SYNTHESIS AND PROCESSING OF POLYMERS AND POLYMERIC COMPOSITES

СИНТЕЗ И ПЕРЕРАБОТКА ПОЛИМЕРОВ И КОМПОЗИТОВ НА ИХ ОСНОВЕ

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RESEARCH ARTICLE

Influence of emulgator nature on dispersity and stability of artificial polymer suspensions based on polycarbonate and polymethyl methacrylate

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Abstract

Objectives. *To create stable artificial polymer suspensions with a positive charge of particles based on polycarbonate and polymethyl methacrylate using cationic surfactants and organosilicon surfactants.*
Methods. *The size of droplets and polymer suspension particles was determined by photon correlation spectroscopy (dynamic light scattering) using a Zetasizer NanoZS laser particle analyzer (Malvern, UK).*
Results. *Domestic cationic surfactants Katamin-AB and Azol-129 were found to be capable of producing stable artificial polycarbonate and polymethyl methacrylate suspensions. Based on the polymer, the optimal surfactant concentration was 6 wt %. The effect of polymer concentration in solution on the stability and particle size of final polymer suspensions was shown. It was determined that the polymer concentration in the solution should not exceed 10%. When obtaining a highly dispersed suspension during dispersion, a higher concentration causes an increase in the viscosity of emulsions. As a result of a synergistic effect formation, we used mixtures of cationic surfactants (Katamin-AB/Azol-138 and Azol-129/Azol-138) to enhance the stability of the final polymer suspensions. The optimal surfactant ratio was 9:1. The total concentration of the mixture is 10 wt %, based on the polymer. Polymer suspensions were stabilized with each of 2:1 mixtures of cationic surfactants Katamin-AB and Azol-129 withan organosilicon surfactant U-851. The total mixture concentration was 9 wt %, based on the polymer.*

Conclusions. *New methods of producing artificial polycarbonate and polymethyl methacrylate suspensions in the presence of domestically produced cationic surfactants, as well cationic-organosilicon surfactants mixtures, were proposed. The colloidal-chemical properties of the obtained polymer suspensions were considered. It was found that using a 2:1 mixture of cationic and organosilicon surfactants produces polymer suspensions that are stable during production and storage.*

Keywords: *organosilicon surfactants, artificial polymer suspension, cationic surfactants, polycarbonate, polymethyl methacrylate, structural and mechanical barrier*

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НАУЧНАЯ СТАТЬЯ

Влияние природы эмульгатора и концентрации полимера на дисперсность и устойчивость искусственных полимерных суспензий на основе поликарбоната и полиметилметакрилата

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Аннотация

Цели. *Создание агрегативно устойчивых искусственных полимерных суспензий с положительным зарядом частиц на основе поликарбоната и полиметилметакрилата с использованием катионных поверхностно-активных веществ (КПАВ), а также их смесей с кремнийорганическим поверхностно-активным веществом (КОПАВ).*
Методы. *Размер капель и частиц полимерных суспензий определяли методом фотонной корреляционной спектроскопии (динамического светорассеяния) с помощью лазерного анализатора частиц Zetasizer NanoZS (Malvern, Великобритания).*
Результаты. *Было установлено, что для получения устойчивых искусственных поликарбонатных и полиметилметакрилатных суспензий могут быть использованы отечественные КПАВ Катамин АБ и Азол-129. Оптимальная концентрация ПАВ составила 6 мас. % в расчете на полимер. Показано влияние концентрации полимера в растворе на устойчивость и размер частиц конечных полимерных суспензий. Определено, что концентрация полимера в растворе не должна превышать 10%. Дальнейшее повышение концентрации приводит к повышению вязкости эмульсий при получении высокодисперсной суспензии в процессе диспергирования. Использованы смеси КПАВ Катамин АБ/Азол-138 и Азол-129/Азол-138 для повышения устойчивости конечных полимерных суспензий за счет образования синергетического эффекта. Оптимальное массовое соотношение ПАВ составило 9:1. Общая концентрация смеси 10 мас. % в расчете на полимер. Получены полимерные суспензии, стабилизированные смесями КПАВ Катамин АБ/КОПАВ U-851 и КПАВ Азол-129/КОПАВ U-851 в соотношении 2:1 каждой смеси в расчете на полимер. Общая концентрация смеси составила 9 мас. % в расчете на полимер.*

