



## Evaluation of Aluminum-Crosslinked Gel Compositions for Controlling Water Cut and Improving Flow Conformance in Oil-Bearing Formations

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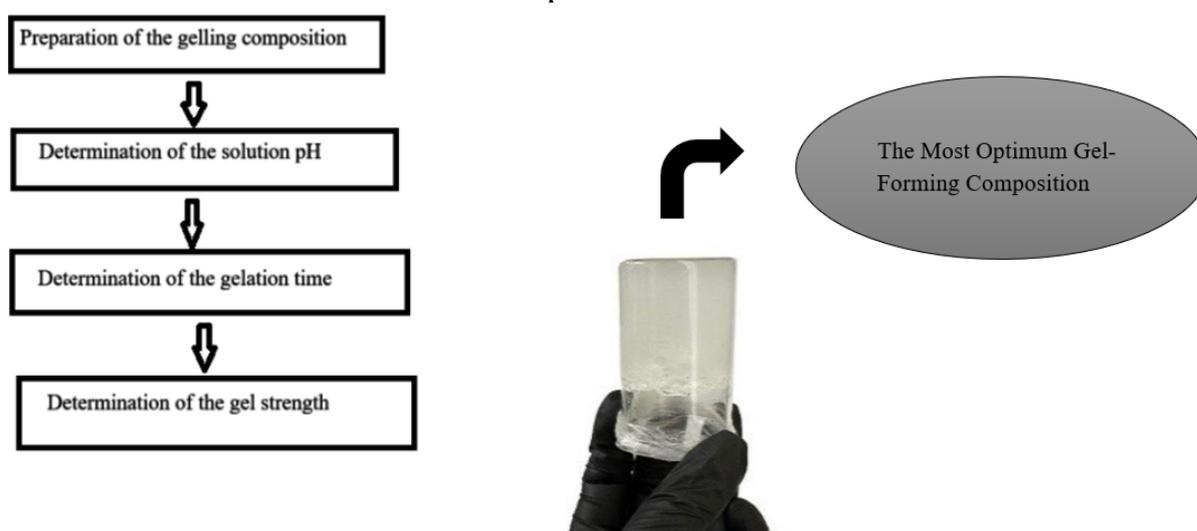
pH

### ABSTRACT

High water cut in produced fluids is a common challenge across most hydrocarbon reservoirs. In this regard, conformance control technologies based on gel-forming compositions are considered one of the most effective methods of enhancing oil recovery. This study investigated a gel-forming composition based on an acrylamide polymer and an aluminum-containing crosslinker. Despite the extensive range of developed crosslinked polymer systems, achieving precise control over the gelation rate without introducing auxiliary retarders remains a significant challenge, particularly when inorganic crosslinkers such as aluminum-based compounds are employed. The aim of the research was to determine the optimal crosslinker-to-polymer ratio required to obtain a gel-forming system with high mechanical strength and adjustable gelation time that meets the technological requirements for reservoir injection. Experimental studies were carried out to determine the gelation time, solution pH, and mechanical strength of the resulting gel. The gelation time reached up to 197 minutes at a polymer concentration of 2 wt.% under standard temperature conditions (20 °C). The measured storage modulus of the gel reached 1350 Pa at a pH value of 4.66. The efficiency of the investigated aluminum-containing crosslinker is compared with existing analogues currently used in the oil and gas industry for water shutoff and conformance control applications.

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### Graphical Abstract



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## 1. INTRODUCTION

Global practice demonstrates that hydrocarbon production is often accompanied by high water cut (1-3). This problem is particularly pronounced in mature oilfields, where the ratio of produced water to hydrocarbons steadily increases over time as the reservoir becomes depleted. High water cut not only reduces the efficiency of hydrocarbon recovery but also leads to an increase in operating costs, as the handling, separation, and disposal of produced water represent a significant burden for the oil industry. A promising approach to improving the oil recovery factor in mature reservoirs is the implementation of conformance control technologies using gel-forming compositions, making their development an urgent task for the petroleum industry (4-6). These technologies are designed to modify reservoir permeability profiles, block high-permeability channels, and restrict the unwanted movement of injected fluids, thereby improving sweep efficiency and extending the productive life of oilfields. Approaches to mineral resource management are outlined by Litvinenko et al. (7), which are also relevant to these technologies. In practice, the application of such methods is considered a cost-effective alternative to large-scale redevelopment or drilling of infill wells, highlighting the strategic importance of conformance control solutions.

