Solution Properties, Stabilizing and Structure Forming Abilities of New Biosurfactants

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ABSTRACT

Biosurfactants are amphipathic molecules with hydrophobic and hydrophilic regions and they can aggregate along the boundary of different phases of liquids. Biosurfactants are produced at the microbial cell surface and have several advantages. Currently the most research has been toward the discovery of biosurfactants from biological natural sources. Biosurfactants are produced by a wide range of microorganisms, and these microorganisms offer considerable promise in producing novel types of biosurfactants. Biosurfactants are environmentally friendly, nontoxic and biodegradable in nature. These potential advantages make them useful in several applications. The purpose of this work was obtaining of new biological surface-active substances and establishing correlations between their surface activity, colloid-chemical properties and stabilizing abilities. The modern methods for separation, determining the surface activity and colloid-chemical properties, stabilizing abilities of new biosurfactants were used in the work. The new biological surfactants were isolated by acid precipitation and purified by solvent extraction. The surface activity and foam forming ability of the obtained new biosurfactants in aqueous solutions were studied depending on concentration and temperature of the system. The obtained results showed that with the increase of the concentration of biosurfactants in water solutions, the surface activities of new biosurfactants were increased. The analysis of results showed good correlations between the foam forming abilities and the surface activity of the studied biosurfactants. Based on the analysis of experimental results it was established that the stabilities of foams were significantly determined by the interaction of biosurfactant molecules in the monolayer. Surfactants are widely used to improve the quality of materials and control technological processes. In this regard, a very urgent problem is the study of new biosurfactants and creation of new compositions to improve the rheological and structure-forming properties of dispersed systems and cement slurries. To effectively solve this problem, it is necessary to investigate new surfactants in disperse systems. Different methods for the measurements of properties and efficiency of new surfactants were used. The obtained experimental data and correlations based on them showed that the colloid-chemical properties of the studied biosurfactants are closely related to the structure of their molecules. It was established that new biosurfactants are effective regulators of the structure formation of cement dispersed systems. It was shown that the introduction of new biosurfactants in combination with marble powder enhances the plasticizing effect and makes it possible to replace part of the binder with marble powder without reducing the final strength of the cement stone. It has been established that with the increase of biosurfactants concentration in water solutions, the surface activity and foam forming abilities of new biosurfactants were increased. The use of modified marble powder with aqueous solutions of new biosurfactants, lengthens the stage of initial structure formation and accelerates the subsequent process of structure formation with an increase in the daily strength of the cement stone.

Keywords: Surfactant, surface activity, regulators of structure formation, plasticizing effect, cement disperse systems.

