How to Cite: Shcherban', M.G., Ivanchina, E.A., Sedusov, I.V., Sosunova, V.V. & Novosyolov, K.P. (2025). Study of the Effect of Potassium Chloride on the Coagulation of PTFE Dispersion by Optical Methods. *Eurasian Journal of Chemistry, 30, 1(117), 50–59.* https://doi.org/10.31489/2959-0663/1-25-11

Article

Received: 6 January 2025 | Revised: 26 February 2024 |
Accepted: 5 March 2025 | Published online: 13 March 2025

UDC 544.77.052.2; 544.42

https://doi.org/10.31489/2959-0663/1-25-11

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Study of the Effect of Potassium Chloride on the Coagulation of PTFE Dispersion by Optical Methods

Electrolyte stability of fluoroplastic-4D dispersion was investigated by optical methods. Potassium chloride (KCl) solutions of various concentrations were used as electrolytes causing coagulation. The kinetic dependences of the influence of KCl concentration on the optical density of polytetrafluoroethylene (PTFE) dispersion were obtained. The maximum in the time dependences of optical density is associated with the particle aggregation processes and the subsequent loss of sedimentation stability of the system. With increasing KCl concentration, this dependence changes: induction period decreases and the peak of the curve narrows. The change in the shape of the kinetic curves with an increase in the electrolyte concentration is associated with the processes of particle dehydration of dispersed phase, desorption of the stabilizer into the solution and the transition of the system from an adsorption-saturated to an adsorption-unsaturated state. The results of measuring the coagulate size of the PTFE dispersion with 2.5 M KCl in different sections of the kinetic curve by the dynamic light scattering (DLS) method correlate with the results of optical density measurements. The data obtained confirm the multi-stage coagulation process, in which the stages of latent coagulation, aggregation, inhibition of coagulation, and loss of sedimentation stability of the system can be distinguished.

Keywords: PTFE, dispersion, surfactant stabilization, coagulation, aggregate stability, optical density, spectrophotometry, dynamic light scattering

Introduction

Highly concentrated dispersed polymer systems have attracted significant interest from both industry and academia owing to their physical and chemical properties. Due to the high electronegativity, low polarizability, and small van der Waals radius of fluorine atom, fluorinated polymers exhibit outstanding properties for a wide range of applications in modern life [1, 2]. Among these, polytetrafluoroethylene (PTFE), widely known as Teflon, is the most useful of the fluorinated polymers.

Owing to the high-energy C–C (346 kJ·mol^{-1}) and C–F (485 kJ·mol^{-1}) bonds, PTFE has a number of unique properties such as wear resistance and corrosion resistance, resistance to high temperatures, low surface energy and low dielectric constant [3, 4]. Because of its properties, PTFE is used in composite coatings [5], membrane technologies [6], fuel industry [7]. It is known that metal particles (for instance, Al) can be coated with a PTFE layer instead of an oxide film on the surface to increase their reactivity and stability [8].

Emulsion polymerization is the primary method used to yield an aqueous colloidal PTFE dispersion in industry, where the tetrafluoroethylene monomer is added into an aqueous medium containing water-soluble initiator, anionic fluorinated dispersant, and other additives (under pressure) [3, 9]. To ensure the stable polymerization and desirable particle size, typically ranging from 100 to 500 nm, the PTFE solid content in dispersion are monitored at approximately 30 % by weight or lower [10]. However, such dilute and unstable as-polymerized dispersion has no practical utility in preparing PTFE articles, and the low solid content also increases the storage and transportation cost. To address this shortcoming, the resulting dilute dispersion should be further concentrated following the polymerization, to typically achieve a content of 60 % by weight [11]. An increase in the solid content leads to an irreversible coagulation of PTFE particles, which renders it useless for various applications. The DLVO theory (abbreviated as Deryagin, Landau, Vervey, Overbeck) explains the aggregate stability of lyophobic dispersed systems, and coagulation is one of the consequences of the aggregation of colloidal particles, in which this stability is lost [12]. Therefore, it is imperative to develop a methodology to concentrate dilute PTFE dispersion with long-term stability and elucidate the stabilization mechanism.

