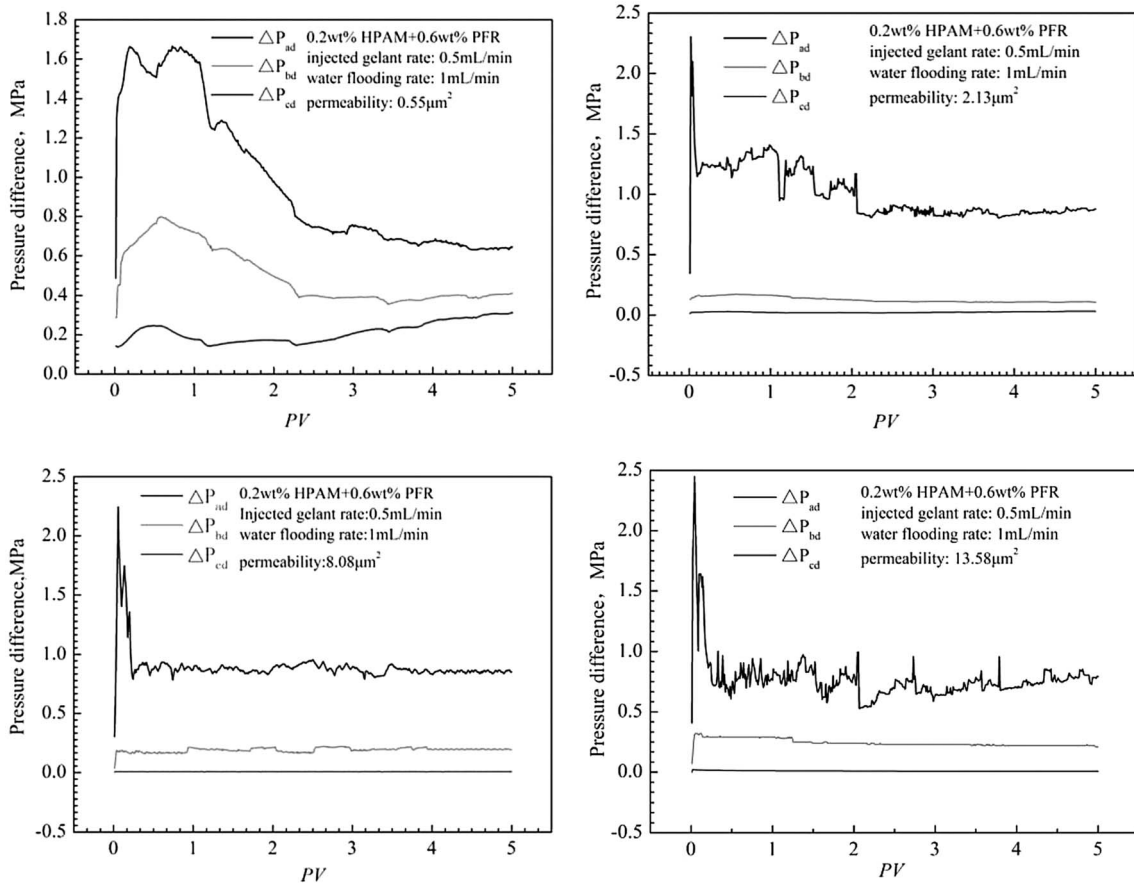


Fig. 6. The change of pressure difference with PV in the process of water flooding after dynamic gelling.

Table 5. Shear rates of PFR gel in the processes of dynamic gelling in porous media under different concentrations and permeabilities.

Code	HPAM, wt%	PFR, wt%	K, μm^2	V, mL/min	Φ	n	C'	γ , s^{-1}
1	0.2	0.3	7.07	0.5	0.347	0.57	2.29	4.25
2	0.2	0.9	8.48	0.5	0.359	0.44	2.29	3.63
3	0.15	0.6	7.22	0.5	0.349	0.64	2.29	4.35
4	0.2	0.6	8.08	0.5	0.359	0.44	2.29	3.71
5	0.25	0.6	8.28	0.5	0.361	0.43	2.29	3.64
6	0.3	0.6	8.99	0.5	0.366	0.35	2.29	3.31
7	0.2	0.6	13.58	0.5	0.379	0.440	2.29	2.86
8	0.2	0.6	8.08	0.5	0.359	0.440	2.29	3.71
9	0.2	0.6	2.13	0.5	0.341	0.440	2.29	7.23
10	0.2	0.6	0.55	0.5	0.338	0.440	2.29	14.22

the pressure difference ΔP_{ad} increased sharply to maximum and then decreased to stability. When the permeability was beyond $2.13 \mu\text{m}^2$, the stable pressure difference ΔP_{ad} was similar, but bigger than that of when the permeability was $0.55 \mu\text{m}^2$. It indicated the polymer gel could block off porous media after dynamic gelation. And the pressure difference ΔP_{ad} was much bigger than ΔP_{bd} , which showed that the main plugging section was the inlet of sand pack. The pressure difference ΔP_{bd} was represented of the ability of in-depth flow diversion of polymer gel. In the range of $0.55 \sim 13.58 \mu\text{m}^2$, all of the pressure difference ΔP_{bd} could reach a certain value.