Introduction

Surfactants are amphipathic molecules that have hydrophobic and hydrophilic regions and they can aggregate along the boundary of different phases of liquids such as oil/water or air/water. When the concentration of these molecules increases beyond a threshold, they form micelles [1-5]. Currently many research works have been towards the discovery of surfactants from biological natural sources and such surfactants are called biosurfactants. The most surfactants that are used today for industrial applications are syntheticallymanufactured via organo-chemical synthesis using different chemicals and they are environmentally incompatible, derived from non-renewable resources, have toxicological effects to humans and other organisms. Well known that nowadays key challenges are to move towards using renewable and sustainable sources for obtaining surfactants. Due to the enormous genetic diversity of microorganisms, they offer considerable promise in producing novel types of biosurfactants for replacing surfactants produced from organo-chemical synthesis. Biosurfactant-producing bacteria can be found in a wide range of habitats, from aquatic (groundwater, fresh and sea water) to terrestrial (soil, sediment and sludge) environments. Biosurfactants are produced by a wide range of microorganisms such as bacteria, fungi, and yeast as secondary metabolites which are either secreted extracellularly or adhered to the cell surfaces [6-11]. Biosurfactants can be used to replace chemical surfactants, as they are environment-friendly, less toxic, biodegradable in nature, have higher foaming ability, and possess lower CMC values [12-14]. These potential advantages make them useful in several applications such as bioremediation, health care, cosmetics, food, and oil industries [15-17]. The chemical composition of biosurfactants varies greatly between different species of microorganisms and broadly can be classified based on their molecular weight or chemical charge. Based on their molecular weight, biosurfactants are classified as low-molecular-weight biosurfactants, which reduce surface tension between two immiscible liquids, and high-molecular-weight biosurfactants, which enable the formation of oil-in-water or water-in-oil emulsions and they are also called polymeric biosurfactants [18-23]. The environment can have a direct influence on the type of biosurfactants that microorganisms produce. The diverse nature of the influence of biosurfactants on the properties of phase interfaces and their use in regulating the characteristics of disperse systems are due to the ability of surfactants to reduce surface energy by being adsorbed at the phase boundaries. By covering the surface of various solids and liquids with the thinnest monomolecular layers, surfactants make it possible to regulate technological processes. Due to this, surfactants have found wide application to improve the quality of various materials [24–28]. Many processes cannot proceed without the participation of surfactants, such as dispersion and stabilization of suspensions and foams, modification and protection of various surfaces, regulation and stabilization of cement mortars and concrete mixtures [29-30]. Increasingly important is the introduction of surfactants into mineral binders to give them the necessary structural and mechanical qualities and control the technological characteristics of these systems. The acceleration and deceleration of the setting time, the control of the flow regime of cement slurries can be provided by the treatment of surfactants. The development of the chemical industry, the growth in the production of synthetic products make it possible to widely use various methods and formulations to control the properties of binders. There are a number of works devoted to the study of the effect of surfactants and the mechanism for regulating the setting time, increasing the mobility and reducing the water loss of cement slurries [31-32]. In this regard, very few studies were reported on the maleic acid derivative surfactants. Currently the implemented in industries plasticizing additives and surfactants are imported from foreign countries and require large foreign exchange costs. Therefore, it is very important and relevant to find new import-substituting surfactants with high plasticizing properties of cement slurries. For these purposes, it is also very important and relevant to create modified mineral additives with polyfunctional surfactants and new cement compositions with improved structure-forming properties and concrete mixtures with high strength for industrial and residential construction. Also very relevant is the study of the relationship between the structure and effectiveness of polyfunctional surfactants on the rheological properties of the concrete composition. Based on the results of these studies, it is possible to develop the scientific basis for the creation of effective surfactants that improve the technological properties of dispersed systems, cement slurries, and concrete compositions. In this regard, it is also important to identify the effectiveness of new biologic surfactants and their composition on cement-concrete mixtures. The purpose of this work was to study new biologic surface-active substances, to investigate their colloidal chemical properties depending on concentration and temperature, and their effectiveness for controlling structure formation in cement slurries.

Methods and materials

Determination of surface tension. The surface tensions of biosurfactant solutions were determined using tensiometer DCAT-9T at different temperatures and concentrations. In order to obtain statistically significant results, each measurement was repeated 5 times.

Extraction and purification of biological surfactants. Biological surfactants were isolated by acid precipitation and purified by solvent extraction. The selected microbes were planted in a medium with nutrients and stored in a thermostat at a constant temperature. Each experiment was conducted at different temperatures in a thermostat. After incubation at a constant temperature for 132 hours in a thermostat, the cells were removed from the cultural liquid by centrifugation for 15 minutes at a constant temperature. The cell-free supernatant thus obtained was acidified with a 10% aqueous solution of acetic acid and the resulting mixture was stored for 16 hours at a constant temperature in an incubator to enhance the precipitation of biological surfactants. The precipitate formed as a result of storage in a thermostat was separated by centrifugation for 20 minutes. The thus isolated precipitate was extracted several times with ethyl alcohol at room temperature. The resulting extract was filtered and then the ethanol solvent in the extract was distilled under reduced pressure. The residue in the flask after distillation was dissolved in acetone and reprecipitated with n-hexane. The biological surfactants isolated after reprecipitation were dried in a thermostat under reduced pressure. Biological surfactants obtained in this way were reddish-brown, viscous substances with a peculiar odor. Depending on the temperature of the medium with nutrients during the experiments obtained biosurfactants have names BDS-1, BDS-2, BDS-3 and BDS-4.

