Passive microrheology for measurement of gelation behavior of a kind of polymer gel P(AM-AA-AMPS)

Hongbin Yang,[1] Wanli Kang,[1,2] Hairong Wu,[1] Zhe Li,[1] Yang Yu,[1] Yao Lu,[1] Liming Zhang,[1] Manying Wang,[1] Yuwei He[3]

[1]Enhanced Oil Recovery Research Institute, China University of Petroleum (Beijing), Changping District, Beijing 102249, China
[2]School of Petroleum Engineering, China University of Petroleum (East China), Qingdao, Shandong 266580, China
[3]Beijing LDS Technology Co. Ltd, Chaoyang District, Beijing 100101, China
Correspondence to: W. Kang (E-mail: kangwanli@126.com and kangwanli@cup.edu.cn)

ABSTRACT: One kind of polymer gel P(AM-AA-AMPS) was prepared by radical aqueous copolymerization, using acrylamide (AM), acrylic acid (AA) and 1-acrylanmido-2-methylpropanesulfonic acid (AMPS) as monomers, N,N-methacrylamide (MBA) as crosslinker and ammonium persulfate (APS) as initiator. The microstructure and molecular structure of the polymer gel were characterized by environmental scanning electron microscope (ESEM), infrared spectrometer (IR) and thermal gravity analysis (TGA). Main factors affecting the gelation behavior of P(AM-AA-AMPS) were qualitatively and quantitatively studied by multi-speckle diffusion wave spectroscopy (MS-DWS) technology, and the elasticity index (EI) and macroscopic viscosity index (MVI) were introduced to evaluate the elasticity and viscosity of the polymer gel. The results show that the synthesized P(AM-AA-AMPS) polymer gel has three-dimensional network structure gel with thermally resistant and salts tolerant groups. The EI and MVI of solution increase abruptly during the gelation time and the two indexes tend to stabilize. Under certain conditions, with the increase of reaction temperature and concentration of monomers and initiator, the gelation time of polymer gel gets shorter and the gel strength increases; with the increase of concentration of crosslinker, the strength of polymer gels increases, while the gelation time remains almost unchanged. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 43364.

INTRODUCTION

Polymer gel, with a three-dimensional cross-linked structure, was widely used in petrochemical, medical treatment and public health, agriculture, forestry, horticulture, architecture and other fields due to the good water absorption and water retention.[1–5] Particularly in recent years, polymer gel becomes more and more important in water shutoff and profile control with the development of oil and gas field.[6–9] Gelling behaviors of the polymer gel affect its swelling ability and plugging performance, on which experts have carried out a lot of researches.[10–15] The widely used evaluation methods in gel-forming properties are the viscosimetry method, strength code method and degree of vacuum breakthrough method.[16–24] Liu et al. studied the influence factors on the gelation behavior of HPAM-complex chromium gel through viscosimetry method, strength code method and degree of vacuum breakthrough method.[20] It was proved that with the increase of polymer mass fraction, crosslinking mass fraction and temperature, gelation time got shorter and BV increased; the gelation time and gel strength of complex chromium gel followed the law of the conventional gel, that is, the shorter the gelation time, the greater the gel strength is. Mortimer et al. defined the gelation time that is required for the elastic and viscous moduli to intersect.[21] Another definition for the gelation time is the time that is needed to reach specific gel strength.[22,23] Similarly, the point that corresponds to the maximum slope in the elastic modulus time curve has been defined as the gelation time.[24]

However, there are some limitations of the conventional methods, especially in the aspect of describing the gelation time accurately. One kind of convenient and accurate method to quantitatively evaluate the gelation time and gel strength is badly needed. Microrheology is a new domain of rheological methods in studying viscoelastic behavior of soft matters such as gels, suspensions, colloidal dispersions, emulsions at the micro length scale. Microrheology applies micron sized particles to measure the local deformation of a sample resulting from an applied stress or thermal energy. When microrheology measurements are performed by measuring the displacement of particles

due to the thermal energy, that is to say the Brownian motion, the method is called passive microrheology.[25] Multispeckle diffusion wave spectroscopy (MS-DWS) technology based on dynamic light scattering used in passive microrheology can overcome the shortcomings of conventional mechanical rheological measurements. The difference of microrheology measurements from traditional macrorheological characterization by rotational rheometors was that the measurement was carried on in the quiescent condition, and had no mechanical shearing force. It was suit for the measurement of particularly vulnerable samples, such as foam, weak gel, foam cement, yoghourt and cream. The results were the raw result without any modification. What's more, the microrheology measurements could get the zero-shear data. Lots of results showed that viscosities obtained by microrheology were in good correlation with the viscosities measured with conventional rheology.[25,26] MS-DWS Technology involves measuring mean displacement of tracer particles such as colloidal particles, droplets, fibers or crystallites contained in the material as a function of time. The plotting of the mean square displacement (MSD) of tracer particles versus decorrelation time provides information on the viscoelasticity of the sample when analyzed appropriately. MSD of tracer particles was detected in a stationary and non-contact measurement. The network structure of the sample could be deduced from the rheological properties by MSD curve. The sample was not deformed and destroyed in the process of measuring the gelation behavior of the polymer gel using microrheometer. In this sense, the gelation time and gel strength could be measured quickly and accurately using the passive microrheology.[27–29]

