Petroleum Science 19 (2022) 3182–3193

Contents lists available at ScienceDirect

Petroleum Science

journal homepage: www.keaipublishing.com/en/journals/petroleum-science

Original Paper

Degradable preformed particle gel as temporary plugging agent for low-temperature unconventional petroleum reservoirs: Effect of molecular weight of the cross-linking agent

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ARTICLE INFO

Article history:
Received 15 February 2022
Received in revised form
16 April 2022
Accepted 29 July 2022
Available online 3 August 2022

Edited by Jia-Jia Fei

Keywords:
Temporary plugging agent
Preformed particle gel
Degradable cross-linking agent
Molecular weight
Low-temperature reservoir

ABSTRACT

The development of unconventional petroleum resources has gradually become an important succession for increasing oil production. However, the related engineers and researchers are paying more and more attention to the application of temporary plugging agents (TPAs) for their efficient development. TPAs can expand the stimulated reservoir volume (SRV) and facilitate the flow of oil and gas to the bottom of the well. Particle-gels used as temporary plugging agents have the characteristics of the simple injection process, good deformation, high plugging strength, and complete self-degradation performance, which have been widely applied in recent years. In this paper, five samples of DPPG polymerized by different molecular weights of cross-linking agents were prepared. In addition, infrared spectroscopy analysis, differential calorimetry scanning (DSC) analysis, static particle gel swelling and degradation performance evaluation experiments, and dynamic temporary plugging performance experiments in cores were conducted at 34 °C. Results show that as the molecular weight of the cross-linking agent (at 0.01 g) in the DPPG molecule decreased from 1,000 to 200 Da, the fewer cross-linking sites of DPPG, the looser the microscopic three-dimensional mesh structure formed. The swelling ratio increased from 7 to 33 times. However, the complete degradation time increased from 40 to 210 min. Moreover, the DSC results confirmed that the higher the molecular weight of the cross-linking agent, the worse is chemical stability and the more prone it to self-degradation. DPPG samples had good temporary plugging performance in reservoir cores. DPPGs prepared by the cross-linking agent with smaller molecular weight has a stronger swelling ratio, higher gel strength, and greater plugging strength in the core permeabilities. Moreover, the degraded DPPG is less damaging to the cores. However, their slower degradation rates take a slightly longer times to reach complete degradation. The results of this paper can provide new ideas and a theoretical basis for the development of particle gel-type temporary plugging agents (TPA) with controllable degradation time in low-temperature reservoirs. It can help to expand the application range of existing DPPG reservoir conditions.

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1. Introduction

With the gradual increase of international oil demand and the gradual decline of proven reserves of conventional reservoir resources, unconventional oil and gas resources have gradually become important replacement resources in the past decade (Zou et al., 2013; Lashgari et al., 2019; Alfarge et al., 2020; Zhu et al., 2021a; Liang et al., 2021). Compared with conventional oil and gas reservoirs, unconventional oil and gas reservoirs are more difficult to be produced due to the unfriendly physical properties of the reservoirs and therefore reservoir stimulation is required (Bai et al., 2013; Ma and Holditch, 2015; Jia et al., 2021a). Fracturing and acidizing are the two main approaches to petroleum reservoir stimulation (Mi et al., 2014; Zhao et al., 2021). Fracturing is to fracture the formation by injecting high-pressure fluids (e.g., water, carbon dioxide, etc.) into the formation and hold up the fracture by

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https://doi.org/10.1016/j.petsci.2022.07.013
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the proppant carried subsequently to enhance the oil and gas inflow capacity (Guo et al., 2021). Alternatively, acidification processes involve injecting strongly acidic substances (e.g., hydrofluoric acid, hydrochloric acid, or their combination) into the formation to acid-etch the formation rocks, thereby creating an easier oil and gas flow path (Zhai et al., 2020; Zhao et al., 2021b). However, when either the above two reservoir stimulation methods is applied to unconventional oil and gas reservoirs, the problems of limited stimulation volume and rapid decline in oil and gas production still occur. Therefore, technologies and methods for increasing the stimulated reservoir volume (SRV) have been extensively studied in recent years to meet the rapidly increasing human demand for hydrocarbon consumption (Mayerhofer et al., 2010; Guo et al., 2014). Among them, the current research hotspots of unconventional petroleum resource development are the techniques and methods of using temporary plugging agents (TPAs) to increase the SRV in the processes of drilling, fracturing, or acidizing (Liu et al., 2020; Jia et al., 2021b; Wang et al., 2021; Zhu et al., 2021b).

After TPAs are injected into the formation, they can temporarily plug the high-permeability petroleum formations or layers so that the reservoir stimulation methods can be applied to the unswept formations with low permeability (Jia et al., 2020; Guo et al., 2021). After the stimulation treatments, the temporary plugging agent can, in principle, degrade in the formation by itself or be removed artificially (Zhu et al., 2021c). According to the different solubility and degradation characteristics of TPAs, they can be divided into the following three categories, namely acid-soluble TPA, oil-soluble TPA, and water-soluble TPA. The acid-soluble TPA takes calcium carbonate ($CaCO_3$) products as the primary raw material, but it needs additional acid for plugging removal, and the process steps are tedious (Zhai et al., 2020). Besides, $CaCO_3$ is a kind of inorganic mineral which is brittle and almost incompressible (Ziad et al., 2016). Therefore, under the effect of positive pressure difference, $CaCO_3$ particles can only play a bridging role at pore throats or fractures, so it is difficult to play an effective plugging through deformation. Oil-soluble TPAs are mainly petroleum resin products with high melting points, such as rosin and oil-soluble phenolic resins (Xiong et al., 2018; Shi et al., 2020). However, their cost is high, brittleness is high, and their application performance is not ideal for wells with large pores and even serious leakage, and the temporary plugging strength needs to be improved (Zhao et al., 2020). The water-soluble TPA mainly uses polymers as raw materials, forms gels with other auxiliary agents. Sometimes, it uses inorganic salts and organic acids and adds an appropriate number of surfactants to make particles form bridge plugging to achieve the purpose of temporary plugging (He et al., 2018). This kind of TPA is usually challenging to degrade by itself, and a gel-breaker needs to be added simultaneously (Wang et al., 2019). Moreover, even if a small amount of gel enters the oil-bearing zones, it will cause formation damage (Seright and Brattekas, 2021). Therefore, in view of the limitations of the above TPAs and the shortcomings of their temporary plugging mechanism, TPAs with excellent self-degrading functions have been developed and applied in recent years (Zhu et al., 2021b, 2021c).

The particulate temporary plugging agent (TPA) with self-degrading function is based on conventional preformed particle gel (PPG) and introduces a kind of cross-linking structure that breaks by itself under a wide range of petroleum reservoir conditions (Zhu et al., 2021c). This kind of TPA can combine the characteristics of conventional PPG of using its deformability to seal or plug large pores with high strength but can also self-degrade into an aqueous solution with low viscosities (Zhao et al., 2021b; Zhu et al., 2021b). It degrades itself from a bulk polymer with a three-dimensional mesh structure to a linear polymer with high water solubility. Zhu et al. (2021c) were the first to develop degradable preformed particle gel (DPPG) as a TPA for drilling and oil recovery operations. Based on the characteristics of conventional PPG, such as a simple pumping process, good deformability, high plugging strength, and good environmental friendliness, the particle incorporates a self-degrading cross-linking structure into its structure. The introduction of the cross-linking agent enables DPPG to change the solid particle gel from a bulk polymer (water swellable) to a linear polymers (water soluble) solution under reservoir conditions. He also studied the effect of monomer, cross-linking agent, and initiator concentrations during polymerization on the swelling and degradation of DPPGs.

