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Organic gels for fluid control in hypersaline and high-temperature oil reservoirs

Geles orgánicos para el control de fluidos en yacimientos petrolíferos hipersalinos y de alta temperatura

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Abstract

Rigid but flexible polymer gels were formed by crosslinking an ionic terpolymer with acrylamide, vinylpyrrolidone, and ionic groups with hexamethylenetetramine, resorcinol, and amino acids (alanine or glycine) to investigate water control in porous media. The rheological properties of the gels were evaluated at 160 °C, achieving storage modulus values between 300 and 1000 Pa. Fluid control experiments were conducted in a Berea-sandstone fractured core saturated with oil at 130 °C. The injection procedure consisted in introducing a 0.5 PV batch of the gel formulation, followed by a 91-h aging period to allow *in situ* gel formation. The fluid control efficiency of the gel formed within the porous medium was determined from differential pressure data. Efficiency greater than 90 % was obtained for all the evaluated brine injection rates (1, 3, and 60 mL/h).

Keywords: Ionic polymer, Berea sandstone, Water control, Bulk gels, Oil well.

Resumen

Geles poliméricos rígidos pero flexibles fueron formados por la reticulación de un terpolímero iónico con grupos acrilamida, vinilpirrolidona y grupos iónicos, con hexametilentetramina, resorcinol y aminoácidos (alanina o glicina) para investigar el control del agua en medios porosos. Las propiedades reológicas de los geles fueron evaluadas a 160 °C, alcanzando el módulo de almacenamiento entre 300 - 1000 Pa. Se llevaron a cabo experimentos de control de fluidos en un núcleo fracturado de arenisca de Berea saturado con petróleo, a una temperatura de 130 °C. El procedimiento de inyección consistió en introducir un lote de 0.5 volúmenes de poro de la formulación de gel, seguido de un período de envejecimiento de 91 h para permitir la formación del gel *in situ*. La eficiencia de control de fluidos del gel formado dentro del medio poroso se determinó a partir de los datos de presión diferencial. Se obtuvo una eficiencia superior al 90 % para todos los caudales de inyección de salmuera evaluados (1, 3 y 60 mL/h).

Palabras clave: Polímero iónico, Arenisca Berea, Control de agua, Geles, Pozo petrolero.

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1 Introduction

Water control in oil wells and reservoirs can be optimized by applying water control agents such as gels, which act as a barrier to fluids, allowing control of water production and at the same time making oil production more efficient. (Cuenca *et al*., 2016; He *et al*., 2015; Sun *et al*., 2020). To control this problem, various types of polymer-based gels have been chosen, such as *in situ* bulk gels, preformed particle gels (PPGs), and pH and temperature-sensitive microgels. (Afsharpour *et al*., 2024).

Bulk gels, formed from cross-linked polymers, are an important treatment for water control. Among the advantages of using bulk gels are the reduction of effective water and oil permeability, high adhesion to porous media, and treatment costs that are lower than those of PPGs (Sun *et al*., 2020; Yin *et al*., 2022). The gelling system, based on an aqueous polymer solution with crosslinking agents, when injected into a porous medium under specific temperature, pH, and salinity conditions, forms a three-dimensional network, which acts as a physical barrier to the passage of fluids (Yadav & Mahto, 2013). Most thermally stable polymer gels are organically crosslinked; examples of these crosslinking agents include hexamethylenetetramine (HMTA) with phenol, resorcinol (RE) or hydroquinone (HQ), and polyethyleneimine; these molecules are considered indispensable for the successful crosslinking process (Sengupta *et al*., 2012; Yin *et al*., 2022). Organically crosslinked gels have replaced inorganically crosslinked gels due to their better thermal stability, higher modulus of elasticity, lower sensitivity to lithology, lower cost, lower toxicity, and ease of execution and control of the placement process in the porous medium due to their flexibility in gelation time during field operations (Liu *et al*., 2016; Sengupta *et al*., 2012; Xu, 2017; Yadav & Mahto, 2013). Polymeric gels exhibit covalent chemical interactions, as well as physical (intermolecular) interactions that promote the formation of a stable gel in high-temperature reservoirs (Aliabadian *et al*., 2019; Pozo Cochea, 2020). To control fluids in the porous medium, plugging agents are injected into the high permeability channels present in the reservoir. Today, the systems that predominate are those with controllable gel time, cost-benefit ratio, and resistance (pH, temperature, or salinity) (Amir *et al*., 2019; Arndt & Gerlach, 2010; Sun *et al*., 2020).