Among the wide range of gel-forming systems, chemical methods based on polymers (8, 9) are most widely applied due to their effective mechanisms of action (10, 11). These systems are capable of forming strong three-dimensional networks that selectively reduce water mobility while leaving oil permeability largely unaffected. Polyacrylamide (PAM) and its derivatives are the most commonly used reagents for the preparation of polymer gels employed in oil reservoirs (12-14). Their advantages include high solubility in water, tunable molecular weight, and the ability to undergo controlled gelation under reservoir conditions. The versatility of PAM-based systems has led to their adoption across different types of reservoirs, ranging from sandstone formations to fractured carbonates. Some polymer systems have been described for water shutoff operations (15, 16), where they are injected to seal water-bearing layers or fractures, while others have been applied for well-killing purposes (17, 18), ensuring safe temporary plugging during workover and intervention activities.

Polymer gels may utilize organic crosslinkers (polyphenol, phenol-formaldehyde, hydroquinone, etc.) (19, 20) or inorganic ones (aluminum, chromium, iron compounds, etc.) (21-23). It is well established that polymer compositions with inorganic crosslinkers are more frequently used for in-situ reservoir isolation due to their selectivity (24, 25) and adjustable gelation time

compared with those containing organic crosslinkers (22, 26).

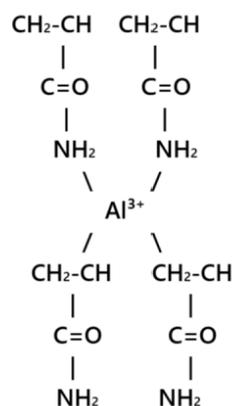
In this study, an aluminum-based crosslinker was selected owing to its ability to form stable gel networks through interactions with amide and hydroxyl groups of polymers and its non-toxic properties (27, 28).

The mechanism of polymer crosslinking with the crosslinker under consideration is shown in Figure 1.

It is well established that aluminum ions are capable of binding several polymer chains simultaneously (29). Nevertheless, the main challenge associated with aluminum-based gel systems lies in controlling the rate of gelation. Therefore, one of the key scientific tasks in such systems is to extend the gelation time without compromising the mechanical strength of the gel and without using toxic or corrosion-active retarders.

Although aluminum as a crosslinking agent has certain limitations, it has been extensively studied in several works, where it has been demonstrated that aluminum exhibits high stability in a mildly acidic medium with a pH range of 4 to 6 and the ability to maintain gel integrity at elevated temperatures up to 200 °C (23, 27). However, as reported in other studies (30,31), at hydrogen ion concentrations below pH 3.5 and above pH 9.0, gel degradation and precipitation occur. The interaction of aluminum ions with the functional groups of polyacrylamide provides the formation of a stable three-dimensional gel network that retains its structural integrity even under high salinity conditions, with sodium chloride concentrations up to 50,000 ppm (31).

Several studies have shown that the structural and chemical stability of inorganic compounds significantly affects their functional performance. Aziz et al. (32) developed Ni/ZnO nanocomposites exhibiting enhanced chemical resistance and catalytic activity due to the



**Figure 1.** Mechanism of cross-linking of acrylic polymer (PAM) with trivalent aluminum ions ( $\text{Al}^{3+}$ ) precise control of interactions between inorganic particles. A similar concept can be applied to aluminum-

based crosslinked gel systems, where the gel strength and stability depend strongly on the bonding interactions between aluminum ions and the amide groups of polyacrylamides. Furthermore, the development of flow-diverting technologies in the oil and gas industry is closely associated with global digitalization and the implementation of innovative approaches. Kongdachudomkul et al. (33) proposed a model for assessing the digital maturity of oil and gas companies, emphasizing the importance of integrating advanced materials and modern process management methods. In the present study, experimental investigations were carried out to analyze the behavior of an aluminum-containing crosslinker and to establish the relationships between its concentration, gelation time, pH and the mechanical properties of the resulting gel-forming composition.

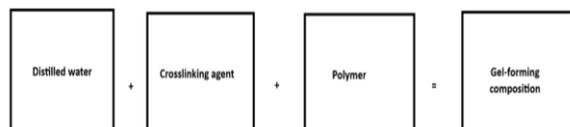
## 2. METHODS AND MATERIALS

**2. 1. Gel Preparation Process** To obtain the gel-forming composition, the procedure shown in Figure 3 was followed. A magnetic stirrer IKA - RT 5 (Figure 2) was used to mix the solution, providing up to 1000 rpm at room temperature.