Zhai et al. (2020) applied DPPGs to drilling fluids and used it in combination with calcium carbonate ($CaCO_3$) fine powder (tens of microns), which has an excellent temporary plugging effect during the drilling process. Zhu et al. (2021b) used a core displacement experimental setup to investigate the temporary plugging effect of the DPPG at different swelling ratios, particle sizes, and injection volumes. They found that DPPGs had good plugging strength and self-degradation ability in porous media. Zhao et al. (2021b) applied the DPPG particles to the field of carbonate acidification. They found that the particle-based TPA also has strong acid resistance, which can further expand the stimulated reservoir volume (SRV) of acid and increase the formation's permeability. Wang et al. (2021) investigated using 1,6-hexanediol diacrylate as a degradable cross-linking agent in PPG as an acidizing temporary plugging agent. For high-temperature petroleum reservoirs (100 °C), Zheng et al. (2021) used azodisisobutyramidine hydrochloride (V-50) and maleic anhydride as raw materials to prepare an azo-type thermosensitive cross-linking agent. Furthermore, based on this, a thermosensitive self-degradation microgel was polymerized. However, the effect of the molecular weight of the cross-linking agent on the performance of the particulate TPA has not been reported yet. Therefore, understanding the influence of the molecular weight of the cross-linking agent will not only help to broaden the applied reservoir temperature range of particulate TPA but also help to screen out particulate TPA with controllable plugging strength and degradation time. Thus, we can explore the intrinsic influence mechanism of their temporary plugging function.

In this paper, we proposed to prepare different DPPGs with different molecular weights of cross-linking agents, which could be used as deformable particulate temporary plugging agents (TPA). Infrared spectroscopy and DSC/TG analysis methods were used to investigate the effect of the molecular weight of the cross-linking agent on the chemical structure of the deformable gel particles. The effects of different molecular weights of cross-linking agents on the gel swelling rate and swelling ratios of the particles were investigated using the particle gel static evaluation method. In addition, the effects of different molecular weights of cross-linking agents on the degradation rate and complete degradation time of the DPPG particles under different swelling ratio conditions were also investigated. The dynamic temporary plugging performance of the DPPG particles prepared with different molecular weight cross-linking agents in cores was investigated using a core flooding device. Finally, based on the above experimental results, the mechanism of the effect of molecular weight of cross-linking agents on the swelling and degradation performance of deformable particle gels was analyzed using a schematic diagram. This study attempted to broaden the scope of application of existing deformable particulate TPAs in petroleum reservoirs and further enrich the mechanism of action between the chemical structure of deformable DPPGs and their temporary plugging performance. It is conducive to further expanding their application prospects in oil fields worldwide.

2. Experimental materials and methods

2.1. Materials

Acrylamide (AM, AR 99%), 2-Acrylamido-2-methyl-1-propane sulfonic acid (AMPS, 98%), persulfate (99.9%), sodium chloride (AR 99.5%), all of the above reagents were purchased from Shanghai Macklin Biological Co., Ltd, China. AM and AMPS were employed as monomers and persulfate was used as the initiator in polymerization. Polyethylene glycol diacrylate (PEGDA, molecular weight 200, 400, 575, 700, and 1000 Da, containing <400 ppm MEHQ stabilizer) using as the crosslinking agent was provided by Beijing Yuanyang Huanyu Petroleum Technology Co., Ltd. Deionized water was prepared in the laboratory.

The core was an artificial carbonate core with a length of ~7 cm and a diameter of ~2.5 cm. The physical formation parameters of the core are shown in Table 1. A hole with a length of 5 cm and a diameter of ~0.5 cm was drilled in the middle of the core (at the inlet) to allow a certain volume of DPPG to be injected in the core to simulate a near-well situation or a large borehole. The water permeability in Table 1 was the result calculated according to Darcy's law when the core was flooded with 1% NaCl solution until the pressure difference kept stable. And the water permeability results were the average value of three experiments.

2.2. Preparation of DPPG

A certain amount of AM and AMPS solid powder was weighed into a beaker filled with deionized water and stirred and mixed at a speed of 600 r/min. AMPS was not neutralized to make it easier to synthesize during large-scale production. The PEGDA liquid was then weighed and dropped into the solution. After stirring for another 10 min, a certain amount of KPS powder was added and then placed in an oven at a constant temperature (45 °C) to react for another 2 h. The composition of DPPG prepared by PEGDA with different molecular weight is shown in Table 2. After gelation, the gel was dried and crushed to particles of different particle sizes. Finally, dry DPPG products with the size of 20–30 mesh were obtained.

2.3. Measurement of swelling and degradation properties of DPPG

The experimental method was described in the literature by Zhu et al. (2021b) as a comparison. 0.1 mL of 20–30 mesh dried DPPG particles were weighed and placed in a dry, high-temperature resistant glass test tube. Then, a certain amount of 1% NaCl solution (simulating formation water) was added, and the variation between the stacked volume of the particle gels and the swelling time was recorded. After 2 h of swelling, the free water in the test tube was removed, the test tube with the swollen DPPG (V_0) was placed in a thermostat at a preset temperature to observe and record the variation between the remaining stacked volume (V_t) of the swollen gel particles and time. The degradation efficiency (E_D) or degradation rate can be calculated by Eq. (1).

$$\text{Degradation efficiency } (E_D) = \frac{V_0 - V_t}{V_0} \times 100\% \quad (1)$$

where E_D is the degradation efficiency, V_0 is the original stacked volume of the swollen DPPG particles before being aged, V_t is the remaining gel stacked volume at aging time t (min).

The temperature of the evaluation experiment in this paper was 34 °C to simulate a low-temperature reservoir temperature in Xinjiang Oilfield. Low-temperature reservoirs need more stringent requirements on the performance of the cross-linking agent in the DPPG system. The application of DPPG in medium and high-temperature reservoirs has been studied in previous works, and their temporary plugging performance can be achieved by adjusting components such as monomer and the type or concentration of cross-linking agent (Zhu et al., 2021c).

Table 2
Synthesis conditions of DPPG prepared from PEGDA with different cross-linking agents.

No.	AM, g	AMPS, g	PEGDA		KPS, g
			M_w	Mass, g	
M1	6	6	200	0.01	0.4
M2	6	6	400	0.01	0.4
M3	6	6	575	0.01	0.4
M4	6	6	700	0.01	0.4
M5	6	6	1000	0.01	0.4
M6	6	6	200	0.02	0.4
M7	6	6	400	0.02	0.4
M8	6	6	575	0.02	0.4
M9	6	6	700	0.02	0.4
M10	6	6	1000	0.02	0.4

2.4. Infrared spectral analysis of DPPG particles

The DPPG particles prepared in Section 2.2 were dried, then ground into 200–300 mesh powder, mixed well with KBr powder, and finally pressed into thin sheets under 20 MPa pressure conditions. After being placed in a vacuum drying oven for 24 h, the sheets were tested by a Fourier transform infrared spectrometer (Nicolet IS20, Thermo Fisher Scientific).

2.5. DSC measurement of DPPG particles

Differential calorimetric scanning (DSC) measurement can be used to study the molecular structure changes of a substance by detecting the change of heat difference (ΔQ) of the sample itself with temperature under programmed temperature control. The DPPG particles prepared in Section 2.2 were dried, and ~5 mg of DPPG samples were weighed for DSC/TG measurements. The testing instrument was a NETZSCH differential calorimetry scanner (STA 449F5, Germany), and the atmosphere was inert nitrogen. The temperature test ranged from 20 °C to 500 °C, and the temperature rise rate was 5.0 °C/min. The DSC range was 500 μV, and finally the heat flow rate versus temperature curve of the DPPG sample was obtained.

2.6. Core displacement experiments

The dynamic temporary plugging performance of DPPG prepared by the different molecular weights of cross-linking agents in porous media was investigated by the core displacement experimental setup. The experimental setup and flow diagram are shown

Table 1
The physical formation parameters of the artificial carbonate core.

