PHARMACIA

Pharmacia 66(1): 19–25
DOI 10.3897/pharmacia.66.e35022

Research Article

Justification of technological parameters of the cream production with sapropel extract

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Received 31 March 2019 ♦ **Accepted** 14 May 2019 ♦ **Published** 7 June 2019

Citation: Strus O, Polovko N, Yezerska O (2019) Justification of technological parameters of the cream production with sapropel extract. Pharmacia 66(1): 19–25. https://doi.org/10.3897/pharmacia.66.e35022

Abstract

The aim of the research was to substantiate the technological parameters of cream production with the sapropel extract and work out the technological scheme for its production. In this study, extract of sapropel from the Prybych deposit, emulsion base, containing corn oil, emulsifier No.1, cetylstearyl alcohol, nisin, euxyl K 100 and purified water were employed. To carry out the research, a set of methods to analyse colloidal and thermal stability was used. Rheological properties of the samples were determined. The method of microscopic analysis was carried out to analyse the stability of the emulsion system. It was proved experimentally that, when producing the cream, it is appropriate to use the phase inversion method, and the emulsification needs to be carried out at 5000 r/m for 20 min. Size and shape of the sample drops, obtained at 5000 r/m, were monodisperse and more uniform, most of which range from 2 to 3 microns that indicates the system stability. The following parameters of technological process were determined: mixing temperature conditions, speed of homogenisation and mixing time.

Keywords

sapropel extract, o/w emulsion cream, emulsification methods, technology

Introduction

The sapropel resources in Ukraine comprise approximately 74.5 million tonnes. There are 234 deposits in Volyn, Rivne, Sumy, Chernihiv and Kyiv regions. The sapropel resources in Volyn region comprise about 63 million tonnes that allows consideration of it as the largest raw material base of sapropel in Ukraine. Prybych deposit, located in Volyn region, occupies the area of 39 ha and yield 212 thousand tonnes of organic type sapropel (Leymunskii et al. 2007; Booth et al. 2007; Bevan and Smith 2011; Stankevica et al. 2012; Zander-Ukraine 2018).

The experimental research has shown in the sapropel composition, extracted from Prybych Lake, the presence of significant amounts of carboxylic acids, amino acids, micro- and macroelements, humic acids (Nikolov 2014; Strus 2015a; Strus 2015b). The technology of sapropel extract production using the cavitation method was improved. It provides better extraction of humic and fulvic acids, since their content exceeds 20% (Strus and Polovko 2016). The occurrence of anti-inflammatory, wound healing and antibacterial effects of the extract was demonstrated (Klöcking and Helbig 2005; Strus et al. 2014, 2015c).

The presence of large amounts of sapropel in Ukraine and the research results suggest that sapropel is a promising raw material for preparing effective remedies for application in medicine, pharmacy and veterinary medicine.

One of the most important factors that affect the quality of semi-solid preparations (SSP) and their activity is the mode of preparation. The technological process of emulsion SSP involves the following operations: heating, stirring, emulsification, homogenisation, cooling etc. (Mori et al. 2000; Ke-chun et al. 2004; ; Bouftira et al. 2008; Zhi-yu et al. 2012; Yurchenko et al. 2013; Kolesnikova and Polovko 2015; Cizauskaite et al. 2018). The order of stirring oil and water phases, mixing temperature, speed of homogenisation and mixing time are of significant importance. Generally, an o/w emulsion is prepared by dividing the oily phase completely into minute globules, surrounding each globule with an envelope of emulsifying agent and finally suspending the globules in the aqueous phase. Conversely, the w/o emulsion is prepared by dividing the aqueous phase completely into minute globules, surrounding each globule with an envelope of emulsifying agent and finally suspending the globules in the oily phase (Barkat et al. 2011).

The common methods used to obtain emulsions in pharmaceutical practice represent direct emulsification and phase inversion methods. In this method, the aqueous phase is first added to the oil phase, so as to form a w/o emulsion. At the inversion point, the addition of more water results in the inversion of emulsion which gives rise to an o/w emulsion (Barkat et al. 2011).

For obtaining an o/w emulsion by the direct emulsification method, the water and oil phases are prepared separately, then the dispersed phase is added gradually to the dispersion medium and emulsified to obtain an emulsion with an appropriate degree of dispersion. Applying the method, a permanent quantitative predominance of the dispersion medium is visualised, that provides the achievement of the required type of emulsion. The phase inversion method also allows receiving coarse dispersion emulsions. Employing the phase inversion method to obtain o/w emulsion, a heated aqueous phase is added in portions to the oil phase (Ruban 2008).

Temperature of the emulsification production affects the physical, chemical and rheological characteristics, stability and cost of SSP.

The aim of this research was to substantiate technological parameters for production of the cream with sapropel extract and the technological scheme for its manufacture.

Materials and methods

Materials

The objects of the research were experimental test samples of the cream with sapropel extract from Prybych deposit, Volyn region, Ukraine (Zander-Ukraine 2018). The sapropel extract was obtained by the method of cavitation (Strus and Polovko 2016). The concentration of the sapropel extract in the test samples was 10%, which was confirmed by the pharmacological research. (Klöcking and Helbig 2005; Strus et al. 2014, Strus 2015c). During our previous investigations of test samples of the cream with sapropel extract, there was worked out the o/w emulsion base,containing 15% corn oil, 6% emulgator no.1 (Lanette SX), 1% cetylstearyl alcohol (CSA), 0.01% nisin, 0.1% euxyl K 100 and purified water to 100 g (Strus et al. 2018, 2019).