In this article, a kind of a polymer gel P(AM-AA-AMPS) with temperature and salt tolerance group was synthesized by radical solution polymerization. P(AM-AA-AMPS) is an promising water shutoff and profile control agent in the oil and gas field, especially the high temperature and salinity reservoirs. The effects of reaction temperature, monomer concentration, crosslinking agent concentration and initiator concentration on the gelation behavior of polymer gel P(AM-AA-AMPS) were studied from the perspective of microrheology. Furthermore, the morphology and molecular structures of polymer gel P(AM-AA-AMPS) were characterized by means of microscopic characterization methods. The purpose of this paper is to provide a new idea and method to study the gelation behavior of polymer gel by introducing microrheology.

EXPERIMENTAL

Materials

Acrylamide(AM), acrylic acid(AA), ammonium persulfate (APS), ammonium chloride (NH_4Cl), polyethylene glycol (PEG-200), sodium hydroxide (NaOH) and acetone (C_3H_6O) were all purchased from Sinopharm Chemical Reagent Co., Ltd(China). N,N'-methylenebisacrylamide (MBA) was obtained from Tianjin Kemi'ou Chemical Reagent Co., Ltd (China). Hydrolyzed polyacryamide (HPAM, relative molecular mass 27.4×10^6) was supplied by Dagang oilfield (China). Latex particles were supplied by Beijing LDS Technology Co., Ltd (China). Anhydrous ethanol was obtained from Xilong Chemical Co., Ltd (China). Acrylamide-2-methyl propane sulfonic acid (AMPS, industrial products) was purchased from XinQuan Chemical Co., Ltd (China). Deionized water was used for the preparation of all aqueous.

PREPARATION OF POLYMER GEL P(AM-AA-AMPS)

Polymer gel P(AM-AA-AMPS) was prepared by radical solution polymerization used in this paper. Typically, a certain quantity of AM, AA and AMPS monomers were added into a 250 mL beaker of deionized water and stirred for 40min. Meanwhile, nitrogen was pumped in to dissolve all the solids, and then a certain quantity of MBA, 15 wt % sodium hydroxide, 15 wt % ammonium chloride and polyethylene glycol were added into the mixed solution and stirred evenly. After that a certain quantity of 15 wt % APS was added to the above solution, stirring for 5min. Finally, the mixture was poured into a cylindrical glass cell and few drops of latex particles were added into the cylindrical glass cell. Shake the vial evenly. The vial was then put in the measuring unit of the micro-rheometer.

GELATION BEHAVIOR MEASUREMENT OF POLYMER GEL P(AM-AA-AMPS)

Gelation behavior measurements of polymer gel P(AM-AA-AMPS) were performed using an optical microrheometer (Formulaction, France). The prepared sample with a few drops of $1 \mu m$ tracer latex particles was kept in a cylindrical glass cell of 20 mL with a 25 mm diameter. The light was multiply scattered many times, by the latex particles in to the sample, leading to interfering backscattering waves (Figure 2). During the measurement, the photon free path should be less than 6000, and the test mode was full characterization, frequency ranging from 10^{-3} Hz to 10^3 Hz.