The polymer concentration in all experiments was fixed at 4%, while the crosslinker concentration varied from 0.1 to 1 wt.%.



**Figure 2.** Illustration of the mixing process on a magnetic stirrer IKA - RT 5



**Figure 3.** Procedure for preparing the gel-forming composition

**2. 2. Determination of Gelation Time** Gelation time is a fundamental parameter when selecting the optimal concentration to form an effective crosslinked

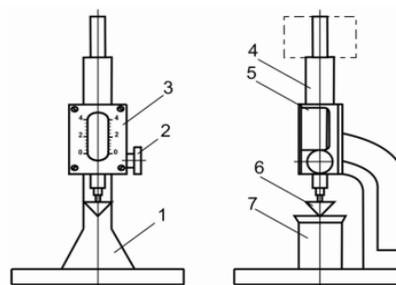
system (34, 35). As in other studies (19, 26, 36), this work employed the bottle test, a rapid and cost-effective method based on visual observation. Additionally, the Sydansk gel code (A–J) was used to classify gel strength (10, 37, 38). For example: Code A – No detectable gel formed; B - highly flowing gel; code C - flowing gel; code D - moderately flowing gel; Code E – barely flowing gel; F- highly deformable nonflowing gel; G - moderately deformable nonflowing gel; code H - Slightly deformable nonflowing gel; Code I – rigid gel; Code J – ringing rigid gel.

**2. 3. Determination of pH** The hydrogen ion concentration is a key parameter influencing gelation in crosslinked polymer systems (16, 39). It is important to identify the pH interval where strong and stable gels form. A Testo-206 pH meter (Figure 4) was used.

**2. 4. Determination of Gel Strength** The efficiency of gel-forming systems is largely determined by their ability to resist water breakthrough, which depends on gel strength. Strength tests were conducted 24 and 48 hours after preparation. The Rebinder method was initially applied, measuring cone penetration under constant load for 10 minutes. (Figure 5) (40, 41).



**Figure 4.** Testo-206 pH meter



**Figure 5.** Scheme of a conical plastometer: 1—frame; 2—clamp; 3—measuring scale; 4—rod; 5—vertical clip; 6—cone; 7—glass beaker with test gel

However, due to the softness of early-stage gels, more accurate rheological testing with an Anton Paar MCR 302 rheometer was employed. The oscillatory test determined the storage modulus ( $G'$ ), a key indicator of gel strength.

After 24 hours of preparation of the gel-forming composition, this test was performed in a rheometer. A plate-plate measuring system with a diameter of 25 mm and a gap of 2 mm was used. The test is called oscillatory and is performed on the basis of an amplitude ramp, conducted in the range from 1 to 1000% at a constant frequency of 1 Hz (within the linear viscoelastic region). The experiments were conducted at a temperature of 25 °C.

### 3. RESULTS AND DISCUSSION

**3. 1. Gelation Time** The authors conducted experimental studies to determine the gelation time at various concentrations of polyacrylamide (PAA) in the range of 1–5 wt.% and an aluminum-based crosslinker within 0.1–1.0 wt.%. The selected concentration range for the crosslinker was based on preliminary tests, which showed that gel formation does not occur at aluminum reagent concentrations below 0.1 wt.% due to an insufficient number of coordination centers for forming a three-dimensional polymer network. Furthermore, at crosslinker concentrations above 0.8 wt.%, precipitate formation and system destabilization were observed, attributed to excessive hydrolysis of aluminum ions and the formation of insoluble hydroxide phases (29). Figure 6 shows the dependence of gelation time on crosslinker concentration. The data indicate that the gelation time increases up to 197 minutes with an increase in the aluminum-based component concentration to 0.7 wt.% and a decrease in polymer concentration to 2 wt.%. This behavior can be explained by the increased formation of oligomeric aluminum species at higher crosslinker concentrations, which are less reactive than monomeric  $Al^{3+}$  ions. This slows down the formation of a uniform network. At the same time, a decrease in polymer concentration reduces the density of active points involved in crosslinking, further contributing to the increase in gelation time (42). The optimal curing time of gel-forming compositions is generally considered to be 30 minutes or longer, depending on specific parameters (temperature, pH) and the particular objectives of each researcher (19, 43). At a low polymer concentration - 1 wt.%, no gel formation was observed during the bottle test, as indicated by meniscus deviation, confirming the system's fluid state. This is probably due to the insufficient number of polymer chains available for interchain coordination with aluminum ions. Several studies (23, 31, 44) report similar gelation time ranges for aluminum-based crosslinkers without additives. According to the Sydansk method, gels obtained at concentrations from 0.1 to 0.7% correspond to Code I (Figure 7).

