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Phase-separation behavior of polyanionic cellulose, sodium humate and their mixtures in calcium-magnesium salt media: implications for salt-resistant drilling fluids

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Фазовое расслоение полианионной целлюлозы, гумата натрия и их смесей в средах на основе солей кальция и магния: значение для солеустойчивых буровых растворов

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Abstract

The objective of this study is to quantify and interpret the phase-separation thresholds of polyanionic cellulose (PAC), sodium humate (Na-GK) and PAC + Na-GK mixtures under calcium-magnesium salt aggression in sodium-chloride-saturated media. The work addresses the stability

reserve required for water-based drilling fluids used in salt-bearing and highly mineralized formations. Polymer solutions of different concentration, PAC substitution degree and polymerization degree were examined by nephelometric titration with CaCl2 and a mixed CaCl2 + MgCl2 titrant after controlled preparation and, where required, thermal pretreatment at 105 °C. The results show that phase separation is controlled not only by the total concentration of divalent cations, but also by polymer molecular architecture, hydration of cation-polyion complexes and the different coordination behavior of Ca2+ and Mg2+. Mixed CaCl2 + MgCl2 titration required approximately 1.5 – 1.8 times more titrant to induce phase separation than CaCl2-only titration. PAC samples with a substitution degree of 115 – 125 and high polymerization degree showed the highest resistance, while heating reduced the limiting concentration by about 4 – 10 %. The combined use of PAC and Na-GK increased resistance to divalent salts and is therefore a rational direction for designing salt-resistant drilling fluids.

Аннотация

Целью исследования является количественная оценка и физико-химическое объяснение порогов фазового разделения полианионной целлюлозы (ПАЦ), гумата натрия (Na-ГК) и смесей ПАЦ + Na-ГК в насыщенной хлоридом натрия среде при воздействии кальций-магниевых солей. Работа направлена на определение запаса устойчивости, необходимого для водных буровых растворов, применяемых в соленосных и высокоминерализованных пластах. Растворы полимеров различной концентрации, образцы ПАЦ с разной степенью замещения и степенью полимеризации исследованы методом нефелометрического титрования растворами CaCl2 и смешанным титрантом CaCl2 + MgCl2 после контролируемой подготовки и, при необходимости, термообработки при 105 °C. Полученные результаты показывают, что фазовое разделение определяется не только общей концентрацией двухвалентных катионов, но и молекулярной архитектурой полимера, гидратацией комплексов «катион-полиион» и различиями координационного поведения Ca2+ и Mg2+. Для инициирования фазового разделения смешанным титрантом CaCl2 + MgCl2 требуется примерно в 1,5 – 1,8 раза больше реагента, чем при титровании только CaCl2. Наибольшую устойчивость показали образцы ПАЦ со степенью замещения 115 – 125 и высокой степенью полимеризации, тогда как нагрев снижал предельную концентрацию титранта примерно на 4 – 10 %. Совместное применение ПАЦ и Na-ГК повышает устойчивость к двухвалентным солям и является рациональным направлением разработки солестойких буровых растворов.

Keywords: polyanionic cellulose, sodium humate, Na-GK, phase separation, calcium chloride, magnesium chloride, salt-resistant drilling fluid, nephelometric titration, polymer-salt interaction, drilling mud stability.

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Ключевые слова: полианионная целлюлоза, гумат натрия, Na-ГК, фазовое расслоение, хлорид кальция, хлорид магния, солеустойчивый буровой раствор, нефелометрическое титрование, взаимодействие полимера с солью, стабильность бурового раствора.

Introduction

Drilling through salt-bearing deposits, clay-carbonate interlayers and intervals containing highly mineralized formation waters creates a chemically aggressive environment for water-based drilling fluids. In such systems, technological performance is determined not only by density, rheology and filtration loss, but also by the ability of the polymer phase to remain soluble and structurally active under strong electrolyte loading. Recent reviews of synthetic drilling-fluid polymers identify high salinity and multivalent-ion contamination as major causes of rheological and filtration deterioration [1]. Standard drilling-fluid practice likewise treats salt and divalent-ion effects as separate performance constraints that must be evaluated quantitatively [13]. Calcium and magnesium salts can neutralize anionic polymer groups, compress the electrical double layer, promote polymer bridging and trigger irreversible flocculation; uncontrolled destabilization therefore increases filtration loss and the risk of wellbore instability.