No.	Length, cm	Diameter, cm	Porosity, %	Gas permeability, 10^{-3} μm^2	Water permeability, 10^{-3} μm^2
50–8	7.521	2.535	21.50	50	36.52
50–9	7.538	2.536	22.50	50	32.73

in Fig. 1. Firstly, 1% NaCl solution was injected into the core by the ISCO pump at a constant flow rate of 0.05 mL/min until the displacement pressure was stabilized for a period of time. The water permeability (K_1 in 10^{-3} μm^2) was calculated by Darcy's law at this time. Then, the core was injected with 20 times swollen DPPG particles at a constant pressure of 20 MPa for 3 min. The method for preparing a swollen gel with a swelling ratio of 20 times was to weigh 1 mL of the dry particle gel and disperse it in 19 mL of the aqueous solution. Finally, 1% NaCl solution was injected into the core again at a constant flow rate of 0.05 mL/min until the replacement pressure was stabilized for a period of time. The water permeability (K_2, in 10^{-3} μm^2) was calculated by Darcy's law at this time. During the displacement process, the pressure changes during the experiment were recorded in real-time. Then, the core holders were kept in an oven at 34 °C for two weeks. After the complete degradation of DPPG, 1% NaCl solution was injected again into the core at a constant flow rate of 0.05 mL/min by the ISCO pump until the displacement pressure stabilized for a period of time. At this time, the water permeability (K_3, in 10^{-3} μm^2) was calculated by Darcy's law. The permeability variation in porous media could be used to compare the plugging strength and temporary plugging performance of DPPG prepared by different molecular weights of cross-linking agents.

3. Results and discussion

3.1. Synthesis of DPPGs with different molecular weights of cross-linking agents

For gels polymerized from monomers, the molecular weight of the cross-linking agent can affect the cross-linking density and strength of the whole gel, which in turn affects the performance of the gel. For example, when the cross-linking density of the gel is small, the size of the microscopic spatial network structure of the formed bulk polymer will be large (Zhao et al., 2021a; Zhu et al., 2021c). The looser the network, the larger the swelling volume will be when it is swollen by water under the action of osmotic pressure, but its degradation mechanism is still unclear. Therefore, Table 1 designs DPPGs prepared from different molecular weights of cross-linking agents and different dosages. The dosage of both AM and AMPS were fixed at 6 g, and the dosage of KPS was 0.4 g. Five different PEGDA solutions with molecular weights of 200, 400, 575, 700, and 1,000 Da were added and placed in a constant temperature oven at 45 °C for 2 h to react. After drying, a series of 20–30 mesh DPPG particle samples was yielded after crushing.

Fig. 2. Photo of the DPPG-1 particle sample.

They were numbered in order from DPPG-M1 to DPPG-M5. For example, the 20–30 mesh DPPG-1 sample prepared from 0.01 g of PEGDA with the molecular weight of 200 Da is shown in Fig. 2. The DPPG-M1 sample exhibited white and translucent crystalline solid particles with uniform particle size at room temperature.

In order to facilitate the analysis of the effect of different molecular weights on the structure of DPPG samples and their macroscopic properties, i.e., to investigate the effect of their molecular weights on the temporary plugging properties, DPPG samples were analyzed by infrared spectroscopy. In this paper, the infrared spectra of the samples were measured using an FTIR spectrometer. The infrared spectra of DPPG-M1 to M5 (see Table 1 for the specific formulation) are shown in Fig. 3.

Taking the DPPG-M1 sample as an example, the correspondence between the main functional groups and the main absorption peaks is as follows. The absorption peak around 3415 cm^{-1} is caused by the stretching vibration of secondary amide (—NH) or primary amide (—NH$_2$). 1637 cm^{-1} is the C=O stretching vibration in the carboxyl (—COOH), which is the characteristic peak of polyethylene glycol diacrylate (PEGDA). 1384 cm^{-1} is the stretching vibration of C—N in the amide group, which belongs to the amide III spectrum. The strong peak at 1730 cm^{-1} and the peaks at 1250 cm^{-1} and

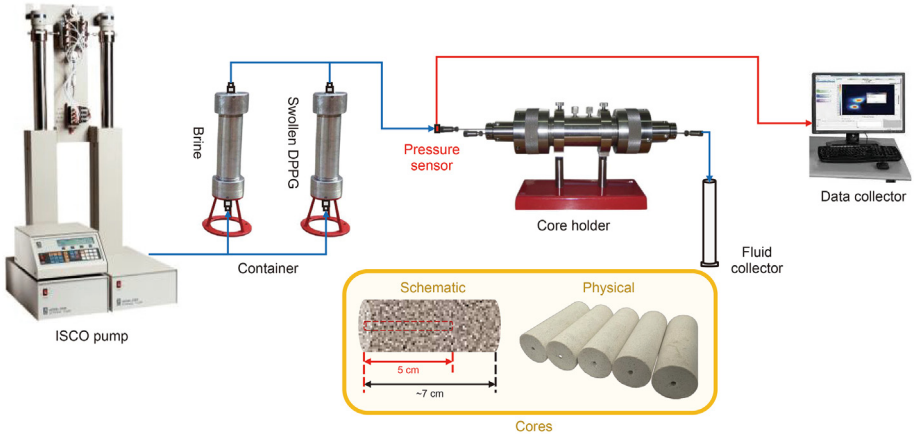


Fig. 1. Schematic diagram of the displacement experiment.

1170 cm^{-1} are the characteristic peaks of the antisymmetric and symmetric stretching vibrations of C—O in PEGDA, respectively. The absorption peak at 1040 cm^{-1} is attributed to the S—O stretching vibration of the sulfonic acid group. 623 cm^{-1} is the out-of-plane bending vibration frequency of —NH.

In addition, comparing the IR spectra of the other four DPPG samples in Fig. 3, it can be found that the positions of those absorption peaks are unchanged. It is mainly because the cross-linking agent is still poly(ethylene glycol) diacrylate, i.e., their chemical composition does not change. Only the molecular weight of the cross-linking agent changes. Therefore, the position of their absorption peaks remains the same. Only the transmittance appears to change in a certain regularity. In general, the transmittance decreases as the molecular weight of the cross-linking agent increases. It is mainly due to the increase in molecular weight of the cross-linking agent and the corresponding increase in the active cross-linking sites, leading to an increase in cross-link density during the reaction (i.e., more cross-linking reactions). The sample as a whole is closer to white, making it slightly less translucent.

In addition, to verify whether the same law of influence still occurs when the cross-linking agent concentration increases, we also compared the experimental results of IR spectra of DPPG samples prepared from PEGDA of different molecular weights when the cross-linking agent concentration changes to 0.02 g, as shown in Fig. 4. The results show that the influence laws are all similar. The transmittance at the maximum absorption peak for DPPG M6, M7, M8, and M10 is approximately 68%, except for M9, which has a transmittance of 53% at the maximum absorption peak. The transmittance corresponding to the maximum absorption peak is significantly lower when compared with Fig. 3. Since PEGDA contains a carboxyl group (—COOH), it corresponds to the maximum absorption peak near 3415 cm^{-1} in Figs. 3 and 4. It shows that the higher the concentration of PEGDA contained in the DPPG sample, the more imine groups are involved in their molecular structures. According to the Lambert-Beer law, the intensity of its absorption peak will be greater, and the transmittance will be smaller. Overall, the results of comparing the IR spectra in Figs. 3 and 4 show somewhat regular variations between the chemical structures of DPPG prepared by different molecular weight cross-linking agents. Moreover, these differences between chemical structures will inevitably affect their macroscopic temporary plugging properties.

3.2. Static swelling properties of different DPPG samples

Through nearly 30 years of experimental and oilfield application studies on PPG, Bai et al. found that the swelling performance of PPG can directly affect the plugging strength when the reservoir conformance is improved (Bai et al., 2015; Zhang and Bai, 2011). Usually, under a certain swelling ratio condition, the plugging strength increases with the swelling ratio. Therefore, to understand the swelling performance of DPPG samples synthesized by PEGDA with different molecular weights, it is necessary to evaluate their static swelling performance by using a static evaluation method of preformed particle gels (Zhu et al., 2021b). In turn, we can explore the mechanism of the effect of different molecular weights on the temporary plugging performance of DPPG, which helps us screen out high-quality DPPG with controllable plugging strength and degradation time. We weighed 0.1 g each of DPPG M1 to M5 samples into test tubes and then added ~10 mL of mass fraction of 1 wt% NaCl solution in the above tubes, and performed swelling experiments in a constant temperature oven at 34 °C. The relationship between the swelling volume and swelling time of DPPG M1 to M5 is shown in Fig. 5.

