



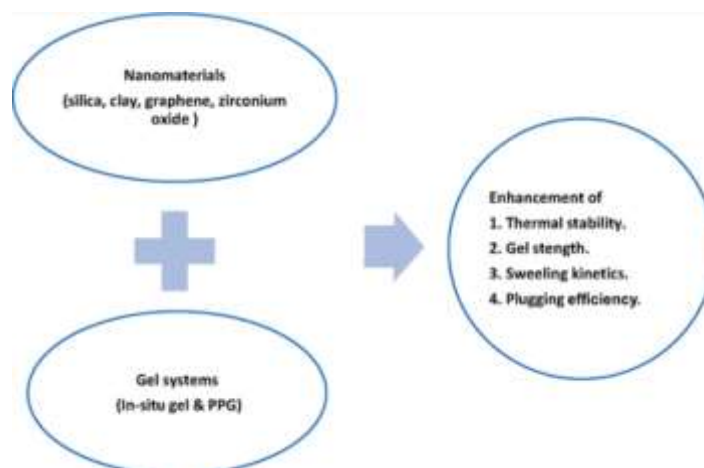
NANOMATERIAL INNOVATIONS FOR WATER SHUTOFF IN HYDROCARBON WELLS: A COMPREHENSIVE REVIEW

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ABSTRACT

The paper discusses the importance of reducing water production from hydrocarbon wells to prolong their lifespan. Gel treatments are commonly used for water shut-off applications, but traditional methods have limitations. The paper proposes enhancing gel performance by incorporating nanomaterials like nanosilica, nanoclay, and graphene. These nanomaterials can improve gel properties such as gelation time and strength. The study reviews the benefits of nanomaterials in different gel systems, including in situ gel, preformed particle-gel, and nanosilica-based fluid. Nanomaterials are found to enhance gel properties significantly, with nanosilica-based gels exhibiting exceptional plugging efficiency. Additionally, modeling is discussed as a tool to overcome operational challenges. The study also explores colloidal gels as an alternative to polymers for addressing high water production in oil fields. Bottle tests were conducted to characterize the gels, and their applicability in porous media was inspected using a dual-patterned glass micromodel. Results indicate that increasing NaCl concentration can alter gel behavior, and rheology tests align with gelation times. The study demonstrates the effectiveness of solid gels in controlling water conformance, with promising results in oil recovery.



INDEX TERMS- Water Production, Gel Treatments, Nanomaterials, Gelation Time, Strength, Nanosilica, Plugging Efficiency, Modeling, Colloidal Gels, Oil Recovery

I. INTRODUCTION

In oil production, the effectiveness of driving fluids in pushing oil toward the production interval is crucial, measured by conformance. However, reservoir heterogeneity may lead to unbalanced fluid movement, resulting in unswept oil and concurrent production of water or gas, termed reservoir conformance problems. Water shut-off, a form of conformance control, targets excessive water production to enhance oil recovery and well longevity. Undesired water production poses economic and environmental challenges, necessitating water shut-off technologies for efficient reservoir management and regulatory compliance. Accurate diagnosis of water production issues is

essential for effective treatment, involving various data analysis techniques. Gel treatments, despite being longstanding, encounter challenges such as instability and control issues. Utilizing nanomaterials like silica and clay in gel compositions presents promising solutions, enhancing gel properties and performance. This study discusses novel gel systems incorporating nanoparticles and highlights their potential in addressing operational challenges during water shut-off treatments, emphasizing the role of gel modeling for improved efficacy.



Chemical Systems		
Chemical System	Name	Chemical Structure
Inorganic Gels	Sodium Silicate	
Monomer Systems	Acrylamide	
Polymers	Polyacrylamide	
	HPAM	
	Xanthan Gum	
Resins	Phenol-formaldehyde	