The thermal energy of particles in Brownian movement was the rheological mechanical stress in microrheology, while the strain was the MSD of particles movement per unit time. Latex particles are a kind of tracer particles, the particles are based on crosslinked polystyrene, with practically no swelling behavior in water based systems and the size is 1.01 µm with low polydispersity. The concentration is 10% w/v of the original solution. Usually, the final concentration of polystyrene particles in the sample is between 0.1 and 0.5% w/v. In this range, there is usually no influence of the particles on the sample viscoelastic properties. The surface contains no functional groups such as hydroxyl or amines, which means, there is no chemical interaction with the sample. MSD of the particles reflected the distance from the initial position at some point and the rheological properties of particles in the small local environment. Under certain conditions, polymer gel could gradually form a three-dimensional network structure, which was similar to a cage-type space. Latex particles could be embedded in that cage-type space. With the increase of decorrelation time, MSD curve can be divided into three stages (Figure 3)[30,31]: the first stage, the particles could move freely in the cage, the MSD grows linear with the increase of decorrelation time; the second stage, the particles are blocked by the cage wall, thus MSD curve hardly change with the increase of decorrelation time, forming a platform area, which is characteristic of the elasticity of the sample; the third stage, at longer time scales, the particles start to break the cage wall and diffuse into another cage-type space and the MSD grows as it would for a viscous fluid. This is
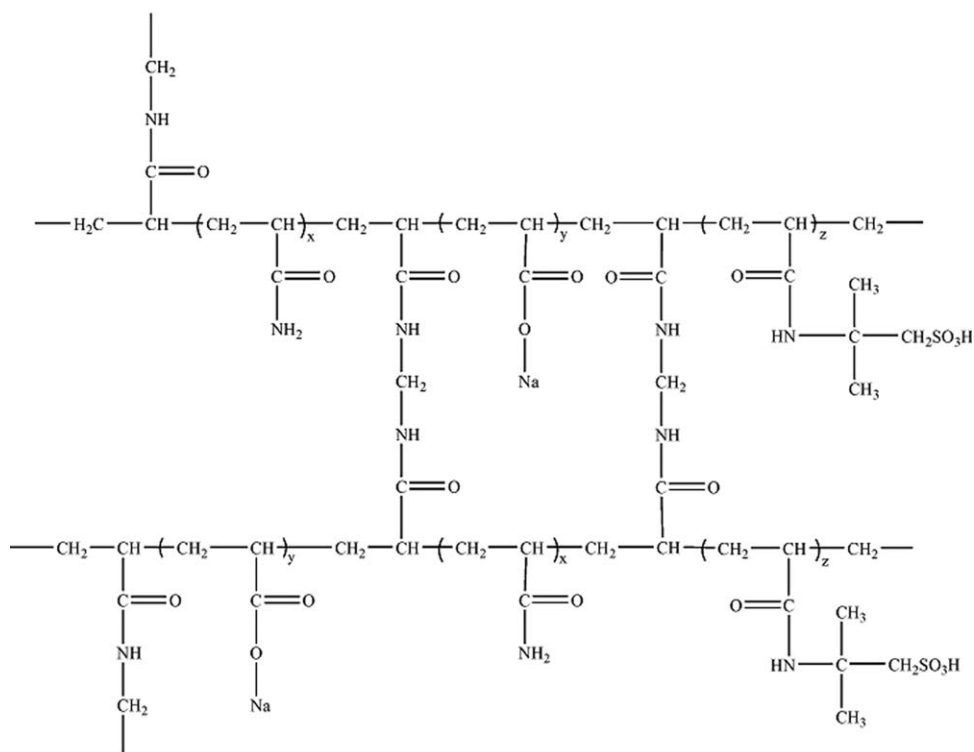
Figure 1. Chemical structural formula of polymer gel P(AM-AA-AMPS).

characteristic of the macroscopic viscosity, as it corresponds to the migration speed of the particles in the sample.

In summary, the MSD is the viscoelastic fingerprint of the analyzed sample. Elastic index (EI) is obtained by the reciprocal of MSD plateau value, corresponding to conventional elastic modulus G' to indicate the size of the flexibility. The lower the elastic plateau is, the bigger the EI is; macroscopic viscosity index (MVI) is the inverse of the slope in the third stage of MSD curve, which can be used to indicate the macroscopic viscosity of the sample. For a fixed distance, if the decorrelation time (t_{dec}) is longer, that means the tracer particle moves slower and the sample has a higher macroscopic viscosity. That is to say, the smaller the slop of MSD is, the bigger the MVI is.

MICROSCOPIC CHARACTERIZATION

Environmental scanning electron microscope (ESEM) images were taken by FEI Quanta 200 FEG (FEI Company, Holland) to observe the structure of polymer gel after swelling. The experiment conditions were as follows: HV=7KV, WD and PA current level.

Molecular structure of polymer gel P(AM-AA-AMPS) was characterized using NEXUS670 infrared spectrometer (NICOLET Company, USA) with a wave number range of 4000-400cm^{-1} and a resolution of 0.01cm^{-1}. The synthesized polymer gel was placed in the thermostat of 70 for 3 d, then shattered, purified, cleaned and dried. The high purity polymer gel powder was obtained. Finally the powder sample was prepared by potassium bromide tablet method. The infrared spectra of P (AM-AA-AMPS) were analyzed compared with HPAM.

Thermal Gravity Analysis of polymer gel P (AM-AA-AMPS) was carried out using TGA/DSC 1 simultaneous thermal analyzer (METTLER TOLEDO Company, Switzerland), with temperature range of 0~800°C and a heating rate of 10°C/min in an argon gas atmosphere.

Figure 2. Measurement principle of MS-DWS. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
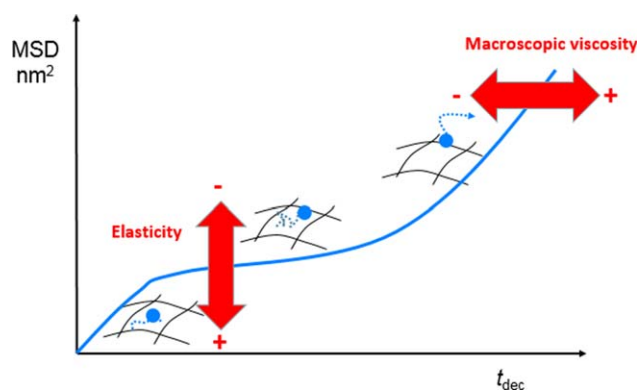
Figure 3. MSD schematic diagram of viscoelastic materials. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

RESULTS AND DISCUSSION

Microstructure

Figure 4 is ESEM image of polymer gel P(AM-AA-AMPS) after swelling. It can be observed that there is three-dimensional network structure inside the polymer gel. As the polymer gel contacts with water, the water molecules can go into the internal network under the osmotic pressure, stretch the molecular chain of the network structure and thereby enlarge the volume of polymer gel. The gel volume expands several times as compared to that of the dry gel. The three-dimensional network structure with pore sizes ranging from 1.29 to 2.95 μm can be seen clearly from the ESEM image. The investigation of microstructure confirms the swelling mechanism of the polymer gel.