Plastic strength and structure formation of cement mixtures. The kinetics of structure formation in cement mixtures was judged by the change in plastic strength. Measurements for the study of plastic strength and structure formation of cement mixtures were carried out on a Bostwick consistometer.

Determination of the density of the cement mixture. The density of the cement mixture was characterized by the ratio of the mass of the compacted mixture to its volume and was expressed in g/cm^3. A steel cylindrical vessel with a capacity of 1000 ml was used for testing.

Foam forming ability. The foam forming abilities of biosurfactants were determined at a temperature of 293K, while 100 ml of a freshly prepared surfactant solution with a certain concentration was shaken in a graduated container for 60 s. Then the height of the foam column at the initial moment in the graduated container was measured.

Density of surfactant samples. The density meter (Easy plus) was used to determine the density of new biosurfactants. The density of the surfactants was measured at a temperature of 293 K.

Refractive indices of aqueous solutions of surfactants. The refractometer (Easy plus) was used to determine the refractive index of aqueous solutions of the new surfactants. The refractive index of aqueous solutions was measured at a temperature of 293 K.

As a micro-filler for cement mixtures marble chips obtained from a marble factory was used. Marble powder (MP) of the required size was obtained by grinding marble chips in a mill for 20 minutes. The biosurfactants were purified by vacuum distillation and recrystallization methods and their water solutions were used for cement mixtures.

Results and discussion

Surfactants are amphipathic molecules that have hydrophobic and hydrophilic regions and they can aggregate along the boundary of different phases of liquids such as oil/water or air/water. When the concentration of these molecules increases beyond a threshold, they form micelles. Due to the enormous genetic diversity of microorganisms, they offer considerable promise in producing novel types of biosurfactants for replacing surfactants produced from organo-chemical synthesis. Biosurfactants can be used to replace synthetic surfactants, as they are environment-friendly, nontoxic, biodegradable in nature, have higher foaming ability and lower CMC values. These potential advantages make them useful in several applications such as bioremediation, health care, cosmetics, food and oil industries. Among the numerous surface-active substances that are widely used in practice, biological surfactants are poorly studied. Biosurfactants combine high surface activity, good biodegradability, low toxicity to warm-blooded animals and relatively low cost. Due to such properties, they can be used in various fields of industry, agriculture and in everyday life. For this study we used new biologic surface-active substances BDS-1, BDS-2, BDS-3 and BDS-4. The new biological surfactants were isolated by acid precipitation and purified by solvent extraction. The physic-chemical properties of new biological surface-active substances are presented in Table 1 below.

Table 1. The physic-chemical properties of new biologic surface-active compounds.

Abbreviation of surfactant	Physical appearance	Optical density, D	Electro-conductivity	Rf	n_D^{20}	d_4^{20}
BDS-1	Brown viscous substance	1.3148	4.7956	0.68; 0.79	1,4925	1.11
BDS-2	Red-brown viscous substance	1.3672	4.4895	0.61; 0.72	1,5385	1.13
BDS-3	Brown, viscous substance	1.3183	4.6185	0.65; 0.81	1,5465	1.15
BDS-4	Darkbrown viscous substance	1.3842	4.6574	0.68; 0.84	1.6124	1.17

*Rf – Thin layer chromatogram retention factor.