The tendency of hydrophobic PTFE particles to aggregate has prompted many of the studies. The surface properties of PTFE depend on the pH and the ionic strength of the electrolyte solution had studied [13]. The motion of Newtonian and well-characterized elastic liquids with constant viscosity under the impulse inclination of hydrophobic PTFE surfaces has been investigated [14]. New methods of modifying the surface of PTFE, that lead to a change from hydrophobic to hydrophilic, and a perfect recovery from hydrophilic to hydrophobic behavior have been developed [15]. Moreover, dynamic contact angles of pure surfactants and surfactants with electrolyte solutions on PTFE surface have been studied [16].

Surfactants play an important role in the stabilization of hydrophobic nanoparticles [17]. It was found that the surfactants could adsorb on the surface of the PTFE particles and remarkably improve their surface wettability, thus significantly improving the interaction with the bulk phase [18]. In addition, polymers are used as stabilizers for PTFE dispersion, for example polymethylmethacrylate [19]. Long Bai and others analyzed four types of surfactants including cationic dodecyltrimethylammonium bromide (DTAB), anionic sodium dodecyl sulfate, zwitterionic lauryl betaine (LB), and nonionic lauryl alcohol polyoxyethylene Brij 30, are used to stabilize 60 % PTFE dispersions from the as-polymerized dilute mixture [20]. As shown in Figure 1, the surfactants show different capabilities for stabilizing PTFE particles in aqueous solutions. While the cationic surfactant causes particle flocculation, the anion, zwitterionic and nonionic surfactants can be used to prepare concentrated dispersions [20, 21].

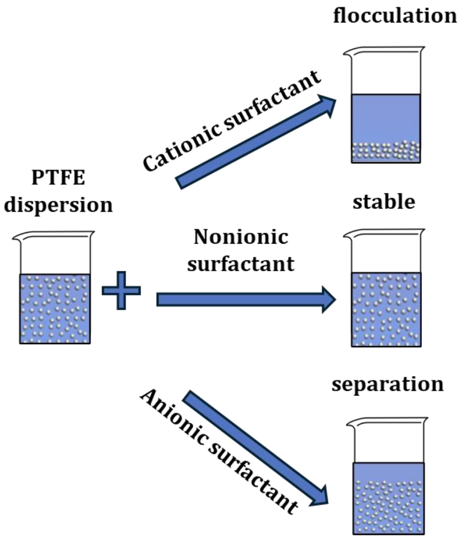


Figure 1. Scheme of the stabilization mechanism of PTFE dispersion with different surfactants.
Adapted and redrawn from Ref. [20] with permission from Elsevier

In addition, the applicability of surfactants to PTFE has been investigated not only in aqueous solutions, but also in oil solutions [22].

Despite the high popularity of PTFE dispersion, their coagulation has not been thoroughly studied. Therefore, it is necessary to investigate the effect of electrolytes on PTFE dispersions in order to develop methods for stabilizing this system. Electrolytic coagulation is a process of particle aggregation, the study of which will lead to the development of effective methods to ensure the long-term stability of the studied dispersions. The aim of this work is to study the coagulation of PTFE dispersion under the action of potassium chloride.

Experimental

The subject of the study was a dispersion of fluoroplastic F-4D with a dispersed phase concentration of 55 % by weight., stabilized with the non-ionic surfactant OP-7, obtained by emulsion polymerization. The main dispersion parameters are shown in Table 1.

Table 1

The main measures of dispersion

pH	Kinematic viscosity, cSt	Dry residue, mg·ml^{-1}	Density, *kg*·m^{-3}
9.82±0.11	7.179±0.001	871.24±8.19	1510.59±9.09

The particle size of the PTFE dispersed phase was studied using a scanning electron microscope (SEM) (Hitachi, S-4800 SEM, Tokyo, Japan). The micrograph of the dry residue of the investigated dispersion is shown in Figure 2.