Polyanionic cellulose (PAC) remains one of the most widely used water-soluble polymers in drilling-fluid technology. Its carboxymethyl substituents provide hydration, viscosity-building and filtrate-reducing functions, but the same anionic groups also act as binding sites for divalent cations. Calcium and magnesium ions can alter chain conformation and promote intermacromolecular association. The salt and calcium sensitivity of PAC-based systems is well documented; PAC-derived nanocomposites have therefore been engineered specifically to improve high-temperature, salt and CaCl2 resistance [2]. Consequently, PAC performance should be assessed in polymineral media rather than only in fresh water or simple NaCl brine.

Sodium humate, designated in this study as Na-GK, is another functional reagent used in mineralized drilling systems. Humate materials contain carboxyl, phenolic and other polar groups capable of interacting with mineral surfaces, dissolved ions and polymer additives [10]. Recent reviews also identify modified natural materials, including humic-acid-derived reagents, as a relevant route for rheology and stability control in water-based drilling fluids [9]. In a PAC-based system, Na-GK may redistribute ion-binding events and modify the hydration shell of polymer aggregates; the practical question is therefore whether the PAC + Na-GK mixture shifts the phase-separation threshold under simultaneous Ca-Mg aggression.

The relevance of this issue is high for engineering practice. In salt formations and formation-water inflow zones, contamination of the circulating fluid is gradual and compositionally variable. The system may first encounter NaCl saturation and then receive Ca2+ and Mg2+ from dissolving minerals or entering formation water. If the polymer package has a narrow stability reserve, a small increase in divalent cation concentration can abruptly transform a functioning drilling fluid into a flocculated suspension. Such failure is particularly dangerous in extended-reach and deep wells, where replacement of the whole fluid system is costly and where wellbore instability can develop faster than corrective treatment is applied.

Accordingly, the stated objective of this article is to determine the limiting concentrations at which PAC, Na-GK and PAC + Na-GK systems undergo salt-induced phase separation in NaCl-saturated calcium-magnesium media, and to identify the polymer-structural factors that increase

resistance to this transition. The working hypothesis is that mixed Ca-Mg aggression cannot be predicted from the total divalent-cation concentration alone because Ca2+ and Mg2+ generate different hydration and coordination states in anionic polymer complexes.

Literature Review and Theoretical Background

The literature on drilling-fluid chemistry shows that water-based fluids are multicomponent colloid-polymer systems whose stability depends on electrolyte composition, clay dispersion, pH, temperature and polymer architecture. Micro-crosslinked polyampholytes retain filtration performance after high-temperature aging in saturated-salt media [3]. Zwitterionic polymers likewise preserve useful drilling-fluid performance under concentrated NaCl and CaCl2 contamination [4]. Ternary polyampholytes have also been developed as salt-tolerant rheology enhancers and fluid-loss additives, with PAC-LV used as a direct comparator [5]. These studies show that molecular architecture, rather than polymer dosage alone, controls tolerance to strong electrolyte loading.

From the viewpoint of polyelectrolyte theory, salt-induced phase separation involves electrostatic screening, counterion condensation, dehydration of macromolecular coils, formation of intra- and intermolecular bridges, and aggregation of complexes that are no longer sufficiently hydrated to remain dispersed. Polymer-chain length, charge density and solvent-mediated interactions determine whether ion binding produces reversible contraction or irreversible aggregation [11]. Surface-force theory further explains why hydration and ion-specific interactions can modify the onset of colloidal instability [12].

The distinction between calcium and magnesium is especially important. Both ions can interact with carboxylate-containing macromolecules, but they differ in ionic radius, hydration energy and water-exchange behavior. Calcium can efficiently promote carboxylate bridging and chain contraction, whereas the more strongly hydrated magnesium ion can retain a larger hydration shell around ion-polymer complexes. On this basis, the working mechanistic interpretation of the present study is that mixed Ca-Mg media cannot be described by total divalent-cation concentration alone; ion-specific hydration must also be considered [11, 12].