Gels, whose three-dimensional network is formed by a crosslinked organic polymer with formaldehyde-based polyphenolic resins or HMTA with phenol or HQ or RE, usually exhibit high gelation performance and high thermal stability (Liu *et al*., 2016; Velazquez-Pineda *et al*., 2025). Even if the polymer network is composed of a polymer such as partially hydrolyzed polyacrylamide, the thermal resistance of the formed gel reaches up to 140 °C (Han *et al*., 2024; X. Li *et al*., 2024; Z. Li *et al*., 2020; Lu *et al*., 2024; Martínez-Vázquez *et al*., 2007; Qiao *et al*., 2022; Zhao *et al*., 2015); for this reason, it has been attempted to reinforce gels by employing graphene oxide nanoparticles (W. Li *et al*., 2025). Copolymers based on polyacrylamide with poly(2-acrylamido-2-methyl-1-propanesulfonic acid) or terpolymer based on polyacrylamide / polyvinylpyrrolidone /poly(2-acrylamido-2-methyl-1-propanesulfonic acid) are stable up to 140 °C and have considerable tolerance to salinity (Bryant *et al*., 1997; Z.-J. Li *et al*., 2024; Yang *et al*., 2025); in order to improve the stability of AMPS-based gels under high salinity conditions at 120 °C, cellulose nanofibers have been added (L. Dai *et al*., 2025; L.-Y. Dai *et al*., 2025). There are also studies on gels based on natural polymers such as guar gum and silicon and bentonite nanoparticles (Dehkohneh *et al*., 2024). In this context, the following conventional gel systems for water-blocking in oil wells have been successfully applied: Schlumberger offers the Organo SEAL gel, a crosslinked gel with metallic salts that seals abandonment zones in oil wells (Schlumberger, 2026); Halliburton has several solutions such as WaterWeb, a polymer-based permeability modifier with high rock adsorption and immediate response without requiring gelation time (up to 163 °C); EquiSeal^{TM}, a rigid organic gel used to lock out water in horizontal or deviated wells (between 60 and 121 °C); and H_2Zero^{TM}, an organic gel with 90 % efficiency even after a year at 143 °C (Halliburton, 2026). However, these water-blocking efficiencies are reported for low-salinity water conditions. However, the search for gel formulations that are stable at temperatures above 150 °C and tolerant to hypersaline formation water (total dissolved solids (TDS) >100 g/L) (Olivares-Xometl *et al*., 2024), which also include divalent cations, remains relevant, especially for the Mexican oil industry (Velazquez-Pineda *et al*., 2025).

Some Mexican oil fields, such as Jujo-Tecominoacán, have formation water salinities up to 385 g/L TDS and temperatures up to 158 °C (Birkle *et al*., 2009). The Grijalva Delta has temperatures around 150 °C and is hypersaline (TDS >120 g/L) (Pérez *et al*., 2015), as well as the Activo Luna Oil Field (Birkle *et al*., 2002, 2009) and Yaxche field (Rios & Roegiers, 2012; Rosales *et al*., 2006), etc.

Based on the foregoing, the goal of this work is to develop rigid, but flexible or resonant bulk gels, stable at temperatures up to 160 °C and formed via organic crosslinking with controlled gelation time. The gel formed *in situ* from the porous medium, represented by the Berea sandstone core, saturated with oil from a Mexican field, was formulated to

tolerate hypersaline brine and temperatures up to 130 °C, effectively blocking the fluids.

2 Experimental part

2.1 Gelling system formulation and gel characterization

Hexamethylenetetramine (HMTA), resorcinol (RE), DL-alanine, glycine, and sodium bisulfite were all at 99 % purity obtained from Merck and used as received. The terpolymer Polyeca^{TM} with molecular weight of 590,140 g/mol and thermal stability greater than 295 °C, determined by high-resolution thermogravimetric analysis (TGA) (Figure 1), was provided by the Mexican Petroleum Institute (Guzmán-Lucero *et al.*, 2022, 2024; Velazquez-Pineda *et al.*, 2025). The terpolymer structure, containing acrylamide, N,N´-methylenebisacrylamide, vinylpyrrolidone, and vinylimidazolium groups, was confirmed by ^{1}H NMR performed on a Bruker Advance Neo 600 MHz spectrometer, using D_2O as a solvent, (Figure 2). To prepare the gelling systems, 4.10 cm^3 of Polyeca^{TM} aqueous solutions (at 1, 2, 3, or 5 wt.%) were placed in quartz vials. Subsequently, 0.20 cm^3 of 10 wt.% HMTA solution, 0.70 cm^3 of RE solution, and 0.25 cm^3 of 1 wt.% sodium bisulfite solution were added. For specific systems, 0.5–1 cm^3 of a 10 wt.% alanine or glycine aqueous solution was also included. The mixture pH was adjusted to 2, 3, or 4 using 1.0 M hydrochloric acid (up to 0.8 cm^3). Dissolved oxygen was removed by bubbling nitrogen for 20 min, after which the vials were hermetically sealed. The sealed vials were placed in an oven (BINDER Model ED 720) at 160 °C to monitor the gelation process over time; all tests were performed in duplicate. Gel strength development was evaluated using the Sydansk bottle test method (Sydansk & Moore, 1992), establishing the relationship between the gel code and its strength description. Rheological tests were performed at 25 °C using a parallel-plate geometry with a 20-mm diameter and a 1-mm gap in an Anton Paar rheometer. The strain sweep test was conducted to assess the structural integrity of the synthesized hydrogels at a constant frequency of 1 Hz, with strain ranging from 0.1% to 1000%. The microstructure of the gel formed after 24 h at 160 °C, and subsequently dried for 24 h at 70 °C, was analyzed using the environmental scanning electron microscopy (ESEM) technique in an environmental scanning electron microscope PHILIPS XL-30, which can generate images in wet mode at high vacuum level, with 1KV-30KV range and 10X-100,000x magnification. TGA was performed by means of a Netzsch STA 409 piece of equipment in nitrogen atmosphere at a heating rate of 5 °C/min,

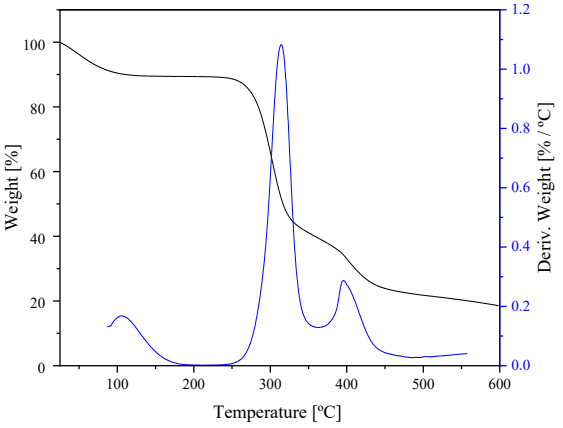

Figure 1. TGA of the terpolymer Polyeca^{TM}.

