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Stability study of emulsions based on modified xanthan gum

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ABSTRACT
Modified xanthan gum is often used in a variety of industries such as pharmaceuticals, the food industry, and the oil industry. The use of modified xanthan gum in emulsions can solve two problems, stabilize and thicken. This work is dedicated to the study of oil-in-water emulsion stability. The continuous phase of the emulsion consists of aqueous solutions of modified xanthan gum (XG-g-MMA), alkyl polyglycoside (APG), and silicon dioxide nanoparticles (SiO_2), and kerosene was used as a dispersed phase. Modified XG was prepared by grafting with methyl methacrylate to improve its properties. To determine the stability, the stratification time was monitored, and rheological properties and droplet sizes of emulsions were determined. The results showed that the emulsions based on XG-g-MMA and APG have better properties than the emulsions based on XG and sodium lauryl ethoxy-sulphate (SLES). Emulsions with 0.3% XG-g-MMA, 5% APG and 0.2% SiO2 demonstrated stability for up to 2 years, whereas emulsions with 0.3% XG stay stable for up to 1 year.

Keywords: emulsion stability, modified xanthan gum, grafted polymer.

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Introduction

Many building materials, paints, lubricants, etc. are being emulsions. In the cosmetic industry, all creams and lotions are W/O, O/W and W/O/W emulsions, which makes it possible to select creams depending on skin type. There are new medical preparations based on emulsions with functional, useful components [[1], [2]].

However, emulsions are thermodynamically unstable systems, undergo coalescence, gravitational separation, flocculation, form water and oil phases, so a stabilizers are required for their long-term stability. Basically, synthetic surfactants are used to stabilize emulsions, but synthetic surfactants are considered aggressive and limit the scope of use of emulsions. In this regard, biological surfactants are proposed which are safe and biodegredable. An example of biosurfactant is alkyl polyglycoside (APG). APG is a nonionic biosurfactant, which is a derivative of glucose and fatty acids [3]. The raw materials for obtaining these surfactants are starch, glucose and fatty alcohols. These products are derived from corn and other grains, therefore considered renewable. The final product of the reaction using acid catalysts is a mixture of compounds containing a hydrophilic sucrose part and a hydrophobic alkyl part [[4], [5]]. APG is used as a foaming agent in detergents, replaces surfactants such as lauryl sulfate, lauryl ethoxysulfate, and has a milder effect on the skin [6]. Biopolymers are often used as stabilizers which are less toxic and biodegradable [[7], [8], [9]]. One of the biopolymers, xanthan gum, is an extracellular polysaccharide of the bacterium Xanthomonas Campestris [10]. Xanthan is widely used in medicine, cooking, construction and other areas, but there are many works proving the great effectiveness of Modified xanthan [[11], [12], [13], [14], [15], [16], [17], [18],]19]]. After modification xanthan gum demonstrates better properties as viscosity, solubility and stability [[20], [21], [22], [23]].

In this work modified xanthan was used to improve the stability of the emulsion. The ratios of the ingredients are shown in Table 1.

Experimental part

Materials

Biopolymer Xanthan gum (XG) as a powder (Sigma-Aldrich Chemical Co., USA), alkylpolyglucoside (APG) as a 50% solution (Spec Chem Industry Inc., China), sodium lauryl ethoxysulfate (SLES) – 70% solution (OMED, China), silicon dioxide nanoparticles (40 nm) as a 40% solution (Sigma-Aldrich Chemical Co., USA) and kerosene (Kloriant, Kazakhstan) were used. Modified xanthan gum was produced in a laboratory.

Emulsion preparation

Aqueous sol solution surfactant, polymer, and silicon dioxide were mixed and distilled water was added till the required volume. The solution was placed on a magnetic stirrer and kerosene was added while stirring. After adding kerosene, the solution was stirred for 15 minutes at a speed of 700 rpm. Prepared emulsion immediately was poured into a measuring cylinder and observed for phase separation over time. Thus, emulsions were prepared with different ratios of components to determine the best concentration of each of the components.

