

# Evaluation of polyacrylamide gels with accelerator ammonium salts for water shutoff in ultralow temperature reservoirs: Gelation performance and application recommendations



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## ABSTRACT

Water shutoff in ultralow temperature reservoirs has received great attention in recent years. In previous study, we reported a phenol-formaldehyde-based gel formula with ammonium salt which can provide a gelation time between 2 hrs and 2 days at 25 °C. However, systematic evaluation and field recommendations of this gel formula when encountering complex reservoirs environment are not addressed. In this paper, how and why such practical considerations as water composition, temperature, pH, weight ratio of formaldehyde to resorcinol and contaminant Fe<sup>3+</sup> to affect the gelation performance are examined. Brookfield DV-III and scanning electron microscopy (SEM) are employed respectively for viscosity measurement and microstructure analysis. SEM results further illustrate the mechanism of the effect of salinity on gelation performance. It reveals that cross-linking done by covalent bond has great advantage for gel stability under high salinity environment. The target gel formula can provide desirable gelation time below 60 °C, perfect for 15–45 °C, while it is unfeasible to use high salinity to delay gelation at 60 °C. We summarized the effect of salinity on gelation performance of different gel formulas from the present study and published literature. The summarized data can provide important guideline for gel formula design before conducting any kinds of experiments. The variation of gelation performance at different salinity may be dominated by the interaction between crosslinker-salt-polymer, not only limited to “charge-screening effect” and “ion association” proposed by several authors. We hope the analysis encouraging further investigations. Some recommendations for field application of this gel are given in the end of this paper.

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## 1. Introduction

With the growth and maturity of oil field worldwide, high water cut is common to see in oil wells. Excessive produced water will restrict oil recovery and cause large economic loss due to the cost of lifting, separating, treating and disposing produced

water [1–5]. Many chemical techniques such as foam, microgel, emulsified asphalt, polymer gel or combined techniques have been proposed and applied in oilfields to improve formation heterogeneity and minimize water output [6–14]. Among these techniques, polymer gel, the mainstream techniques of the chemical water shutoff and conformance control method, has proven to be efficient and cost-effective [15,16]. Polymers can crosslink with metal salt cross-linkers, such as Al<sup>3+</sup>, Cr<sup>3+</sup>, Ti<sup>4+</sup> and Zr<sup>4+</sup> etc. [17,5,18–20]. However, the long-term stability of gel may be not good as well as the difficulty to control gelation time due to the ionic bond crosslinking mechanism [21,22].

Polymers can also crosslink with organic crosslinkers like phenolformaldehyde, hydroquinone-hexamethylenetetramine, methenamine, terephthalaldehyde, polyethyleneimine (PEI) etc. The gelation between polymer and organic crosslinker done by

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covalent bonding is much more stable than ionic bonds [23–26]. Phenol-formaldehyde based polymer gel has predominant thermal stability and is served in petroleum industry over several decades [27]. However, this gel having activation temperatures above 70–80 °C are often used for conformance control in high-temperature reservoirs [28]. There are huge number of low temperature reservoirs worldwide needed for water shutoff treatments. However, the reaction and gel-strength-development rate is rather slow at low temperatures for many kinds of gel formulas, resulting in long gelation time of over 10 days and weaker gel strength, even exhibiting gelation failure in reservoir condition [29].

In previous study, we have examined the HPAM gel formula with ammonium salt, a kind of promising conformance control agent used in ultralow temperature reservoirs [30]. In consideration of better field application, systematic evaluation of the target gel is conducted and the purpose of this work falls into the following: (1) investigating the compatibility of gelling solution with mixed water, (2) examining the effect of inorganic salts, temperature, initial pH, weight ratio of formaldehyde to resorcinol and corrosion products  $\text{Fe}^{3+}$  on the gelation performance; (3) providing a guideline for screening the potential gel formulas at different situations from the discuss of the effect of salinity on gelation performance, and (4) giving recommendations for field implementation of the target gel formula. To achieve the above objectives, the present experimental study mainly combines SEM spectroscopy, Brookfield viscometer DV-III and observation work.

## 2. Experimental studies

Soluble HPAM with a molecular weight of  $1.8 \times 10^7$  Da and a hydrolytic ratio of 25%–35% was furnished by Hengju Chemical, Beijing, China. All of the rest of the materials are supplied by Kelong chemical, Chengdu, China. Standard reagent-grade formaldehyde and resorcinol, the former furnished as a 37 wt.% aqueous solution, the latter furnished as A.R. grade with net weight more than 99.5 wt.%, were employed. Inorganic salts examined included:  $\text{NH}_4\text{Cl}$ ,  $\text{NaCl}$ ,  $\text{KCl}$ ,  $\text{CaCl}_2$ , and  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ . All the salts are A.R. grade and used as received.