### 3. 2. Effect of pH

The optimal pH range is determined by the polymer nature and crosslinker type, as the acid-base environment affects both the complexation kinetics and bond stability. For chromium-containing systems, studies (16, 45) indicate optimal crosslinking near neutral pH, where chromium ions maintain coordination activity without premature hydrolysis. In contrast, aluminum-based systems exhibit different behavior. Research conducted by Shamlooh et al. (31) and Altunina et al. (46) demonstrates that stable aluminum-gel compositions form across a broader pH range (0.5–14), depending on the aluminum compound and complexing additives. Specifically, gelation rate decreases with increasing pH, suggesting that mildly acidic conditions (4–5) promote active coordination and stable bond formation, leading to robust polymer networks (31). As noted in work (29), higher pH values induce aluminum ion hydrolysis, forming larger polymeric structures and altering crosslinking mechanisms. pH is a key driver of aluminum speciation in all environments. In this study, the pH range of the systems was 3.54–5.6. Figure 8 illustrates the pH dependence on aluminum crosslinker concentration: increasing crosslinker content (0.1–1.0 wt.%) and decreasing polymer concentration (5–1 wt.%) reduce pH due to enhanced aluminum ion hydrolysis and  $H^+$  release.

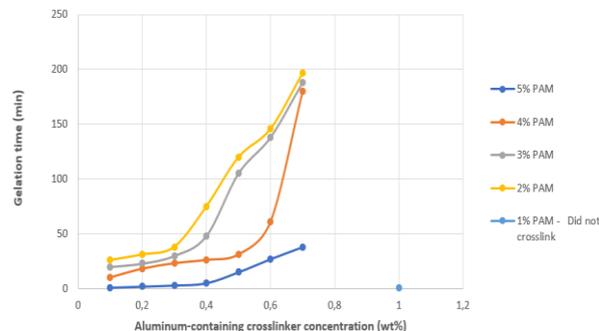


Figure 6. Determination of gelation time

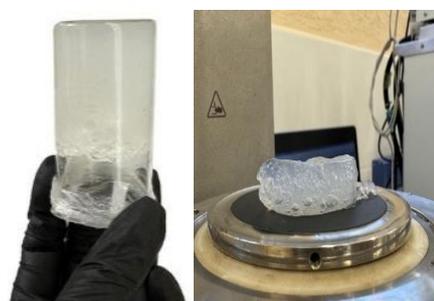


Figure 7. Gel obtained with code I

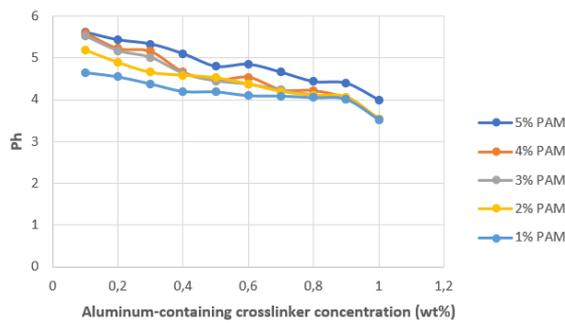


Figure 8. pH of gel-forming composition

In research by Berger et al. (42) further confirm that monomeric aluminum hydroxy species dominate at low pH, transitioning to oligomeric/polymeric forms as pH rises, reducing free H<sup>+</sup> concentration. Thus, literature and experimental data confirm that the optimal pH range for aluminum-gel systems is approximately 4–5. This interval balances reaction rate, aluminum hydrolysis, and coordination complex stability, ensuring the formation of strong and durable gel structures.

### 3. 3. Effect of Crosslinker Concentration on Gel Strength

At the initial stage, the strength of the gel-forming compositions was assessed using the Rebinder method. However, the experiments revealed that the gels exhibited low mechanical rigidity during the early stages of gel network development, resulting in strength values below the detection threshold of the method. The cone penetrated completely into the sample under its own weight within 24 hours of preparation, making it impossible to obtain reliable data. This indicates that the Rebinder method has limited applicability for the characterization of soft gel-forming systems, particularly during the first day after preparation. Therefore, for subsequent evaluations, a more precise, sensitive, and reliable approach was adopted—rheological testing using an Anton Paar MCR 302 rheometer (31, 47) (Figure 9). As shown in Figure 10, the storage modulus ( $G'$ ) increases with higher concentrations of both the aluminum-based crosslinker and polyacrylamide. This is attributed to increased crosslink density between macromolecules, forming a more robust three-dimensional network. Increasing the crosslinker concentration from 0.1 to 0.7 wt.% raised  $G'$  to 1350 Pa, indicating the development of a robust three-dimensional polymer network capable of withstanding substantial mechanical stresses during water shutoff operations in the reservoir. At PAM concentrations of 4-5 wt%, maximum  $G'$  values of 1100-1350 Pa were achieved after 24 hours (Figure 10). Additional measurements after 48 hours showed further increase to 1990-2200 Pa, confirming strength development through slow bond reorganization and ongoing aluminum chain hydrolysis. These results align with literature values for