Выводы. *Предложены новые способы получения искусственных поликарбонатных и полиметилметакрилатных суспензий, полученных в присутствии КПАВ отечественного производства, а также их смесей и смесей КПАВ с КОПАВ. Рассмотрены коллоидно-химические свойства полученных полимерных суспензий и показано, что при использовании смеси КПАВ и КОПАВ, взятых в объемном соотношении 2:1, образуются устойчивые в процессе получения и хранения полимерные суспензии.*

Ключевые слова: *кремнийорганические ПАВ, искусственная полимерная суспензия, катионные ПАВ, поликарбонат, полиметилметакрилат, структурно-механический барьер*

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INTRODUCTION

Recently, artificial polymer suspensions have become increasingly important because they are used in a wide range of industries, including the rubber industry (films, coatings, gloves, rubber threads, etc.), light industry (adhesives and textile materials), food industry (protective coatings on food), pulp and paper industry, construction industry (binders, sealants, compounds), agricultural industry (soil protection against erosion), and other industries.

Artificial polymer suspensions (artificial latexes) are obtained by emulsifying polymer solutions in an organic solvent in the presence of different surfactants, followed by replacing the organic phase with an aqueous phase, solvent distillation, and concentration to attain the required polymer content. In the preparation process, it is essential to maintain stability at all stages. For this, cationic surfactants of various structures are used. It is well established in the literature that combining different types of surfactants is the best way of obtaining stable polar polymer suspensions. The preparation of artificial polymer suspensions has not been described in the literature. It was best to rely on the fact that synthetic polar polymer suspension stability is characterized by high stability when using a mixture of different surfactants.

In this work, mixtures of different surfactants were used to enhance polymer suspension stability. This ensured the formation of structural-mechanical and electrostatic stabilization barriers, according to Rebinder [1].

This study aims at obtaining stable artificial polycarbonate and polymethyl methacrylate suspensions with a positive particle charge in the presence of cationic surfactants and their mixtures with other surfactants.

MATERIALS AND METHODS

The feedstocks selected were granular polycarbonate from the Makrolon brand (*Bayer*, Germany) and polymethyl methacrylate from the Acrypet VH 001 brand (*Mitsubishi Chemical Corporation*, Japan).

Without additional purification, chloroform (reagent grade) was used as a solvent.

The surfactants used were cationic surfactants synthesized at *Kotlas Chemical Plant*: Katamin AB (alkyldimethylbenzylammonium chloride, where alkyl is a mixture of normal C10–C16 alkyl radicals); Azol-129 (quaternary ammonium base of coconut oil *tert*-alkylamine acids and benzyl chloride with a hydrocarbon radical of coconut oil fatty acids C8–C14 as a substituent; active substance content is 75%); Azol-138 (*N*,*N*,*N*-trimethyl-*N*-(alkyl 12–14) ammonium methyl phosphite); Azol-139 (quaternary ammonium base from dicocoalkyldimethylamine and dimethyl phosphonate with a hydrocarbon radical of coconut oil fatty acids C12–C14). We also used an organosilicon surfactant (U-851 α,ω-bis[3-methylsiloxy]polydimethylmethyl-(10-carboxydecyl) siloxane) synthesized at the N.S. Enikolopov Institute of Synthetic Polymeric Materials of the Russian Academy of Sciences.

The size of droplets and polymer suspension particles was determined by photon correlation spectroscopy (dynamic light scattering) using a Zetasizer NanoZS laser particle analyzer (*Malvern*, UK) [2].

Dynamic light scattering (DLS) is a combination of the following phenomena: a change in frequency (Doppler shift) and an intensity and in motion direction of light transmitted through a medium of moving (Brownian) particles. It is a method for measuring particles that are up to 6 μm in diameter. The Brownian motion of particles is measured by DLS and correlated with their size. Elastic (Rayleigh) scattering occurs when a light beam passes through a suspension. In DLS, coherent and monochromatic laser radiation is used. The autocorrelation function, which is determined from the time variation of scattered radiation intensity, is the quantity being measured:

$$G(t_d) = 1/N \sum_i I(t_i)I(t_i - t_d) = (I(t)I(t - t_d)),$$

where $G(t_d)$ is the autocorrelation function; N is the number of measurements performed at time t_i; $I(t_i - t_d)$ is the light scattering intensity after a certain delay time t_d.

