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Impact sodium salts on colloidal and rheological characteristics of nanosilica/water/electrolyte system

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The influence of sodium salts (chlorides, iodides, nitrates) on the behavior of nanosilica particles in aqueous medium was studied. The dependences of key physicochemical characteristics of the nanosilica/water/electrolyte colloidal system (zeta potential, particle size distribution, rheological characteristics) on the electrolyte and solid phase contents were determined. The zeta potential for all the systems is not below -20 V. As the salt concentration varies, there is a decrease in the ζ modulus approaching zero. It has been revealed that the adsorption and ion exchange processes due to the interaction of Na^+ with a nanosilica surface can alter the system's behavior at electrolyte concentrations of 0.001-0.1 M, which manifested in the coagulation of primary nanoparticles and the peptization of agglomerates. The presence of sodium salts leads to additional structuring of the solvent and an increase in the viscosity. The highest viscosity for concentrated silica suspensions in the presence of NaI correlates to the data on an increase in the hydrodynamic radius of aggregates in low-concentration nanosilica suspensions. In the presence of sodium nitrate, silica suspensions show the lowest viscosity. In the colloidal system with nanosilica/water/electrolyte, when the solid phase concentration exceeds 7.5 wt.% and is influenced by sodium chloride and iodide, the rheopexy becomes evident after just three days of storage.

Keywords: Nanosilica; Electrolytes; Aqueous suspension; Zeta potential; Particle size distribution.

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Introduction

Nanosilica finds wide applications in aquatic systems, particularly in medicine [1]. The adsorption processes occurring on a surface of silica nanoparticles are crucial in regulating or influencing the migration of metal ions within biosystems [2]. In medicine, nanosilica is often utilized in suspensions with saline (0.9 wt.% NaCl) or other buffers. Buffered solutions can contain various cations and anions, the specific contents and composition of which depend on intended applications. Consequently, understanding the behavior of suspended silica nanoparticles in the presence of the monovalent alkali metal ion Na^+ and different anions holds practical significance [3-5].

Comprehensive investigations have been previously performed on aqueous suspensions of fumed silica in the presence of various electrolytes [3, 5-13]. The majority of these studies focused on silicas characterized by relatively large particle sizes ranging from 0.5 to 50 μm. Although some works delve into aqueous suspensions of nanosilicas [1,10,11], a systematic exploration of the influence of specific cations, coupled with a diverse array of anions, on the physicochemical characteristics of silica nanoparticles in aqueous media is lacking. It is imperative to consider that, beyond the charge state, ionospecific interactions can lead to chaotropic and kosmotropic effects, ultimately determining the behavior of the colloidal system consisting of a water dispersion medium and a solid dispersed phase [2]. Notably, the majority of works are devoted to studying interactions and characteristics of nanosilica/water/electrolyte systems with electrolyte concentrations up to 0.001 M. Therefore, there exists a compelling interest in extending these investigations at higher salinity levels and exploring essential parameters of the colloidal systems, such as zeta-potential, particle size distribution (PSD), and rheological characteristics, revealing the impact of solid phase concentration, the presence or absence of electrolytes, as well as their nature and concentration.

In the present study, electrolytes such as NaCl, $NaNO_3$, and NaI have been investigated in aqueous environment, where they dissociate into ions, namely Na^+ and Cl^- for NaCl, Na^+ and NO_3^- for $NaNO_3$, and Na^+ and I^- for NaI. In this case, sodium ions are classified as kosmotropic (enhancing water structure), chloride ions (Cl^-) are typically considered as weakly kosmotropic, while nitrate ions (NO_3^-) and iodide ions (I^-) are generally chaotropic. The latter can disrupt the hydrogen bonding network in water, leading to decreased water structure order and increased disorder [7]. Indeed, the presence of these ions can exert a significant influence on the zeta potential.