Measurements

The stability of the emulsions was determined by visual observation, the time of the kerosene phase appearance (the formation of a layer of kerosene on the surface of the emulsion) was noted over time. To determine the effect of salinity on stability, sodium chloride solution was added to the emulsion. The content of sodium chloride in the emulsions was 5, 15, and 30 g/L.

The samples that showed the highest stability were analyzed on a Bohlin rheometer, Rheolab QC to study the rheological properties. Rheological tests (viscosity and shear stress versus shear rate) were carried out at 30, 60, and 80°C.

The sizes of the dispersed phase droplets were measured on a scanning electron microscope Auriga Crossbeam 540 Carl Zeiss. To obtain images before analysis, the samples were applied to a film and dried at room temperature. In this way, photographs of capsules of volatilized kerosene were obtained.

Table 1 - Content of emulsions

Sample №	XG, %	XG-g-MMA, %	SiO_2, %	APG, %	SDS, %	Distilled water, ml	Kerosene, ml	Salinity, g/l
1	0.1		**2**	5		7	3	
2	0.1		**1**	5		7	3	
3	**0.1**		**0.2**	5		7	3	
4	0.1		**0.1**	5		7	3	
5	0.1		**0**	5		7	3	
6	0.2		**2**	5		7	3	
7	0.2		**1**	5		7	3	
8	**0.2**		**0.2**	5		7	3	
9	0.2		**0.1**	5		7	3	
10	0.2		**0**	5		7	3	
11	**0.3**		0.2	5		7	3	
12	0.2		0.2	**2**		7	3	
13	0.2		0.2	**3**		7	3	
14	0.2		0.2		**3**	7	3	
15	0.2		0.2		**5**	7	3	
16		**0.2**	0.2	5		7	3	
17		**0.3**	0.2	5		**7**	**3**	
18		0.3	0.2	5		**9**	**1**	
19		0.3	0.2	5		**6**	**4**	
20		0.3	0.2	5		7	3	**5**
21		0.3	0.2	5		7	3	**15**
22		0.3	0.2	5		7	3	**30**

Results and discussions

Emulsion stability

Observations have shown that the stability of the emulsion depends on the amount of each component. Emulsions prepared with 5% APG were more stable than with SLES (the same ratio). Also, emulsions with 0.3% XG-g-MMA stayed stable for more than 2 years, while emulsions with XG with the same amount stayed stable for about 1 year. However, with an increase in the polymer content, the emulsion became a gel-like state. The nanoparticles showed stabilizing effect at a concentration of 0.2%.

Also, the statement proved that with an increase in the ratio of the oil phase, the stability of an emulsion increases, so with the increase of the kerosene volume increased the stability of the emulsion. Salinity stability testing showed that emulsions with XG-g-MMA stay stable at salinities up to 30 g/l, while XG emulsions break down already at 5 g/l salinity. The visual observations are shown in Table 2.

Table 2 - Results of emulsion stability by visual control

Sample №	Stability
1	Not stable
2	Not stable
3	Not stable
4	Not stable
5	Not stable
6	Not stable
7	Not stable
8	Stable for 1 month
9	Not stable
10	Not stable
11	Stable for over 1 year
12	Not stable
13	Not stable
14	Not stable
15	Not stable
16	Not stable
17	Stable for over 2 years
18	Not stable
19	Stable for over 2 year
20	Stable for over 1 year
21	Stable for over 1 year
22	Stable for over 1 year

Rheology properties

To determine the rheological properties, the most stable emulsions with the composition XG(0.3)-APG(5)-SiO2(0.2) and XG-g-MMA(0.3)-APG(5)-SiO_2(0.2) were chosen. The results of viscosity and shear stress measuring showed that at 30°C with a change of shear rate from 1 to 100 s^{-1}, the shear stress of the XG-g-MMA(0.3)-APG(5)-SiO_2(0.2) emulsion reaches 16 Pa, XG(0.3)-APG(5)-SiO_2(0.2) reaches 9 Pa. And the viscosities of XG-g-MMA(0.3)-APG(5)-SiO_2(0.2) and XG(0.3)-APG(5)-SiO_2(0.2) decrease to 0.16 Pa·s and 0.09 Pa·s respectively.