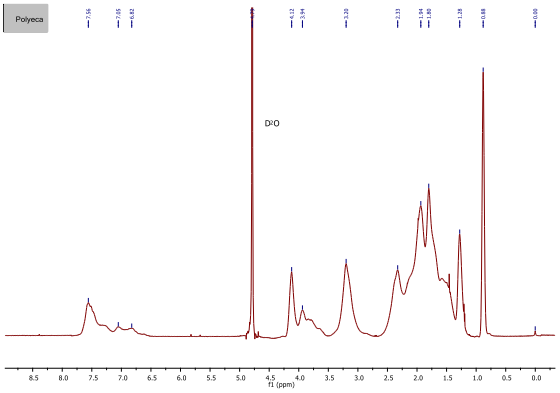

Figure 2. ^{1}H NMR spectrum of the terpolymer Polyeca^{TM}.

employing a gel sample dried at constant weight at 70 °C. Likewise, the dry sample of formed gel was submitted to FTIR analysis on a Thermoscientific Nicolet 8700 Spectrometer piece of equipment.

2.2 Fluid control test

The experimental procedure followed these stages:

Preparation: A Berea Sandstone^{TM} core (Cleveland Quarries) was utilized as the porous medium. The cleaned and dried core (length = 0.05 m, diameter = 0.124 m) exhibited porosity of 22 % and water permeability 126 mD. After inducing a longitudinal fracture (Figure 3), the core water permeability reached 154 mD. The displacement system was then heated to 130 °C under constant confinement pressure of 3000 psi (Figure 4). Finally, Mexican crude oil (20.1 °API, density of 0.9317 g/cm^3 and dynamic viscosity of 544 mPa.s) was injected to reach connate water saturation (12.5 %).

Figure 3. Fractured sandstone rock core before the fluid control test.

Secondary Recovery: the synthetic brine (250,000 ppm TDS: 150,000 ppm NaCl and 100,000 ppm $CaCl_2$), serving as the displacement fluid, was injected at 1 cm^3/h for at least 5 pore volumes (PVs) until reaching residual oil saturation.

Gel Treatment: A 0.5 PV slug of the gel formulation was injected. The system was then shut-in for 91 h at 130 °C and 3000 psi to allow for gelation.

Evaluation: Brine injection was resumed at different flow rates. The differential pressure was monitored to calculate the fluid control percentage using Equation 1 (Jennings *et al*., 1971; Wu *et al*., 2014):

$$\%E = (\Delta P1 - \Delta P0) * 100\ \%/\Delta P1 \qquad (1)$$

where $\Delta P1$ is the differential pressure reached by the injected synthetic brine and steady flow conditions were established through the porous medium; in this case, $\Delta P0$ is the differential pressure in the core before the formation of the gel *in situ*.

3 Results and discussion

The strength of the gel, its gelling time, is directly related to its composition. The polymer is a crucial component in the polymer gel system. Using a water-soluble terpolymer with long chains and molecular weight around half a million g/mol, which is not excessively high, containing ionic charges and thermal stability up to 295 °C (Figure 2), results in a good combination for forming a dense cross-linked network. The strength of the formed polymer gels also depends on the crosslinking agents. The main organic crosslinking agents are polyethyleneimine and polyphenolic resin, especially for polymers other than hydrolyzed polyacrylamide.

The gel strength of the crosslinking system composed of terpolymer/HMTA/RE with a different pH setting (2, 3, and 4) is shown in Figure 5. The gels, which consisted of 1 or 2 wt.% terpolymer (while the concentration of HMTA and RE was fixed) with pH adjusted up to 2 with 0.1 M hydrochloric acid, achieved the longest gelation time, up to 3 h and the best gel strength; according to the Sydansk code, the letter "I" was assigned. Although gels formed with 3 wt.% terpolymer and pH 4 gelled in 1 h, they, nevertheless, exhibited cavities in their structure, while gels formed with lower terpolymer concentration at pH 2-3 and longer gelation times (2-3 h) exhibited a more solid structure (Figure 5).