Suspensions were prepared by combining a hydrocarbon phase (a polymer dissolved in a solvent) with a surfactant aqueous solution in a 1:1 ratio. The first stage involved using a magnetic stirrer to prepare a low-dispersed emulsion. The droplet size ranged from 20 to 100 μm. The second stage involves using a rotor-stator homogenizer DIAX-900 (*Heidolph*, Germany) to disperse a low-dispersed emulsion to obtain a highly dispersed emulsion. Dispersion speed: 24000 rpm, dispersion time: 7–10 min. A rotary evaporator RV 10 (*IKA*, Germany) was used to distill the solvent.

RESULTS AND DISCUSSION

According to the published data, the emulsion dispersity (expressed by the polydispersity index, *PdI*), average particle diameter (d_{av}), and charge (ζ-potential) are the main parameters that determine suspensions stability [3–5].

Investigations began with the study of the colloidal-chemical properties of the polycarbonate (PC) and polymethyl methacrylate (PMMA) suspensions obtained in the presence of different cationic surfactants.

Artificial polymer suspensions were obtained using a 5% polymer solution. For the polymer, the concentration of the surfactant was 6 wt %.

Tables 1 and 2 and Figs 1 and 2 show the colloidal-chemical properties of the polycarbonate and polymethyl methacrylate suspensions obtained using Katamin AB and Azol-129 in different concentrations.

Table 1 and Fig. 1 show the number-average particle diameters of polycarbonate artificial polymer suspensions determined using Katamin AB, Azol-129, Azol-138, and Azol-139. The polymer suspension sample obtained using Katamin AB as a stabilizer had great stability, a narrow particle size distribution, and particle diameters ranging from 500 nm to 700 nm (80% of the particles by number). The samples stabilized with Azol-129 and Azol-138 had the largest diameter and particle size distribution. In the presence of Azol-139, it is impossible to obtain stable artificial polymer suspensions. A significant

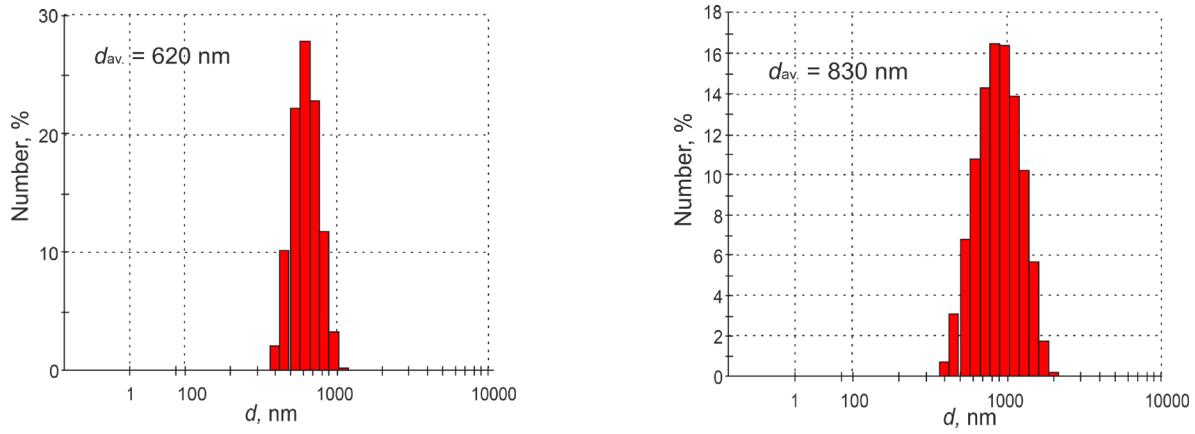
Table 1. Comparative analysis of the colloidal-chemical properties of artificial polycarbonate polymer suspensions stabilized by Katamin AB, Azol-129, Azol-138, and Azol-139, taken at different concentrations

Polymer	Surfactant	d_{av}, nm	*PdI*	ζ-potential, mV	Coagulum, wt %
PC	Katamin AB	620	0.140	+30	–
	Azol-129	830	0.290	+24	–
	Azol-138*	1050	0.330	+25	10
	Azol-139	–	–	–	100

*The size of the coagulum-free polymer suspension.