Influential Factors on Gelation Behavior of P(AM-AA-AMPS)

In the measurement of gelation behavior of polymer gel P(AM-AA-AMPS) using microrheometer, EI, and MVI were used to evaluate the elasticity and viscosity of the polymer gel. Gelation time should be defined as the turning point, before which EI and MVI suddenly increase and become flat afterward, and gel strength should be defined as the relative stable value of EI and MVI. By the analysis and comparison of EI and MVI values under different experimental conditions, influential factors of reaction temperature, monomer, crosslinker, and initiator concentration on gelation behavior of polymer gels were studied systematically.

(1) **Reaction Temperature.** Reaction temperature mainly affects the polymerization rate, relative molecular quality and structure of polymers. Figure 5 shows EI curve and MVI curve of polymer gel P(AM-AA-AMPS) at different reaction temperature. As a contrast curve, EI and MVI curve of 1000 mg/L HPAM in Figure 5 is shown. EI and MVI stable values of samples after gelation are far greater than the values of HPAM solution, indicating the formation of polymer gel. Moreover, with the increase of temperature, the time of sudden rise of EI and MVI becomes shorter and EI and MVI stable value becomes larger. This indicates that the higher the reaction temperature is, the shorter the gelation time and the greater the gel strength are. APS is a thermal initiator. The polymerization reaction rate can be accelerated with the increase of reaction temperature. The

initiator can generate more free radicals, which will contribute to the chain growth and the increase the molecular weight of the polymer gel, thus the EI stable value of polymer gel after gelation increases. However, excessively high temperature can lead to rapid decrease of half-life of APS, so that the increase of chain initiation rate is much greater than that of the chain growth rate. Free radicals decomposition speed increases and a large number of free radicals jump over the barrier, making an increase of polymerization rate and a decrease of gelation time. EI and MVI show a sudden increase in shorter time. Furthermore, the side reaction is promoted while the polymerization rate is accelerated. It is shown in decline of polymerization degree, polymer molecular weight, and the gel strength.

(2) **Monomer Concentration.** Monomer ratio of AM:AA is always kept as 1:1 in the synthetic experiment. EI and MVI of different monomer concentrations are shown in Figure 6. It can be seen that with the increase of monomer concentration, the time needed to reach the sudden increase of EI and MVI decreases, while the EI and MVI stable values increase. That indicates that the gelation time of polymer gel decreases and the gel strength increases. This is because the typical redox free-radical polymerization could be divided into the elementary reaction of chain initiation, chain growth and chain termination.[26] With the increase of monomer concentration, the intramolecular or intermolecular crosslinking points increase and the radical active segment collision probability also increases. Thus, the crosslinking reaction occurs rapidly and the time needed to achieve the sudden increase of EI and MVI decreases. Meanwhile, the molecular weight of the formed polymer gel increases and EI and MVI stable values increase as well.

(3) **Crosslinker Concentration.** The crosslinker MBA with two identical and extremely lively reactive functional groups could change the structure of high molecular weight polymer from linear to three-dimensional network. The crosslinker concentration could affect the strength and viscoelasticity of polymer gel.
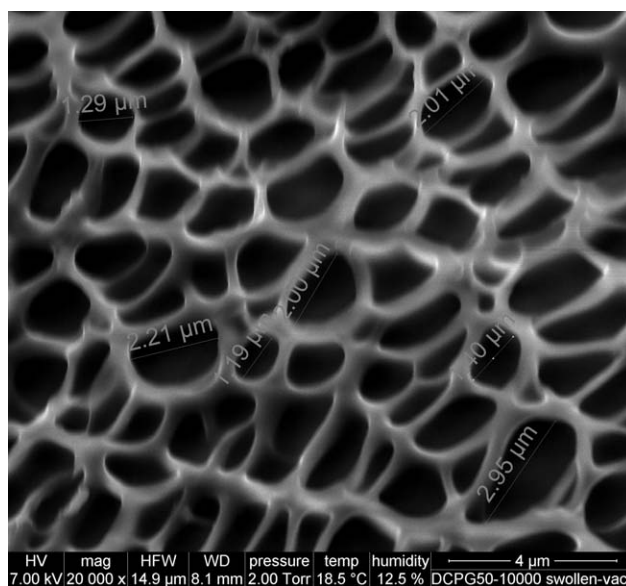