The rheology performance decreases with the rise of temperature, which can be seen in Diagrams 1, 2, and 3. At 60°C the shear stress of the XG-g-MMA(0.3)-APG(5)-SiO_2(0.2) emulsion reaches 12.5 Pa, XG(0.3)-APG(5)-SiO_2(0.2) reaches 7 Pa. The viscosity of both emulsions changes slightly, showing 0.13 Pa s and 0.07 Pa·s. At 80 ºC the maximum values drop to 9.5 Pa for XG-g-MMA(0.3)-APG(5)-SiO_2(0.2) and to 4.7 Pa for XG(0.3)-APG(5)-SiO_2(0.2).

Thus, it can be noted that emulsions with XG-g-MMA(0.3)-APG(5)-SiO_2(0.2) showed better results compared to XG(0.3)-APG(5)-SiO_2(0.2).

Emulsions prepared using modified xanthan have demonstrated resistance to salinity. With the addition of salt up to 30 g/l the rheological parameters of the XG-g-MMA(0.3)-APG(5)-SiO_2(0.2) emulsions did not differ much (Diagram 4), showing 14-16 Pa.

Drop sizes

SEM images showed that the kerosene capsules emulsified with XG had diameters ranging from 2 to 45 µm, with an average diameter of ~20 µm (Figure 1). Kerosene capsules emulsified with XG-g-MMA had diameters ranging from 2 to 20 µm, with an average diameter of ~10 µm (Figure 2). Thus, it can be understood that the stability of the emulsion with XG-g-MMA is due to the uniform formation of smaller capsules compared to the emulsion with XG.

Traces of nanoparticles can be seen on the walls of the capsules which also have a stabilizing effect, forming a layer at the oil-water interface (Figure 3).

