THE STUDY OF THE PROPERTIES OF EMULSIONS BASED ON SEPIPLUS 400

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Key words: emulsion; emulsifier; rheological properties; structural viscosity

Emulsifying properties of Sepiplus 400 and emulsions on its basis obtained by the method of cold emulsification have been studied. It has been found that the given emulsifier being in the concentration of 1-5% without adding co-emulsifiers and structure-forming agents allows to obtain stable emulsions within the range of the oil phase concentrations of 10-40%. Organoleptic, physicochemical, technological, structural and mechanical properties of the experimental samples have been investigated; they prove their belonging to the structured systems with satisfactory thixotropic properties, sensory characteristics and spread capacity. It has been proven that the mass fraction of the emulsifier, as well as the mass fraction of the oil contribute to the increased viscosity index of the samples examined. The results of the studies conducted are the evidence of the prospectivity of Sepiplus 400 in development of medicinal and cosmetic emulsions of various kinds of actions.

Currently polyanionic surface-active agents are widely used in manufacture of medicinal and cosmetic products. They are especially interesting as effective emulsion stabilizers [4, 9, 10].

One of the methods of emulsion stabilization is the use of micelles of block copolymers colloidal dispersed in oil and having high emulsifying properties. Colloidal particles of the micelles accumulate at the oil-water interface, form mechanically firm structured films and thus stabilize the emulsion. The composition and the structure of such films are diverse and depend on the chemical structure of a stabilizer-emulsifier [6, 7].

One of the new promising emulsifiers for production of medicinal and cosmetic emulsions is the substance with a commercial name of Sepiplus 400 (Sepiplus™ 400, Seppic, France) and international unlicensed name of Polyacrylate – Polyisobutene – Polysorbate-20 (INCI).

Sepiplus 400 is a liquid polyanionic surface-active agent, which does not require any preliminary operations for its preparation: heating, neutralization, dispersion, etc. This is a ready-made emulsifying mixture, which is an invert emulsion where polymer chains are tightly convoluted in the internal aqueous phase. The mechanism of the emulsifying action of Sepiplus 400 is explained by the theory of “unwrapping drops”. The essence of this phenomenon consists in the phase inversion during which the polymer chains of Sepiplus 400 are unwrapping into the external aqueous phase, generating a stabilizing gel structure [5, 8].

The aim of our work was to study the emulsifying properties of Sepiplus 400, prepare the experimental samples of emulsions on its basis and study the organoleptic, structural and mechanical, as well as technological properties of the emulsions obtained.

Materials and Methods

The sets of experimental samples with different consistency were prepared by the method of cold emulsification with mineral and vegetable (corn) oil, their concentration varied between 5 and 50%. Sepiplus 400 was introduced as a monoemulsifier within the range of concentrations of 0.5-5% recommended by the manufacturer [2, 11]. The finished emulsions were tested by the following characteristics: appearance, colloidal and thermal stability, pH value, type of emulsion, some rheological properties, sensory characteristics (the rate of absorption, subjective sensations, stickiness, the absence of white traces when applying).

Colloidal and thermal stability were determined in accordance with the methodology of GOST “Cosmetic creams” [3]. The pH values of the experimental samples were determined potentiometrically in 10% water extraction of the cream with the help of a pH 150 MI pH-meter (Russia) according to the I-st ed., p.2.2.3. of the SPhU. Rheological studies were conducted with a BROOKFIELD HB DV-II PRO viscosimeter (USA) within the range of the shear rate from 0.1 sec\(^{-1}\) to 150 sec\(^{-1}\) (SC4-21 spindle for a chamber of 8.3 ml volume) at the temperature of 20 and 34ºC. The type of emulsion was determined by the method of dilutions according to the SPhU [1].

Results and Discussion

The studies conducted allowed to select thermostable and collooidally stable formulations, which were oil-in-water emulsions of different consistency – from...
The composition of the model samples of emulsions

<table>
<thead>
<tr>
<th>No. of the sample</th>
<th>1</th>
<th>14</th>
<th>15</th>
<th>23</th>
<th>25</th>
<th>31</th>
<th>34</th>
<th>54</th>
<th>55</th>
<th>64</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mineral oil, %</td>
<td>0</td>
<td>–</td>
<td>10</td>
<td>15</td>
<td>–</td>
<td>15</td>
<td>20</td>
<td>–</td>
<td>30</td>
<td>–</td>
</tr>
<tr>
<td>Corn oil, %</td>
<td>–</td>
<td>10</td>
<td>–</td>
<td>–</td>
<td>15</td>
<td>–</td>
<td>–</td>
<td>30</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Sepiplus 400, %</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
<td>1</td>
<td>2</td>
<td>5</td>
<td>1.5</td>
<td>1.5</td>
<td>2</td>
<td>1.5</td>
</tr>
<tr>
<td>Purified water</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Colloidal stability at 6000 rpm</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>Colloidal stability at 8000 rpm</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>Thermal stability</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>pH</td>
<td>6.18</td>
<td>6.20</td>
<td>6.18</td>
<td>6.12</td>
<td>6.25</td>
<td>6.5</td>
<td>6.22</td>
<td>6.21</td>
<td>6.38</td>
<td>6.25</td>
</tr>
<tr>
<td>Structural viscosity (mPa·s) at 20 rpm</td>
<td>3700</td>
<td>3420</td>
<td>3300</td>
<td>4700</td>
<td>7000</td>
<td>9100</td>
<td>4480</td>
<td>5680</td>
<td>6000</td>
<td>7940</td>
</tr>
</tbody>
</table>

Note: «+» – the emulsion is stable; «–» – the emulsion is unstable; n=5.

It was found that Sepiplus 400 was effective as a monoemulsifier in the concentration of 1.5-5%, and it allowed to obtain stable emulsions.