4 Discussion

4.1 The relationship between static gelation time and dynamic gelation time

The gelation time is one of the most important performances. It is very crucial to establish the quantitative relationship between static gelation time and dynamic gelation time. Contrast static gelation in ampoule bottles and in porous media, the initial gelation time and final gelation time in porous media was 1 ~ 1.5 times and 1.5 ~ 2 times of that in ampoule bottles separately. It might result from adsorption and retention of polymer and crosslinker molecules in porous media. When dynamic gelation in porous media, the HPAM/PFR gelant suffered from the shear of porous media. And the shear rate was calculated by equation (1) (Savins, 1969; Hirasaki and Pope, 1974; Camilleri *et al.*, 1987; Fabbri *et al.*, 2020). The shear rates under different concentrations and permeabilities were listed in Table 5:

$$\gamma = \frac{3n+1}{n} \cdot \frac{v}{\sqrt[0.5]{8C'K\phi}} \quad (1)$$

Equation (1) solved the shear rate in porous media. γ – shear rate, s^{-1} ; n – Rheological index, MPa s^n ; V – injected speed, mL/min; C' – tortuosity, decimals; K – permeability, μm^2 ; Φ – porosity, decimals. Contrast static gelation and dynamic gelation, the initial dynamic gelation time in porous media was 2 ~ 2.5 times and 1.5 ~ 2 times of initial

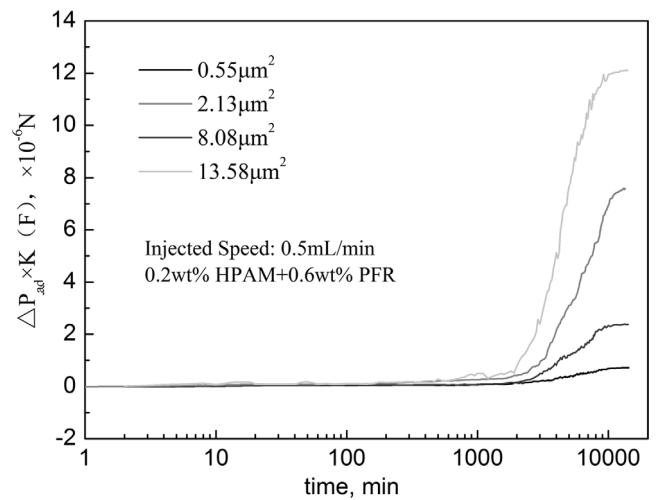
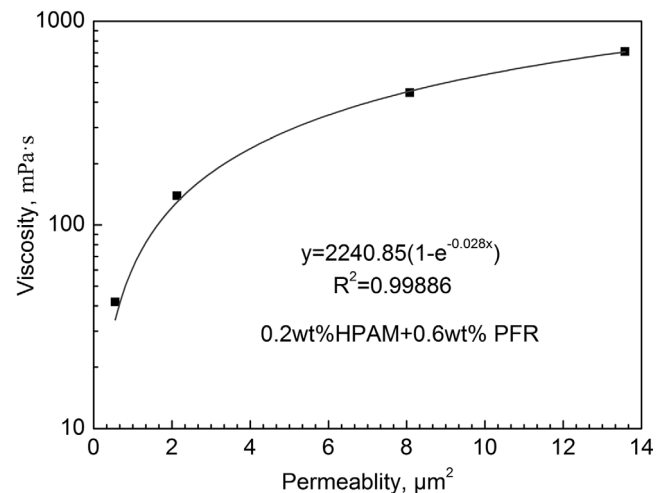
**Fig. 7.** The change of $\Delta P_{\text{ad}} \times K$ with time in the processes of dynamic gelling in sandpacks.**Fig. 8.** Relationship between sandpack permeability and apparent viscosity.

Table 6. Apparent viscosity calculated by Darcy equation in the processes of dynamic gelation in porous media under different concentrations and permeabilities.

Code	HPAM, wt%	PFR, wt%	Permeability, μm^2	Injected speed, mL/min	Shear rate, s^{-1}	Apparent viscosity*, mPa s
1	0.2	0.3	7.07	0.5	4.25	323.67
2	0.2	0.9	8.48	0.5	3.63	567.19
3	0.15	0.6	7.22	0.5	4.35	215.38
4	0.2	0.6	8.08	0.5	3.71	445.78
5	0.25	0.6	8.28	0.5	3.64	507.32
6	0.3	0.6	8.99	0.5	3.31	525.03
7	0.2	0.6	13.58	0.5	2.86	710.51
8	0.2	0.6	8.08	0.5	3.71	445.78
9	0.2	0.6	2.13	0.5	7.23	139.24
10	0.2	0.6	0.55	0.5	14.22	41.77

* Apparent viscosity calculated by Darcy equation was represented of the strength of gel restricted in sandpack.