III. LITERATURE REVIEW

There are two types of water produced with oil: good water, which is part of the fractional flow process and does not compete with oil production, and bad water, which hinders oil production and requires immediate treatment. Excessive water production can stem from various sources, categorized as wellbore-related or reservoir-related. Wellbore-related sources include flow behind the pipe, casing leaks, migration of the oil-water contact (OWC), and barrier breakdowns. Reservoir-related sources include fractures between injector and producer, fissures from water layers, water coning, watered-out layers, channels through high permeability zones, and fingering. To address these issues, mechanical and chemical methods are employed. Mechanical methods involve placing high-strength tools or cement in the wellbore to shut off water sources, suitable for near-wellbore problems. Chemical methods, like gel injection, target both near-wellbore and reservoir-related sources, improving reservoir conformance and sweep efficiency. Chemical solutions can last for months to years but depend on reservoir properties and compatibility. The study focuses on chemical techniques utilizing

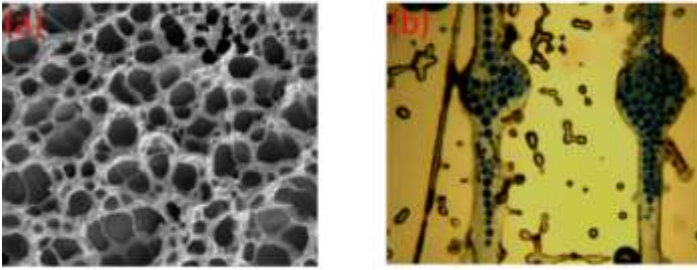
II. HYPOTHESIS

Excessive water production in oil wells, whether due to water coning from strong aquifers or channeling during injection into fractured reservoirs, presents significant challenges. High water cuts result in pipeline corrosion, separation costs, and energy consumption, potentially halting oil production for economic reasons. Variations in permeability and viscosity differences between fluids contribute to excess water production. Mechanical and chemical solutions, including polymer and gel treatments, have been introduced to mitigate this issue. Gel systems, composed of polymers or colloidal particles dispersed in a liquid phase, exhibit both liquid- and solid-like behaviors. Colloidal gels, formed by particle aggregation in a liquid, rely on interparticle attractions and can exhibit unique properties such as non-zero yield stress. Various mechanisms govern colloidal gelation, including particle aggregation and cross-linking, influenced by factors like temperature and pH. Despite limited literature on colloidal particle gels for water conformance control in oil reservoirs, promising results have been reported. Colloidal silica gel, for instance, has shown effectiveness in reducing water permeability in laboratory studies. Compared to other methods like polymer gels, colloidal gels offer advantages such as environmental friendliness, thermal stability, and injectivity. To explore their potential, bottle tests were conducted to assess gelation regions under different salinities, followed by characterization of gel properties. Furthermore, a novel dual-patterned glass micromodel was developed to simulate porous media for investigating solid gel efficiency in water conformance control, providing insights into potential applications in oil reservoir management.

gels as blocking agents, discussing their operational challenges and the role of nanomaterials like nanosilica. Various chemical systems have been developed over the years, including inorganic gels, monomer systems, polymer gels, un-gelled polymers, resins, and viscous flooding (polymers), each with distinct structures and applications for water shut-off.

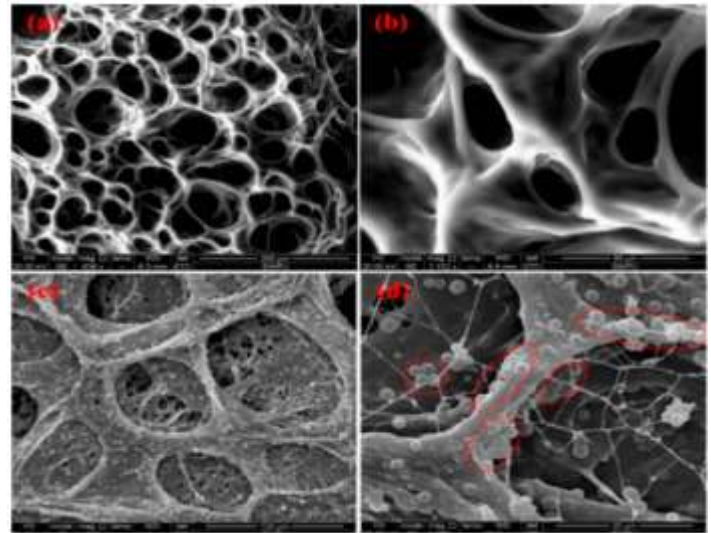
IV. METHODOLOGY

The review begins by addressing the inherent challenges associated with three distinct types of gel treatments: in situ polymer gel, preformed particle gel, and silicate gel. Figure 9 outlines the systematic methodology employed to evaluate the impact of nanomaterials on gel treatment, with nanomaterials categorized according to their effects on each gel type's properties. Notable enhancements have been observed in thermal stability, gelation time, gel strength, and swelling performance, attributed to the utilization of silica, clay, graphene, and zirconium oxide nanomaterials.