Figure 4. ESEM image of polymer gel P(AM-AA-AMPS) after swelling.
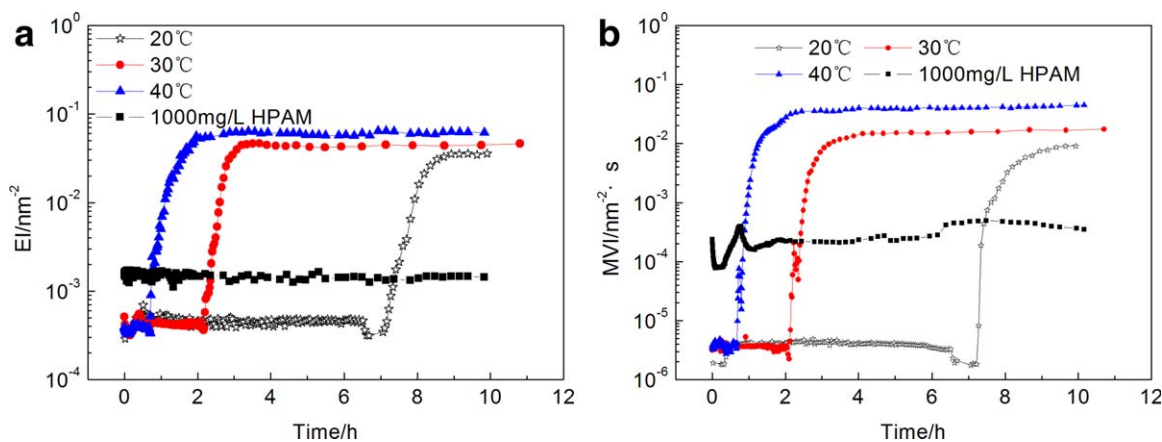
Figure 5. EI curve and MVI curve of polymer gel P(AM-AA-AMPS) at different reaction temperature. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

If the crosslinker concentration is too low, the polymer gels cannot crosslink effectively and form the network. When the crosslinker concentration is too high, the stretching motion of the polymer molecules and the rotational motion of molecular clew will be limited, and the structure of polymer gel can be damaged easily under external force. Figure 7 shows the EI curve and MVI curve of polymer gel P(AM-AA-AMPS) at different crosslinker concentrations. With the increase of crosslinker concentration, EI and MVI stable values increase and the time needed to reach the sudden increase of EI and MVI is almost unchanged. This indicates that the increased crosslinker concentration can improve the gel strength of polymer gel, but the gelation time keeps stable. This is mainly because the increased crosslinker can improve the intermolecular crosslinking density and form more network structure during radical polymerization. In the same volume, the individual space of polymer gel network structure decreases and the skeletal structure becomes more obvious. Therefore, EI stable value increases and gel strength increases as well. However, the crosslinker concentration does not affect the gelation rate. It is concluded that the crosslinker concentration does not have much effect on gelation time of polymer gel.

(4) Initiator Concentration. Initiator is a material that can generate active center in radical polymerization. Initiator concentration mainly affects the polymerization degree of monomers. The aggregate state of the initiator determines the structure of polymer gel, which has an impact on the gelation behavior of polymer gel. EI curve and MVI curve of polymer gel P(AM-AA-AMPS) at different initiator concentrations are shown in Figure 8. It can be seen that under the low initiator concentration conditions, with the increase of Initiator concentration, the time needed to reach the sudden increase of EI and MVI decreases, indicating that the gelation time becomes shorter. With the increase of initiator concentration, EI and MVI stable values increase suggesting that the gel strength increases. This is mainly because the higher initiator concentration can generate more free radicals and activity center, which will shorten the gelation time and enhance the gel strength. But extremely high initiator concentration could make initiation rate increase too fast and shorten the gelation time. According to kinetic chain length formula $\gamma \propto [I]^{1/2}$, the high radical concentration in the system could decrease the kinetic chain length and molecular weight of the polymer gel. This is not conducive to the formation of the three-dimensional network structure. As a result, gel
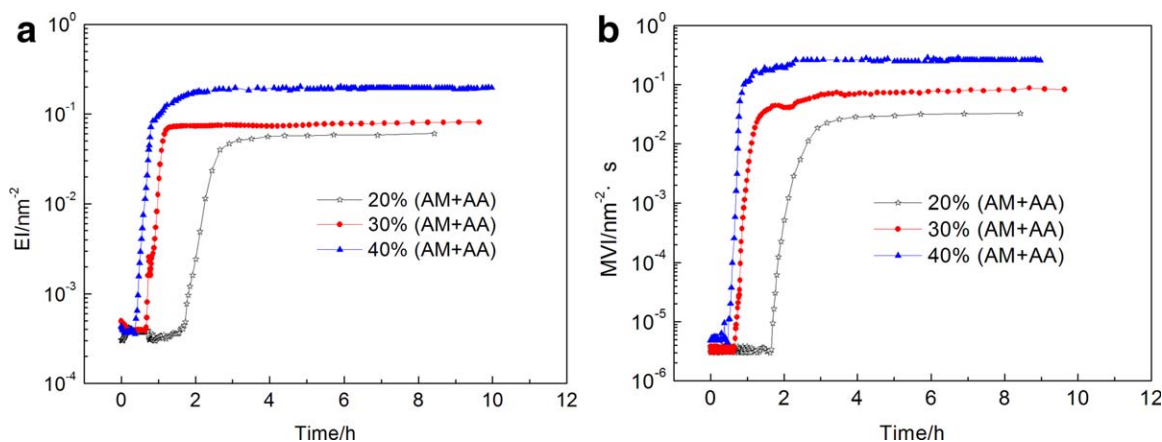