Table 2. Comparative analysis of the colloidal-chemical properties of artificial polymethyl methacrylate PS stabilized by Katamin AB, Azol-129, Azol-138, and Azol-139

Polymer	Surfactant	d_{av}, nm	*PdI*	ζ-potential, mV	Coagulum, wt %
PMMA	Katamin AB	260	0.270	+39	–
	Azol-129	300	0.359	+28	–
	Azol-138	310	0.375	+23	–
	Azol-139	–	–	–	100



a b

Fig. 1. The number-average particle size distributions of polycarbonate artificial polymer suspensions when used in the production process as a surfactant: (a) Katamin AB, (b) Azol-129.

part of the polymer coagulated when the solvent was removed under vacuum. This could be due to the poor stability of the particles in the presence of the atypical cationic surfactant with two alkyl radicals with a chain length of C12–C14 in the hydrophobic part of the surfactant molecule. The most stable suspensions were obtained using Katamin AB. The fact that it has a higher surface activity than the other stabilizers presented [6] explains this outcome.

Data on the stability of the polymethyl methacrylate polymer suspensions using different surfactants revealed that Katamin AB allows for the narrowest particle size distribution (Table 3 and Fig. 2).

The polymer concentration effect in the initial chloroform solution at all the preparation stages was evaluated using the final properties of the polymer suspensions. The polymer concentration in the solution was varied from 5% to 20% (Table 4).

As shown, the suspensions obtained using polymer solutions with concentrations of 5% and 10% are stable. The average particle diameters and *PdI* increase as polymer concentration increases.

The viscosity of solutions with a polymer concentration of more than 10% was high. As a result, polymer suspension emulsification, degassing, and concentration was difficult.

According to the literature, a synergistic effect, which significantly affects the colloidal-chemical properties of polymer suspensions, can occur when using mixtures of surfactants in a certain volume ratio of components [6].

Azol-129/Azol-138 and Katamin AB/Azol-138 were selected as the surfactant mixtures. The colloidal-chemical properties of the polymer suspensions obtained in the presence of these mixtures are presented in Tables 5 and 6 and Fig. 4.