Materials and Methods

The methodological basis of the study is a comparative physicochemical evaluation of water-soluble polymer reagents intended for drilling fluids operating under salt aggression. The tested systems included PAC samples differing in degree of polymerization and degree of substitution, sodium humate (Na-GK), and PAC + Na-GK mixtures. The base liquid phase was a saturated sodium chloride solution prepared from analytical-grade NaCl and distilled water at 25 ± 1 °C. Excess NaCl was contacted with water for 24 h, the liquid was filtered from undissolved crystals, and the final NaCl content was taken as 26.4 wt.% at the preparation temperature.

The principal aggressive components were calcium chloride and a mixed calcium-magnesium chloride titrant. The CaCl2-only titrant was prepared as a 1.000 mol/L solution. The mixed titrant was prepared from CaCl2 and MgCl2 solutions in a final composition of 0.500 mol/L CaCl2 + 0.585 mol/L MgCl2; the total concentration of divalent cations was therefore 1.085 mol/L, and the

Ca2+:Mg2+ molar ratio was 0.46:0.54. This composition was selected to model polymineral aggression while preserving the experimentally observed condition that the mixed titrant contains approximately 8.5 % more divalent cations than the calcium-only titrant.

Polymer concentration was considered as a key variable because dilute and concentrated polymer solutions do not have the same resistance to salt loading. The experimental series distinguished 0.7 wt.% and 1.4 wt.% solutions of Na-GK, PAC and PAC + Na-GK mixtures. For each determination, 100 mL of polymer solution was titrated stepwise with 0.05 – 0.10 mL portions of titrant. The phase-separation threshold was fixed at the first persistent turbidity break followed by visible opalescence or flocculation. The limiting titrant volume was recalculated as mL of titrant per 100 mL of polymer solution.

The molecular architecture of PAC was evaluated using degree of polymerization (DP) and degree of substitution (DS). The experimental series included low-polymerization specimens around DP 110 – 136, medium specimens around DP 410 – 450 and high-polymerization specimens around DP 1310 – 1400; DS covered approximately 84 – 100, 115 – 125 and 155 – 160. DP is used here as the available chain-length descriptor. Size-exclusion/gel-permeation chromatography was not part of the present dataset, so molecular-weight distribution parameters (Mn, Mw and dispersity, Đ) are not reported and no conclusion is drawn about distribution breadth.

Thermal history was also examined. Some PAC solutions were titrated without prior heating, while others were preheated at 105 °C before calcium-magnesium titration. The inclusion of thermal pretreatment is important because drilling fluids often circulate at elevated bottomhole temperatures. Heating may change polymer conformation, reduce molecular weight through partial degradation or alter the balance between hydrated and ionically cross-linked forms.

Table 1.

Experimental variables considered in the phase-separation assessment

Variable group	Specific parameter	Technological meaning
Polymer reagent	PAC, Na-GK, PAC + Na-GK mixture	Determines the chemical nature of hydration, adsorption and ion binding
Base electrolyte	NaCl-saturated aqueous medium	Models salt-bearing rock intervals and high-mineral formation-water contamination
Aggressive salts	CaCl2 and CaCl2 + MgCl2 mixture	Represents monomineral and polymineral divalent-cation aggression
PAC architecture	Degree of substitution and degree of polymerization	Controls solubility, chain conformation and resistance to cation bridging
Thermal history	Untreated and preheated at 105 °C	Models thermal aging during circulation in deeper wells
Analytical criterion	Nephelometric titration threshold	Indicates onset of turbidity, aggregation and phase separation

Results

The first major result is that phase separation in the studied polymer systems follows the same general regularity under mixed CaCl2 + MgCl2 aggression as under CaCl2-only aggression, but the quantitative thresholds are different. The measured values are presented as mean ± standard deviation in Table 2, and they provide the numerical basis for comparing experimental and calculated behavior.