The isolated and purified biological surfactants were weighed and aqueous solutions with different concentrations were prepared from them. The surface activity and disperse systems stabilizing ability of new biological surfactants in water solutions were investigated. The results of a study of the surface tension of aqueous solutions of the new biologic surface-active substances and the influence of the concentration and temperature of the system on the surface-active properties of new biosurfactants are presented in Table 2 below. Analysis of the obtained experimental data in Table 2 showed that the studied biosurfactants are located in the following order in terms of increasing surface activity: BDS-4>BDS-3>BDS-2>BDS-1. It can be seen from Table 2, that with an increase in the surfactants' concentration, the surface activity of new biosurfactants has increased, which was related with the increase adsorption of biosurfactant molecules.

Table 2. Surface tension of biosurfactant water solutions depending on concentration and temperature.

Surfactant	T, K	Surface tension σ (mN/m) of surfactant solutions at different concentrations $(C\cdot10^2)$ mol/l								
		0,02	0,04	0,08	0,16	0,31	0,62	1,25	2,51	5,22
BDS-1	293	72,1	71,2	68,6	64,8	54,9	46,9	40,9	36,1	34,8
	303	71,5	69,8	67,7	62,6	53,2	45,7	40,2	35,5	33,7
	313	70,9	68,9	66,8	58,8	52,4	44,5	38,5	34,8	32,5
	323	69,8	67,6	65,6	57,5	51,2	43,4	37,8	33,9	31,6
	333	68,1	66,5	61,4	55,8	50,3	42,8	35,9	33,1	30,8
BDS-2	293	71,5	70,7	67,8	62,7	52,8	45,6	39,5	35,3	33,8
	303	70,9	69,5	66,6	61,5	51,6	44,3	38,8	34,7	32,6
	313	70,1	68,5	65,4	59,7	50,4	43,8	36,5	33,6	31,9
	323	68,6	66,7	61,3	56,4	49,5	42,4	35,9	33,8	30,6
	333	67,4	65,9	60,7	52,6	48,8	41,5	34,8	32,4	30,4
BDS-3	293	69,5	67,3	63,2	58,6	53,2	41,9	34,7	31,9	31,2
	303	67,6	65,8	62,8	57,5	48,6	40,8	33,4	30,7	29,4
	313	66,9	64,3	59,3	54,8	45,4	39,9	32,1	29,1	28,9
	323	65,7	62,7	58,1	52,3	44,5	38,8	30,9	28,5	27,8
	333	62,2	60,2	56,9	50,4	42,8	38,6	30,4	27,1	26,5
BDS-4	293	68,5	65,8	61,6	55,5	51,8	40,9	32,4	30,6	29,8
	303	66,3	63,4	60,5	54,3	46,7	39,8	30,9	29,1	27,5
	313	64,7	62,5	57,5	52,2	43,6	38,6	30,1	26,8	26,8
	323	62,1	61,7	56,6	50,8	42,2	37,6	29,7	25,2	25,5
	333	59,8	58,9	55,8	48,6	41,8	36,5	28,2	25,4	24,8

On the basis of analysis of the surface tension isotherms found for aqueous solutions of new biosurfactants the thermodynamic parameters of adsorption at the liquid-gas interface were calculated. The calculated thermodynamic parameters of adsorption showed that with the increase of the biosurfactants concentration in the system, the adsorption capacity and surface activity of the studied biosurfactants were increased. The analysis of the calculated thermodynamic parameters of biosurfactant adsorption at the liquid-gas interface showed that the adsorption process proceeded with absorption of energy and with low enthalpy values. The values of the enthalpy and entropy of adsorption increase with the increase of the content of studied biosurfactants in the compositions.

Biosurfactants are of great interest as stabilizers and regulators of the properties of the different dispersed systems. The effectiveness of biosurfactants in many cases depends on the structure and composition of their molecules. In this regard, the study of the stabilizing properties of new biosurfactants is of great scientific and practical importance. The stabilizing abilities and effect of new biosurfactants on foam dispersions, as well as the effect of system temperature and biosurfactants concentrations on foam forming abilities were studied. The results of a study of the foam forming ability of aqueous solutions of the studied new biosurfactants are presented in Table 3.