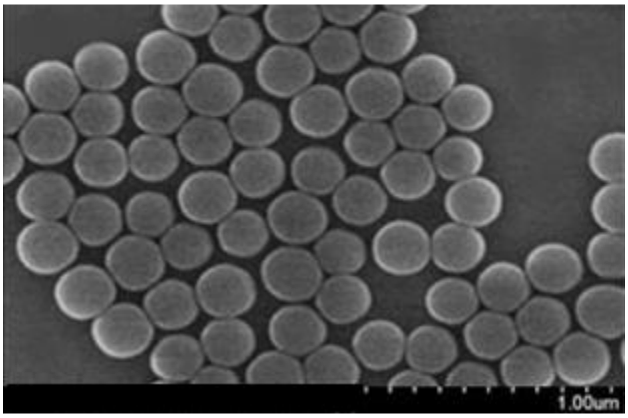


Figure 2. Micrograph of the dry residue of the dispersion

The degree of polydispersity is an important characteristic of such systems. In addition to the predominant number of particles of a certain specific size, there are usually also particles of other, noticeably different sizes due to difficult polymerization conditions. Secondary particles of adhered primary globules can form in stable systems in a number of cases. In order to ensure their absence, as well as to determine the degree of polydispersity of the system, we used the method of dynamic light scattering (DLS), based on the analysis of the spectrum of scattered laser radiation on the sample under investigation. The coagulum particle size was determined using a particle size analyzer (Delsa Nano C, Beckman Coulter, Inc., Fullerton, CA, USA) at 160° scattering angle.

For dispersions obtained by the method of emulsion polymerization, the issues of aggregation stability and coagulation are extremely important, which can lead to the destruction of the dispersion system and the release of PTFE in the macroscopic state. The presence of an insignificant amount of aggregates and coagulum can have an extremely negative effect on the functional properties of coatings based on fluoroplastic dispersions. To prevent aggregation of PTFE particles during polymerization, a stabilizer is introduced into the system. These are usually non-ionogenic surfactants such as OP-7 or Neonol.

Disruption of stability and coagulation of dispersions can be caused by various methods: the introduction of an electrolyte, mechanical interaction, freezing, a change in pH, etc.

We investigated the case of electrolyte stability of the fluoroplastic F-4D dispersion by a spectrophotometric method based on measuring the degree of reflection or absorption of monochromatic light rays [23]. Optical density of nanoparticle dispersions was measured with KFK-3KM spectrophotometer (Russia) in a cuvette with an optical path length of 10 mm (λ=400 nm). Potassium chloride solutions of various concentrations were used as electrolytes causing coagulation. The KCl solutions were prepared using bidistilled water from a reagent produced by Sigma–Aldrich (St. Louis, Missouri, USA). Unfortunately, photometry imposes limitations on the optical density of the studied solutions and this allows observations to be carried out only in low-concentration dispersion systems. However, according to the literature data, the kinetic curves of coagulation obtained for dilute and concentrated dispersions do not have fundamental differences. This does not mean that the processes occurring in concentrated and dilute systems are completely identical, since dilution not only reduces the number of possible collisions per unit volume, but also changes the state of the protective adsorption surfactant layer on the surface of the particles, its saturation and structuring, changes the surface charge, the degree of hydration, and so on. This indicates the commonality of the main regularities of coagulation. All experiments were carried out at 20 °C.

Results and Discussion

The distribution of particles in dispersion obtained using the DLS method is shown in Figure 3.

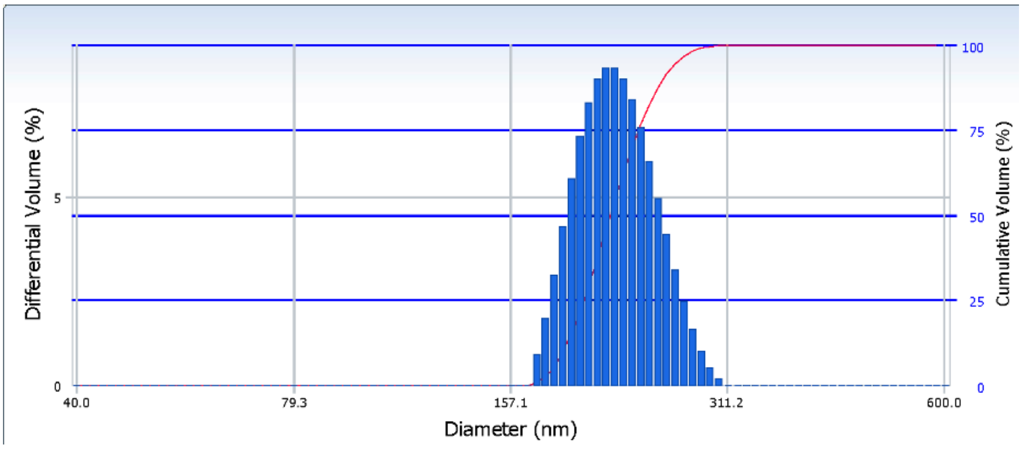


Figure 3. Particle distribution in dispersion

According to the results obtained, the average particle size in the system was 220±26 nm with a polydispersity index of 0.002, which gives reason to consider the studied system as monodisperse. No agglomerates of particles were detected either by microscopy or by the DLS method. Therefore, this dispersion can be used as a model to study aggregation processes.