Figure 6. EI curve and MVI curve of polymer gel P(AM-AA-AMPS) at different monomer concentrations. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
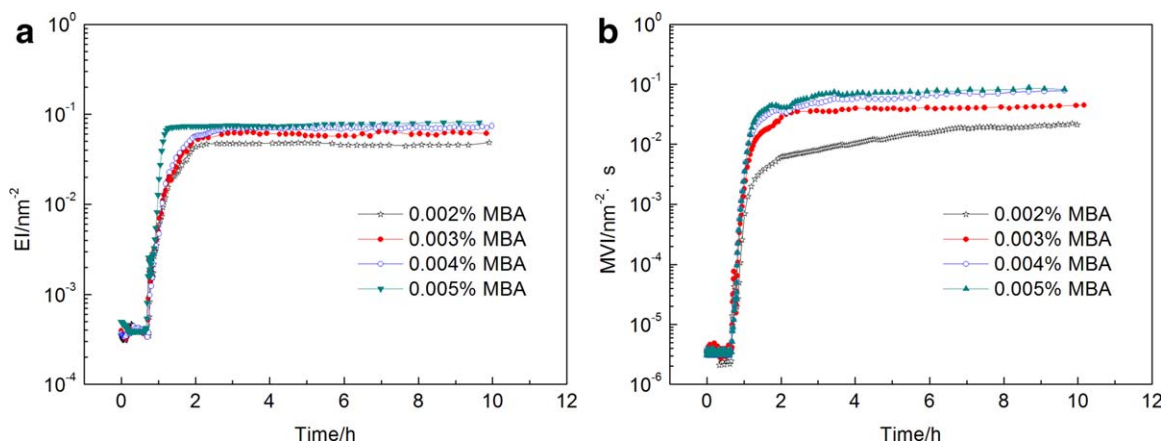
Figure 7. EI curve and MVI curve of polymer gel P(AM-AA-AMPS) at different crosslinker concentrations. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

strength of polymer gel decreases, as is indicated by the decrease of EI and MVI stable values.

To summary, we can conclude that the time needed to get the sudden increase of EI and MVI (gelation time) of polymer gel P(AM-AA-AMPS) decreases with the increase of reaction temperature, monomer and initiator concentration, and the EI and MVI stable values (gel strength) increase with the increase of reaction temperature, monomer, and initiator concentration. The following parts are the microstructure characteristics of a kind of polymer gel P(AM-AA-AMPS). The formula of P(AM-AA-AMPS) is: 30%(AM+AA) +5%AMPS+0.15%APS+0.003%MBA, and the reaction temperature of 40°C.

Microscopic Characterization
Infrared Spectroscopic Analysis. Figure 9 shows the FTIR spectra of HPAM and polymer gel P(AM-AA-AMPS). From the two FTIR spectra, it can be seen that the intensity of CN stretching vibration absorption peak ($1415 cm^{-1}$) and the intensity of an NH bending vibration peak ($1475 cm^{-1}$) of polymer gel P(AM-AA-AMPS) significantly decrease as compared with the FTIR spectrum of HPAM. It indicates that the amide group is partial hydrolysis, but it has not disappeared completely. Due to the carboxyl contained in acrylic acid and carboxyl hydrolyzed from

amide, the C=O inner bending vibration absorption peak appears near $1740 cm^{-1}$ and $1190 cm^{-1}$ is the –S=O stretching vibration absorption peak of the synthetic polymer gel. The FTIR spectrum analysis shows that the three kinds of monomer AM, AA, and AMPS are involved in the polymerization reaction.