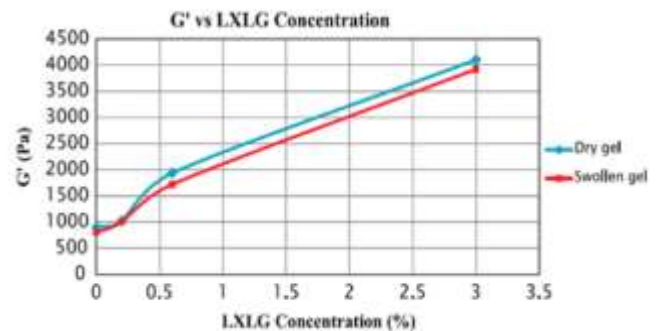


Gel treatments represent widely adopted chemical methods for mitigating water production in mature reservoirs, offering cost-effective solutions. Polymer gels effectively control water mobility by reducing permeability or blocking high-permeability zones and fractures, thereby improving sweep efficiency and increasing oil production. Depending on where gelation occurs, treatments are classified as in situ gel or preformed gel. In situ gel forms downhole, involving a mixture of polymer solution, cross-linker, and additives injected into the target zone. Conversely, preformed gels, such as PPGs, are generated at the surface before injection into the reservoir as particles, primarily addressing high-permeability zones and fractures. Silicate gels, with a mechanism akin to other gelling materials, form a brittle gel through chemical bonding between silicate solution and an activator, addressing water or gas production concerns. While silicate gels boast environmental friendliness and low cost, challenges include rapid gelation time and sensitivity to divalent ions.

The study focuses on overcoming operational challenges faced by these gel systems, necessitating modifications to composition. Recent advancements introduce nanomaterials like nanosilica, nanoclay, and nanographene to enhance gel properties. Accurate diagnosis of water intrusion root causes is crucial for effective treatment, with tracers and logging services aiding in source identification. Successful field applications highlight the efficacy of in situ polymer gels and silicate gels for addressing near-wellbore issues like casing and tubing leaks, while preformed particles excel in treating fractures, particularly in high-permeability zones.



Gelation Time (GT) serves as a crucial parameter in devising effective water shutoff treatments. Most mathematical models for polymer gels typically involve one dependent variable, GT, and three independent variables



: temperature, polymer concentration, and crosslinker concentration. To analyze the gelation kinetics of in-situ gels for water shutoff, researchers widely employ steady shear rate measurements.

The Arrhenius equation describes the impact of absolute temperature on reaction rate and elucidates the mechanism of chemical reactions. Hurd and Letteron developed an empirical model correlating the gelation time of silicic acid gels with temperature, resembling the Arrhenius equation. This correlation was validated using experimental data under certain assumptions



$$\ln k = \ln A - \frac{E_a}{R} \frac{1}{T}$$

$$\ln t = \ln c - \ln k - (n - 1) \ln a$$

$$GT = 38.4333 - \frac{13}{75}T + \frac{19}{30}S - \frac{67}{30}C + \frac{1}{100}TC - \frac{1}{300}$$

The developed model by Hurd and Letteron was further verified by Jorden et al., who examined the effect of temperature on GT using the PAM/Cr(III) gel system. They demonstrated that the Arrhenius equation can be applied to analyze various chemical reactions. Similarly, Broseta et al. validated the relationship between GT and temperature, focusing on the PAM/Cr (III) acetate system. They highlighted that temperature significantly influences GT and follows the Arrhenius equation.

Marfo et al. investigated a water shutoff gel comprising an acrylamide-acrylate copolymer crosslinked with a polyamine crosslinker. Using statistical analysis software, they developed a predictive GT model considering temperature, cross-linker concentration, and water salinity.