Diagram 1 - Rheological properties at 30ºC

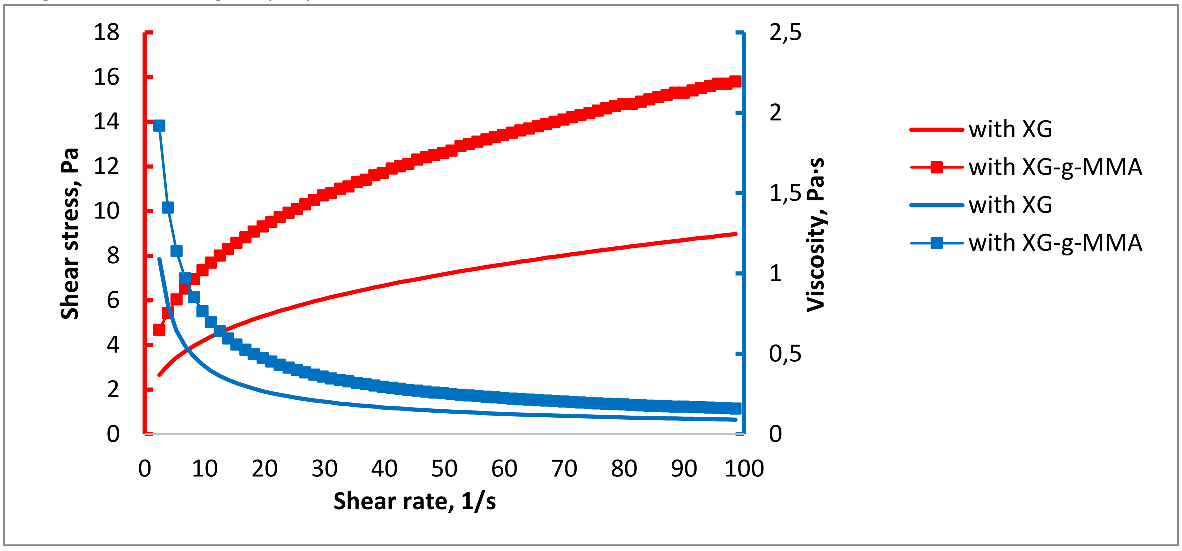


Diagram 2 - Rheological properties at 60ºC