Zeta potential (or electrophoretic mobility) is affected by a set of factors such as the surface charging (due to dissolution of surface functionalities or ion adsorption), contents of various ions within the electrical double layer (EDL) and bound to a surface, and pH value of the dispersion medium. It serves as a metric for electrostatic interactions among colloidal particles and stands as a significant parameter influencing the stability of dispersed systems. Analyzing the zeta potential vis-à-vis these parameters enhances our comprehension and facilitates better control of the mechanisms of dispersion, aggregation, and flocculation [3, 4, 6].

Changes in the EDL characteristics and surface charging in aqueous suspensions due to pH and salinity variations strongly affected by ionogenic compounds (acids, bases, and salts), profoundly impact the behavior of colloidal systems. The characteristics and properties of nanoparticulate colloids, including aggregative stability, viscosity, turbidity, aging effects, etc., hinge on factors like concentration, aggregation degree, particle-particle interactions, particle size distributions, and particle shapes [3, 4, 10, 11, 14]. Based on our previous researches, related to the effects of various cations on aqueous suspensions of nanosilica [3, 4, 10, 11], this paper extends the inquiry to scrutinize the influence of different anions using three sodium salts. Thus, this study assumes significance in refining the characteristics and properties of dispersions, colloids, emulsions, and suspensions during both developmental and production stages.

I. Experimental procedure

1.1. Materials

Fumed silica (nanosilica) A–300 (pilot plant at the Chuiko Institute of Surface Chemistry, Kalush, Ukraine, purity 99.8 %, specific surface area $S_{BET} = 232$ m^2/g) was used for preparing the aqueous suspensions with the concentrations of 0.5, 1, 2.5, 5, 7.5, and 10 wt.%

1.2. Zeta potential and particle size distribution

Electrophoretic mobility and particle size distributions were studied using a Zetasizer Nano ZS (Malvern Instruments) apparatus using a universal dip cell (ZEN1002) and a disposable polystyrene cell (DTS0012) for zeta potential measurements. Aqueous suspensions with bidistilled water and certain amounts of dissolved salt (0.001-0.1 M NaCl, $NaNO_3$, or NaI) and nanosilica A-300 were sonicated for 2 min using an ultrasonic disperser (Sonicator Misonix, power 500 W and frequency 22 kHz). The suspensions were equilibrated for 24 h.

According to the Smoluchowski theory, there is a linear correlation between the electrophoretic mobility (U_e) and the ζ potential, expressed as

$$U_e = A \cdot \zeta \qquad (1)$$

where *A* is a constant applicable to a thin EDL, when $\kappa a >> 1$ (where *a* is the particle radius and *κ* is the Debye–Hückel parameter). In the case of a thick EDL ($\kappa a < 1$) and pH close to the isoelectric point (IEP), a more suitable equation involves the Henry correction factor:

$$U_e = 2\varepsilon\zeta/(3\eta) \qquad (2)$$

where ε represents the dielectric permittivity and η is the liquid viscosity [3, 4].

1.3. Rheometry

The rheological characteristics of the nanosilica suspensions were studied using a rotational viscometer Rheotest 2.1 (cylinder system S/N) at a shear rate of 9-1312.2 s^{-1}. To determine the thixotropic properties of the suspensions, the rheological measurements were performed with stepwise increasing shear rate and exposure at maximal shear rate (1312 s^{-1}) up to a constant viscosity, and finally stepwise decreasing shear rate. To compare different suspensions, the viscosity at the maximum shear rate (i.e., the effective viscosity of the suspension with the most destroyed structure) was chosen.

II. Results and discussion

The behavior of the aqueous suspensions of nanosilica with addition of various salts has a complex character strongly dependent on several factors. Figure 1 shows the zeta potential of nanosilica particles in the aqueous medium and the value of effective diameter of aggregates vs. content of A-300. An increase in the amount of A-300 alone (i.e., without addition of any electrolyte) in the suspension is accompanied by a decrease in the negative value of *ζ* (Fig. 1, *curve 1*). This decrease can be attributed to two primary effects: (i) aggregation of nanoparticles that leads to increasing average particle diameter (Fig. 1, *curve 2*) and (ii) a reduction in the total charge (resulting from the deprotonation of silanols to form ≡SiO−) on the silica surface, coupled with changes in the EDL under the slipping plane due to variations in the concentration of solvated protons with increasing silica content. Furthermore, the increase in the viscosity with increasing silica content, along with the formation of aggregates, contributes to a reduction in the electrophoretic mobility of the formed secondary particles compared to individual nanoparticles.