Modeling Water Shutoff Performance: Xianchao et al. predicted water shutoff performance in horizontal wells by modifying the PCGEL simulator, integrating gel flow physics, and considering gel degradation. They achieved reliable prediction results, aligning with actual field scenarios.

Alghazal and Ertekin proposed an artificial neural network (ANN) for deep polymer gel conformance treatments in fractured reservoirs. Their developed ANN module surpassed commercial simulators in speed and computational complexity, utilizing reference simulation modules for dataset construction.

Meshalkin et al. presented a three-dimensional computer model simulating water shutoff performance in high water cut oil zones. Their model, considering geophysical characteristics and injected water control solutions, demonstrated accuracy in predicting well performance after treatment.

Ferreira et al. focused on creating a neural network model to predict well performance post-gel treatment, aiding in ranking treatment candidates and optimizing resource use.

Limitations of Proposed Models: Existing coupled numerical models for water shutoff treatments lack integration of dynamic models between the wellbore and reservoir, simplifying gel blocking effects and dynamic interactions.

Despite available simulation tools, there exists a gap between prediction accuracy and field performance due to inadequate consideration of fluid composition and reservoir properties, impacting treatment efficiency and economic viability.

Field Cases in Oil Wells: Water shutoff treatments, including polymer gels, resins, and nanosilica, have been employed in various reservoir conditions, each with its advantages and limitations. Field cases analyzed different treatments, highlighting successes and challenges based on reservoir temperature.

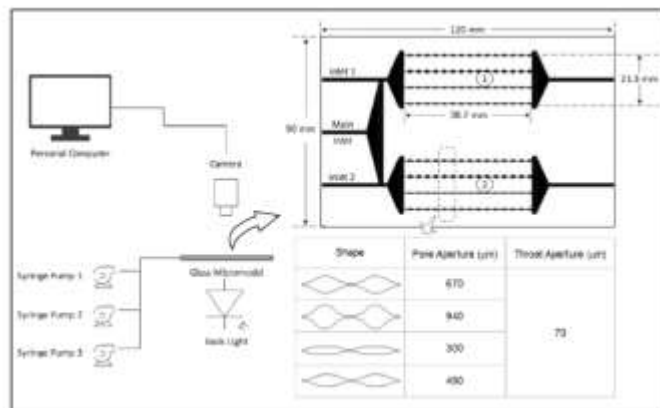
Materials and Methods

Materials: Aqueous hydrophilic silica with a SiO₂/Na₂O ratio of 75–100, comprising 30 wt% and characterized by bluish stock dispersion with a pH of 9–10 and particle sizes ranging from 10 to 30 nm (average 20 nm), was utilized in this study. Sodium chloride (NaCl) and magnesium chloride hexahydrate (MgCl₂·6H₂O) of ACS reagent grade (purity ≥ 99%) were procured from Sigma Aldrich (United States of America, USA) to adjust the salinity of dispersions. Crude oil from an Iranian reservoir with high water cut issues was also employed, and its properties are outlined in Table 1.

Methods: Determination of Gelation Regions: Various concentrations of silica (1–6 wt%) were prepared by diluting the 30 wt.% stock dispersion with deionized water (DIW), followed by the addition of varying concentrations of NaCl (1–11 wt%). After stirring for 30 minutes at room temperature (20–25 °C), the pH of all dispersions was measured using an 86502 AZ pH meter (AZ Instrument Corp., Taiwan). The dispersions were then transferred to glass vials, capped, and observed over 24 hours at room temperature to detect any visual changes in appearance. Gelation was assessed by gently rotating the sample vials by 180°. **Gelation Time:** The gelation time of solid gels was determined using both bottle tests and UV–Vis spectroscopy. The absorbance of particles was measured at 400 nm using a Dynamica DB-20S spectrophotometer (UK). Bottle tests provided the time of complete solid gelation, while spectroscopy indicated the onset of gelation. Fresh dispersions forming single-phase solid gels were prepared following similar procedures as the bottle tests. Absorbance measurements were recorded over time, and the onset of gelation was identified from the plot of absorbance versus time. **Effect of Salt Type on Gelation:** To examine the effect of cation valence on gelation, magnesium chloride replaced sodium chloride in bottle tests conducted in the "Determination of Gelation Regions" section. Visual changes were recorded over time.