Experimental normalized dependences of the relative optical density on time (Fig. 4) were obtained using the spectrophotometric method.

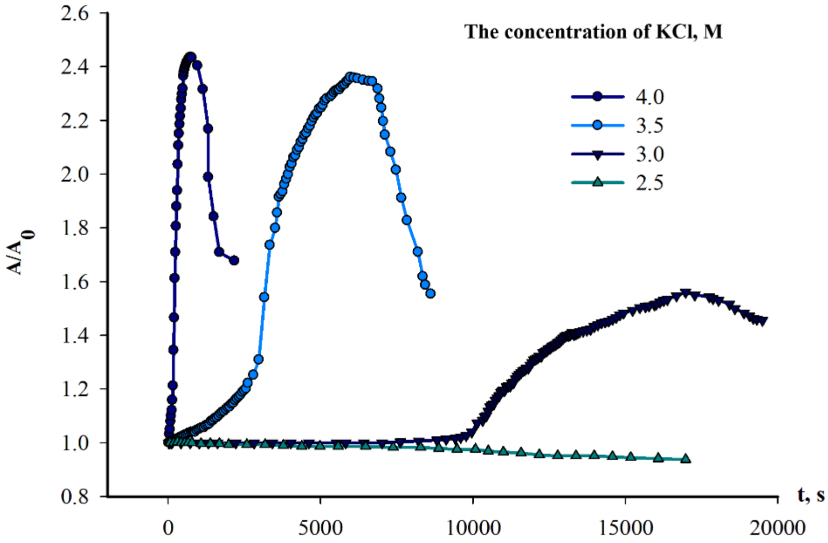


Figure 4. Normalized curves of coagulation of PTFE aqueous dispersion under the action of KCl

The obtained dependences allow us to conclude that the concentration of the electrolyte causing coagulation has a significant effect on aggregation processes occurring in dispersion. The coagulation curve under the action of a 2.5 M solution can be identified as the coagulation curve of an adsorption saturated system — a long (10.000 sec) induction period is observed on it, during which no visible changes occur in the system. The increase of the electrolyte concentration leads to its reduction and almost complete disappearance in a 4 M solution. A similar extreme type of turbidity dependence on time, as in the case of 3.5 and 4 M KCl solutions, was observed by the authors of [24] for copolymers of N,N-dimethylaminoethyl methacrylate with N-vinyl caprolactam. However, a decrease in turbidity did not lead to the formation of a coagulum, as in our case, when this behavior is the result of aggregation of dispersed particles and their sedimentation.

At 2.5 M, the constancy of optical density during the induction period suggests the absence of particle aggregation or their flocculation through the medium interlayer. In more concentrated solutions, the steepness of the initial sections increases, as a result of which a significant increase in interparticle interaction can be assumed due to an increase in the dehydration of the stabilizing layers with an increase in the electrolyte concentration.

A narrowing and an increase in the height of the peak with an increase in the concentration of the electrolyte is observed (the kinetic characteristics of the curves are shown in Table 2).

Table 2

Kinetic parameters of electrolyte coagulation curves for aqueous dispersion of PTFE-KCl

The concentration of KCl, M	Coagulation rate, sec^{-1}		Peak width at half height, sec
	I section	II section	
4.0	$1.2 \cdot 10^{-3}$	$5.5 \cdot 10^{-3}$	600
3.5	$5.5 \cdot 10^{-5}$	$8.7 \cdot 10^{-4}$	3300
3.0	$2.7 \cdot 10^{-7}$	$1.3 \cdot 10^{-4}$	7600
2.5	$2.0 \cdot 10^{-8}$	$1.2 \cdot 10^{-7}$	–

Figure 5 shows the initial sections of the coagulation curves, the determination of the angular coefficients of which makes it possible to obtain the dependence of the coagulation rate on the concentration of electrolyte (Fig. 6).