Table 3. The foam-forming ability and stability of foams in aqueous solutions of new biosurfactants.

Surfactant	T, K	Foam-forming ability (ml)/Foams stability at different biosurfactant concentrations (g/l)					
		0,1	0,5	0,62	1,25	2,5	5,0
BDS-1	293	181/84	217/87	226/89	271/89	289/91	337/92
	313	195/83	238/84	251/84	293/85	329/86	354/87
	333	206/46	249/49	265/54	309/55	339/57	359/56
BDS-2	293	185/86	221/89	229/94	277/94	297/94	345/94
	313	199/84	244/85	256/85	299/86	335/89	359/89
	333	210/48	257/53	269/54	318/56	346/59	365/58
BDS-3	293	189/88	226/91	234/95	281/94	299/95	349/95
	313	203/86	248/86	259/87	302/87	338/90	365/92
	333	214/51	261/55	275/56	321/57	349/60	368/59
BDS-4	293	195/90	232/93	239/95	287/95	304/96	354/96
	313	208/87	251/87	265/88	307/88	342/91	368/94
	333	218/58	265/58	278/58	324/58	354/62	371/61

The analysis of the research results in Table 3 showed that, with the increase in the concentration of biosurfactants in water solutions and the temperature of the system, the foam forming abilities of new biosurfactants were increased. The increase of the foam forming abilities of new biosurfactants with the increase in water solutions temperature (Table 3) was associated with a change in the kinetic parameters of the adsorption of biosurfactant molecules, and, accordingly, the parameters of the dielectric layer at the interface. However, it should be noted that the increase in the volume of the formed foams was accompanied by a decrease in their stability. Such results were probably due to an increase in liquid drainage from foam films, and, accordingly, an increase in the rate of foam destruction. The analysis of the data presented in Table 3 showed that, at the temperature of 333K the slowly falling foams turn into the rapidly falling foams. At lower temperatures from 293 to 313K, the foams stabilities were very high and equal to 0.88–0.96. Apparently, such results were due to the formation of a highly viscous structured films of biosurfactant molecules at the solution–air interfaces. The obtained results of the foam forming abilities of new biosurfactants were compared with some physical and colloid-chemical properties of their water solutions. As can be seen from the research results (Table 3), an increase in the biosurfactant concentrations leads to the increase in foam forming abilities of biosurfactants. Apparently, these results were due to the fact that with the increase of biosurfactant concentrations in the system, the adsorption of biosurfactants molecules were increased, and, accordingly, the foam forming abilities of biosurfactants solutions increased. The increase in foam forming capacity with the increase of concentrations of new biosurfactants were also associated with the processes of hydration of biosurfactants hydroxyl groups, strengthening of the structure of dense and saturated monolayers due to the network of hydrogen bonds. It has been found that there is a good correlation between the foam forming ability and the surface activity of the studied biosurfactants. It should be noted that the surface activity and foam forming abilities of new biosurfactants were interrelated, and such results were due to the similarity of interfacial processes for the parameters considered, which were important in the formation and stabilization of foams, and the formation of monolayers at the liquid/gas interfaces.