Rheology of Gels: Fully solidified gels from bottle tests were rheologically analyzed using an Anton Paar (UK) cone/plate method at 25 °C. Fresh gellants were prepared with identical silica and salt concentrations and left undisturbed for 24 hours at room temperature to ensure gelation stability. Viscosity, storage modulus (G'), and loss modulus (G'') were measured under different shear strains/stresses.



Displacement Study by Glass Micromodel: A novel double-patterned glass micromodel was designed and constructed to assess the efficacy of solid gels for water conformance control in oil reservoirs. This setup, not previously utilized, involved three syringe pumps (LA-30, LANDGRAVE HLL, Germany), a back light source, a glass micromodel, and a camera connected to a computer. The glass micromodel comprised two identical porous media segments with separate inlets, simulating water-bearing zones overlying oil layers in reservoirs. Prior to the main experiment, control tests were conducted to validate the functionality of the micromodel components. The main experiment involved saturating one porous medium with a solid gel dispersion and injecting deionized water (DIW) through the main inlet to assess the gel's ability to divert flow towards the oil-saturated zone.

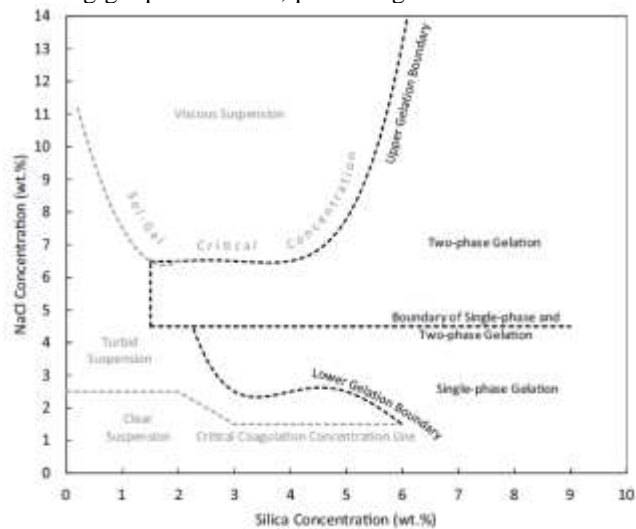
Control Experiment: Before the main experiment, the functionality of the glass micromodel was verified by saturating both porous media with crude oil and subsequently injecting DIW to ensure uniform displacement from both segments.

Main Experiment: A dispersion forming a solid gel after 24 hours at room temperature was chosen for saturating one porous medium in the glass micromodel. After saturation, DIW was injected through the main inlet to assess the gel's effectiveness in diverting flow towards the oil-saturated zone. Macro- and microscopy techniques were employed to analyze fluid displacement during the experiment, and the amount of recovered oil was determined using image analysis of porous medium images.

V. RESULTS

Water shutoff treatments in oil and gas wells employ various methods and materials, each with its own set of advantages and limitations. Polymer gels, resins, and nanosilica have been utilized across a broad spectrum of reservoir temperatures. Preformed gels

offer stability under thermal conditions but necessitate precise placement. Resins boast exceptional mechanical strength but may face challenges regarding injectability. Nanosilica treatments exhibit versatility and applicability even in ultrahigh temperatures. Further research and field studies are imperative to optimize water shutoff treatments tailored to specific reservoir conditions. The advancement in nanomaterial utilization holds promise for enhancing gel performance, presenting several future challenges:



SAMPLE A



SAMPLE B

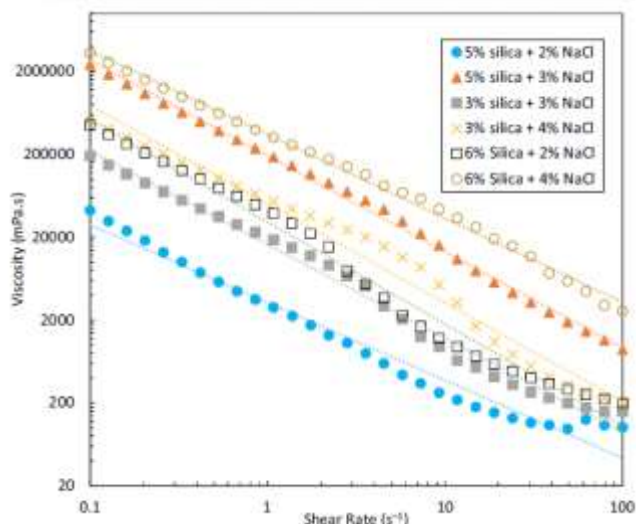
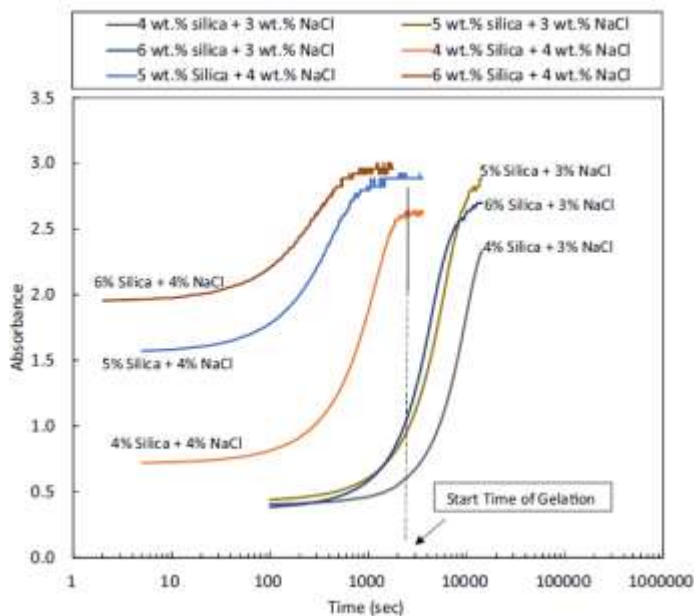


SAMPLE C

6.1. In-Situ and Preformed Particle Polymer Gel: While studies have showcased the beneficial impact of nanomaterials like nano silica, nanoclay, and nanographene, future investigations should explore a broader array of nanomaterials approved for their applicability in EOR applications, such as Aluminum Oxide (Al_2O_3), Titanium dioxide (TiO_2), and Zinc Oxide (ZnO). Understanding the compatibility with different polymers and compositions is an area requiring further exploration. Determining the most effective nanomaterial-polymer combinations can amplify the applicability of these gels. Long-term stability and performance, along with potential degradation, necessitate in-depth investigation. Understanding the behavior of these gels over extended periods within reservoirs is crucial. This involves evaluating the longevity of gel stability and identifying potential degradation mechanisms to ensure sustained effectiveness in preventing water flow. Nanomaterials significantly bolster the mechanical properties of gels while ensuring thermal stability, rendering them suitable for use in harsh environments with temperatures exceeding 300 °C and high salinity levels. Further inquiries are warranted in this domain. The transition from laboratory experiments to field applications poses a significant



research gap. Extensive lab work and field trials are essential to validate the practicality and efficacy of nanomaterial-enhanced gels in actual reservoirs with varying characteristics.



6.2. Nanosilica-Based Gel: Studies have indicated the sensitivity of gelation time to temperature variations. Future research should delve deeper into the mechanisms underlying this sensitivity and explore additives to mitigate the impact of temperature on gelation time. This is crucial for ensuring successful placement of silicate gels across a wide temperature range. While research has confirmed that nanosilica enhances the plugging efficiency of silicate gels, additional studies can focus on assessing the long-term stability and endurance of these gels under reservoir conditions. This includes evaluating their injectivity, stability, and plugging efficiency over extended periods to affirm their effectiveness in preventing unwanted water flow. With its remarkable plugging efficiency of 100%, nanosilica gel emerges as a viable alternative to polymer in situ gels. Its low viscosity and

environmental friendliness further enhance its appeal for various scientific applications.