The second and most technologically important result is counterintuitive: the amount of CaCl2 + MgCl2 titrant required to initiate phase separation is considerably higher than the amount required when only CaCl2 is used. At equal titrant volume, the total concentration of divalent cations in the CaCl2 + MgCl2 mixture is about 8.5 % higher than in the calcium-only titrant. Nevertheless, precipitation of the polymers requires approximately 1.5 – 1.8 times more of the mixed titrant. This observation proves that magnesium does not simply intensify the salt aggression in proportion to its charge; instead, it changes the hydration and binding mechanism of the polymer-cation complex, delaying the loss of solubility.

The third result concerns the role of PAC structure. The most salt-resistant PAC samples were those with a degree of substitution in the interval of approximately 115 – 125 combined with a high degree of polymerization. In the graphic data corresponding to the PAC-(CaCl2 + MgCl2) system, high-polymerization PAC samples display the highest limiting concentrations before phase separation. Medium-polymerization samples show lower resistance, while low-polymerization samples are much less stable. At very high substitution degrees, the limiting concentration declines sharply. Therefore, the relationship between substitution and stability is not linear: insufficient substitution weakens hydration and solubility, but excessive substitution appears to facilitate more intensive cation binding and unfavorable conformational changes.

The fourth result relates to thermal pretreatment. Heating PAC solutions at 105 °C before titration decreases the limiting titrant concentration by approximately 4 – 10 %. This reduction means that the resistance of PAC to calcium and magnesium salts becomes weaker after thermal exposure. The decrease is not identical for all samples. PAC with substitution degrees around 84 – 100 appears less sensitive to heating than some other specimens, but the general trend remains negative.

The fifth result is the beneficial effect of combining PAC with Na-GK. The joint use of PAC and sodium humate increases the resistance of polyanionic cellulose to calcium and magnesium salts. This improvement has practical value because it allows the design of salt-resistant drilling-fluid reagents with a wider stability margin. The stabilizing effect can be explained by intermolecular association between PAC and Na-GK macromolecules or aggregates, as well as by the distribution of cation-binding events across chemically different functional groups. In other words, the mixture may prevent divalent cations from concentrating their cross-linking action on PAC alone.

Table 2.

Experimental phase-separation thresholds for PAC, Na-GK and PAC + Na-GK systems (mean ± SD, n = 3)

System	CaCl2 threshold, mL/100 mL	CaCl2 + MgCl2 threshold, mL/100 mL	Mixed/CaCl2 ratio	RSD range, %
Na-GK 0.7 wt.%	0.68 ± 0.03	1.12 ± 0.04	1.65	3.6-4.4
Na-GK 1.4 wt.%	0.94 ± 0.04	1.58 ± 0.06	1.68	3.8-4.3
PAC DS 90 / DP 120	0.50 ± 0.02	0.82 ± 0.03	1.64	3.7-4.0
PAC DS 120 / DP 1350	1.44 ± 0.05	2.44 ± 0.08	1.69	3.3-3.5
PAC DS 158 / DP 1350	0.82 ± 0.03	1.32 ± 0.05	1.61	3.7-3.8
PAC + Na-GK 1:1	1.25 ± 0.04	2.05 ± 0.07	1.64	3.2-3.4

Table 3.

Generalized experimental findings and their drilling-fluid interpretation

Observed result	Interpretation	Engineering implication
Experimental titrant consumption exceeded calculated values by about 5-10% in mixed Ca-Mg systems	Phase separation is not strictly additive or stoichiometric	Use experimental thresholds instead of simple salt-balance estimates
CaCl2 + MgCl2 required 1.5-1.8 times more titrant than CaCl2 alone	Magnesium changes hydration and coordination of polymer-cation complexes	Mixed-salt tests are mandatory for realistic contamination screening
PAC with substitution degree 115-125 and high polymerization degree showed highest resistance	Balanced hydration and limited cation bridging produce maximum salt tolerance	Select PAC by molecular parameters, not only commercial grade name
Preheating at 105 °C reduced limiting titrant concentration by 4-10%	Thermal exposure reduces polymer stability reserve	Include thermal aging before field approval of reagent packages
PAC + Na-GK improved resistance to Ca and Mg salts	Mixed macromolecular aggregates distribute ion-binding and delay insolubilization	Use PAC + Na-GK as a base direction for salt-resistant drilling-fluid systems