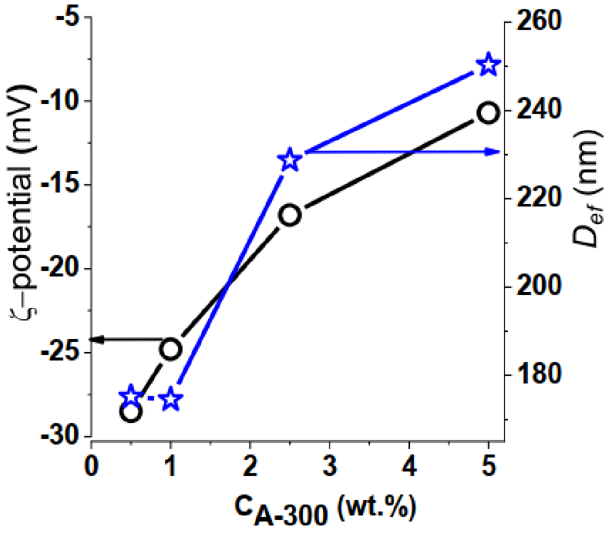

Fig. 1. Zeta potential and effective diameter as a function of nanosilica content in the aqueous suspensions without addition of any electrolyte (C_{A-300} = 5 wt.%).

Correlation of the values of the ζ-potential and average particle diameter (Fig. 1) corresponds to small changes in the EDL thickness, according to Eq. (2).

In general, the introduction of any electrolyte to suspensions of inorganic oxides results in a reduction of the absolute value of the zeta potential, bringing it closer to zero. Consequently, this leads to a decline in the sedimentation stability of the colloidal system due to an increased aggregation of particles [15] and some other characteristics (Figs. 2 -6).

The addition of electrolytes, such as NaCl, $NaNO_3$, and NaI, induces noteworthy changes in the electrokinetic potential of silica particles (Fig. 2), particle size distribution, and effective diameter of particles (Figs 3 and 4). These characteristics are significantly affected by both the nature of the anions (Cl^-, NO_3^-, I^-) and the content of the solid phase. Importantly, the pH value was not adjusted by adding an acid or base (HCl or NaOH), but it is caused by the rapid exchange of protons from the silanol groups on the surface of silica nanoparticles with alkali metal cations that is especially noticeable at low electrolyte concentrations (Fig. 5)

Within the examined concentration range, the absolute value of the zeta potential for all systems does not exceed 20 V and has a negative sign. A variation in salt concentration, ranging from 0.001 to 0.1 M, results in a decrease in the ζ value approaching zero (Fig. 2).

This could be attributed to the influence of the nature and size of hydrated anions and Na^+ cations distributed in different layers of the EDL, consequently affecting PSD and average diameter of silica aggregates (Fig. 3-4).

The effective diameter values are similar for the suspensions with different salts used at C_{A-300} = 1 wt.% and C_{salt} < 0.05 M (Fig. 3a). It means that all anions have the similar effects on the aggregation of the silica nanoparticles. The minimal aggregation is observed at C_{NaCl} = 0.1 M and it is maximal at C_{NaNO3} = 0.1 M (Fig. 3a).