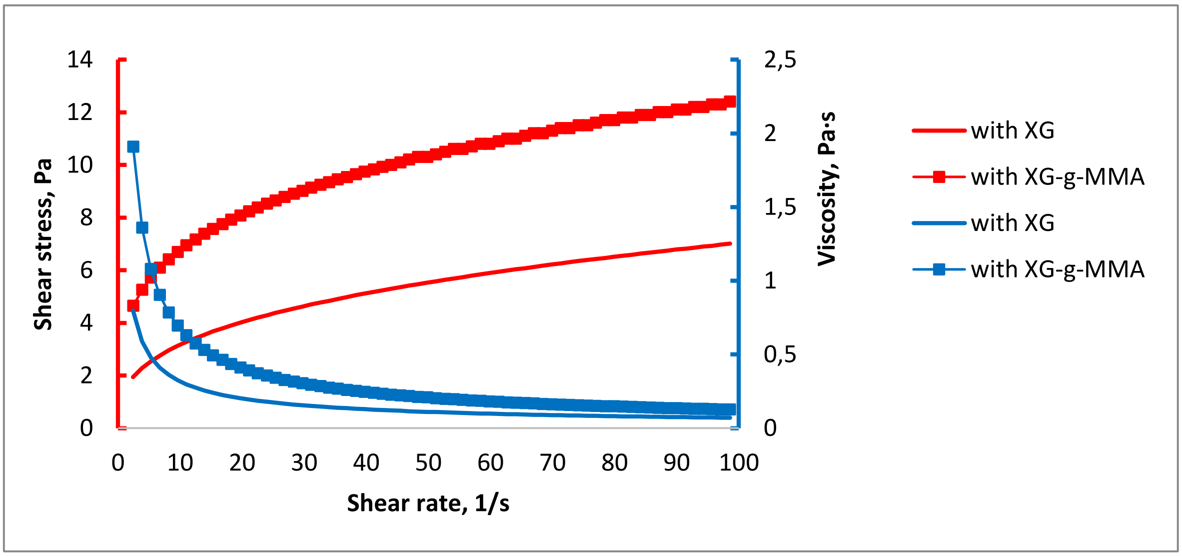


Diagram 3 - Rheological properties at 80ºC

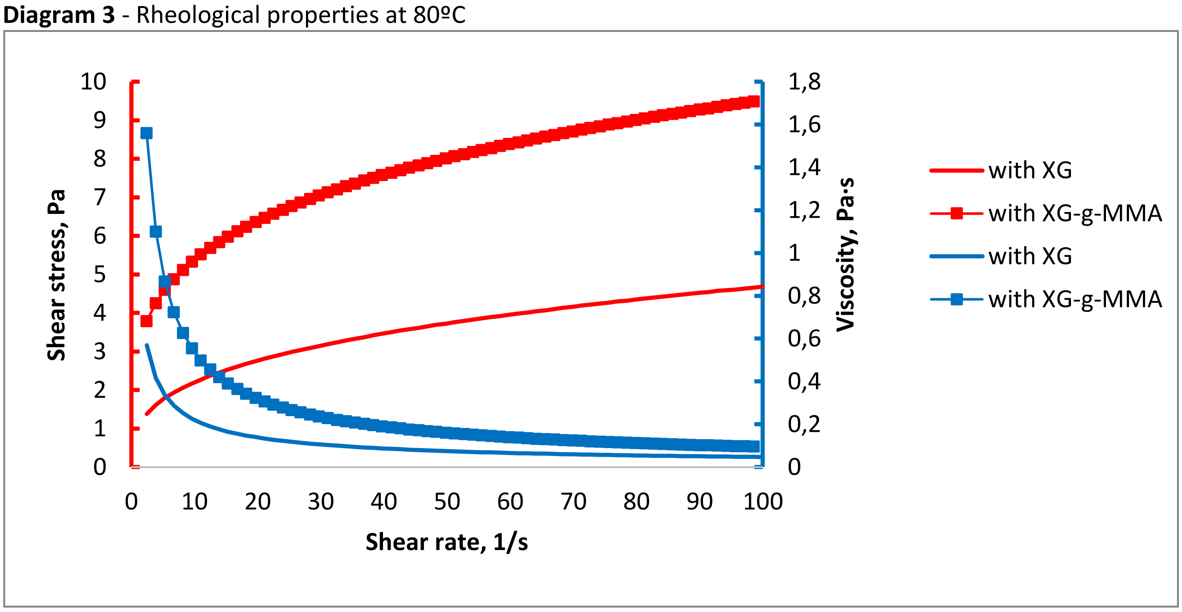
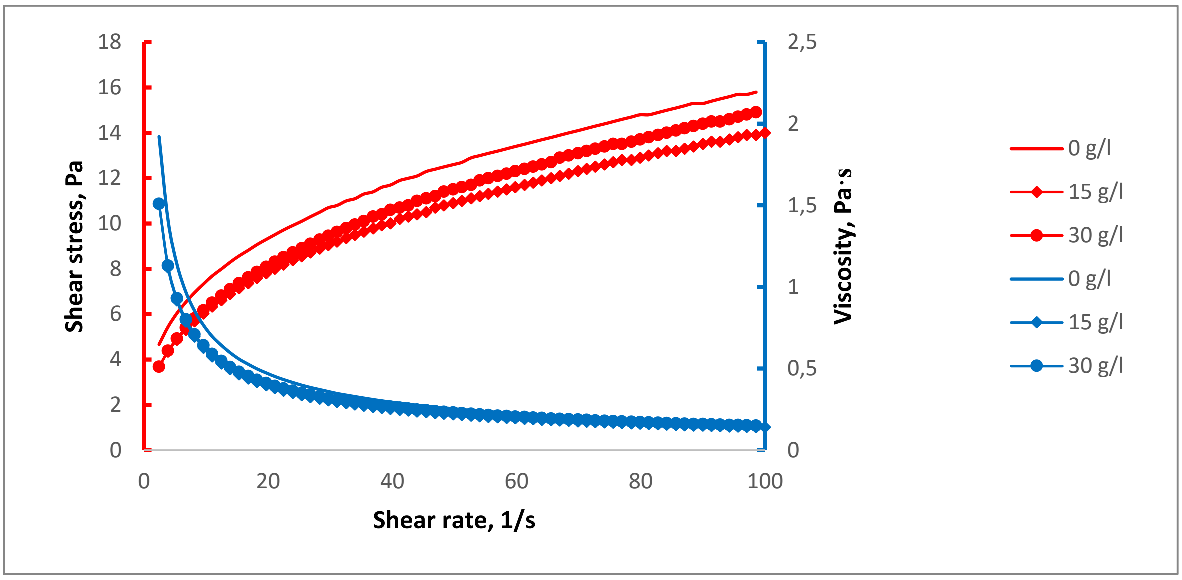