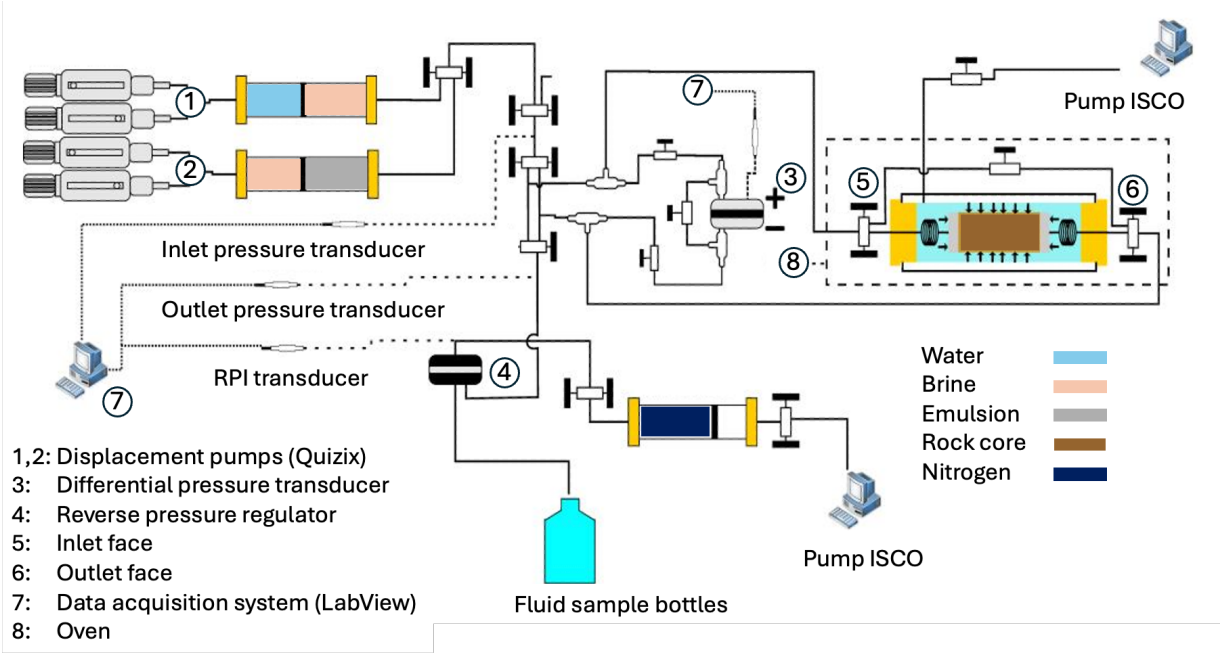

Figure 4. Experimental setup of a rock core injection system.

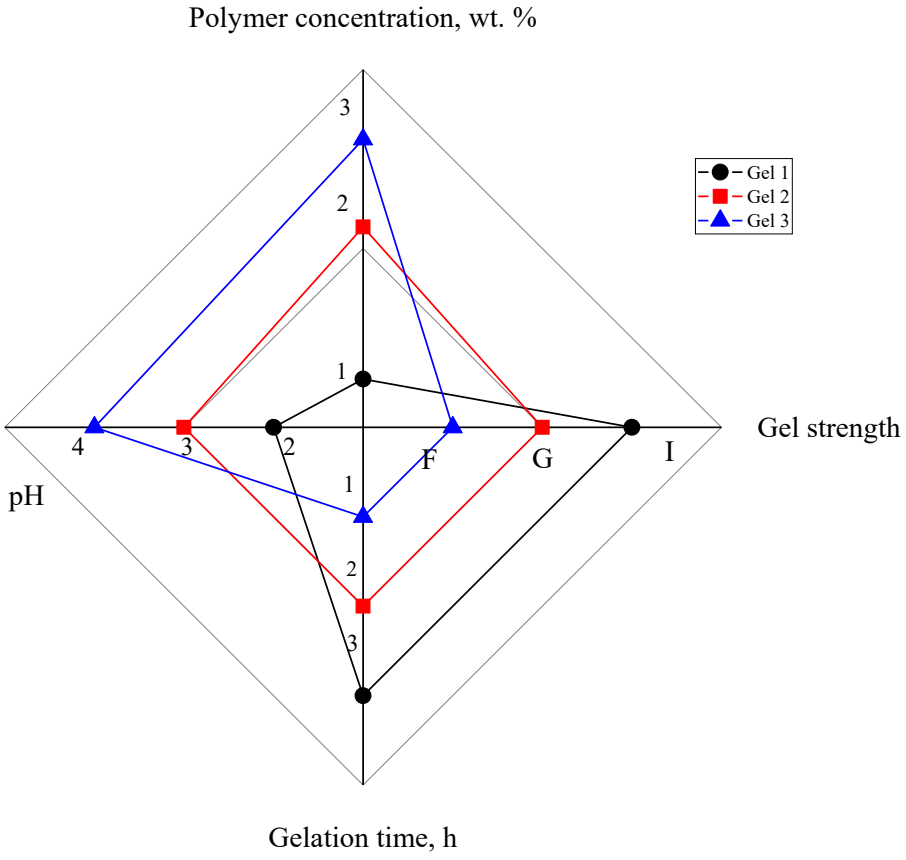

Figure 5. "Blank" gels prepared with different terpolymer concentrations and pH: (a) Gel 3 at pH 4; (b) Gel 2 at pH 3; (c) Gel 1 at pH 2.

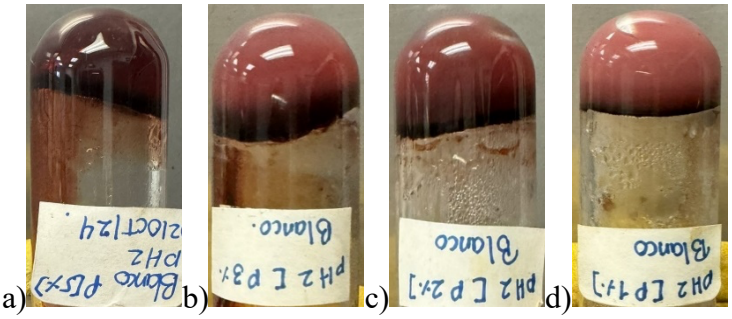

Figure 6. Appearance of "blank" gels prepared at pH 2 with different terpolymer concentrations: (a) 5 wt.%; (b) 3 wt.%; (c) 2 wt.%; (d) 1 wt.%.