The basic gelling solutions were formulated with a combination of 0.3 wt.% HPAM, 0.2 wt.% formaldehyde, 0.02 wt.% resorcinol and 0.2 wt.%  $\text{NH}_4\text{Cl}$ . The initial pH of the basic gelling

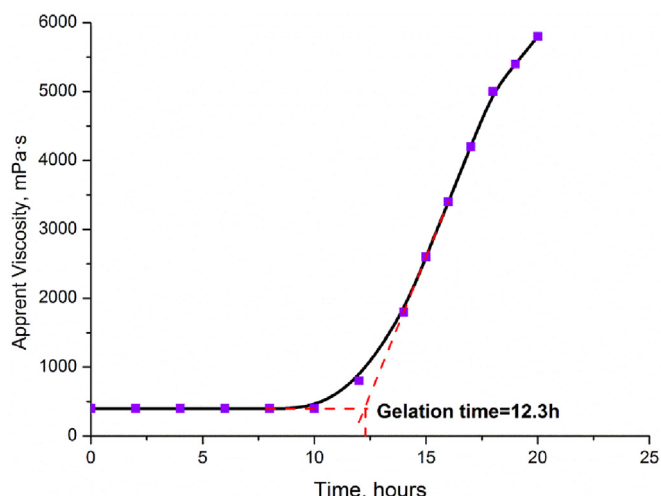


Fig. 1. Graph of gelation time measurement.

solution was found to be 7.5. No attempts were made to adjust the initial pH of the gelling solution, unless otherwise indicated.

## 3. Measurement of gelation time

The gelation time is defined as the time needed to reach the inflection point on the viscosity vs. time curve (Fig. 1). This method corresponds to the onset of the gel formation and has been widely used by several authors to determine the gelation time of water shutoff treatments [17,31,25]. In this paper, a Brookfield DV-III viscometer was used to measure the viscosity. Gelation performance studies were initiated at low temperatures from 15 to 60 °C.

## 4. Results and discussion

### 4.1. Effect of mixing water on gelation time

In field application, gels always are prepared using fresh water, formation water, and even seawater. These waters contain various ions can greatly impact the gelation performance [23,22,32,33]. Therefore, it is important to test the gelation performance for the target gel prepared in different water composition. Table 1 shows the chemical analysis of the formation water. For most study, people often use single salt to investigate the effect of salinity on gelation performance [34,32,29,33]. However, the injection water often has complex composition, hence the single salt cannot reflect the real field application requirements. Fig. 2 shows the viscosity evolution for two basic gelling solutions formulated with a combination of 0.3 wt.% HPAM, 0.2 wt.% formaldehyde, 0.02 wt.% resorcinol and 0.2 wt.%  $\text{NH}_4\text{Cl}$ . The first solution was prepared in distilled water (initial pH is 7.5); whereas the second solution was prepared using formation water (initial pH is 8.3). Viscosity measurements were conducted at 30 °C. Chemical analysis of the formation water was given in Table 1. The gelation time was 7.8 hrs for the gel prepared in distilled water, while 12.8 hrs for prepared in formation water. High salinity can shield the accelerating gelation function of  $\text{NH}_4\text{Cl}$ , accordingly reduce the chance for crosslinker interacting with amide groups. Moreover,  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  can affect polymer performance even to form precipitation, hence, the crosslinking chance decreased, resulting in delayed gelation and poor gelation performance. Perhaps the accelerating gelation function of  $\text{NH}_4\text{Cl}$  has some relationship with  $-\text{NH}_2$ . However, this is beyond the scope of this paper. More detail discussion about the effect of salinity composition on gelation performance will be presented in subsequent section.

Table 1  
Chemical analysis of the formation water.

Variable	Value
$\text{Na}^+$	17,753
$\text{K}^+$	807
$\text{Ca}^{2+}$	594
$\text{Mg}^{2+}$	2324
$\text{Cl}^-$	35,237
$\text{SO}_4^{2-}$	993
$\text{HCO}_3^-$	181
TDS	57,889
pH	8.0

All concentrations are in mg/L; Total dissolved solids (TDS) are determined by the addition of variables.

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