At higher content of silica (2.5 wt.% or more), the effect of anions on the aggregation is changed. The values of D_{ef} weakly change *vs.* C_{salt} (Fig. 3b). At C_{salt} = 0.1 M, the values of D_{ef} are changed according to the line $Cl^- < NO_3^- < I^-$. A similar tendency is observed at C_{A-300} = 5 wt.% (Fig. 3c); however, the values of D_{ef} increase. The anion type effect on the value of D_{ef} is also visible for the particle size distribution, PSD (Fig. 3d), which is bimodal. However, the PSD can be monomodal for some systems (Fig. 4), which, however, become bimodal with increasing salt content. As a whole, the salt addition leads to an increase in size of small aggregates of primary silica nanoparticles (from 16 nm in the water to 40-50 nm in the presence of electrolyte) and a decrease in

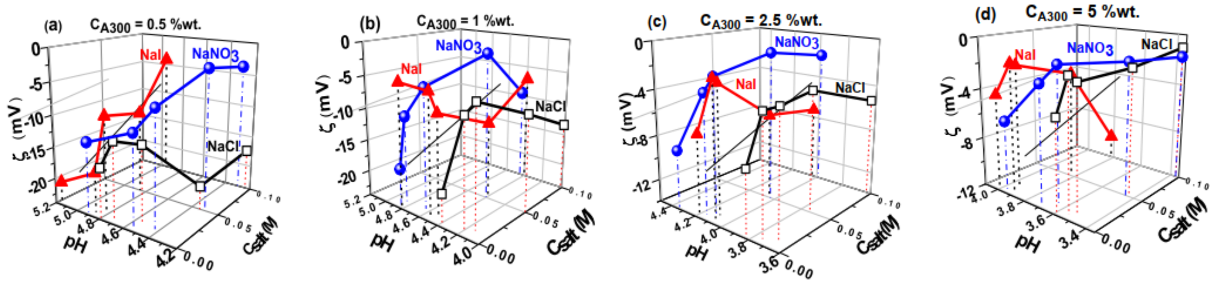

Fig. 2. Zeta potential as a function of silica content (C_{A-300} = 0.5-5 wt.%) in the aqueous suspensions with addition of different electrolytes: NaCl (□), $NaNO_3$ (○), NaI (▲).

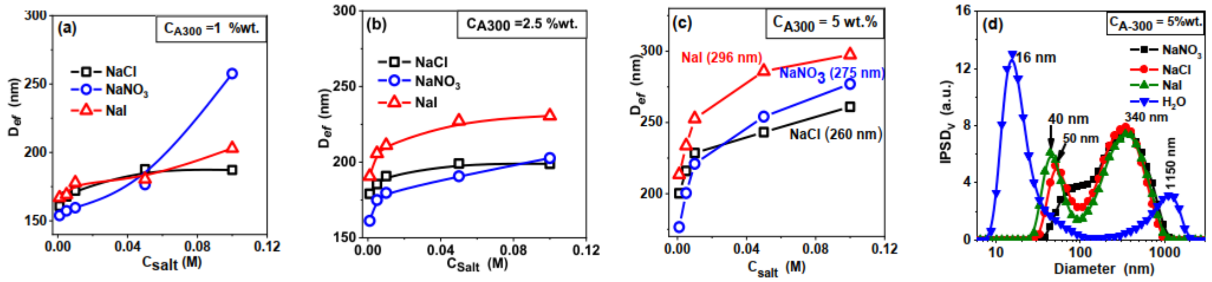

Fig. 3. Effective diameter of particles (determined from the light scattering intensity) as a function of the salt content (0.001-0.1 M) for NaCl, $NaNO_3$, and NaI at different content of A-300 (a) 1, (b) 2.5, and (c) 5 wt.%) in the suspensions, and (d) particle size distributions with respect to particle volume (C_{salt} = 0.01 M, C_{A-300} = 5 wt.%).

size of larger aggregates (from 1000 nm to 340 nm, Fig. 3d). Thus, the processes of aggregation and peptization take place.