As shown in Fig. 5, the changes of the swelling volumes of all DPPG samples roughly exhibit two trend regions of change. The first is the rapid growth region of swelling volume, followed by the slow growth region. In addition, there is a large discrepancy in the swelling volume variation between DPPG prepared by different molecular weight cross-linking agents. Among them, DPPG-M5 has the smallest swelling volume and swelling ratio. It shows a maximum expansion volume of 0.6 mL at 20–25 min, i.e., it increases from 0.5 to 0.6 mL and remains the same at the later stage. However, the M1 sample has the largest swelling volume and swelling ratio. Moreover, it changes more rapidly before 20–25 min than that after 25 min. M2-M5 also reaches a plateau area in the time range of 20–25 min, and the swelling volume remains the same after water absorption in 25–60 min, with a slight increase later. The larger the relative molecular weight of the cross-linking agent PEGDA, the smaller the cross-link density, the more sites to form a spatial three-dimensional network structure, and the more sufficient polymerization reaction (Zhu et al., 2021c). Therefore, when DPPGs start to absorb and swell with water, their corresponding swelling volume and swelling rate will be slower because the cross-linked mesh is very dense, and water is difficult to enter.

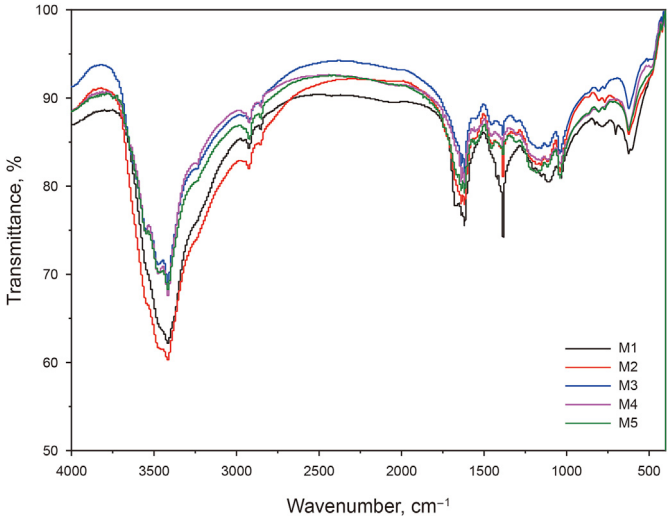


Fig. 3. Comparison of IR spectra of DPPG M1 to M5 samples (0.01g PEGDA).

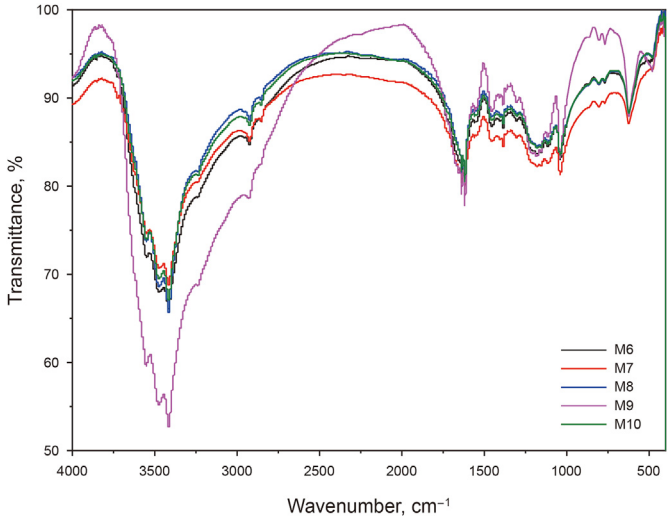


Fig. 4. Comparison of IR spectra of DPPG M6 to M10 samples (0.02 g PEGDA).

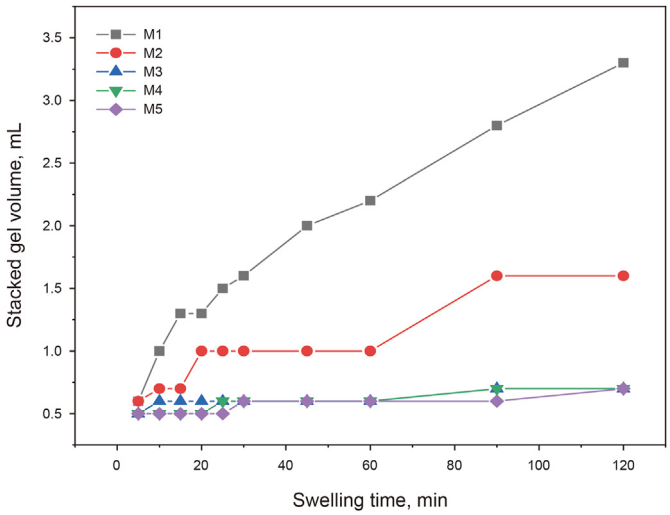


Fig. 5. Swelling performance of DPPG M1 to M5 samples at low formation temperature (34 °C).

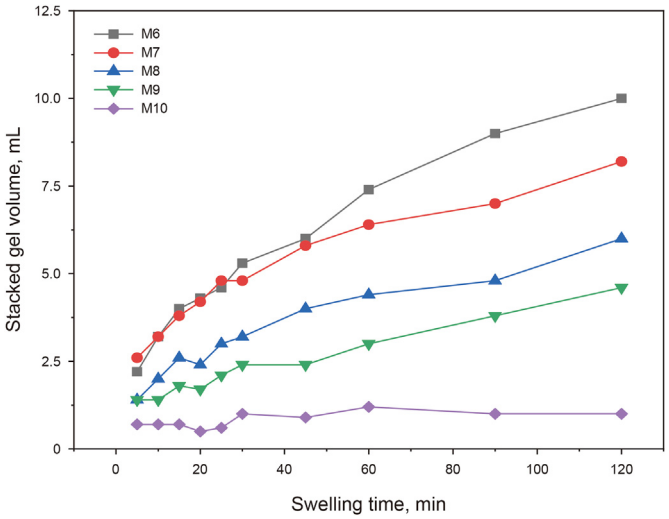


Fig. 6. Swelling Performance of DPPG M6 to M10 samples at the temperature of 34 °C.

As the effect of osmotic pressure continues to weaken, water will slowly enter the network structure so that a region of slow growth in the subsequent swelling volume occurs. Some of them even almost ceased to change. For example, the swelling volume of sample M1 remained unchanged in the range of 30 to 90 min of swelling time. However, as the swelling time increases, the swelling volume of M2 to M5 no longer shows significant changes with time after increasing to a certain range.

Furthermore, in the middle and late stages of the rapid swelling region of DPPG (i.e., in the range of 20 to 25 min), the swelling volume of M4 and M5 changes from 0.5 to 0.6 mL, and M3 remains unchanged at 0.6 mL. However, the swelling volume of M2 suddenly increases from 0.7 to 1.0 mL, and M1 increases from 1.3 to 1.55 mL. The swelling volume does not change much as the molecular weight decreases from 1000 to 575 Da (i.e., changing from M5 to M3). Nevertheless, the swelling volume suddenly increases when it changed to M2 (i.e., molecular weight 400). Moreover, eventually, when it changes to M1, the swelling volume increases and is much higher than that of M2 to M5. Therefore, as the molecular weight decreases, the water absorption and swelling ratio increases instead at the same time. It is because there are fewer cross-linking sites on the molecular structure as the molecular weight of the cross-linking agent decreases. As a result, the spatial network structure formed during the polymerization reaction is not dense enough, i.e., the size of the microscopic lattice is large. When DPPG is exposed to water, it will absorb water and swell rapidly under the action of osmotic pressure. It is worth noting that DPPG M1 continues to swell after 60 min of water absorption, which inevitably affects the plugging performance of the particle gel. Therefore, the swelling volume (or ratio) should be optimized before DPPG applications.