The rheological studies conducted showed the increase in viscosity of the samples when increasing the emulsifier concentration (Fig. 1). Thus, Samples No. 23, 25 had the viscosity of 4700 and 7000 mPa·s (at 20 rpm and 20°C) with the concentration of the emulsifier of 1.5%, 2%; when increasing the emulsifier concentration up to 5% the viscosity considerably increased in Sample No. 31 and it was 9100 mPa·s.

When Sepiplus with the concentration of less than 1.5% was introduced into formulation 5 and 10% emulsions, destruction of the model samples occurred. It is explained most probably by the lack of polymer both for binding the particles of the internal phase and for creating colloidal protection by macromolecules.

At the same time Sample No. 31, which showed a positive test result for colloidal and thermal stability, decayed during the rheological studies breaking a general tendency of correlation of viscosity and the emulsifier quantity. In our opinion, it is connected with disturbance of the phase inversion behaviour as for this very process the dispersion medium–internal phase relation...
ship is important. It is known that there is a limiting (critical) relationship of volumes for each stabilizer in a certain concentration, exceeding of which results in formation of the invert emulsion. And the latter one is often unstable due to the low activity of the emulsifier in the changed conditions [9, 10].

Increase of the mass fraction of the oil phase also resulted in considerable increase of viscosity indices. Thus, the samples with the similar Sepiplus 400 content (1.5%), but with a different oil content (10, 20, 30 and 40%) showed the rising indices of viscosity: 3420 mPa·s, 4480 mPa·s, 5680 mPa·s and 7940 mPa·s, respectively (see Fig. 2). It should be also noted that corn oil slightly changed the viscosity of the bases obtained compared to the formulations with the similar content of liquid petrolatum. The sample without the oil showed a higher index of viscosity of 3700 mPa·s.

In order to estimate the viscous and plastic properties of model formulations their rheological parameters were determined. Based on the data obtained the complete rheograms of the model samples behaviour were plotted in such coordinates as the shear rate – shear stress (Fig. 3).

The rheograms built have a type of behaviour, which is typical for plastic systems, and a yield strength, which is within the following range of the shear stress: 14.7 Pa (Sample No.14) to 150.2 Pa (Sample No.64). The presence of the hysteresis loop in the rheograms confirms the presence of thixotropic properties of the samples studied.

The next step of our work was the study of the correlation between the structural viscosity of the samples studied and the shear rate (Fig. 4).

The result of the study showed a gradual decrease of the indices of the structural viscosity as the shear rate increased. The 58-96 s\(^{-1}\) range of the shear rate is notable for practically linear dependence of the structural viscosity on the shear rate, and it confirms the ability of emulsion systems to regenerate gradually after deformation.

CONCLUSIONS

The model samples of the emulsions with different consistency based on Polyacrylate – Polysobutene – Polysorbate-20 have been developed.

It has been found that the given emulsifier being in the concentration of 1-5% without adding co-emulsifiers and structure-forming agents allows to obtain stable emulsions within the range of the oil phase concentrations of 10-40%.

Organoleptic, physicochemical, technological, structural and mechanical properties of the experimental samples have been investigated; they prove their belonging to the structured systems with satisfactory thixotropic properties, sensory characteristics and spread capacity.

It has been proven that the mass fraction of the emulsifier, as well as the mass fraction of the oil contribute to the increased viscosity index of the samples examined.

The results of the studies conducted are the evidence of the prospectivity of Sepiplus 400 in development of medicinal and cosmetic emulsions of various kinds of actions.

REFERENCES


ВИВЧЕННЯ ВЛАСТИВОСТЕЙ ЕМУЛЬСІЙ НА ОСНОВІ СЕПІПЛЮС 400
Т.М.Ковальова, Н.П.Половко

Ключові слова: емульсія; емульгатор; реологічні властивості; структурна в’язкість
Досліджені емульгуючі властивості Сепіплюс 400 та емульсії на його основі, одержані методом холодного емульгування. Встановлено, що даний емульгатор у концентрації 1-5% без додавання співемульгаторів та структуроутворювачів дозволяє отримати стабільні емульсії в діапазоні концентрації масляної фази 10-40%. Вивчені органолептичні, фізико-хімічні, структурно-механічні властивості експериментальних зразків, які підтверджують їх принадлежність до структурованих систем з задовільними тиксотропними властивостями, сенсорними характеристиками, намазуванням. Доведено, що масова частка емульгатора та масова частка масла сприяють підвищенню показника тиксотропності досліджуваних зразків. Результати проведених досліджень свідчать про перспективність Сепіплюс 400 для розробки лікарських та косметичних емульсій різного спрямування.

ИЗУЧЕНИЕ СВОЙСТВ ЭМУЛЬСИЙ НА ОСНОВЕ СЕПИПЛЮС 400
Т.Н.Ковалева, Н.П.Половко

Ключевые слова: эмульсия; эмульгатор; реологические свойства; структурная вязкость
Исследованы эмульгирующие свойства Сепиплюс 400 и эмульсии на его основе, полученные методом холодного эмульгирования. Установлено, что данный эмульгатор в концентрации 1-5% без добавления соэмульгаторов и структурообразователей позволяет получить стабильные эмульсии в диапазоне концентраций масляной фазы 10-40%. Изучены органолептические, физико-химические, структурно-механические свойства экспериментальных образцов, подтверждающие их принадлежность к структурированным системам с удовлетворительными тиксотропными свойствами, сенсорными характеристиками, намазываемостью. Доказано, что массовая доля эмульгатора и массовая доля масла способствуют повышению показателя вязкости исследуемых образцов. Результаты проведенных исследований свидетельствуют о перспективности Сепиплюс 400 в разработке лекарственных и косметических эмульсий различной направленности действия.