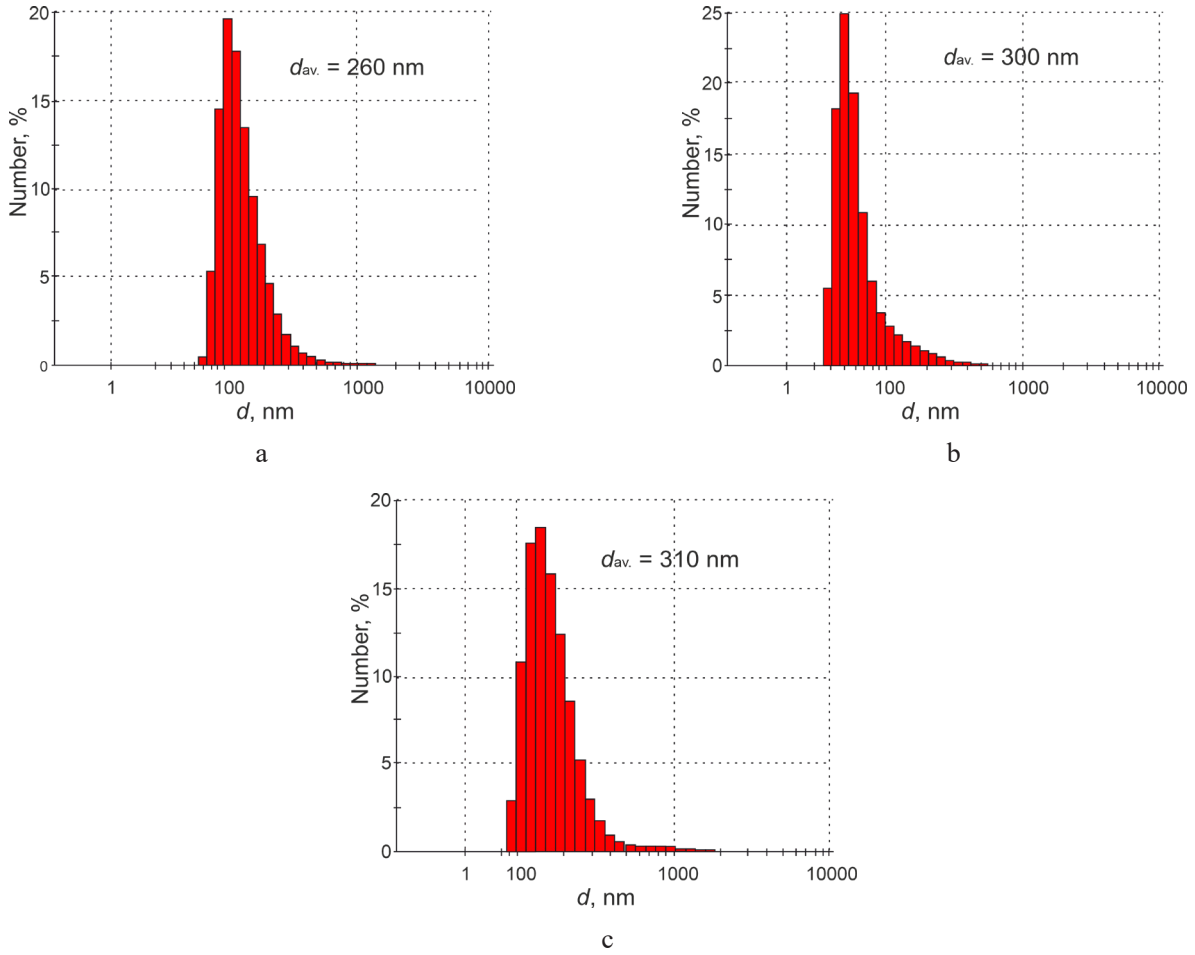


Fig. 2. The number-average particle size distributions of polymethyl methacrylate artificial polymer suspensions when the following surfactants were used in the production process: (a) Katamin AB, (b) Azol-129, and (c) Azol-138.

Table 3. The stability of artificial polycarbonate and polymethyl methacrylate polymer suspensions depending on the polymer concentration

Polymer concentration in solution	Stability in time	
	PC	PMMA
5	+	+
10	+	+
20	+/–	+/–

The conducted study reveals that it is necessary to use a mixture of surfactants Azol-129/Azol-138 and Katamin AB/Azol-138 in a ratio of 9:1, respectively, to achieve the maximum effect (to reduce the particle diameter). When using Azol-129/Azol-138 and Katamin AB/Azol-138 mixtures, there is a significant increase in stability compared to polymer suspensions where these surfactants are used separately. Polymer suspensions stabilized with Azol-129 were characterized by low stability and an average particle diameter of about 800 nm. By adding one mass part of Azol-138 to the system, the polymer suspension stability was greatly improved, and the particle diameter was reduced to 580 nm. The addition of AB Azol-138 to Katamin AB allowed for a reduction in the average diameter (from 620 nm to 400 nm).

Table 4. Colloidal-chemical properties of polycarbonate suspensions stabilized using Katamin AB and obtained at various polymer concentrations in solution

Concentration, %	d_{av}, nm	*PdI*	Coagulum, wt %
5	620	0.140	–
10	1260	0.330	–
20	840	0.270	50