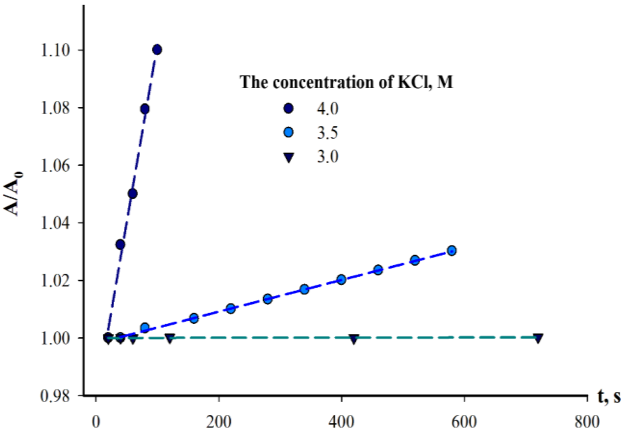


Figure 5. Initial sections of coagulation curves

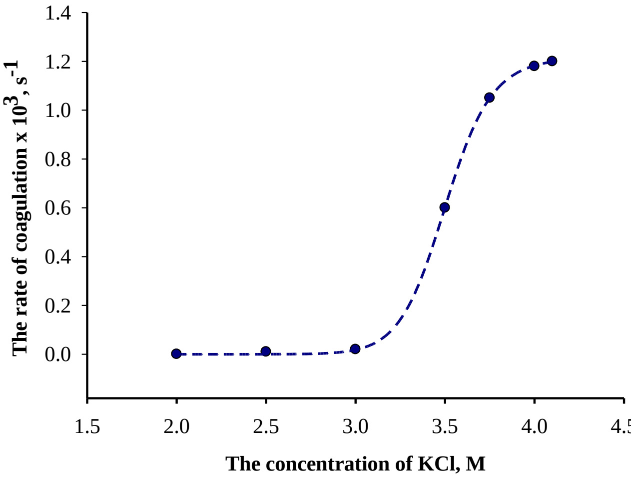


Figure 6. Effect of the KCl concentration on the aggregative stability of PTFE aqueous dispersion based on results of processing of initial sections of kinetic coagulation curves

Based on the obtained dependence, we can define the concentration range up to 3.5 M KCl as a zone of latent coagulation, 3.5–4.0 — as a zone of slow coagulation, when the coagulation rate depends on the electrolyte concentration, and the concentration of 4.0 M, presumably as the threshold of rapid coagulation.

The patterns mentioned above are preserved for the following sections of the kinetic curves, on which the growth of aggregates occurs (Fig. 7, 8).

Concentration dependences characterized by the presence of plateaus (Figs. 6 and 8) were observed in [25] for aqueous dispersions of crosslinked poly-N-vinyl caprolactam stabilized with hydrophobically modified polyacrylamide. The values of the critical concentration of coagulation for sodium chloride of this system varied in the range of 1,500–2,500 mmol/l and were explained by the peculiarities of the stabilizing effect and the charge of the stabilizer.

Apparently, in our case, the concentration of 2.5 M is inadequate in reducing the energy barrier. However, it has been observed to disrupt the stability of the adsorbed solvation layers of particles, which aggregate and form coagulation contacts in the affected areas of the layer. Thus, the processes of thinning of the hydration shells and the weakening of the forces of structural repulsion persist, albeit at an undetectable rate. The rate of coagulation slows down significantly and the curve reaches a plateau after 3.5 hours from the moment of initiation. Presumably, the primary aggregation of particles at the sites of disruption of the hydration shells leads to the outflow of the surfactant from the contact zone and the compaction of the surfactant adsorption layers on the growing aggregates. Consequently, hydration increases temporarily, the structural component of the wedging pressure increases, which inhibits coagulation. The presence of electrolyte leads to a decrease in the thickness of the interlayers in the inter-aggregate space, again weakening the structural component, and the aggregates increase in size. As a result, at some point in time, the system loses its sedimentation stability, which is accompanied by a drop in optical density.