These effects are observed due to the influence of anions on the EDL thickness, since the main fraction of anions is located in the diffusive layer. The EDL consists of an area near an interface, in which the net charge density is not equal zero. As compared to the bulk solution, the counterions (ions with charge opposite to that of the solid surface) are present in the EDL at higher concentration, while the co-ions (ions with charge of the same sign of the surface) are present at a lower concentration in the EDL due to the electrostatic repulsive interactions.

The interactions of the electrolytes with nanosilica particles have a complex character, since the zeta potential (Fig. 2), D_{ef} and PSD (Figs. 3, 4) demonstrate complex dependences on $C_{A\text{-}300}$ and C_{Salt}. Additionally, the values of pH decrease with increasing $C_{A\text{-}300}$ and C_{salt} (Fig.5). However, the changes in pH are linear with one exception for NaI at $C_{A\text{-}300}$ = 0.5 wt.% (Fig. 5c).

Such kind of a non-monotonic curve can be explained by: (i) iodide anions have the smallest hydration degree and tent to be adsorbed on the silica surface; (ii) an increase in the deprotonation degree of the surface silanols with increasing concentration of NaI.

Silanols are the main active sites at the silica surface. Their concentration is 2-2.5 μM/m^2 for dry fumed silica, but it may be twice larger in the aqueous media. The dissociation degree of silanols is low (only several percent) for nanosilica at pH 3-6 [1, 16, 17]. Therefore, adsorption of cations Na^+ leads to increasing acidity of the silica suspension (Fig. 5):

$$\equiv SiOH^- + Na^+ + Cl^- = \equiv SiONa + HCl \qquad (3)$$

The adsorption capacity of nanosilica is relatively efficient for metal cations (which can be located in narrow voids between neighboring nanoparticles in their aggregates) because the concentration of surface hydroxyls is relative high. Thus, nanosilica can be used for extracting metal cations at a low concentration by the following accumulation and analysis of the solid phase.

The introduction of electrolytes into the disperse system leads to additional structuring of solvent or partial destroying of its structure [16-17]. Consequently, ions characterized by a small size and a strong charge, such as Na+, have a structuring effect on water, but larger ions with a small charge (I^- and NO_3^-) *vs.* restructuring effect. Chloride ions are the smallest ones among these anions.

They tend to interact favorably with water molecules, forming a stable hydration shell and promoting water structure. As a result, chloride ions are classified as kosmotropic ones. Thus, the addition of these sodium salt solutions to the disperse system will result in the rearrangement of the solvate shells of ions in the case of their interaction with the nanoparticle surface and changes

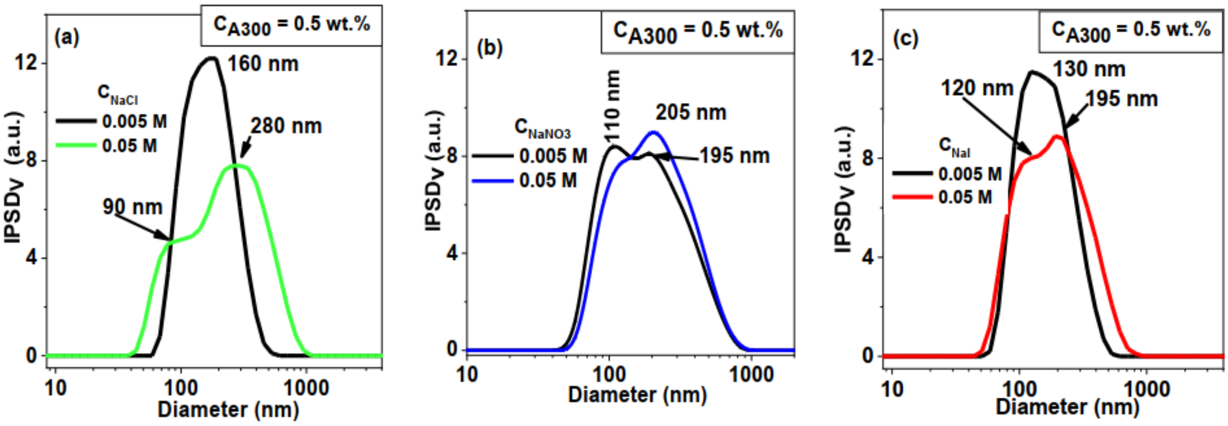

Fig. 4. Particle size distributions with respect to the volume of particles (determined from the light scattering intensity) at $C_{A\text{-}300}$ = 0.5 wt.% and C_{salt} = 0.005 and 0.05 M for the systems with addition of NaCl (a), NaNO$_3$ (b), NaI (c).