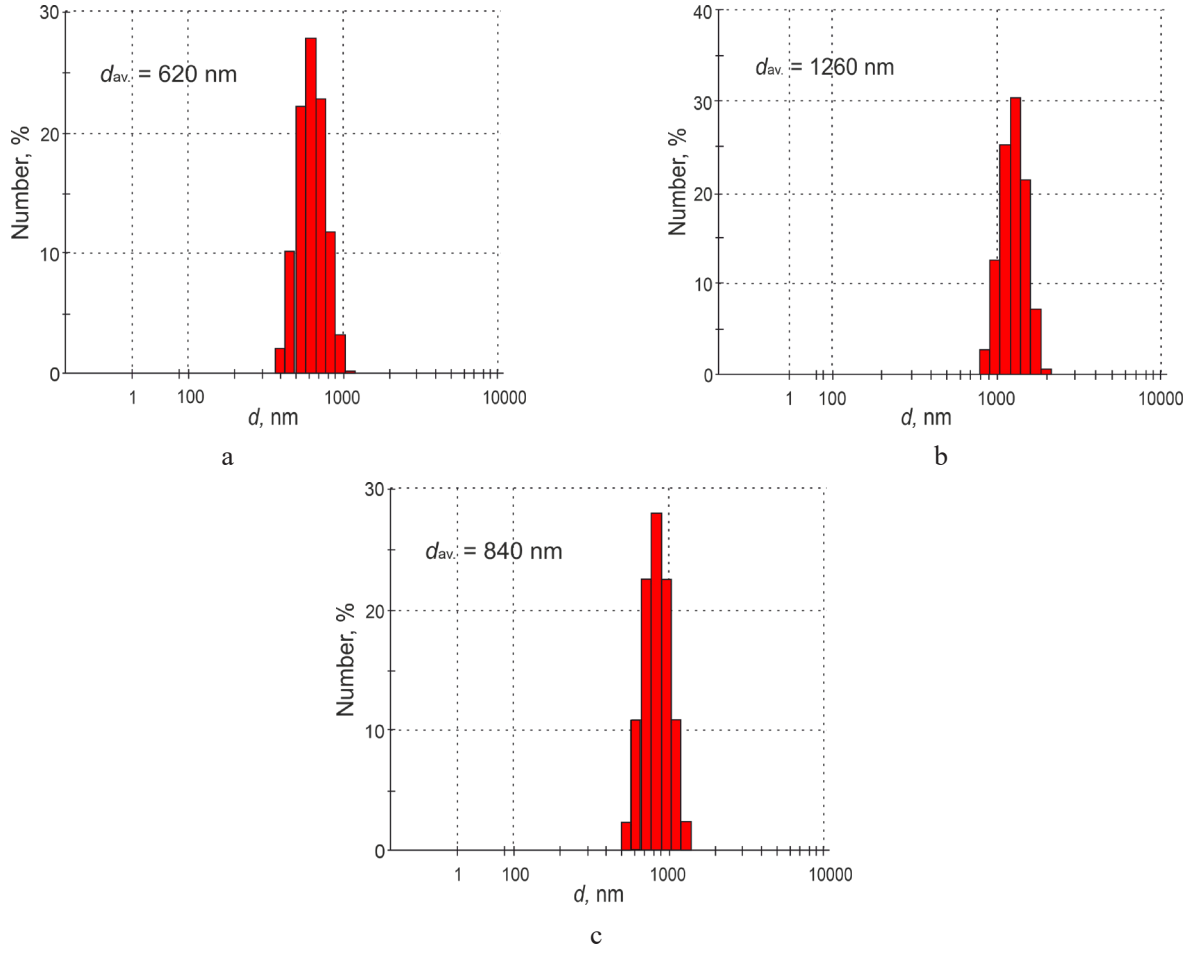


Fig. 3. Numerical distributions of particle size in polymer suspensions for the suspensions presented in Table 3: (a) 5%, (b) 10%, and (c) 20%.

Table 5. Colloidal-chemical properties of polycarbonate polymer suspensions obtained using varied ratios of the surfactants Azol-129/Azol-138

Surfactants	Surfactants ratio	d_{av}, nm	*PdI*	ζ-potential, mV	Coagulum, wt %
Azol-129/Azol-138	9:1	588	0.101	+22	нет no
Azol-129/Azol -138	3:1	770	0.331	+16	–
Azol-129/Azol -138	2:1	1510	0.255	+6	10

Table 6. Colloidal-chemical properties of polycarbonate polymer suspensions stabilized with Katamin AB/Azol-138 in different mass ratios

Surfactants	Surfactants ratio	d_{av}, nm	*PdI*	ζ-potential, mV	Coagulum, wt %
Katamin AB/Azol-138	9:1	402	0.136	+32	no
Katamin AB/Azol-138	3:1	607	0.320	+13	5
Katamin AB/Azol-138	2:1	694	0.382	+7	10