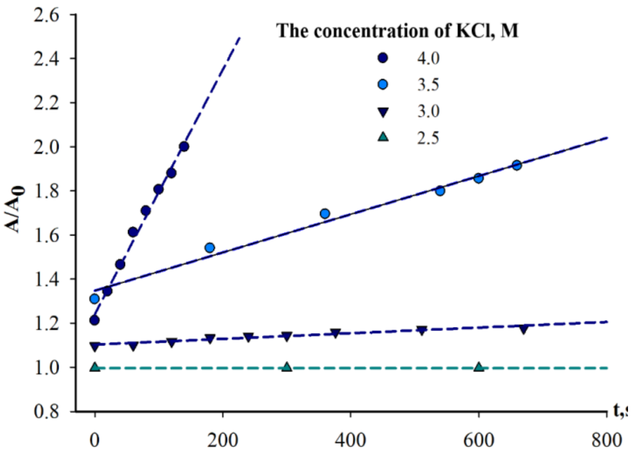


Figure 7. Effect of the concentration of KCl on the rate of aggregation of PTFE dispersion

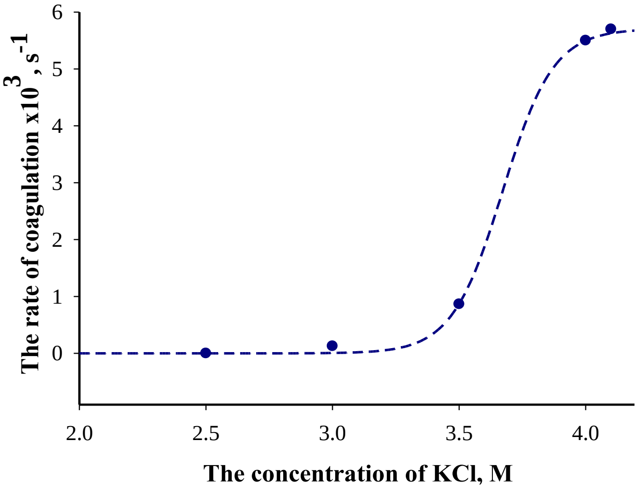
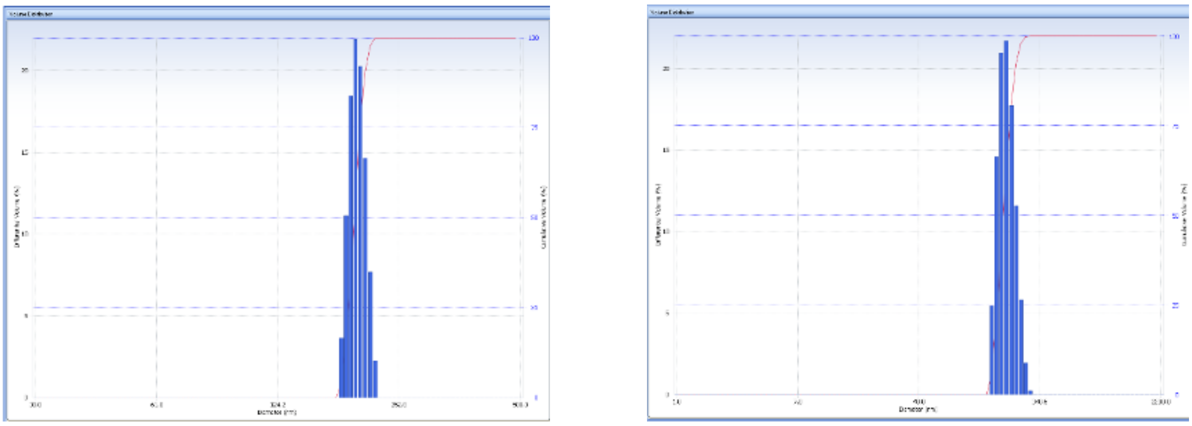


Figure 8. Effect of the KCl concentration on the aggregative stability of PTFE aqueous dispersion based on results of processing of second sections of kinetic coagulation curves

As the electrolyte concentration increases, the dehydration processes occur at a higher rate, and the inhibition of coagulation proceeds with a greater degree of particle aggregation, while the duration of the inhibition stage decreases, as evidenced by the narrowing of the peaks of the kinetic curves.

To confirm the above assumptions, the DLS method was used to determine the sizes of coagulum particles in different parts of the kinetic curve of coagulation of the dispersion with 2.5 M KCl.