The effect of new biosurfactants and mineral additives on the rheological properties and structure formation processes in cement slurries has been studied. The effect of new biosurfactants and a mineral filler such as marble powder on the kinetics of structure formation in cement stones is presented in Table 4 below. The obtained results of studies in Table 4 showed that the addition of new biosurfactants to cement suspensions prolongs the setting time, however, daily strength gains were below the benchmark. To increase the daily strength, marble powder filler was introduced into the system, both in pure form and in a modified form. Modification of marble powder was carried out with aqueous solutions of new biosurfactant BDS-3 and as a result, modified marble powder (MMP) was obtained. The obtained experimental results are presented in Table 4 below. Analysis of the experimental results in Table 4 showed, that the introduction of marble powder into the system lengthens the initial period of structure formation several times, and the daily strength index turned out to be lower than the control one. The use of marble powder together with the new biosurfactant BDS-3 in cement slurry gave similar results. Apparently, marble powder, adsorbing part of the surfactant additive, reduces its active concentration, on the one hand, and is a substrate for emerging neoplasms, accelerates the further hydration of the binder and, accordingly, increases the concentration of the structure-forming element in the system, on the other. Such a negative effect of the filler on the plasticizing effect of the additives was eliminated by pre-treatment of the surface of the marble powder with solutions of new biosurfactants. Such pre-treatment of the marble powder surfaces made it possible to lengthen the stage of initial structure formation up to 2.4-2.6 hours, with a sharp acceleration of the subsequent process of structure formation with an increase in the daily strength of the cement stone up to 1.6 -1.8 times higher than in the control sample. Apparently, this result is associated with the manifestation of surface-active, wetting, dispersing and modifying properties of the new synthesized surfactants when interacting with marble dust. An increase in the dispersion of the system leads to an increase in the number of hydrate-filler contacts, and accordingly, the increase in the number of neoplasms in the form of carbo-aluminates, forming a structure with increased strength. The introduction of additives of new biosurfactant BDS-3 in combination with a mineral filler marble powder enhances the plasticizing effect and makes it possible to replace part of the binder with marble powder without reducing the final strength of the cement stone.

Table 4. The effect of biosurfactants and fillers on the kinetics of structure formation in cement stones.

MP, %	BDS - 3, %	MMP, %	Plastic strength, (P_m, мPa).						
			Hours				Days		
			1	2	4	6	1	7	14
6			1,68	7,21	59,83	115,44	1221,8	1438,2	1843,1
12			2,72	7,22	41,64	125,36	994,3	1224,0	1472,2
24			2,61	6,94	29,25	108,81	805,5	1006,4	1034,3
6	0,2		0,48	0,54	4,855	24,315	514,33	1445,0	1591,0
6	0,4		3,24	2,72	5,915	10,455	235,11	1375,0	1545,0
6	0,8		6,85	8,41	10,40	11,536	45,925	964,15	944,2
6	1,6		5,40	16,5	20,13	30,154	54,386	781,26	755,3
		6	0,36	0,57	60,46	128,15	1644,0	2114,0	2574,0
		12	0,45	0,68	18,24	64,185	1476,0	1985,1	2454,0
		24	0,38	0,54	14,32	62,346	1361,41	1786,0	2293,0

Preliminary modification of the surface of the mineral filler marble powder with the solutions of new biosurfactants makes it possible to significantly lengthen the terms of the initial structure formation, followed by a rapid strength gain of the daily structure of the cement stone. By choosing the concentration of the introduced complex additives, it is possible to regulate both the setting time and the strength of the cement stone, which will be widely used in the production of cement compositions and building materials.

Conclusion

The modern methods for separation, determining the surface activity and colloid-chemical properties, stabilizing abilities of new biosurfactants were used in this research work. The new biological surfactants were isolated by acid precipitation and purified by solvent extraction. The surface activity and foam forming ability of the obtained new biosurfactants in aqueous solutions were investigated. The effects of biosurfactants concentration and temperature of the system on the surface-activity and foam forming abilities of new biosurfactants were studied. It has been established that with an increase in the biosurfactants concentration and temperature of the system, the surface activity and foam forming abilities of new biosurfactants were increased. It has been shown that new biologic surface-active substances were effective regulators of the structure formation of cement dispersed systems. It has been established that the introduction of new biosurfactants in combination with marble powder enhances the plasticizing effect and makes it possible to replace part of the binder with marble powder without reducing the final strength of the cement stone. The use of modified marble powder with aqueous solutions of new biosurfactants lengthened the stage of initial structure formation and accelerated the subsequent process of structure formation with the increase in the daily strength of the cement stones. By choosing the concentration of added biosurfactant additives, it has been possible to regulate the strength of cement stone, which will find application in the production of cement-concrete compositions and building materials.

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