Table 7. RRF of polymer gel after dynamic gelation in sand packs with 2.50 cm ID and 100.00 cm length under different permeabilities at 75 °C.

Code	Permeability before plugging, μm^2	Permeability before plugging, μm^2		RRF	
		Section ad	Section bd	Section ad	Section bd
1	0.550	0.0530	0.058	10	9
2	2.130	0.0390	0.216	55	10
3	8.080	0.0395	0.121	205	67
4	13.580	0.0441	0.110	308	123

static gelation time in ampoule bottles and porous media separately as well as the final dynamic gelation time was 4 times and 2 times of final static gelation time in ampoule bottles and porous media separately. The appropriate polymer gel system can be selected more accurately by the quantitative relationship between dynamic and static gelation time.

4.2 The quantitative equation between dynamic gelling strength and permeability

There were many forms of permeability heterogeneities in petroleum reservoirs. Lateral heterogeneity resulted from local depositional environments or fractures in the rock, either natural or induced. And vertical heterogeneity might be caused by so many different layers in a laminated reservoir (Todd *et al.*, 1991). Permeability heterogeneities led to poor volumetric sweep efficiencies in many water flooding and other displacement processes. Based on the relationship of pore throat and permeability given by Carman–Kozeny equation, the size of pore throat increased with increasing permeability. In the process of dynamic gelation in porous media, the growth of gel network structure was limited by the size of pore throat. So permeability was a significant factor to the process of dynamic gelling in porous media.

Effect of permeability on dynamic gelling of polymer gel was listed in Figure 7. Due to eliminate the effect of initial permeability to pressure difference, vadose force (F , unit N) was introduced, which was defined as the force that fluid flowing through the special core needed. Numerically, F was equal to the product of pressure difference ΔP_{ad} and initial permeability. The vadose force increased with permeability increasing, which resulted from bigger size of aggregates were formed in larger size of throat. The apparent viscosity calculated by Darcy Law could be represented of gel strength and plugging ability, listed in Table 6. Therefore, the relationship between apparent viscosity and shear rate or permeability could be established, shown in Figure 8. When permeability was nearly zero, the apparent viscosity was zero. While the permeability increased to infinite, the apparent viscosity reached a certain value, which was determined by static gelation in ampoule bottles. Therefore, the relationship between permeability and apparent viscosity was in accordance with equation (2):

$$y = a(1 - e^{-bx}). \quad (2)$$

Equation (2), the value of parameter a was represented of viscosity of polymer gel. The value of parameter b was represented of shear resistance. A big apparent viscosity

could be obtained at a certain injected speed only the permeability was beyond $2 \mu\text{m}^2$.

4.3 The effect of permeability on water flooding after dynamic gelation

The pressure difference of water flooding after dynamic gelation in porous media was a significant performance. It was the key to success of conformance control and water shut-off used polymer gel. Typically, Residual Resistance Factor (RRF) was used to represent the plugging ability, listed in Table 7. The RRF of both section ad and section bd increased with increasing permeability. The RRF of section ad could reach 55 when the permeability was $2.13 \mu\text{m}^2$, but the RRF of section bd was 10. If realized the in-depth flow diversion, it needed a appropriate permeability. It was crucial how to select a suitable permeability used for dynamic gelation in the process of conformance control and water shut-off. It should include good injectivity, high apparent viscosity and high RRF in deep reservoir.

5 Conclusion

The experiments of dynamic gelation of HPAM/Phenol Formaldehyde Resin (PFR) in porous media were conducted at 75°C , the most important conclusions were summarized in the following:

1. The relationship of static gelation time and dynamic gelation time was established. The dynamic gelation time in porous was much longer than static gelation time in ampoule bottles and in porous media. And the static gelation time in porous media was much longer than that in ampoule bottles.
2. With concentration of polymer and crosslinker increasing, dynamic gelation time decreased and gel strength increased. The concentration of polymer and crosslinker was an important factor on the migration of polymer gel after dynamic gelation in porous media at certain injected speed and permeability.
3. With permeability increasing, the dynamic initial gelation time increased but the dynamic final gelation time varied inconspicuously. The relationship between permeability and apparent viscosity was in accordance with equation $y = a(1 - e^{-bx})$.
4. With permeability increasing, residual resistance factor of water flooding after dynamic gelation increased. And the appropriate permeability for polymer gel treatment depended on injectivity of polymer gelant, apparent viscosity and residual resistance factor after dynamic gelation.

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