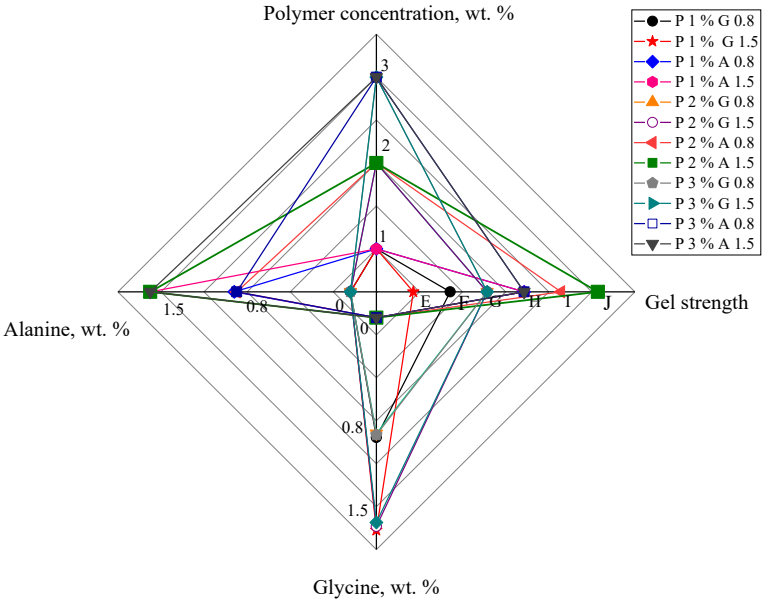

Figure 7. Gels prepared with different terpolymer (P) concentrations at pH 2 with addition of alanine (A) or glycine (G).

Based on the obtained results, the three gel formulations were prepared at four terpolymer concentrations (1-5 wt.%) at pH 2 (Figure 6), and their gelation times were measured. The gels with 1 wt.% and 2 wt.% terpolymer were formed at 160 °C in 3 h, while the gels with 3 and 5 wt.% terpolymer were formed in 2 h. Therefore, it was advisable to use the formulations with 1 or 2 wt.% terpolymer at pH 2 because the gelation time was 3 h, which was sufficient time to place the gelling system in a desired interval of the oil well.

Taking into account the results presented in Figure 6, it was decided to improve the composition of the gelling system to seek the greatest gel strength and deformation resistance so that it was of flexible type. For this purpose, in order to increase the crosslinking process, amino acids such as glycine or alanine were added to the gelling formulation, and its pH was adjusted to 2. As shown in Figure 7, the composite crosslinking system formed by the terpolymer at 2 wt.% and alanine at 1.5 wt.% (in addition, HMTA, RE and sodium bisulfite), as described in part 2.1 "Gelling system formulation and gel characterization" of this article, proved suitable for forming the rigid but flexible gel, to which it was possible to assign the letter "J" according to the Sydansk scale.

Despite the addition of the amino acid glycine to the gelling formulation, rigid but flexible gels were formed, especially when the glycine concentration was 1.5 wt.%, however, the gels exhibited imperfections in their structure, such as fractures and cavities (Figure 8).

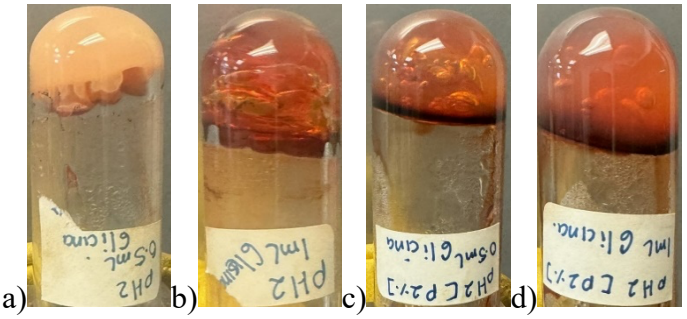

Figure 8. Appearance of the gels formed at 160 °C and pH 2 with the addition of glycine and the following percentages of terpolymer (P)/glycine (G) in the formulation: (a) P1%G0.8; (b) P1%G1.5; (c) P2%G0.8; (d) P2%G1.5.

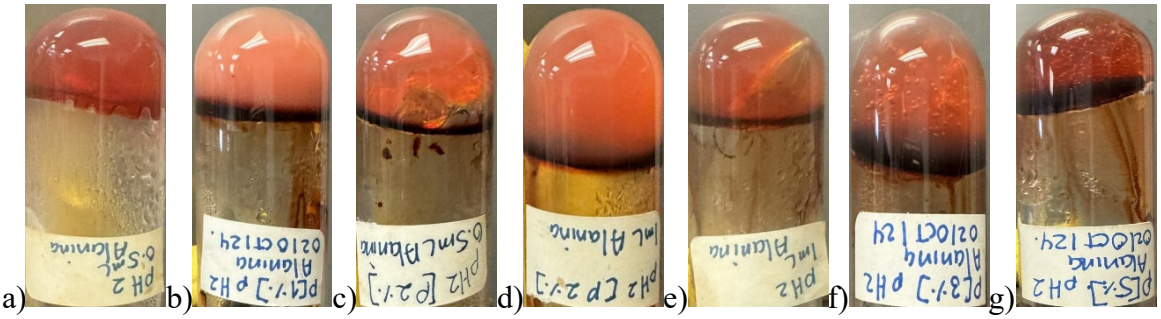

Figure 9. Appearance of the gels formed at 160 °C and pH 2 with the addition of alanine and the following percentages of terpolymer(P)/alanine (A) in the formulation: (a) P1%A0.8; (b) P1%A1.5; (c) P2%A0.8; (d) P2%A1.5; (e) P3%A0.8; (f) P3%A1.5; (g) P5%A1.5.