Thermal Gravity Analysis. Figure 10 shows TG/TGA curves of polymer gel P(AM-AA-AMPS), which can be observed that the TG/TGA curves of polymer gel P(AM-AA-AMPS) can be decomposed into three stages. In the first stage, the temperature ranged from 25°C to 185°C and the weightlessness rate was 4.61%. This stage is a physical filtration process, which might be caused by the evaporation of adsorbed water of amide, carboxyl and sulfonic acid groups in polymer gel. In the second stage, the temperature varied from 185°C to 339°C while the weightlessness rate was 21.01%. This stage can be divided into two sub-parts, the weightlessness rate of first sub-part was 10.01%, which can be attributed to the elimination reaction of side groups such as amide and carboxyl groups in molecular chain of polymer gel; the weightlessness rate of second sub-part was 11%, and it can be attributed to the elimination reaction of sulfonic acid groups in molecular chain of polymer gel. In the third stage, the temperature ranged from 339°C to 476°C and
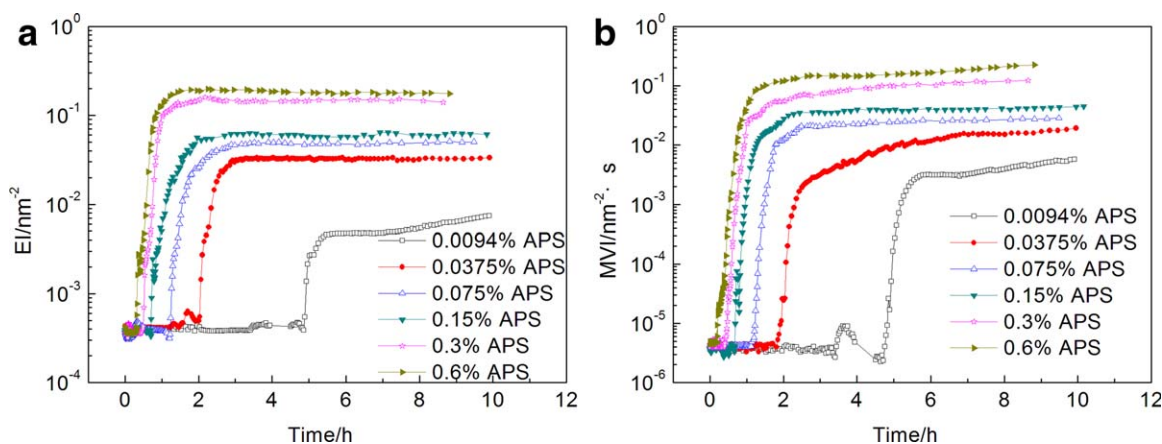

Figure 8. EI curve and MVI curve of polymer gel P(AM-AA-AMPS) at different initiator concentrations. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
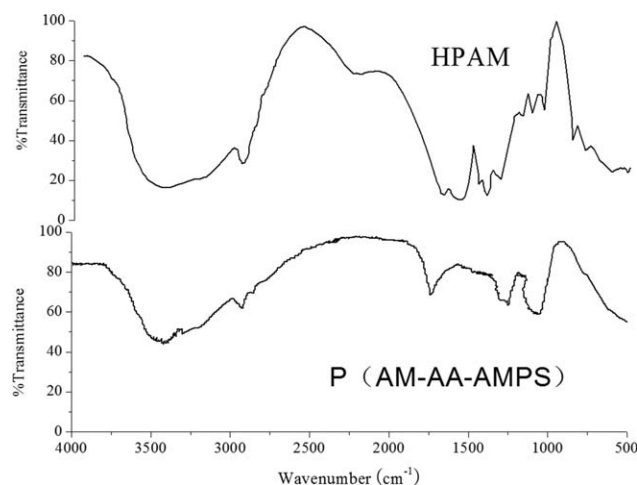
Figure 9. FTIR spectrums of HPAM and polymer gel P(AM-AA-AMPS).

the weightlessness rate was 50.35%, which can be attributed to the decomposition and rupture of the main chain of polymer gel. The TG/TGA data show that the polymer gel P(AM-AA-AMPS) displays a significant weight loss when temperature gets to 339°C, implying that the polymer gel P(AM-AA-AMPS) show temperature resistance to 339°C and has better thermal stability.

CONCLUSIONS

Gelling behavior is an important property of polymer gel in the application of profile control technology. In this work, one kind of polymer gel P(AM-AA-AMPS) with three-dimensional network structure was prepared by radical aqueous copolymerization, and the resisted temperature was up to 339°C. A novel evaluation method using passive microrheology has been put forward. The influential factors on elastic index and macro viscosity index of polymer gel P(AM-AA-AMPS) were studied by micro rheometer systematically. Under a certain condition, the higher the reaction temperature is, the higher concentration of monomer and initiator is, the shorter sudden increase of EI and MVI time and the gelling time is, the greater EI and MVI stable
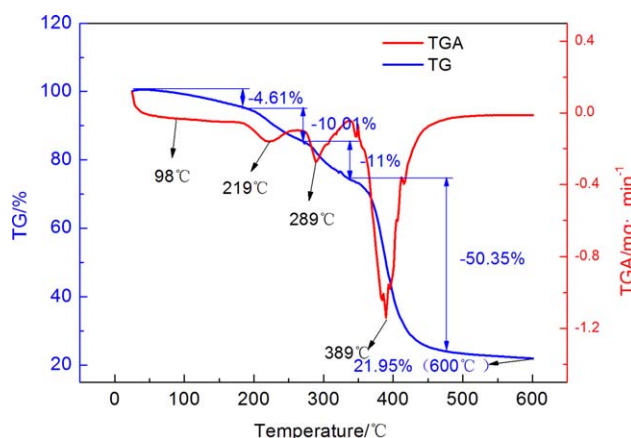
values and gelling strength are. The higher the concentration of crosslinker is, the greater EI and MVI stable values and gelling strength are, while the gelling time remained unchanged at the same time. This work provides a new evaluation method to investigate the gelling behavior of similar gels.