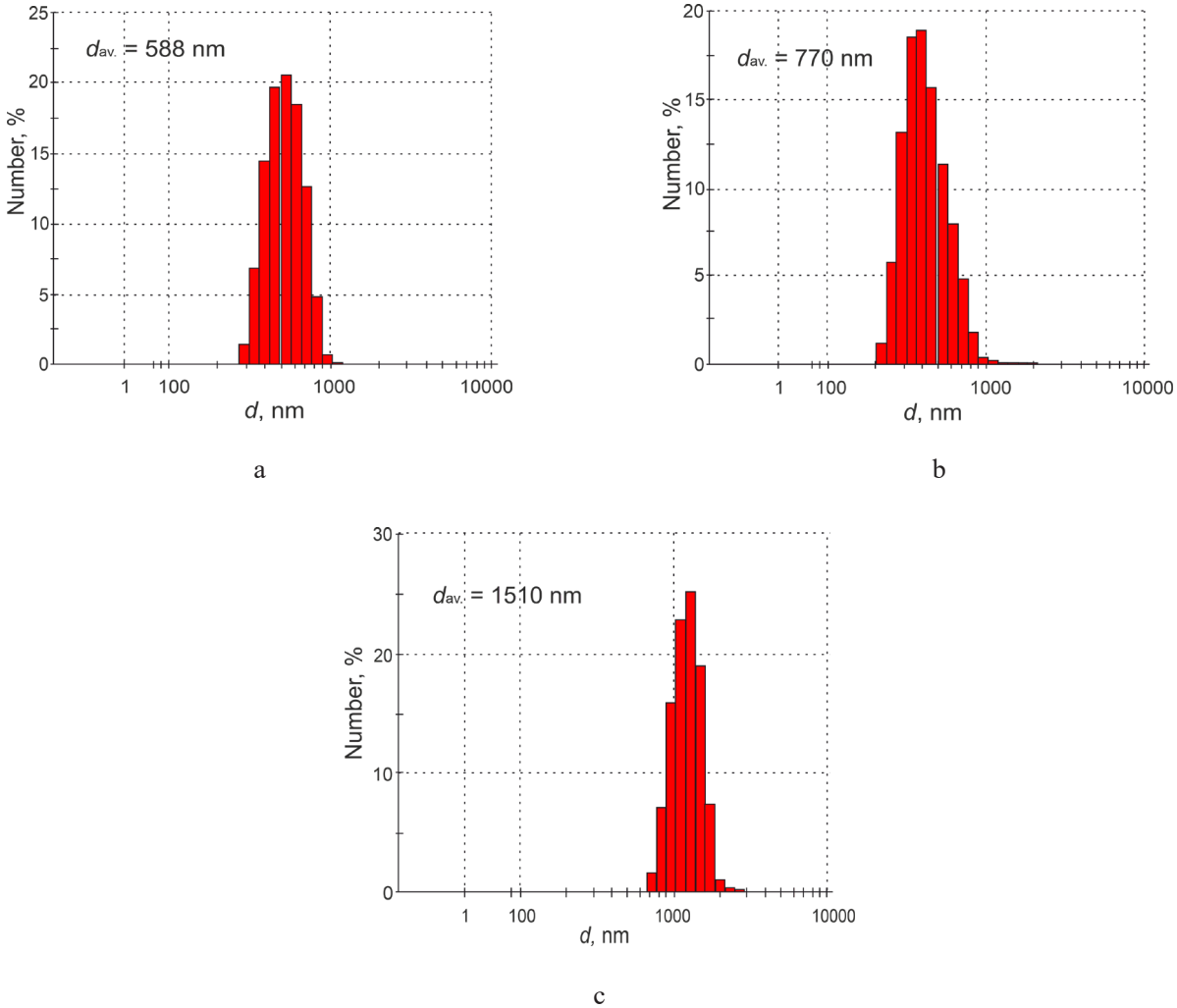


Fig. 4. Numerical distributions of polymer suspension particle size for suspensions presented in Table 4: (a) 9:1, (b) 3:1, and (c) 2:1.

Recently, there have been interests in water-insoluble organosilicon surfactants, owing to the possibility of obtaining aggregate-stable polymer suspensions through the polymerization of vinyl monomers [7, 8].

The formation of an interfacial adsorption layer on particle surfaces in their presence was found to be different from that observed in the presence of water-soluble surfactants.

This difference is due to the formation of a thick strong interphase layer, which is due to the adsorption of a water-insoluble surfactant from the monomer phase. These results were demonstrated when studying the colloidal-chemical properties of different organosilicon surfactants [9–16].

Carboxyl-containing organosilicon surfactant α,ω-bis(trimethylsiloxy-oligodimethylmethyl-(10-carboxydecyl)siloxane (U-851) and its mixture with the cationic surfactants Azol-129 and Katamin AB were used.

The surfactants mixture had a concentration of 9 wt % calculated for the polymer, with a cationic surfactant/organosilicon surfactant ratio of 2:1. The concentration of U-851 was calculated to be 3 mass fractions for the polymer.

In these works, it was found that the formation of an interfacial adsorption layer on the surface of particles in their presence is fundamentally different from that observed in the presence of water-soluble surfactants [3,7].

To obtain stable polymer suspensions, we used a mixture of the cationic surfactants Katamin AB and Azol-129 with the carboxyl-containing organosilicon surfactant U-851.

The surfactants mixture had a concentration of 9 mass fractions calculated for the polymer, with a cationic surfactant/organosilicon surfactant ratio of 2:1. The concentration of the surfactant U-851 in the suspension stabilized only with it was 6 mass fractions calculated for the polymer.

The stability properties of the obtained suspensions and their colloidal-chemical properties are presented in Tables 7 and 8 and Fig. 5.