Discussion

The results show that polymer phase separation in mineralized drilling-fluid systems is a coupled colloid-chemical process rather than simple stoichiometric charge neutralization. If total divalent-cation concentration were the only controlling factor, the mixed CaCl2 + MgCl2 titrant would precipitate PAC and Na-GK faster than CaCl2 alone; the opposite tendency was observed. Recent amphoteric-polymer studies likewise show that salinity response depends strongly on polymer architecture and hydration state [6]. Core-shell salt-resistant additives provide another example in which interfacial architecture stabilizes water-based systems under severe electrolyte loading [8]. The present data therefore support an ion-specific interpretation of the mixed Ca-Mg threshold rather than a simple total-cation model.

Calcium has a strong tendency to create ionic bridges between carboxylate groups, which can connect polymer segments and reduce solubility. Magnesium differs because its hydration shell is stronger; a larger fraction of coordinated water can remain associated with Mg-containing polymer complexes before dehydration and aggregation become dominant. This difference provides a chemically plausible explanation for the larger titrant consumption observed in the mixed calcium-magnesium system. The interpretation is mechanistic rather than spectroscopic: no FTIR measurement of Ca- or Mg-bound PAC/Na-GK complexes was performed in the present experimental series.

The optimum substitution degree of PAC around 115 – 125 is also chemically meaningful. A low substitution degree gives fewer anionic hydration centers and therefore weaker solubility in brine. A moderate substitution degree provides sufficient hydration and steric-electrostatic stabilization. However, excessive substitution can increase the density of carboxylate sites, making the chain more susceptible to divalent-cation bridging. In addition, highly substituted chains may adopt conformations that reduce the effectiveness of steric shielding. The best PAC for salt-resistant

drilling fluids is therefore not the most substituted polymer, but the polymer whose substitution and polymerization degrees produce the most favorable balance between hydration, chain extension and resistance to ionic cross-linking.

The effect of degree of polymerization is equally important. High-polymerization PAC samples resisted phase separation better than low-polymerization samples. This may be explained by stronger macromolecular continuity, more effective formation of a hydrated network and a greater ability to distribute ion-binding events over a longer chain. Low-polymerization fragments, by contrast, are less able to maintain a protective hydrated structure and may form compact complexes more readily. In drilling fluids, this means that thermal or mechanical degradation of PAC is not only a viscosity problem; it also reduces resistance to calcium-magnesium contamination.

The negative effect of preheating at 105 °C confirms this interpretation. Heating can change the coil-to-aggregate balance, expose carboxylate groups, reduce polymerization degree or promote a transition toward more ionically bound structures. Even a 4 – 10 % reduction in the limiting titrant concentration is significant in field practice, because drilling-fluid contamination often develops cumulatively. A system operating close to its stability threshold may fail after only a modest additional inflow of mineralized water. Therefore, laboratory screening of polymer reagents should include thermal aging before salt-tolerance testing.

The PAC + Na-GK mixture is promising because it introduces chemical heterogeneity into the polymer phase. Sodium humate contains multiple functional groups and macromolecular fragments capable of interacting with both ions and PAC. This can distribute calcium and magnesium binding across different molecular domains, reduce the probability of direct PAC-PAC bridging and preserve dispersion stability. The mixture may also improve adsorption on mineral surfaces, thereby supporting filtration control and shale inhibition. However, this advantage will be realized only if the dosage is optimized. Excess organic reagent may increase viscosity, promote unwanted flocculation or complicate rheological control.

For practical formulation, the findings imply that salt-resistant drilling fluids should be designed around stability reserve rather than initial rheological values alone. A polymer package can display acceptable viscosity and filtration before contamination yet approach a phase-separation boundary under Ca-Mg stress. Accordingly, screening should combine NaCl saturation, CaCl2 + MgCl2 titration, thermal aging and turbidity monitoring. Rheological characterization is a complementary, not interchangeable, endpoint; current API guidance treats viscosity, yield behavior and hydraulics as dedicated quantitative performance variables [13].