At the first stage, the particle size of the initial dispersion was determined at a similar dilution to ensure its stability (Fig. 9).



d = 197.6 ± 9.3

(*a*)

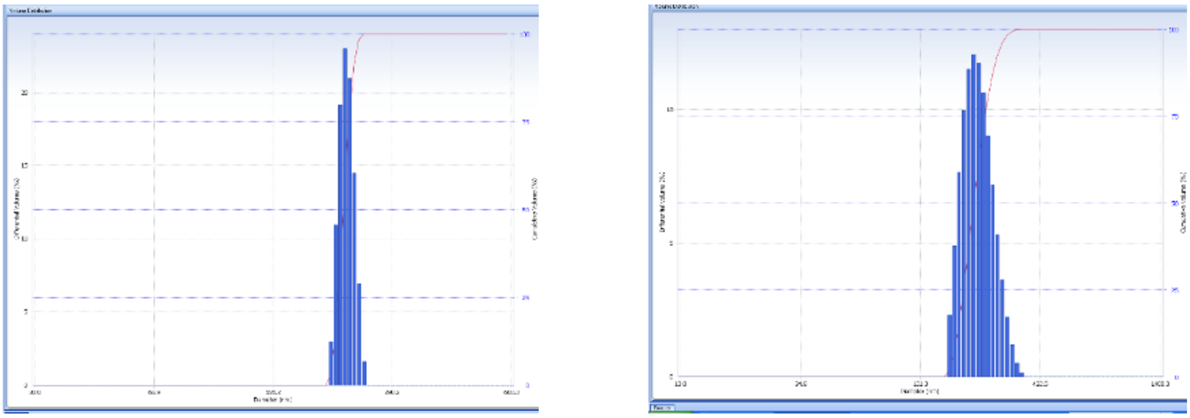
d = 199.4 ± 26.5

(*b*)

Figure 9. Study of the aggregate stability of the initial diluted dispersion. The lifetime of system: (*a*) — 10 minutes, (*b*) — 360 minutes

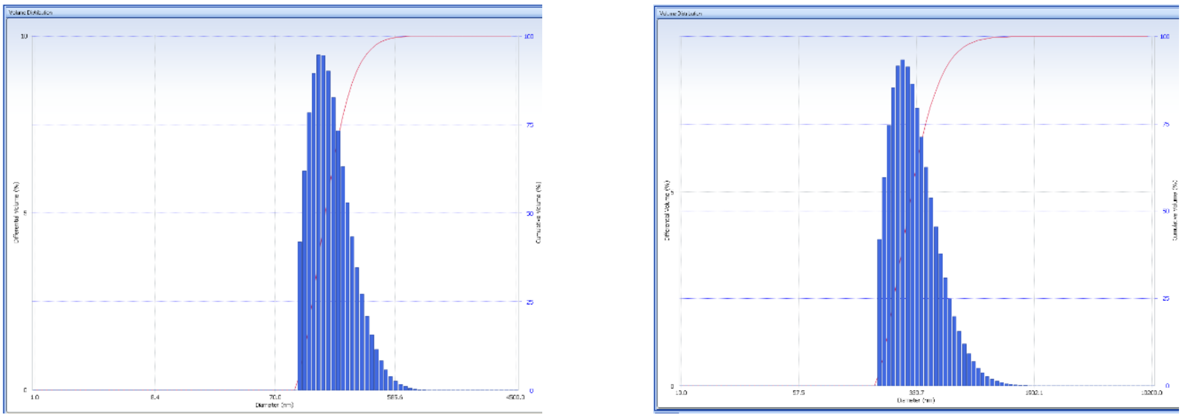
The obtained distributions confirm the aggregate stability and monodispersity of the system. The increase in polydispersity over time can be associated with the desorption of a part of the stabilizer into the bulk of the solution, loosening of the stabilizing layers, a slight decrease in the structural component of the wedging pressure, but it is not critical, and the diluted dispersion can be considered aggregatively stable during the observation period.

In the presence of KCl, visible changes in the distribution are observed after 30 minutes. They are expressed in a slight enlargement of particles and an increase in the width of the maximum (Fig. 10, *a*, *b*).