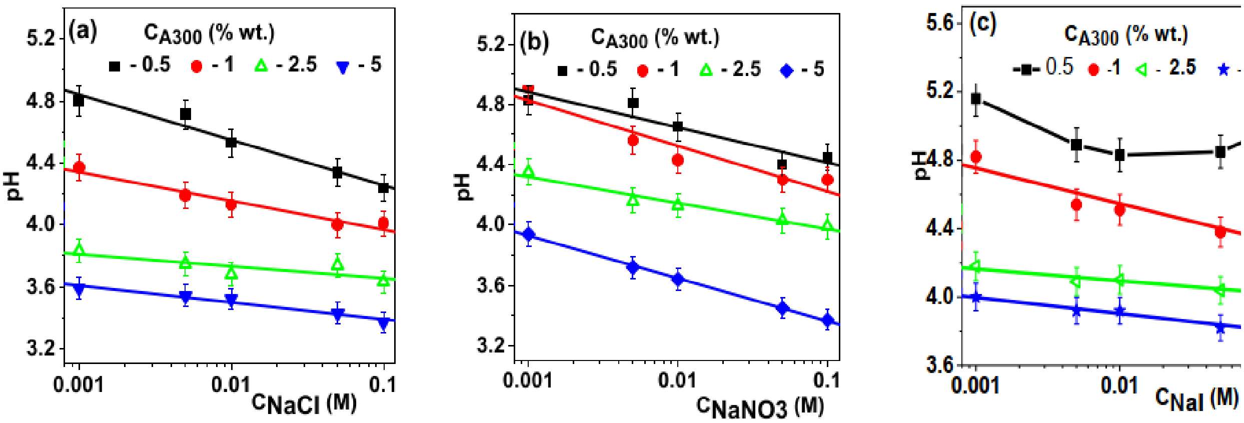

Fig. 5. The values of pH as a function of electrolyte concentration at $C_{A\text{-}300}$ = 0.5-5 wt.%: (a) NaCl, (b) NaNO$_3$, and (c) NaI.

in the viscosity of the disperse system, where interparticle interactions are suppressed by an electrolyte addition. Thus, in the presence of sodium salts with different anions, the effective viscosity of nanosilica suspensions increases in the $NO_3^- < Cl^- < I^-$ series due to the formation of coagulation structures upon contact of initial silica particles in the secondary energy minimum through the interlayer of the dispersion medium. (Fig. 6a,b,c). The formation of such structures depends both on the influence of counterions, which in the case of the studied electrolytes are sodium ions, and on the influence of co-ions, which are Cl^-, I^- and NO_3^-, on the DEL structure. These coagulation structures typically exhibit the thixotropic behavior, *i.e.*, the ability to break under shear stress and recover upon stress removal [18]. All investigated suspensions demonstrate typical thixotropic behavior within the concentration range of the solid phase of 5-10 wt.% This is manifested in the destruction of the suspension structure during the rheological studies by increasing shear rate (γ) and restoring it upon reducing the shear rate.