We also examined the water absorption and swelling properties of DPPG prepared with different molecular weights of PEGDA at the addition amount of 0.02 g, as shown in Fig. 6. We can see that the trends of the swelling volume and swelling rate of DPPG are consistent with those in Fig. 5. As the molecular weight of PEGDA in the composition of DPPG decreases, the swelling volume and rate of the particles keep increasing. In addition, with the increase of the PEGDA concentration, the swelling ratio of DPPG particles also increases accordingly. It is mainly because, with the increase of the cross-linking agent content, the competition between the cross-linking points increases, and thus the cross-linking density decreases. Hence, the size of the formed spatial network structure increases instead. It can also be seen from Fig. 6 that when the molecular weight of PEGDA is 200 Da and the addition amount is 0.02 g, its water absorption is maximum, and swelling ratio can reach 100 times. However, it will inevitably affect its plugging performance and degradation properties, so the swelling ratio of DPPG needs to be optimized before oilfield application.

3.3. Static degradation performance of different DPPG samples

The temporary plugging performance of the gel may damage the formation permeability, and the degree of gel damage to the formation is related to the molecular weight or the molecular structure of the gel particles. Therefore, we did experiments to evaluate the static degradation performance of DPPG samples prepared by different molecular weights of PEGDA. We fixed the amount of AM, AMPS, KPS, and deionized water and varied the molecular weight of the cross-linking agent PEGDA (i.e., 200, 400, 575, 700, and 1000 Da) to prepare different DPPG samples. The static degradation performance experiments were performed at 45 °C and placed in a constant ventilated oven for evaluation. The results of the experiments are shown in Fig. 7.

As shown in Fig. 7(a), the time to reach the maximum degradation rate (i.e., 100% degradation) for M1 was 210 min. Within 10–150 min, the degradation rate and degradation time exponentially increased. After 150 min, the degradation rate slowed down. It can be seen that as the molecular weight of the cross-linking agent PEGDA increased, the degradation rate became faster, and the slope of the curve in the figure became steeper. Among them, the time required for the complete degradation of M4 and M5 was only 40 min, the time required for the complete degradation of M3 was 50 min, and the time required for the complete degradation of M2 was 65 min. In summary, as the molecular weight of the cross-linking agent PEGDA added to the DPPG samples decreased, the more time it took for the DPPG samples to reach complete degradation.

To further verify the reliability of the above conclusions, we also compare the degradation at three other different swelling ratios (i.e., complete swelling, swelling 20 times, and swelling half of the full volume). The results are shown in Fig. 7(b-d), respectively. It can be seen that the patterns are similar to those in Fig. 7(a), except that there is a difference in the absolute time required for complete

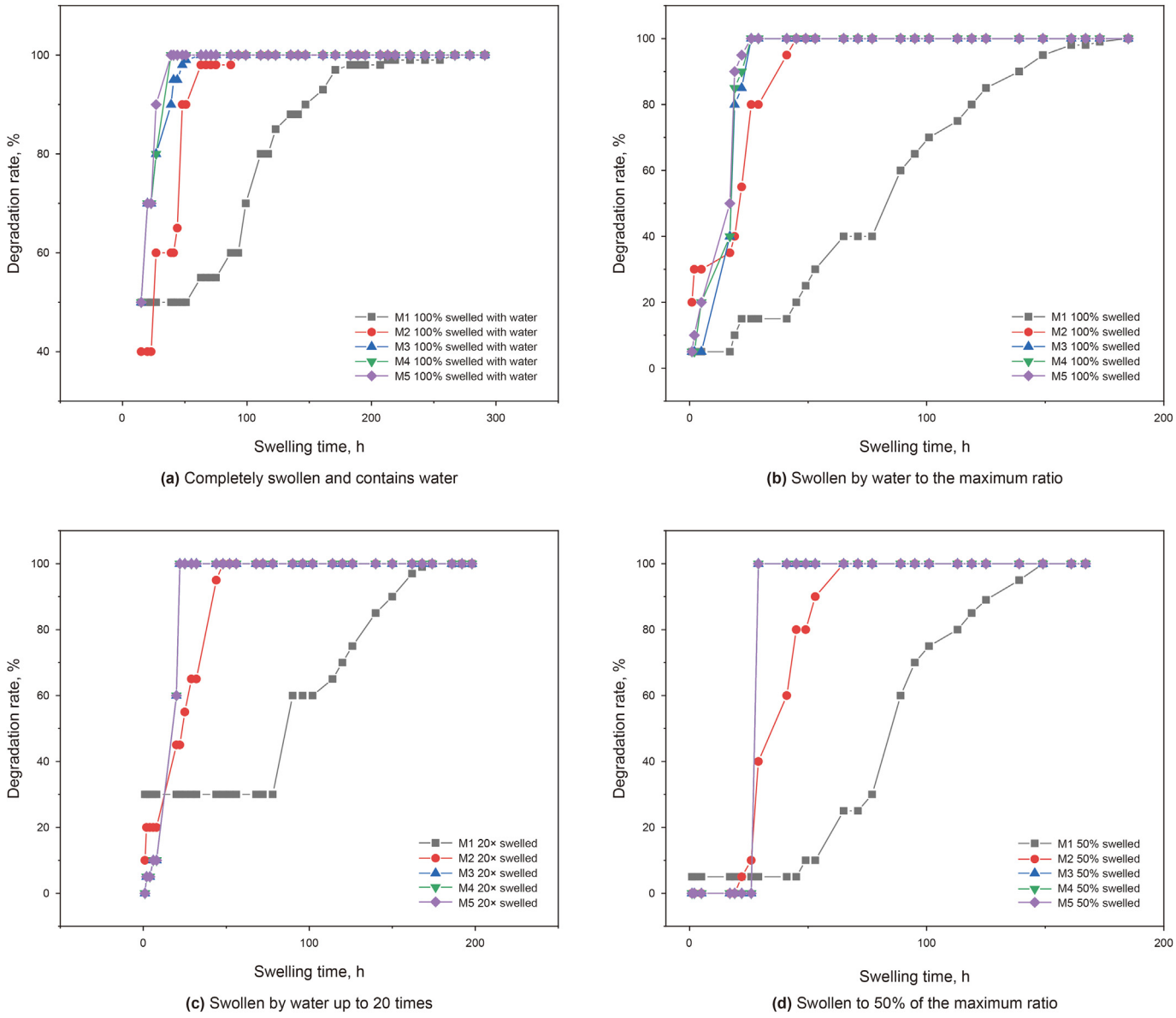


Fig. 7. Degradation performance of DPPG samples M1 to M5 at the different swelling ratio.

degradation. Taking M1 as an example, the time required for complete degradation was 185 min when it was fully swollen (i.e., the swelling ratio was 33 times), while the time required for complete degradation reduced to 175 min when it was swollen 20 times, and further reduced to 150 min at half the swelling multiple. It can be seen that for the same DPPG sample, the degradation time gradually increases with the increase of the water absorption and swelling ratio of DPPGs.

In addition, as the molecular weight of the cross-linking agent PEGDA increases, the time required for complete degradation becomes shorter. Moreover, all DPPGs can self-degrade completely. Therefore, the increase of the molecular weight of the cross-linking agent PEGDA decreases the swelling ratio (as shown in Figs. 5 and 6), but its degradation time is also significantly reduced. It is mainly due to the fact that the cross-linking agent PEGDA in the microscopic network structure of DPPG particles is an important component of the bulk polymer and that it is degradable. Thus, the higher the molecular weight of PEGDA, the faster the breaking rate, the faster the conversion of bulk polymer (swelling type) to linear polymer (dissolving type), and the shorter the required degradation time. However, it is worth noting that although the higher the molecular weight of the cross-linking agent is, the shorter the degradation time is, its swelling performance is also limited. Therefore, in order to reduce the damage of temporary plugging agents to the formation and improve the effectiveness of water control and oil enhancement treatments, we can select DPPG products with swelling volume and degradation time that meet the reservoir stimulation requirements in advance. Therefore, it can meet the need of plugging strength or protection of the formation in the actual reservoir stimulation processes.