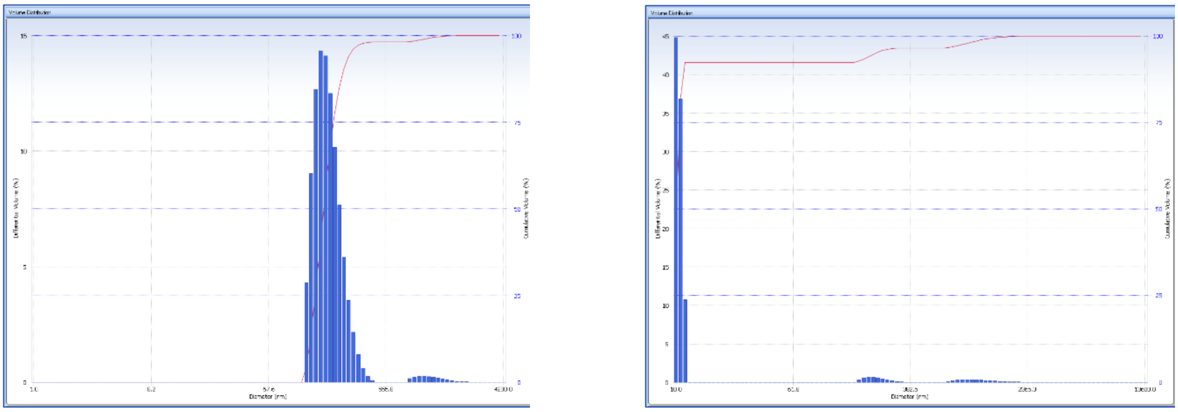
d=216.5±10.4 nm
a

d=221.6±34.9 nm
b



d = 210.9 ± 86.8 nm
c

d = 340.5 ± 141.4 nm
d



d = 217.3 ± 155.3 nm
d = 1233.5 ± 307.2 nm
e

d = 10.5 ± 0.5 nm
d = 227.7 ± 38.4 nm
d = 1112.0 ± 347.2 nm
f

Figure 10. Study of the aggregate stability of PTFE dispersion under the action of 2.5 M KCl.
The lifetime of system: *a* — 10 minutes; *b* — 30 minutes; *c* — 180 minutes; *d* — 240 minutes;
e —300 minutes; *f* — 330 minutes

The symmetry of the system is lost three hours after the introduction of the electrolyte, of the system is lost the end of the induction period. A more than one and a half-fold increase in the particle size against the background of an even more significant increase in the polydispersity of the system occurs after four hours (Fig. 10, *c*, *d*) and an increase in the kinetic curve is observed.

Another maximum appears in the inhibition zone (300 min) on the distribution. It is rather weakly expressed, but, nevertheless, it indicates the presence of sufficiently large aggregates in the system with a size of 1.0–1.5 microns (Fig. 10 *e*).

After 30 minutes, the maximum corresponding to the primary particles almost completely degenerates, and a new narrow peak appears, characterizing particles with a size of about 10 nm. These may correspond to micelles formed by the stabilizer desorbed from the surface of the dispersed phase particles during the coagulation process (Fig. 10, *f*). This time interval corresponds to a gradual decrease in optical density and visually recorded separation of the system into a dispersion medium and a dispersed phase.

Thus, the results obtained by the DLS method correlate with the results of measuring the optical density and confirm the multistage process of coagulation, in which the stages of latent coagulation, aggregation, inhibition of coagulation and loss of sedimentation stability of the system can be distinguished.

Conclusions

The studied aqueous dispersion of PTFE is an adsorption saturated aggregatively stable system. An insignificant increase in the polydispersity of the system during its dilution is caused by the desorption of the stabilizer and a decrease in the density of the adsorption layer of the stabilizer layer on the particles of the dispersed phase. The change in the shape of the kinetic curves with the increase in the concentration of the electrolyte is associated with the processes of dehydration of particles of the dispersed phase and the transition from the adsorption-saturated to the adsorption-unsaturated state of the system.

The results obtained by the DLS method correlate with the results of measuring the optical density and confirm the multistage process of coagulation, in which the stages of latent coagulation, aggregation, inhibition of coagulation and loss of sedimentation stability of the system can be distinguished. The process of inhibition of coagulation is associated with the rearrangement of the adsorbed solvation layer on the surface of the aggregates.

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Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. CRediT: **Marina Grigoryevna Shcherban'** conceptualization, supervision, data curation, investigation, methodology, validation, visualization, writing-review & editing; **Ekaterina Alekseevna Ivanchina** data curation, formal analysis, visualization; **Ilya Vladislavovich Sedusov** data curation, formal analysis; **Valeriya Vasil'evna Sosunova** data curation, **Konstantin Pavlovich Novosyolov** literature review, design.

Conflicts of Interest

The authors declare no conflict of interest.

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