If upon application of shear stress, the equilibrium between the destruction and formation of interparticle bonds is shifted toward bond formation, resulting in a strengthened structure and increased viscosity, it is referred as rheopexy [19]. To determine the presence of thixotropy or rheopexy, rheological studies were carried out in the mode of increasing the shear rate, holding at a constant maximum shear rate and in the mode of reverse decrease of the shear rate. This mode of measurement enables the characterization of the capacity to disrupt and restore coagulation bonds, according to the hysteresis loop on the viscosity curve [20]. As can be seen in Fig. 6d,e,f such a hysteresis loop is vividly expressed for the nanosilica suspension in the presence of sodium chloride and iodide, and the viscosity determined in the mode of reverse shear rate decrease is significantly higher than the initial viscosity determined during the shear rate increase, which indicates rheopexy properties. In nanosilica suspensions with a solid phase concentration surpassing 7.5 wt.% in the presence of electrolytes, the rheopexy is observed after already three days of storage. In the presence of sodium nitrate, the viscosity values determined in the modes of increasing and decreasing shear rate align, indicating the absence of a hysteresis loop. However, for this system, a decrease in the viscosity is observed as the shear rate increases. This behavior implies that the system behaves more like a structured liquid without pronounced thixotropic or rheopexy properties.

The value of the effective viscosity is the largest for the suspensions of nanosilica in the presence of NaI – at a shear rate of 1312 s ^{1}, it is 433.25 mPa*s, which indicates its greatest influence on the ELD structure and, consequently, on the strength of the formed coagulation structure. The highest viscosity value for concentrated silica suspensions in the presence of NaI correlates with data on an increase in the hydrodynamic diameter of particles (aggregates) in the presence of NaI in low-concentration silica suspensions [21]. At the same time, in the presence of sodium nitrate, silica suspensions show the lowest viscosity (13.40 mPa*s at a shear rate of 1312 s^{-1}).

Conclusions

Thus, the chemical nature of the anions in the composition of sodium salts affects the zeta potential and dispersity of nanoscaled silica particles. The modulus of the zeta potential decreases with an increase in the concentration of sodium salts from 0.001 M to 0.1 M, as well as with the solid phase due to a