Diagram 4 - The dependence of rheology of emulsions on salinity at 30ºC



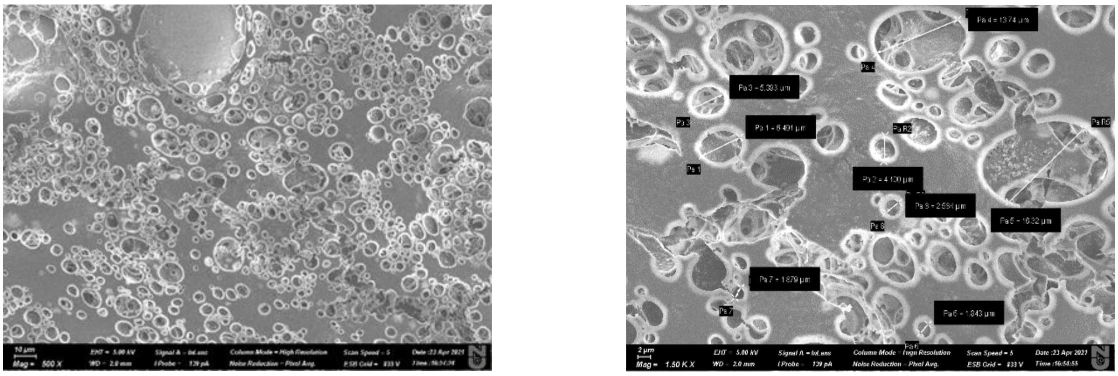


Figure 1 - Capsules of kerosene in emulsion XG(0.3)-APG(5)-SiO_2(0.2)

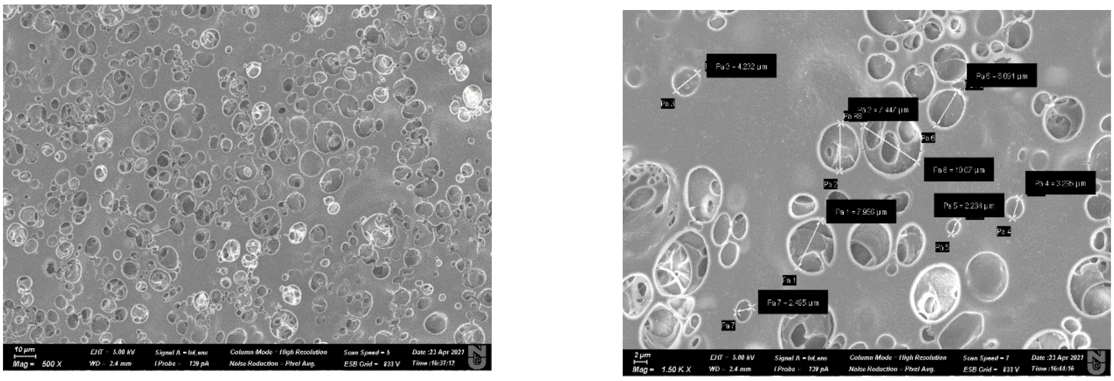


Figure 2 - Capsules of kerosene in emulsion XG-g-MMA(0.3)-APG(5)-SiO_2(0.2)

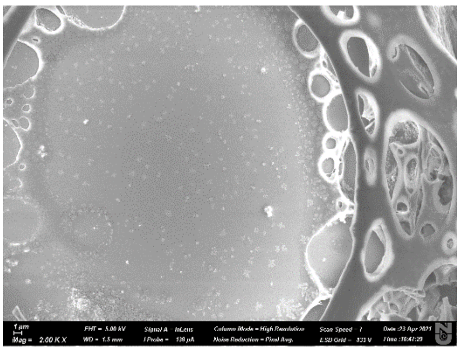


Figure 3 - Nanoparticles of SiO_2 at oil/water interface

Conclusions

The results of the stability study showed that the emulsion prepared with the addition of modified xanthan gum (XG-g-MMA(0.3)-APG(5)-SiO_2(0.2)) remains stable for more than 2 years, while the emulsion with the addition of unmodified xanthan gum (XG(0.3)-APG(5)-SiO_2(0.2)) remains stable for more than 1 year. When the alkylpolyglycoside is replaced with sodium ethoxysulfate, the stability drops markedly.