Conclusion: Polymer and silicate gels stand as the most widely applied chemical systems for water shut-off applications. The incorporation of nanomaterials in these treatments has the potential to substantially enhance efficiency and effectiveness. Gelation time, along with its impact on reservoir characteristics, plays a pivotal role in the placement process. Therefore, modeling treatments will aid in enhancing agents' performance as water shut off agents. Ultimately, the impact of nanomaterials can be summarized as follows:

1. Nanomaterials, such as nanoclay, nanosilica, and nanographene, can significantly adjust the properties of in situ gels, including control of gelation time, enhancement of gel strength, and maintenance of thermal stability at high temperatures.
2. Incorporating nanomaterials into preformed particle gels (PPG) could lead to adjustments in swelling ratio and improvements in gel strength to withstand reservoir conditions.
3. Experimental results of nanosilica gel demonstrate enhanced plugging efficiency, reinforcing its potential usage as a plugging material alongside other advantages of silicate systems.

Determining Gelation Regions: Figure S-1 in the Supporting Information confirms that particles alone (without salt) cannot form a gel within the concentration range studied here. Gelation is solely attributed to the effect of added ions on particle interactions. Figure S-2 illustrates the outcomes of various dispersions containing different concentrations of silica and NaCl in deionized water (DIW) at room temperature for 24 hours. These results have been incorporated into the plot in Figure 2, offering a comprehensive depiction of gelation concerning silica and salt concentrations. The boundaries depicted in this figure are fitted models ($R^2 > 0.8$) to the data presented in Figure S-2.

Five potential states based on particle and NaCl concentrations are discernible in Figure 2: clear suspension, turbid suspension, viscous suspension, single-phase solid gel, and two-phase gel (gel + liquid). Figure S-3 in the Supporting Information displays a typical photo of different classes obtained after 24 hours in glass vials. Clear suspensions exhibit no visual change in color compared to the initial bluish hue. Turbid suspensions show some degree of aggregation, while viscous suspensions appear milky and viscous. Single-phase gels are entirely solid with no liquid phase, whereas two-phase gels consist of solid gel topped with a liquid phase. Completely solid gels do not flow upon rotating the vials. Metin et al. found that two-phase gelation occurs at low silica concentrations, contrasting with the findings of this study, where single- and two-phase gelation depend more on salt concentration than particle concentration.

Critical Coagulation Concentration (CCC): The critical coagulation concentration (CCC) denotes the salt concentration above which the dispersion becomes unstable. At CCC, salt ions



screen particle surface charges, causing bare charged particles to aggregate and render the dispersion unstable. Colloidal particles can form gels above CCC, with gel formation occurring due to particle entrapment. At short distances, Van der Waals (VdW) attractions between particles may overcome electrostatic repulsions, leading to the aggregation of particles into larger structures. However, if the salt concentration is well above CCC, gelation may not occur, resulting instead in a viscous suspension without particle stranding.

Gelation Time: The gelation time, i.e., the time of complete gelation, was visually evaluated in bottle tests. Figure 5 illustrates the gelation time in hours for different silica and sodium chloride concentrations in DIW. Increasing salt concentration at a fixed silica concentration decreases gelation time, particularly for particle concentrations ≥ 4 wt%. Conversely, gelation time decreases with increasing particle concentration at both salt concentrations (3 and 4 wt%), following a power law ($R^2 > 0.91$). The decrease in gelation time is more pronounced at the highest salt concentration. The gellant containing the highest silica and NaCl concentrations (6 wt% and 4 wt% NaCl in DIW) exhibits the shortest complete gelation time.

Start Time of Gelation: The start time of gelation was determined by UV-Vis spectroscopy at 400 nm for dispersions forming single-phase solid gels. Figure 6 depicts the absorbance of different gellants at 400 nm over time. The first point where the diagrams plateaued was considered the start time of gelation. The difference between complete gelation time (determined by bottle tests) and start time of gelation (determined by absorbance measurements) indicates the time required for each dispersion to gel completely. Reducing salt concentration by 1 wt% at a fixed silica concentration significantly delays gelation time, while the required time for gelation remains relatively unchanged for varying particle concentrations at a given salt concentration.