In this paper, the degradation effect of the DPPG system with different molecular weights of PEGDA added at 0.02 g was also investigated, as shown in Fig. 8. The law between the degradation of DPPG and the molecular weight of the cross-linking agent PEGDA is similar to that of Fig. 7. The relationship between the degradation rate and the degradation time is approximately exponential. As shown in Fig. 8(a), the complete degradation time for both M6 and M7 was 880 min at the maximum swelling ratio. The complete

degradation time for M8 and M9 was 440 min, while the time required for the complete degradation of M10 was 80 min. With the increase of molecular weight of PEGDA during the preparation of DPPG, the degradation time of DPPG particles that are completely swollen in an aqueous solution is decreasing at the low temperature of 34 °C. As can be seen from Fig. 8(b-d), the degradation times of DPPG particles with different swelling multiples all decrease with the increase of molecular weight of the cross-linking agent. In addition, comparing the degradation rates of the other three different swelling ratios, it is evident that as the swelling ratio of DPPG decreases, the degradation rate decreases, and the complete degradation time is correspondingly and significantly shortened.

During the oilfield application, we should fix the particles to maintain a certain swelling ratio to ensure that they have a certain plugging capacity. It can also be seen from Fig. 8 that the molecular weight of the cross-linking agent also affects the degradation properties of DPPG when the swelling ratio of DPPG is given. As the molecular weight of PEGDA in the structure of DPPG increased, the degradation time of the particles after swelling shortened. Of course, the gel strength differs between different types of DPPG, which can have an impact on its plugging performance in porous media, which will be investigated in Section 3.4.

Fig. 9 compares the maximum swelling stack volume and complete degradation time of DPPG prepared with different PEGDA molecular weights in the presence of water. The cross-linking agent PEGDA was added at 0.01 g and 0.02 g, respectively. As can be seen from Fig. 9(a), the molecular weight of the cross-linking agent in M1 to M5 increased from 200 to 1000 Da, the swelling volume of the particles after water swelling decreased from 3 mL to 0.7 mL, and the corresponding complete degradation time decreased from 240 min to 40 min. Thus, the molecular weight of the cross-linking agent PEGDA in the DPPG formulation affected both the swelling ratio of the particles (i.e., it affects the plugging strength of the system) and the complete degradation time of the particles. Although the addition of PEGDA to the DPPG system in Fig. 9(b) increased to 0.02 g, the effect of PEGDA molecular weight on the

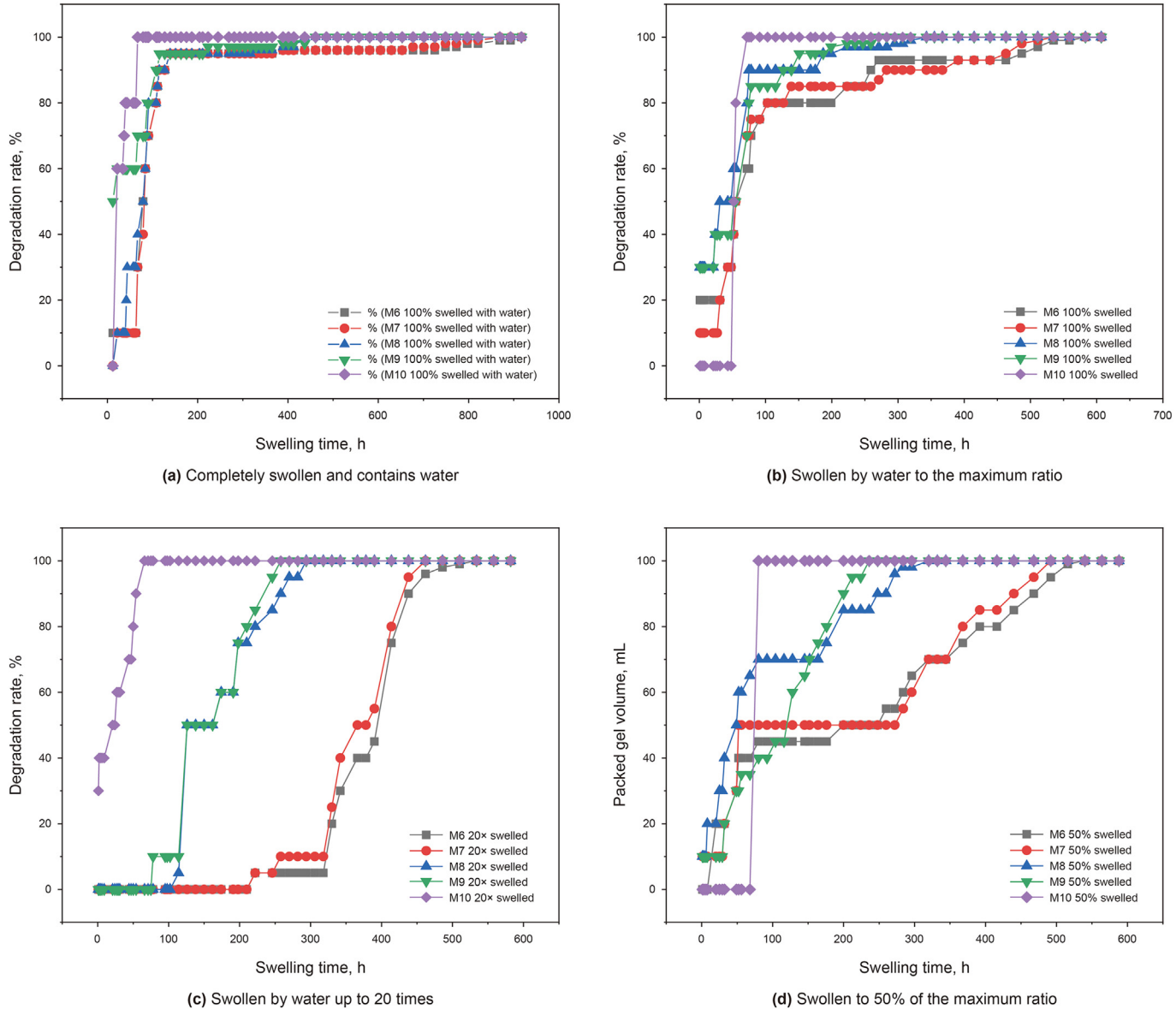
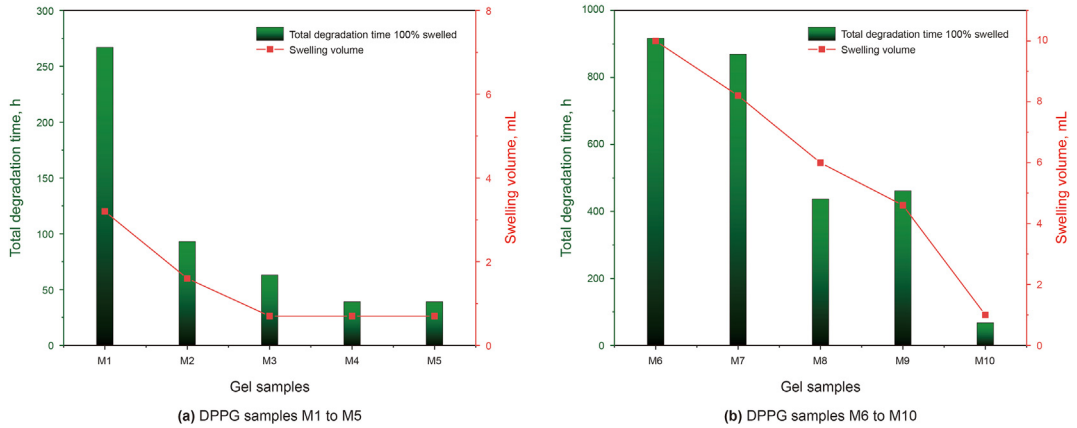


Fig. 8. Degradation performance of DPPG samples M6 to M10 at the different swelling ratio.



(a) DPPG samples M1 to M5 **(b)** DPPG samples M6 to M10

Fig. 9. Degradation performance of DPPG samples M1 to M10 at maximum swelling multiplier.

degradation rate follows the same law as in Fig. 9(a), except that there is a very significant overall increase in the degradation time. It is mainly because as the cross-linking agent increases, the cross-linking sites increase correspondingly and significantly, so the time required to totally break these bonds also increases.