According to the results of the study of rheological properties, it was found that the emulsion with the addition of XG-g-MMA has a shear stress value of 16 Pa at 30ºC, 12.5 Pa at 60ºC and 9.5 Pa at 80ºC, while the emulsion with the addition of XG reaches 1.8 times less value - 9 Pa at 30ºC, 7 Pa at 60ºC and 4.7 Pa at 80ºC. Also, the emulsion with the addition of modified xanthan gum shows resistance to salinity up to 30 g/l, while the emulsion with the addition of xanthan gum breaks down at a salinity of 5 g/l.

Thus, the use of an emulsion with modified xanthan gum XG-g-MMA(0.3)-APG(5)-SiO_2(0.2) can become one of the effective methods used in enhanced oil recovery or other industries.

Conflict of interest. On behalf of all authors, the correspondent author declares that there is no conflict of interest.

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Модификацияланған ксантан шайыры негізіндегі эмульсиялардың тұрақтылығын зерттеу

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ТҮЙІНДЕМЕ
Модификацияланған ксантан шайыры фармацевтика, тамақ және мұнай өнеркәсібі сияқты әртүрлі салаларда жиі қолданылады. Модификацияланған ксантан шайырын эмульсияларда қолдану, тұрақтандыру және қоюландыру сияқты екі мәселені шешуі мүмкін. Бұл жұмыс мұнайдың судағы эмульсиясының тұрақтылығын зерттеуге арналған. Эмульсияның үздіксіз фазасы модификацияланған ксантан шайырының (XG-g-MMA), алкилполигликозидтің (APG) және кремний диоксиді нанобөлшектерінің (SiO_2) сулы ерітінділерінен тұрады, ал дисперсті фаза ретінде керосин қолданылған. Модификацияланған XG, оның қасиеттерін жақсарту үшін метилметакрилатты егу арқылы алынды. Тұрақтылықты анықтау үшін қатпарлану уақыты бақыланды, эмульсия тамшыларының реологиялық қасиеттері мен өлшемдері анықталды. Нәтижелер XG-g-MMA және APG негізіндегі эмульсиялардың XG және натрий лаурил этоксисульфаты (SLES) негізіндегі эмульсияларға қарағанда жақсы қасиеттерге ие болатынын көрсетті. Құрамындағы 0,3% XG-g-MMA, 5% APG және 0,2% SiO2 бар эмульсия 2 жылға дейін тұрақты болатынын көрсетті, ал құрамындағы 0,3% XG бар эмульсиялар тек 1 жылға дейін тұрақты болып қалды.

Түйін сөздер: эмульсия тұрақтылығы, модификацияланған ксантан сағызы, егілген полимер.

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Исследование стабильности эмульсий на основе модифицированной ксантановой камеди

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АННОТАЦИЯ

Модифицированная ксантановая камедь часто используется в различных отраслях промышленности, таких как фармацевтика, пищевая промышленность и нефтяная промышленность. Использование модифицированной ксантановой камеди в эмульсиях может решить две проблемы: стабилизировать и загустить. Данная работа посвящена исследованию стабильности эмульсии нефти-в-воде. Непрерывная фаза эмульсии состоит из водных растворов модифицированной ксантановой камеди (XG-g-MMA), алкилполигликозида (APG) и наночастиц диоксида кремния (SiO_2), а в качестве дисперсной фазы использован керосин. Модифицированная XG была получена путем прививки метилметакрилата для улучшения его свойств. Для определения стабильности контролировали время расслоения, определяли реологические свойства и размеры капель эмульсий. Результаты показали, что эмульсии на основе XG-g-MMA и APG обладают лучшими свойствами, чем эмульсии на основе XG и лаурилэтоксисульфата натрия (SLES). Эмульсия с 0,3 % XG-g-MMA, 5 % APG и 0,2 % SiO2 показала стабильность до 2 лет, тогда как эмульсии с 0,3 % XG остаются стабильными до 1 года.

Ключевые слова: стабильность эмульсии, модифицированная ксантановая камедь, привитый полимер.

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