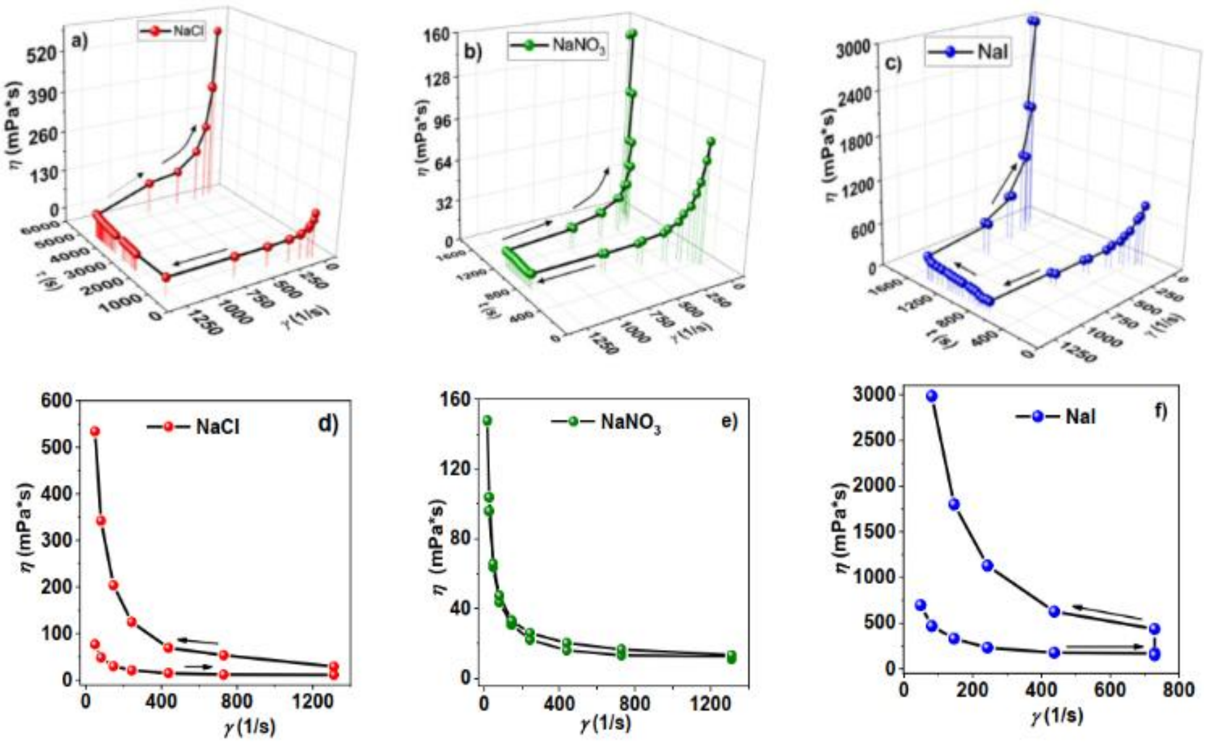

Fig. 6. The dependence of viscosity on shear rate in time (a, b, c) and on shear rate gradient (γ, s^{-1}) for silica suspension (10 wt.%) in solution of 0.1 M NaCl (a, d), $NaNO_3$ (b, e), NaI (c, f) after 14 days.

reduction in the thickness of the electrical double layer, leading to suspension instability and an increase in the aggregation of primary silica nanoparticles from an initial size of 16 nm to 40-50 nm. At the same time with the addition of electrolytes, the peptization processes also occur – the destruction of particles agglomerates with a diameter of about 1150 nm to aggregates of 340 nm at $C_{A\text{-}300} = 5$ wt.%

All studied anions have the same effect on the aggregation and peptization of silica nanoparticles. These processes non-monotonically affect the electrokinetic potential of the nanosilica/water/salt system. Experiments reveal that the behavior of silica nanoparticles differs from that of larger particles of the same material in inert electrolytes. For nanodisperse materials like nanosilica, aggregation/peptization phenomena occur depending on the concentration of both the electrolyte and silica, manifesting as additional extrema on curves illustrating the dependence of the zeta potential on the concentration of electrolyte and nanosilica in the aqueous medium. Moreover, with the same Na^+ cation and varying anion nature, these extrema appear at different concentrations of silica. This indicates complex interactions in the system emphasizing the need for further research to gain a deeper understanding of these phenomena.

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Вплив солей натрію на колоїдні та реологічні властивості системи нанокремнезем/вода/електроліт

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Досліджено вплив солей натрію (хлоридів, йодидів, нітратів) на поведінку частинок нанокремнезему у водному середовищі. Визначено залежності ключових фізико-хімічних характеристик колоїдної системи нанокремнезем/вода/електроліт: дзета-потенціалу, розподілу частинок за розмірами та реологічних властивостей, від вмісту електроліту та твердої фази. Дзета-потенціал для всіх систем не нижче -20 В. Зі зміною концентрації солі відбувається зменшення значення ζ, що наближається до нуля. Виявлено, що процеси адсорбції та іонного обміну внаслідок взаємодії катіонів Na^+ з поверхнею нанокремнезему можуть змінювати поведінку системи при концентраціях електроліту в межах 0,001-0,1 М. що проявляється в коагуляції первинних частинок та пептизації агломератів. Присутність електролітів призводить до додаткового структурування розчинника та збільшення в'язкості суспензії порівняно з суспензією нанокремнезему без солі. Найбільше значення в'язкості для концентрованих суспензій кремнезему в присутності NaI корелює з даними про збільшення гідродинамічного радіуса частинок (агрегатів) у присутності NaI у низькоконцентрованих суспензіях кремнезему. Водночас у присутності нітрату натрію кремнеземні суспензії мають найменшу в'язкість. У колоїдній системі нанокремнезем/вода/електроліт, коли концентрація твердої фази перевищує 7,5 мас.% і під впливом натрію хлориду та йодиду, реопексія спостерігається вже через три дні зберігання.

Ключові слова: Нанокремнезем; Електроліти; Водні суспензії; Дзета потенціал; Розподіл частинок за розмірами.