3.4. Dynamic temporary plugging performance of different DPPGs in cores

In order to investigate the influence of the molecular weight of the cross-linking agent PEGDA on the dynamic temporary plugging performance of the prepared DPPG in porous media, we selected two DPPG samples (i.e., M1 and M5) for comparisons based on the experiments of static swelling and degradation performance studied in Sections 3.2 and 3.3. These two DPPGs prepared from PEGDA with molecular weights of 200 and 1000 Da, respectively, were examined using a core displacement device (Fig. 1). During the experiments, swollen DPPGs with the swelling ratio of 20 were prepared as described in Section 2.3 and then injected at the constant pressure of 20 MPa to simulate the high-pressure condition during stimulation operations such as fracturing. Experimental results are shown in Figs. 10 and 11, respectively.

The temporary plugging performance of DPPG-M1 in a core with the air-measured permeability of 50×10^{-3} μm^2 (Core 50-8) after 20 times of swelling is given in Fig. 10. First, the water-measured permeability of the core was tested with 1% NaCl solution with a pressure difference of 34 kPa, and the water-measured permeability at this point was calculated to be 36.52×10^{-3} μm^2 according to Darcy's law (Jacob, 1946). Then, DPPG with 20 times swelling was injected into the side of the core with long holes at a constant pressure difference of 20 MPa, and the injection time was set to 3 min. Then, 1% NaCl solution was injected again to determine the plugging strength of DPPG in the core at this time. As shown in Fig. 10, the injection pressure difference between the two ends of the core could reach 7.638 MPa at this time, and the converted permeability was only 0.16×10^{-3} μm^2. Therefore, the DPPG-M1 swollen by 20 times could form a high-strength plug in the core. Then, the core holder was placed in the constant temperature 34 °C oven for two weeks. And finally, the 1% NaCl solution was injected again to measure the degradation of DPPG. As shown in Fig. 10, the injection pressure difference dropped to 39 kPa during the second water flooding, i.e., the water permeability recovered to 31.84×10^{-3} μm^2, which is very near to the permeability before treatment. It indicates that DPPG-M1 has an excellent temporary plugging ability. It has a strong strength plugging effect during the injection process, and it can degrade itself in the formation so that the damage to the formation is minimal. That is, the permeability of the formation after DPPG treatments can be largely recovered.

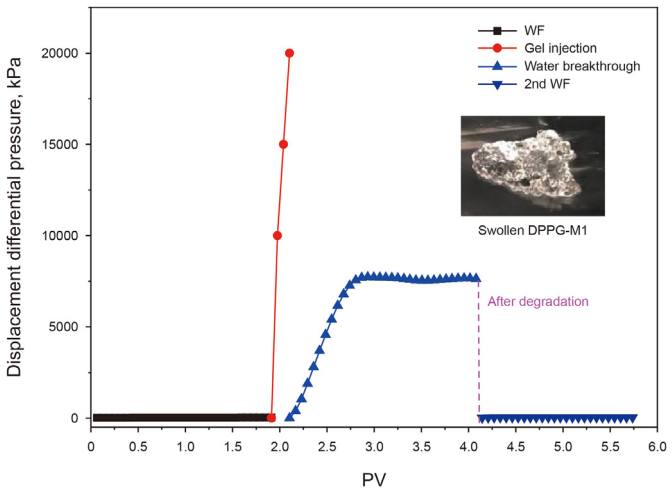


Fig. 10. Displacement pressure dynamics of DPPG-M1 sample in Core 50-8.

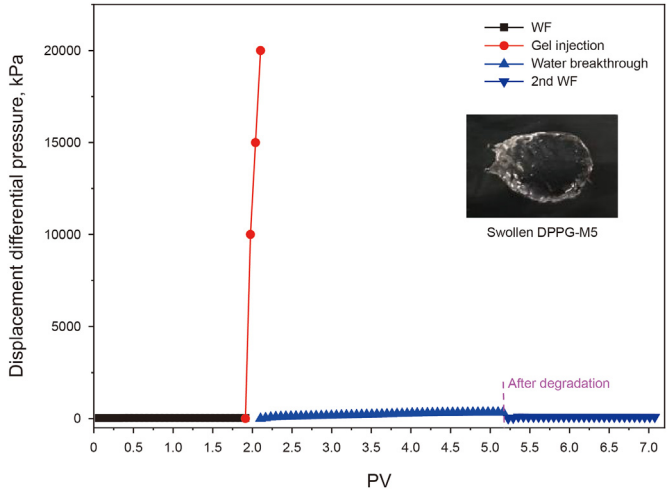


Fig. 11. Displacement pressure dynamics of DPPG-M5 sample in Core 50-9.

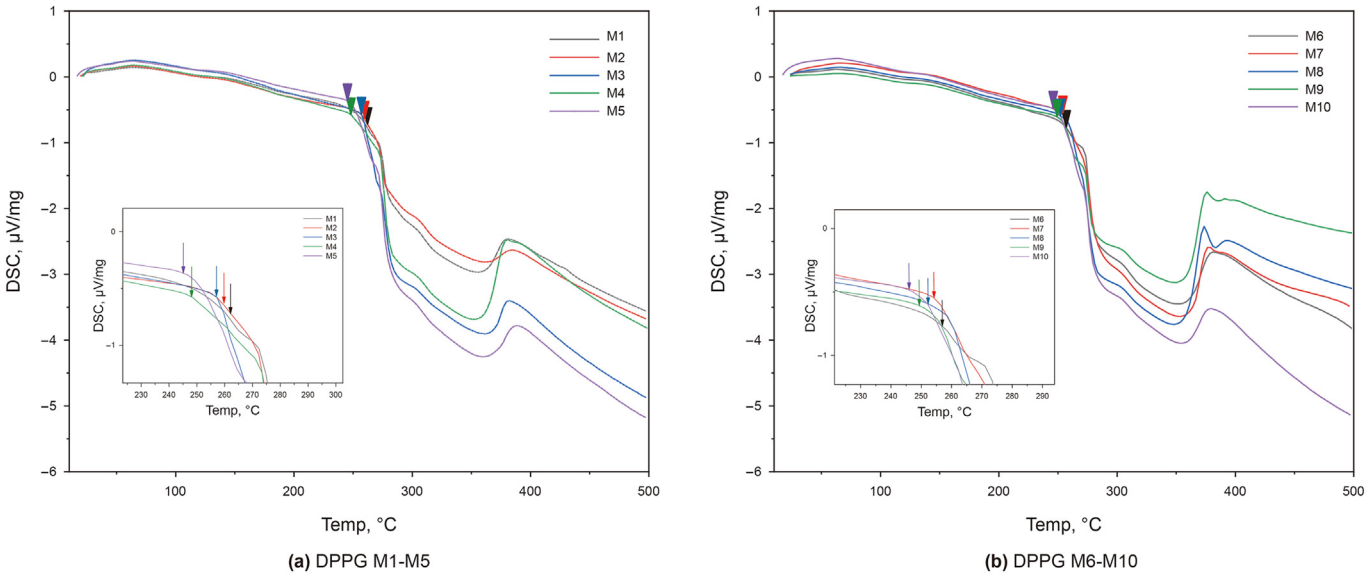


Fig. 12. DSC analysis of DPPG samples M1-M10.