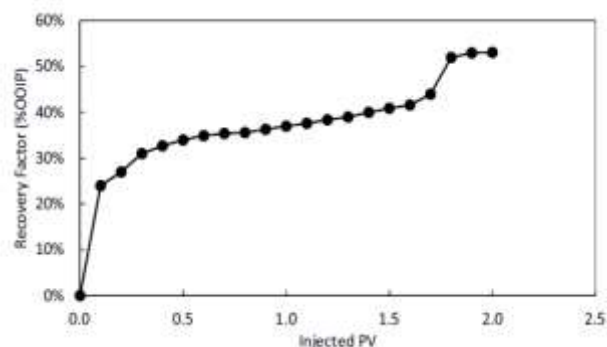
Effect of Type of Salt on Gelation: To investigate the influence of the cation type on gelation, monovalent Na^+ was replaced with divalent Mg^{2+} in some random dispersions. CCC is inversely proportional to the valence of the coagulating ion in the dispersion. Comparing NaCl and MgCl_2 , the higher valence cation can destabilize the dispersion more effectively, as evidenced by a 64-fold reduction in CCC when using Mg^{2+} . Gelation type depends solely on salt concentration; increasing salt concentration at a fixed particle concentration does not necessarily lead to gel formation. At high salt concentrations, viscous suspensions are formed instead of gels.

CONCLUSION

The study investigated the formation of colloidal gels at room temperature by introducing various concentrations of salt to hydrophilic silica dispersions for water conformance control in oil reservoirs. Bottle tests were conducted to define the gelation window concerning different particle and salt concentrations, as well as to examine gelation time, the effect of salt type, and rheology and viscosity. Subsequently, the strength of the solid gel

was evaluated in porous media using a novel dual-patterned glass micromodel designed by the research group to simulate a water producer layer overlying oil layers. The main findings of this study are summarized as follows:

- Bottle test results yielded well-classified data with clear boundaries based on different silica (1–6 wt%) and NaCl (1–11 wt%) concentrations. Various classes were identified after 24 hours at room temperature, including clear suspensions, single- or two-phase gels, and (non)viscous suspensions. It was concluded that single- or two-phase gelation primarily depends on salt concentration rather than particle concentration. The findings suggest that a high salt concentration is not necessarily required for gel formation (single- or two-phase gelation), and higher NaCl concentrations at a constant silica concentration may result in viscous suspensions. Thus, determining the gelation window for a specific salt is crucial. The machine learning-based decision tree developed in this study effectively predicted the fate of dispersions after a 24-hour resting time at room temperature, with 39% of total observations in bottle tests indicating single- and two-phase gels.



- The type of salt significantly affects the final state of dispersions. Replacing monovalent sodium cations with divalent magnesium at the same ionic strengths in some random dispersions led to notably different behavior, mainly due to the higher coagulating ability of divalent cations in colloids. This highlights the need for comprehensive studies on MgCl_2 as future research.
- Gelation times of solid gels were visually determined in bottle tests (complete gelation time) and further examined through UV-Vis spectroscopy (start time of gelation). The difference between these two times provided good estimates of the time required for each gellant to gel completely. In the single-phase solid gelation window, the highest silica and NaCl concentrations resulted in the shortest gelation time, i.e., 6 wt% silica and 4 wt% NaCl in DIW. Additionally, an additional 1 wt% salt reduced the gelation time by 2.5 times.
- Rheology and viscosity analyses corroborated the gelation time results, showing the same parametric dependence on particle and salt concentrations. At high shear rates (100 s⁻¹), the formed solid gels exhibited viscosities, storage moduli, and loss moduli of 100–2600 mPa s, 2–57 Pa, and 7–430 Pa, respectively, which are sufficient to withstand pressure gradients applied in wells. The gel formed by 5 wt%



silica and 2 wt% NaCl demonstrated the highest elasticity. However, it was observed that strong gels may become fragile under shear stresses and should be avoided for water conformance controls.

- Injection of DIW into a dual-patterned glass micromodel initially saturated with crude oil in the top porous medium and a solid gel in the bottom porous medium demonstrated the effective performance of the solid gel in blocking the bottom porous medium. This allowed the injected DIW to divert its flow toward the oil-saturated zone for potential enhanced oil recovery (EOR). A total oil recovery of 53% original oil in place was achieved, which would not have been possible without the blockage of the bottom porous medium by the gel. This promising observation in a visualization study underscores the potential applicability of colloidal gels for water shut-off applications.
- Other parameters such as temperature, ion type, particle size, and particle surface wettability may also be important in gelation and should be considered in future research efforts.

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