Comparison with commonly used and recently reported additives. Cross-study numerical comparison must be interpreted cautiously because published studies use different mud compositions, aging protocols and endpoints. Nevertheless, two direct industrial reference points are available. Wei et al. reported that sodium alginate maintained rheological and filtration performance under concentrated salt contamination better than the commonly used PAC-LV and Na-CMC comparators [7]. Mukhametgazy et al. reported lower fluid loss for a ternary polyampholyte than for PAC-LV in high-salinity bentonite systems [5]. PAC-derived nanocomposites have also demonstrated strong CaCl2 tolerance at elevated temperature [2], while a zwitterionic polymer maintained useful performance in 20 wt.% NaCl and 5 wt.% CaCl2 after

thermal aging [4]. The PAC + Na-GK system studied here should therefore be viewed as a simpler mixed-reagent route whose demonstrated endpoint is delayed phase separation, not yet full equivalence to these formulations in API rheology and filtration tests.

Scope limitations: pH, molecular-weight distribution and near-threshold rheology. The role of pH cannot be quantified from the present matrix because pH was controlled/monitored but not intentionally varied. Likewise, the PAC series is characterized by DP and DS, not by SEC/GPC-derived Mn, Mw and dispersity. Finally, apparent viscosity, plastic viscosity, yield point and viscoelastic moduli were not measured immediately around the nephelometric phase-separation threshold. This distinction matters because high-salinity amphoteric-polymer systems can show substantial rheological changes before macroscopic precipitation [6]. These three items are therefore stated as experimental limitations and as priorities for the next validation stage rather than being inferred from unavailable data.

Several failure modes should be considered. First, a PAC grade with too low polymerization may pass a short-term viscosity test but fail under divalent-cation titration. Second, excessive substitution may create a false expectation of improved salt tolerance while actually increasing susceptibility to bridging. Third, temperature can reduce the safety margin of polymer stability. Fourth, magnesium cannot be treated merely as an additional source of divalent charge; it changes the precipitation pathway. Fifth, the PAC + Na-GK mixture improves tolerance, but this improvement should be verified for each brine composition and temperature range instead of being assumed universal.

Conclusion

The phase separation of PAC, Na-GK and their mixtures under calcium-magnesium salt aggression is governed by a combination of polymer architecture, ion-binding mechanism and hydration stability. The experimental protocol specifies NaCl saturation conditions, CaCl2 and CaCl2 + MgCl2 titrant composition, thermal pretreatment, replicate measurement and statistical reporting. The mixed CaCl2 + MgCl2 titrant produces a different destabilization pathway from CaCl2 alone: although it contains a higher total divalent-cation concentration at equal volume, approximately 1.5 – 1.8 times more mixed titrant is required to reach the phase-separation threshold.

The most resistant PAC samples are those with a degree of substitution around 115 – 125 and a high degree of polymerization. Prior heating at 105 °C decreases the phase-separation threshold by about 4 – 10 %, indicating that thermal history should be included in all screening programs for salt-resistant drilling-fluid reagents. The combination of PAC and Na-GK increases resistance to calcium and magnesium salts and can serve as a scientifically justified basis for creating drilling fluids suitable for salt-bearing formations and intervals affected by highly mineralized formation waters.

Practical Recommendations

1. PAC grades selected for salt-resistant drilling fluids should have a controlled substitution degree near the experimentally favorable interval of 115 – 125 and a sufficiently high degree of polymerization.

2. PAC + Na-GK mixtures should be considered as a more robust polymer package for calcium-magnesium contaminated systems, but the optimum ratio must be determined experimentally for each brine composition.

3. Laboratory screening should include NaCl saturation, mixed CaCl2 + MgCl2 titration, thermal aging at relevant temperatures and turbidity-based determination of phase-separation thresholds.

4. Formulation decisions should not be based only on viscosity or filtration loss in uncontaminated mud; they must include a quantitative stability reserve against divalent-cation aggression.

5. Future research should combine nephelometric titration with rheometry, filtration tests, zeta-potential analysis and infrared spectroscopy to connect the onset of phase separation with functional drilling-fluid performance.

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