The temporary plugging performance of DPPG-M5 in another core (Core 50-9) with an air-measured permeability of 50×10^{-3} μm^2 after 20 times of swelling is given in Fig. 11. As described in Table 1, the molecular weight of the cross-linking agent PEGDA in the DPPG-M5 system was 1000 Da, which is larger than that of DPPG-M1, but the static swelling performance and degradation performance were poor. However, it can be seen from the photos in Fig. 11 that the strength of DPPG-M5 after 20 times of swelling was significantly weaker than that of DPPG-M1. Same as the experimental procedure of Core 50-8, firstly, 1% NaCl solution was injected into the core, and the pressure difference between the two ends of the core was 38 kPa. That is, the water-measured permeability of the core was 32.73×10^{-3} μm^2. Then, DPPG-M5 with 20 times swelling was injected into the core for 3 min at a constant differential pressure of 20 MPa, but because the strength of the dissolved DPPG-M5 was weaker at this time, 2.1 mL of liquid came out of the exit end of the core, indicating that some of the DPPG should have entered the core matrix. After that, 1% NaCl solution was injected again, and the plugging strength of DPPG-M5 in the core was measured at this time. Compared with the injection pressure difference of DPPG-M1, it was significantly reduced to 346 kPa (i.e., the water permeability was 0.36×10^{-3} μm^2). It is mainly because the strength of DPPG-M5 was significantly weakened. After two weeks of aging, the 1% NaCl solution was injected again, and the injection pressure difference decreased to 89 kPa (i.e., the permeability only recovered to 1.40×10^{-3} μm^2). The experimental results show that swollen DPPG-M5 had some temporary plugging ability, but it could enter the core matrix under the action of high-pressure difference due to its weak strength, and it would still cause some damage to the formation even after degradation.

In summary, it can be seen that the molecular weight of the cross-linking agent can significantly affect the dynamic temporary plugging performance of the prepared DPPG. Therefore, in the actual temporary plugging construction operations, it is hoped that the temporary plugging agent has both strong plugging performance and can self-degrade after a certain period of stability (to meet certain construction requirements). Therefore, during the development and screening of the temporary plugging agent of

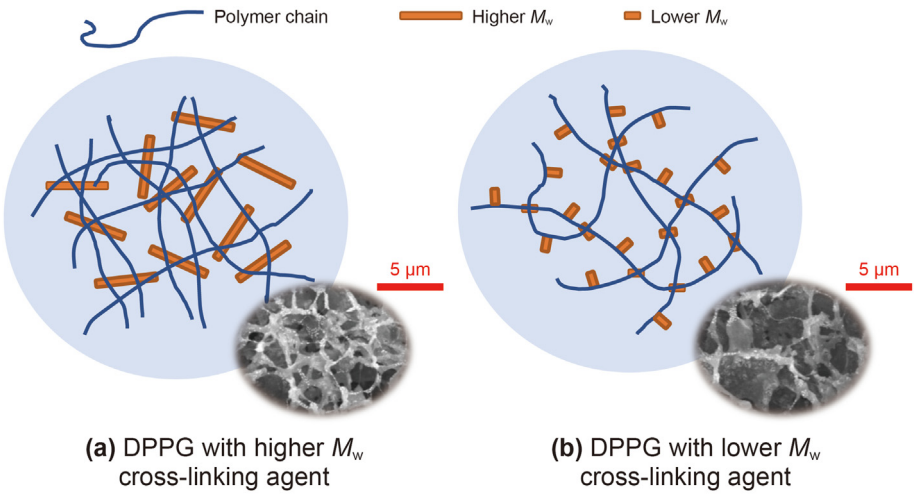


Fig. 13. Schematic diagram of the principle of swelling and degradation of DPPG prepared from PEGDA of different molecular weights.

particle gel type, its swelling performance should be considered, and the molecular weight of the cross-linking agent in the system should also be paid attention to.

3.5. Mechanistic analysis of the role of molecular weight of cross-linking agents

This paper intended to analyze the influence mechanism based on the above experimental results of static and dynamic temporary plugging of DPPGs prepared by different molecular weights of cross-linking agents. Prior to the analysis, differential calorimetric scanning (DSC) analysis of the dried DPPG particles was performed using the NETZSCH differential calorimetric analyzer, and the experimental results are shown in Fig. 12.

As shown from Fig. 12(a), the heat flow rate of the dried DPPG samples was significantly different with increasing molecular weight of the cross-linking agent PEGDA when 0.01 g of the cross-linking agent was used for the preparation of DPPGs. With the increasing temperature, the DPPG-M5 sample first showed a significant exothermic change at around 245 °C. It shows that the chemical structure of the DPPG-M5 sample was firstly damaged, which is consistent with its macroscopic degradation phenomenon in Sections 3.2 and 3.3. With the further increase of temperature, DPPG-M4 was also followed by exothermic changes, and the last one was DPPG-M5. The destruction mentioned above the law of the chemical structure of DPPG samples is consistent with its macroscopic degradation law and the influence law of its dynamic temporary plugging effect in the core (Section 3.4). As the molecular weight of the cross-linking agent PEGDA in the DPPG sample increased, its chemical structure was more easily disrupted.

A schematic diagram of the corresponding reaction structure is given in Fig. 13. The degradation effect of DPPG is water-thermal dissolution. Specifically, the DPPG swollen to a certain number has a three-dimensional network structure, which makes it have good viscoelasticity and plugging ability. Subsequently, the cross-linked structure in DPPG (i.e., PEGDA) can be self-degraded. After the cross-linked structure is broken, the swollen DPPG changes from a three-dimensional network structure to a linear polymer (Zhu et al., 2021c), and the DPPG changes from a swollen particle state to a solution state, causing its viscosity to drop sharply. In addition, it can be seen that the DPPG particles prepared from cross-linking agents with higher molecular weights have more cross-linking sites, and each cross-linking agent can form multiple sites, so the microscopic network structure formed is denser. Therefore, when they swell in contact with water, their swelling volume and swelling rate significantly reduce, as shown in Figs. 5–8. Moreover, since the chemical structure of the DPPG prepared at the higher molecular weight of the cross-linking agent is more easily destroyed, the faster its degradation rate is, and the less time it takes to reach complete degradation. Besides, the weaker the gel strength after swelling, the weaker the plugging strength in the porous media of the core, and it is more likely to enter the porous media and cause harm to the formation permeability. To sum up, when designing a particle gel-type temporary plugging agent, we should select the cross-linking agent with moderate molecular weight to achieve suitable plugging strength, meet a reasonable degradation rate, and minimize its harm to the formation.

4. Conclusions

(1) By adjusting the molecular weight of the cross-linking agent PEGDA, a series of particle-type temporary plugging agents with controlled degradation time at low-temperature reservoir temperature (34 °C) can be prepared.

(2) The results of IR spectroscopy and DSC analysis show significant differences between the chemical structures and their thermal stability of DPPGs prepared by different molecular weights of cross-linking agents. The differences in chemical structures will affect their macroscopic temporary plugging properties. Moreover, as the molecular weight of cross-linker in DPPG samples decreases from 1000 to 200 Da, the chemical stability of its structure is more robust.

(3) The static evaluation results of particle gel DPPG show that the swelling volume and swelling rate of the DPPG after water absorption and swelling will gradually increase as the molecular weight of the cross-linker in the DPPG sample decreases, but its degradation rate will be slowed down accordingly.

(4) DPPG has good temporary plugging performance in cores, DPPGs prepared by cross-linking agents with smaller molecular weights has greater plugging strength and is less damaging to cores. However, the degradation rate is slower, and the time required to reach complete degradation is longer.

(5) The cross-linking agent PEGDA can be used to prepare temporary plugging agents because it can be self-breaking under formation conditions. However, its molecular weight can significantly affect the chemical structure, static swelling, degradation performance of DPPG, and even its temporary plugging performance in the core. For different reservoir conditions (e.g., reservoir temperature, brine salinity, etc.), screening of the molecular weight of the cross-linker is needed to obtain the best performance of a particle-type temporary plugging agent.

Acknowledgments

This work was supported by the Research Foundation of China University of Petroleum-Beijing at Karamay (No. YJ2018B02002 and XQZX20200010), the Natural Science Foundation of Xinjiang Uygur Autonomous Region (No. 2021D01E23 and 2019D01B57), the University Scientific Research Project of Xinjiang Uygur Autonomous Region (No. XJEDU2019Y067), the Xinjiang Uygur Autonomous Region Innovation Environment Construction Project (No. 2019Q025), and the Sichuan Province Regional Innovation Cooperation Project (No. 2020YFQ0036), and the CNPC Strategic Cooperation Science and Technology Project (ZLZX2020-01-04-04).

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