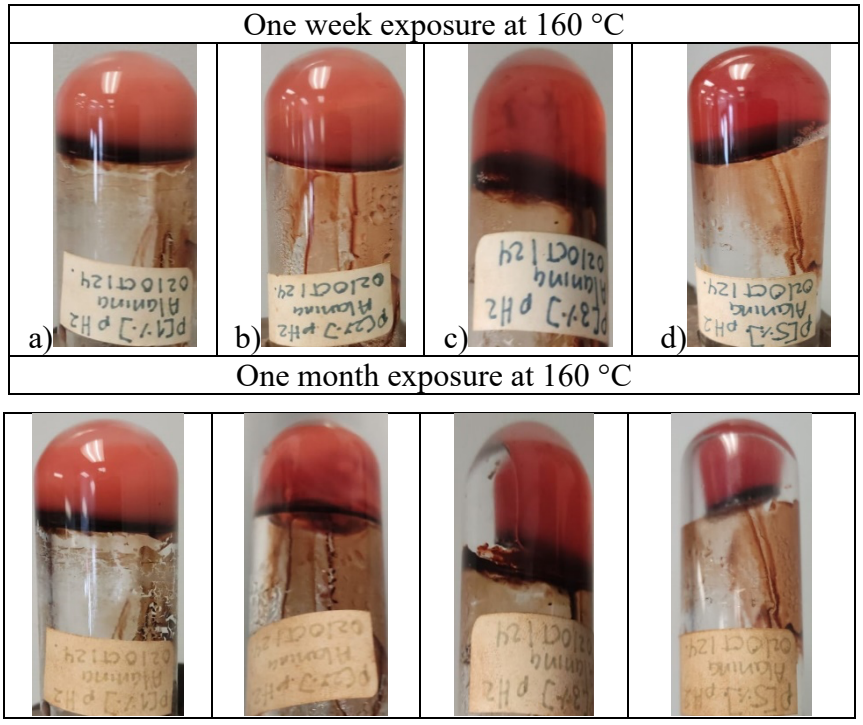

Figure 10. Change in appearance of gels with 1.5 wt.% alanine exposed to a temperature of 160 °C for one week and one month, with the following percentages of terpolymer(P) in the formulation: (a) P1%; (b) P2%; (c) P3%; (d) P5%.

The addition of alanine to the gelling formulation promoted the formation of rigid, but flexible and transparent gels (Figure 9), where with the increase in alanine concentration, the gels became more flexible, notwithstanding, the increase in terpolymer concentration greater than 2 % did not significantly influence the increase in strength of the formed gel.

Gels with 1 and 2 % terpolymer and alanine showed good thermal stability for 1 month at 160 °C, while gels with 3 and 5 % terpolymer in their composition exhibited water expulsion and gel shrinkage (Figure 10).

The 2% terpolymer and 1.5% alanine resulted in a good combination for forming a rigid gel with an internal cross-linked network structure, but flexible enough not to be brittle, and in turn with a gelation time of 3 h (Figure 11).

For use in porous reservoirs, gels must retain their viscoelastic properties, as they maintain in pores under high pressure and temperature, and withstand continuous, prolonged deformation (L. Dai *et al*., 2025; Zhou, Kang, *et al*., 2025; Zhou, Li, *et al*., 2025; Zhou, Zhao, *et al*., 2025). Therefore, the analysis of rheological properties of the samples is essential. During the deformation sweep test, the elastic (G') and viscous (G'') moduli were evaluated over a deformation interval ranging from 0.1 % to 1000 %, maintaining a constant frequency of 1 Hz. Figure 12 shows the linear viscoelastic region (LVR) of the alanine, glycine, and terpolymer (blank) crosslinked

gel, in which the modulus remained constant up to strains of 100, 27, and 5%. Polymer networks lacking alanine (P2%A) and glycine (P2%G) may not provide sufficient resistance, leading to structural weakness and hydrogel failure at strains exceeding 5% (P2%). In all cases, the elastic modulus was always greater than the viscous modulus, indicating the formation of a three-dimensional crosslinked network caused by chemical bonds between the functional groups of the terpolymer and crosslinking agents. The gels obtained by crosslinking with alanine and glycine showed higher elastic moduli than the terpolymer without these two agents. The alanine and glycine gels reached 2580 Pa and 738 Pa, respectively, compared to 512 Pa for the gel without crosslinking agent. In addition, the intersection of the curves when G'=G' indicates fluid-like behavior and represents an irreversible change in the gel; this is far more pronounced for the gel formed with alanine, where the crossover point occurs at 629%.