ACKNOWLEDGMENTS

REFERENCES

1. Thivaios, I.; Bokias, G. *J. Appl. Polym. Sci.* **2010**, *116*, 1509.

2. Bajpai, S. K.; Johnson, S. *React. Funct. Polym.* **2005**, *62*, 271.

3. Zou, X. X. Super Absorbent; Chemical Industry Press: Beijing, **2002**.

4. Hua, Z.; Lin, M. Q.; Guo, J. R.; Xu, F. X.; Li, Z. Y.; Li, M. Y. *J. Petrol. Sci. Eng.* **2013**, *105*, 70.

5. Cavallieri, A. L. F.; Cunha, R. L. D. *Food Hydrocolloid.* **2008**, *22*, 439.

6. Zhao, G.; Dai, C. L.; Zhao, M. W.; You, Q. *J. Appl. Polym. Sci.* **2014**, *131*, 2231.

7. Dai, C. L.; Zhao, G.; You, Q.; Zhao, M. W. *J. Appl. Polym. Sci.* **2014**, *131*, 4127.

8. Chen, Y.; Liu, Y.; Tan, H.; Jiang, J. *Carbohydr. Polym.* **2009**, *75*, 287.

9. You, Q.; Dai, C. L.; Tang, Y. C.; Guang, P.; Zhao, G. *J. Energy Resour. Technol.* **2013**, *135*, 1.

10. Adewunmi, A. A.; Ismail, S.; Sultan, A. S. *J. Appl. Polym. Sci.* **2015**, *132*, 4127.

11. Shibayama, M.; Ozeki, S.; Norisuye, T. *Polymer* **2005**, *46*, 2381.

12. He, H.; Wang, Y. F.; Zhang, J. *J. Disper. Sci. Technol.* **2015**, *36*. 633.

13. Nguyen, N. T. B.; Tu, T. N.; Bae, W.; Dang, C. T. Q.; Chung, T.; Nguyen, H. X. *Energ. Source Part A* **2012**, *34*, 1305.

14. Dadvand Koohi, A.; Zarringhalam Moghaddam, A.; Vafaie Sefti, M.; Mousavi Moghadam, A. *J. Macromol. Sci. A* **2011**, *50*, 1905.

15. Civan, F.; Vasquez, J.; Dalrymple, D.; Eoff, L.; Reddy, B. R. *Petrol. Sci. Technol.* **2007**, *50*, 353.

16. Winter, H.; Mours, M. *Adv. Polym. Sci.* **1997**, *134*, 170.

17. Tang, J. X.; Hai, Y. Z.; Sheng, H. Y.; Luo, J. M.; Huang, H. Q. *Chem. Eng. Oil Gas* **2013**, *42*, 392.

18. Bai, B. J.; Li, L. X.; Liu, Y. Z.; Liu, H.; Wang, Z. G.; You, C. M. *SPE Reserv. Eval. Eng.* **2007**, *10*, 415.

19. Zhao, M. Y.; Zhang, S. B.; Ding, S. B.; Ou, Y. J.; Zhao, Q. *Oilfield Chem.* **2010**, *21*, 69.

20. Liu, L. B.; Dai, C. L.; Zhao, J.; Zhao, G.; Lv, X. R.; Zhao, F. L. *Oilfield Chem.* **2010**, *27*, 153.

Figure 10. TG/TGA curves of polymer gel P(AM-AA-AMPS). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary. com.]

21. Mortimer, S.; Ryan, A. J.; Stanford, J. L. *Macromolecules* **2001**, *34*, 2973.

22. Sydansk, R. D. *SPE Adv. Technol. Ser.* **1993**, *1*, 146.

23. Te Nijenhuis, K.; Mensert, A.; Zitha, O. L. *J. Rheol. Acta* **2003**, *42*, 132.

24. Prud'homme, R. K.; Uhl, J. T. SPE12640 Presented at SPE Enhanced Oil Recovery Symposium, **1984**.

25. Bellour, M.; Skouri, M.; Munch, J. P.; Hebraud, P. *Eur. Phys. J. E* **2002**, *8*, 431.

26. Mason, T. G. *Rheol. Acta* **2000**, *39*, 371.

27. Chen, Y. Q.; Chou, P. L.; Cheng, C. Y.; Chiang, C. C.; Wei, M. Z.; Chuang, C. T.; Chen, Y. S.; Chiou, A. *J. Biophotonics* **2012**, *5*, 777.

28. Chen, Y. Q.; Kuo, C. Y.; Wei, M. T.; Wu, K.; Su, P. T.; Huang, C. S.; Chiou, A. *J. Biomed. Opt.* **2014**, *19*, 61.

29. Selvaggi, L.; Salemme, M.; Vaccaro, C.; Pesce, G.; Rusciano, G.; Sasso, A.; Campanella, C.; Carotenuto, R. *Methods* **2010**, *51*, 20.

30. Tisserand, C.; Fleury, M.; Brunel, L.; Bru, L.; Meunier, G. *Prog. Colloid Polym. Sci.* **2012**, *139*, 101.

31. Sarmiento-Gomez, E.; Montalvan-Sorrosa, D.; Garza, C.; Mas-Oliva, J.; Castillo, R. *Eur. Phys. J. E* **2012**, *35*, 1.