Table 7. Stability of polycarbonate suspensions over time when a mixture of a cationic surfactant and an organosilicon surfactant is used

Surfactant	Stability over time
Katamin AB/U-851	+
Azol-129/U-851	+
U-851	–

Table 8. Colloidal-chemical properties of artificial polycarbonate suspensions obtained using U-851 and its mixture with a cationic surfactant

Surfactant	d_{av}, nm	*PdI*	ζ-potential, mV	Coagulum, wt %
Katamin AB/U-851	360	0.240	+32	–
Azol-129/U-851	410	0.287	+25	–
U-851	–	–	–	100

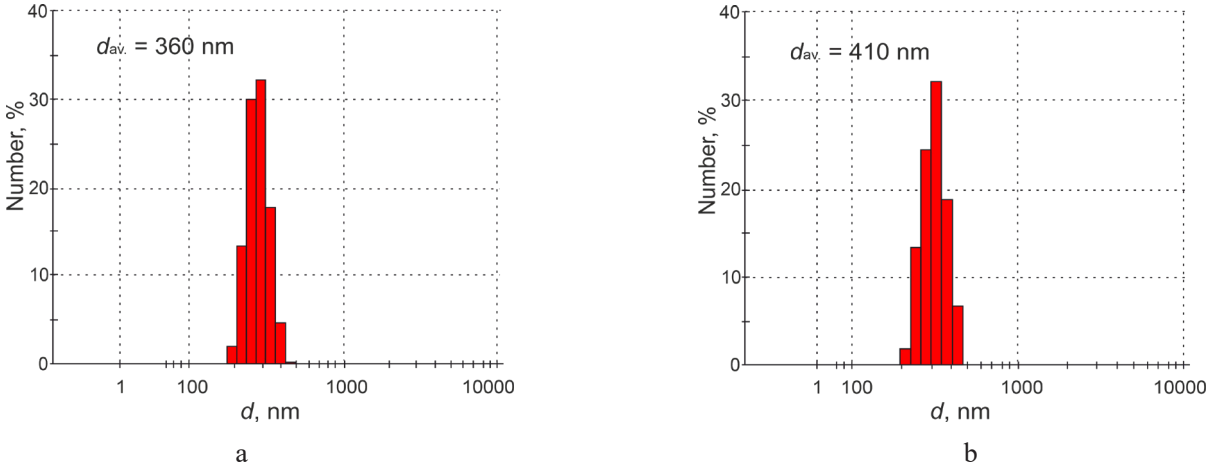


Fig. 5. Numerical distributions of polycarbonate suspensions particle sizes depending on the choice of surfactant mixtures: (a) Katamin AB/U-851 and (b) Azol-129/U-851.

Both suspensions were characterized by a rather narrow particle size distribution, with an average particle diameter of 360 nm and a charge of +32 mV for the Katamin AB/U-851 mixture, and an average particle diameter of 410 nm and a charge of 25 mV for the Azol-129/U-851 mixture.

CONCLUSIONS

The article proposed a methodology for obtaining stable artificial polycarbonate and polymethyl methacrylate polymer suspensions with a positive particle charge. It was shown that to obtain them, domestically produced cationic surfactants with a concentration of 6 wt % calculated for the polymer can be used. It is proposed to use a 9:1 mixture of surfactants: Azol-129 and Azol-138; Katamin AB and Azol-138, with a total surfactant concentration of 10 wt % calculated for the polymer to form a more durable electrostatic stability barrier in the interfacial adsorption layers. It is possible to increase stability over time using Katamin AB/U-851 and Azol-129/U-851 mixtures because of the formation of structural-mechanical and electrostatic stability barriers in the interfacial adsorption layers of polymer particles.

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Authors' contributions

A.N. Stuzhuk – development of a method for obtaining aggregate-stable artificial polymethylmethacrylate and polycarbonate suspensions with a positive charge of particles, writing and editing the text of the article, and preparing a manuscript for publication;

A.V. Shkolnikov – collecting and processing the material, synthesis of aggregate-stable artificial polymethyl methacrylate and polycarbonate suspensions with a positive particle charge;

P.S. Gorbatov – synthesis of artificial polymethylmethacrylate suspensions with a positive particle charge, and statistical processing of research results;

I.A. Gritskova – developing the scientific concept, offering consultation on the research methodology, writing and editing the text of the article.

The authors declare no conflicts of interest.

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