unmodified aluminum-complexed systems, where  $G'$  ranges from 125 Pa to 2000 Pa were reported in some works (29,31). As noted by Raupov et al. (16) optimal high-strength gels with low initial viscosity and delayed gelation can be achieved using chemical modifiers such as gel-time regulators, retarders and radiation-treated polymers. Figure 11 shows a graph showing the dependence of elastic modulus ( $G'$ ) on strain for a crosslinker concentration of 0.7% wt.% at a polymer ratio - 4 wt.% obtained by rheometer.

Table 1 shows the comparison of the investigated gel-forming composition with analogs.



Figure 9. The process of investigating the strength of gel-forming composition using Anton Paar MCR 302 rheometer

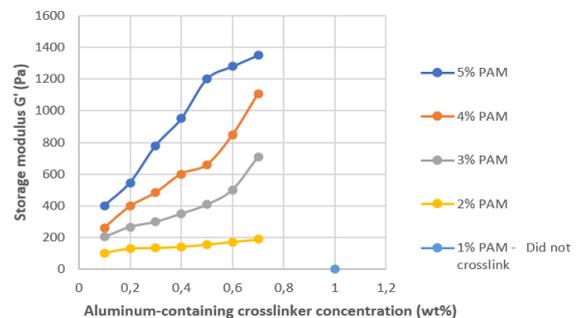


Figure 10. Storage modulus of the obtained gel as a function of the crosslinker concentration

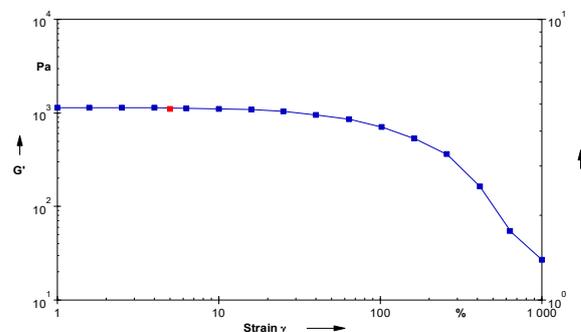


Figure 11. Obtained function of elastic modulus ( $G'$ ) on strain by Anton Paar MCR 302 rheometer

**TABLE 1.** Comparison of the investigated gel-forming composition with analogs

Gel-forming composition	Gelation time (min)	pH range with gel formation	Storage modulus G' (Pa)	Specific characteristics	References
Investigated gel-forming composition based on polyacrylamide and aluminum-containing crosslinker	<197	3.54-5.6	1350	Without additives or retarders; high strength under natural pH conditions	The authors' composition under study
Polyacrylamide + tris(2-aminoacetate) aluminum	Not specified	0.5-10	<125	Requires pH control; reaction proceeds more slowly	(31)
Polyacrylamide + aluminum/acetate	50	0.5-8.5	2000	Requires pH control	(31)
Polyacrylamide + aluminum/zirconium	40-60	4.0-6.5	<100	Complex structure with zirconium ions; suitable for high reservoir temperatures up to 200 °C	(23)
Polyacrylamide + aluminum/nitrate	Not specified	2.5-14	2000	Narrow pH range	(31)
Polyacrylamide + aluminum	150	Not specified	Not specified	Various salinity ranges	(44)

Table 1 highlights the advantages of the investigated gel system, confirming its practical potential.

#### 4. Future Research Directions and Recommendations

Future work will focus on expanding the study of the developed gel-forming composition through detailed characterization of the chemical composition and crosslinking mechanism of the aluminum-based agent. A key objective is the selection of a gelation kinetics modifier to increase the gelation time, which is essential for large-volume treatments in conformance control applications. To enhance data accuracy, rheological tests will be conducted to determine the gelation induction period at specific shear rates, simulating the mechanical stresses experienced during field injection. Furthermore, the adhesive properties of the gel towards oil- and water-saturated rocks of varying lithology will be investigated. This will assess the gel's ability to adhere to and remain stable on pore walls, a critical factor for long-term isolation performance. Additional studies will examine gel performance across different temperature ranges and core model test to evaluate the composition's effectiveness in isolating high-permeability channels.