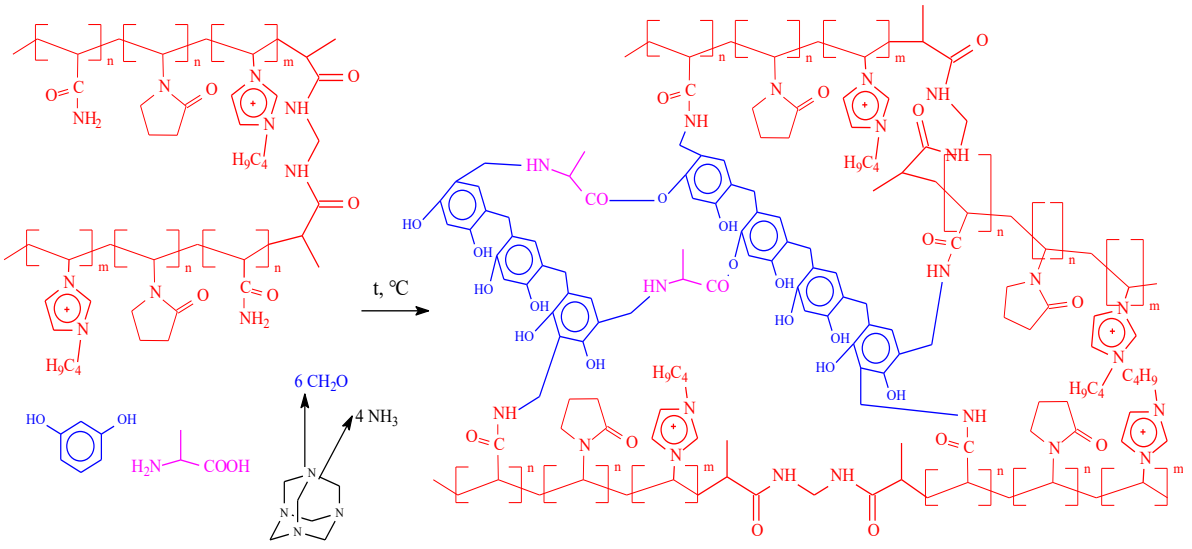

Figure 11. Schematic illustration of the polymer gel network.

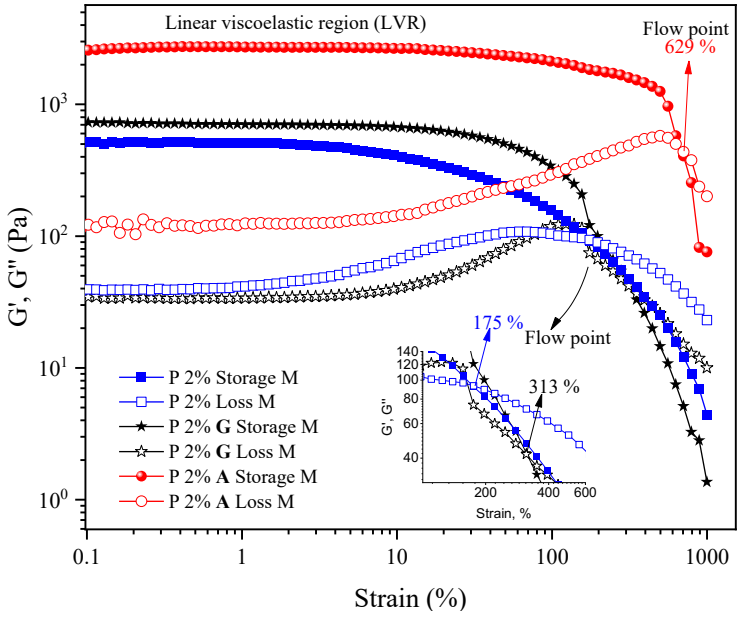

Figure 12. Gel strength: blank (P2%), with 1.5 % of alanine (P2%A) or 1.5 % of glycine (P2%G).

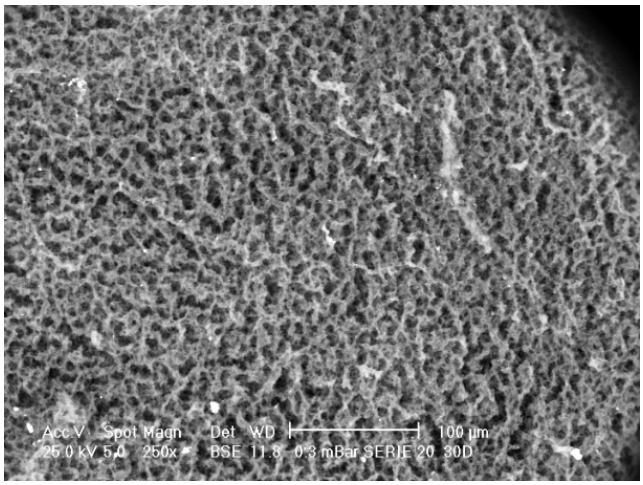

Figure 13. ESEM micrograph of the gel with 1.5 % of alanine (P2%A).

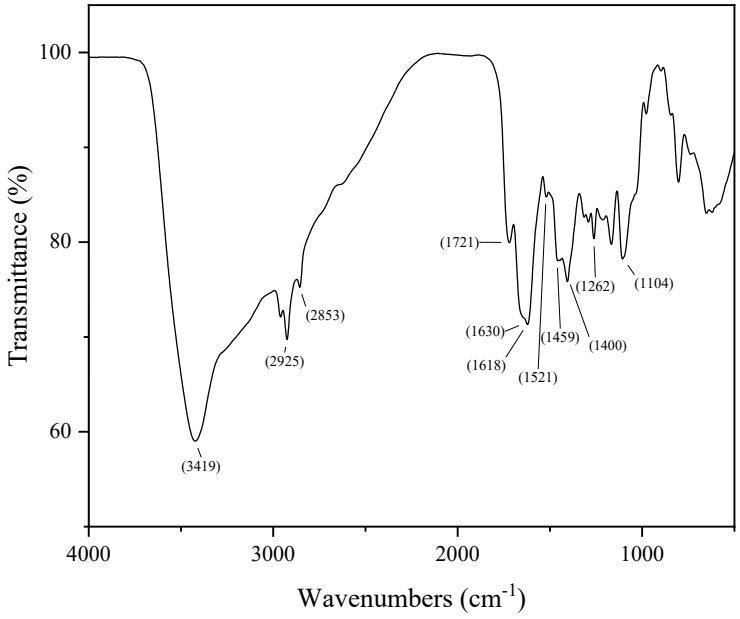

Figure 14. FTIR of dry gel with 1.5 % of alanine (P2%A).