#### 5. CONCLUSIONS

The study presents a set of laboratory investigations aimed at developing gel-forming systems, including determination of gelation time by visual inspection, measurement of pH using a Testo-206 pH meter, and evaluation of gel strength by both the Rebinder method and rheological analysis with an Anton Paar MCR 302 rheometer. The following conclusions can be drawn from the research:

- The maximum gelation time of the studied composition based on polyacrylamide and an aluminum-based crosslinker, determined by the bottle test, is 197 minutes at a polymer concentration of 2 wt.% and standard temperature.
- Gel formation for the studied composition occurs within a pH range of 3.54 to 5.6.
- At the initial stage, the Rebinder method was used to determine strength characteristics; however, its limitations were identified. Consequently, a methodology based on an oscillatory test using an Anton Paar MCR 302 rheometer was employed.
- An increase in the crosslinker concentration to 0.7 wt.% leads to a significant rise in the storage modulus (G') to 1350 Pa, measured 24 hours after preparation at a polymer concentration of 5 wt.%.
- The experimental results and a comparison of the studied composition with similar systems from the literature confirm its competitiveness.

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#### Ethics Approval and Consent to Participate

This study did not involve human participants or animals. Therefore, ethical approval and informed consent were not required.

## Competing Interests

The authors declare that they have no known financial or organizational conflicts of interest that could have influenced the work reported in this paper.

## Data Availability

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

## Declaration of Generative AI and AI-Assisted Technologies in the Writing Process

The authors used artificial intelligence-based tools to assist with the accurate scientific translation of selected phrases from Russian into English. All translated content was carefully reviewed, edited, and validated by the authors, who take full responsibility for the integrity, originality, and accuracy of the final manuscript.

## Author Contributions

**D. G. Podoprigora**, Ph.D. in Petroleum Engineering: Conceptualization, problem definition, article writing, and execution of experimental studies.

**F. D. Markushina**, Ph.D. candidate: Literature review, experimental studies, data systematization, and article writing.

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**Persian Abstract****چکیده**

درصد بالای آب در سیالات تولیدی یکی از چالش‌های متداول در اغلب مخازن هیدروکربنی به شمار می‌رود. در این زمینه، فناوری‌های کنترل انطباق (Conformance Control) مبتنی بر ترکیبات ژل‌زا به‌عنوان یکی از مؤثرترین روش‌های ازدیاد برداشت نفت شناخته می‌شوند. در این پژوهش، ترکیب ژل‌زایی بر پایه‌ی پلیمر آکریل‌امید و عامل شبکه‌ساز حاوی آلومینیوم مورد بررسی قرار گرفت. با وجود گستره‌ی وسیعی از سامانه‌های پلیمری شبکه‌ای توسعه‌یافته، کنترل دقیق نرخ ژلاسیون بدون استفاده از مواد بازدارنده‌ی کمکی همچنان یک چالش اساسی محسوب می‌شود، به‌ویژه هنگامی که از عامل‌های شبکه‌ساز معدنی نظیر ترکیبات پایه آلومینیوم استفاده گردد. هدف از این تحقیق تعیین نسبت بهینه‌ی عامل شبکه‌ساز به پلیمر برای دستیابی به سامانه‌ای ژل‌زا با استحکام مکانیکی بالا و زمان ژلاسیون قابل تنظیم بود، به‌گونه‌ای که نیازهای فناوری تزریق در مخزن را برآورده سازد. آزمایش‌های تجربی به‌منظور تعیین زمان ژلاسیون، مقدار pH محلول و استحکام مکانیکی ژل حاصل انجام گرفت. زمان ژلاسیون در غلظت پلیمری ۲ درصد وزنی و در شرایط دمایی استاندارد (۲۰ درجه سانتی‌گراد) تا ۱۹۷ دقیقه اندازه‌گیری شد. مدول ذخیره‌ی ژل در مقدار pH برابر ۴٫۶۶ به ۱۳۵۰ پاسکال رسید. بازده عامل شبکه‌ساز حاوی آلومینیوم بررسی شده با نمونه‌های مشابه موجود که در صنعت نفت و گاز برای عملیات کاهش آب‌دهی و کنترل انطباق به‌کار می‌روند مقایسه گردید.