ESEM was used for studying the microstructure of the gel systems. The micrograph is shown in Figure 13; it is worth noting that the gel sample exhibited a uniform three-dimensional structure, which can provide excellent stability to the swelling gels. It can be concluded that gels crosslinked with alanine and glycine have more compact structures than gels without these crosslinkers, thereby providing greater stability to the gelling systems.

Figure 14 shows the FTIR spectrum of the dry gel sample (P2%A), where the methylene groups of the polymer chain exhibit asymmetric stretching vibrations at 2925 and 2853 cm^{-1}. The acrylamide part of the polymer chain appears as bands at 1630, 1521, 1400 cm^{-1} (Dumitrescu *et al*., 2015); the stretching vibrations of the carbonyl group of alanine linked in the ester bond are evidenced by bands at 1721, 1262, 1104 cm^{-1}, while the resorcinol fragments have vibrations at 1618, 1459 and 1400 cm^{-1} (Shao *et al*., 2017).

For the water blocking test, a fractured Berea sandstone core with porosity of 22 % was used. The core was characterized (initial characterization) by injecting water at different flow rates: 60, 120, and 180 cm^3/h (Figure 15), giving permeability of 154 mD. After saturating the core with oil and undergoing secondary oil recovery through brine injection at 130 °C, the gelling formulation was injected into the core, occupying 0.5 of the porous volume. Under the effect of temperature, crosslinking occurred between the

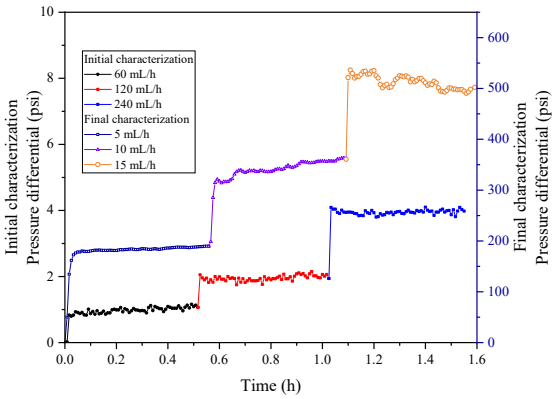

Figure 15. Differential pressure versus injection time during characterization of the rock core.

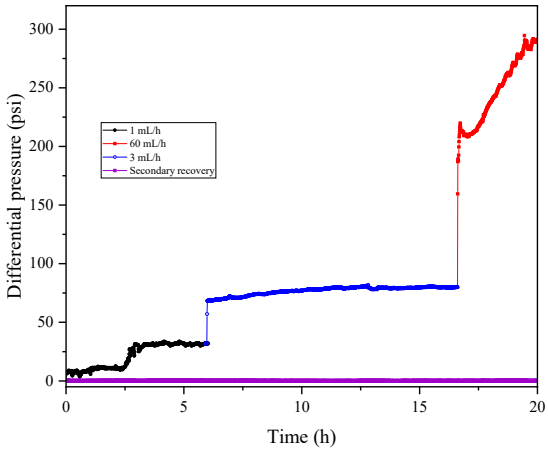

Figure 16. Differential pressure versus injection time during brine injection at secondary recovery without gel (purple line), and after the gel aging process in the core: at 1 cm^3/h (black line), 3 cm^3/h (blue line), and 60 cm^3/h (red line).

terpolymer, crosslinking agents, and alanine, forming a rigid but flexible gel. The differential pressure during the secondary recovery process by brine injection was no greater than 1 psi (Figure 16). After *in situ* gel formation for 91 h at 130 °C and brine injection at a rate of 1 cm^3/h, the differential pressure was established at around 30 psi. Increasing the brine injection rate to 3 cm^3/h raised the pressure to 80 psi. The greatest increase in differential pressure was observed when the injection rate was increased to 60 cm^3/h, reaching 265 psi and where the test was stopped (Figure 17). The gel formed *in situ* promoted an increase in resistance to brine flow, showing fluid control percentages of 96, 98, and 99 % for flow rates of 1, 3 and 60 cm^3/h, respectively. At the end of the test, the core was left without fluid injection for 24 h at 130 °C and was subsequently characterized again (final characterization), injecting water at different flow rates (5, 10, and 15 cm^3/h) (Figure 15), reaching permeability of 0.07 mD. The effective sealing of the core was shown by the gel formed *in situ* of the porous medium.

Based on the rheological observation, bottle test method, and rock-core fluid control results, alanine was shown to be a suitable organic crosslinking agent for high temperature conditions in the presence of high salinity brine and crude oil.

Conclusions

(1) Gels were developed from a Polyeca^{TM} terpolymer with a crosslinking system composed of HMTA and RE/alanine or glycine. Gels with gelation time of 3 h were rigid but flexible and were assigned to the letters G-J according to the Sydansk code.

(2) Gels with added alanine and terpolymer concentrations of 1 % and 2 % exhibited excellent long-term stability for one month and good rheological properties.

(3) The formulation of the gelling system with 1.5 % alanine was selected and the water control test was performed through the porous medium plugging process by injecting 0.5 PV of the gelling system and the *in situ* gel formation at 130 °C, confinement pressure at 3000 psi, presence of residual oil in the core and brine at 250,000 ppm.

(4) The gels exhibited fluid blocking efficiency up to 96 % with an injection rate of 1 cm^3/h and up to 99 % with an injection rate of 60 cm^3/h.

Future research perspectives

For treatments in deep wells, further research on additional retarders to extend the gelation time beyond 3 h is needed.

A cost-benefit analysis of the gelation system is presented.

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Nomenclature

Hexamethylenetetramine	HMTA
Hydroquinone	HQ
Resorcinol	RE
Preformed particle gels	PPGs
Total dissolved solids	TDS

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