Methods

Experimental test samples of emulsifying bases were obtained by different techniques (the direct method and the phase inversion method) with application of several speeds of homogenisation.

Basic principles of European Pharmacopoeia (EP) 8.0., 2013 (Monograph 04/2010:0132) and the State Standard of Ukraine (SSU) No. 4765:2007 "Cosmetic creams. General technical conditions," 2008 were considered during development of the cream composition with sapropel extract (European Pharmacopoeia 2002; State Standard of Ukraine 2008).

Organoleptic (appearance, odour, colour), physical stability and consumer properties of test samples were evaluated.

Organoleptic characteristics and physical stability study

Organoleptic characteristics of the tested samples (appearance, odour, colour and consumer properties of the base) and possible signs of physical instability (coalescence, phase separation, aggregation of particles and coagulation) were studied in accordance with the SSU No. 4765:2007 "Cosmetic creams. General technical conditions," 2008 (SSU 4765:2007).

Colloidal and thermal stability

For determination of colloidal stability, the laboratory centrifuge ("Mechanìka precyzyjna," Poland) was applied. The test tubes were filled with 2/3 volume (approximately 9 g) by investigated test samples and weighed with an accuracy of 0.01 g. The samples then were centrifuged for 5 min at a speed of 5000 rpm (the relative centrifugal force was approximately 5,000 g) (SSU 4765:2007).

For determination of the thermal stability, 6 glass tubes with a diameter of 15 mm and a height of 150 mm were used, filled with 2/3 the volume of the subjected samples and placed in the thermostat TC-80M-2 at 40–42 °C for 24 h. If the formation of an aqueous phase was not observed in any glass tube, the bases were considered as stable (SSU 4765:2007).

Rheological study

The determination of the structural and mechanical (rheological) parameters was carried out on the rotating viscometer «Rheolab QC» («Anton Paar», Austria) with

coaxial cylinders CC27/S-SN29766. The measurement device allows the determination of the shear stress (τ) in the range of 0.5–3.0/104 Pa, shear rate (Dr) - from 0.1 to 4000/s and viscosity (η) - from 1 to 106 Pas/s. The measurements were performed as follows: an accurately weighed portion of the sample (about 17.0 ± 0.5 g) was placed in a container of the external fixed cylinder; the required temperature of the experiment was thermostatically controlled and set for 20 min. Using the software with the equipped devices, the following experimental conditions were set: shear rate from 0.1 to 350.0/s, the duration of measurement at each point of the curve (1 s) and the number of points (35) on the curve of the flow pattern (European Pharmacopoeia 2002; British Pharmacopoeia 2005; State Pharmacopoeia of Ukraine 2015).

Microscopic analysis

To determine the diameter of the dispersed-phase particles and their fractional composition, the laboratory microscope "Konus-Akademy" with an eyepiece camera SpoTek DCM510 was applied. ScopePhoto Software ™ was used for visualisation of the images. Approximately 0.02 g of cream was placed on the glass and size of the drops, with appropriate resolution, was determined.

Preparation of the samples

Experimental cream samples with SE were prepared by both the direct method and phase inversion methods. Using the direct method, emulsifiers (emulsifier no.1, CSA) were melted with corn oil at a temperature of 70 ± 5 °C, taking into account their melting temperatures. Separately, the aqueous phase (water, SE, preservatives) was heated to the same temperature of 70 ± 5 °C. An oily phase was added to the aqueous phase. The mixture was emulsified using the homogeniser Polytron PT 3100 D ("Kinematica AG," Switzerland) for 10 minutes at speeds of 2000, 3000, 5000 and 10000 r/m. Stirring was continued until the emulsion cooled to room temperature.

To obtain emulsions by the phase inversion method, the aqueous phase (water, SE, preservatives) was heated to a temperature of 70 ± 5 °C and was added to the oil phase (emulsifier no.1, CSA, corn oil) in the amount of 10%. The mixture was emulsified using the homogeniser Polytron PT 3100 D ("Kinematica AG," Switzerland) for 5 minutes at speeds of 2000, 3000, 5000 and 10000 r/m to receive an emulsion of w/o type. At a temperature of 70 ± 5 °C the rest of the aqueous phase at the same temperature was added, then emulsified for 5 minutes at the speeds of 2000, 3000, 5000 and 10000 r/m. There was a phase inversion, stirring was continued until the emulsion cooled to room temperature.

Results and discussion

During development of composition of the cream with sapropel extract, test samples were examined for organoleptic characteristics (appearance and consumer properties). The evaluation tests for colloidal and thermal stability and structural-mechanical parameters of the developed composition were performed. Their results are summarised in the Table 1.

The experimental cream samples, obtained at 10000 r/m, using the direct and phase inversion methods, had a creamy consistency, stable, but with the presence of a significant number of air bubbles inside that possibly in the future could lead to the phase separation of the emulsion and indicates the inappropriate use of a such mixing speed.

Samples, obtained at 3000 r/m and 5000 r/m, using the direct and phase inversion methods, according to the data of Table 1 are stable. The method of attaining them does not affect the parameters of colloidal and thermal stability and the viscosity indices of the studied samples.

The sample, obtained at 2000 r/m by the direct method, does not withstand the colloidal stability test and is stratified during storage.

To determine the optimal technology and the speed of emulsification, microscopic examinations of the cream samples using two methods were carried out. Cream samples obtained at 2000 and 10,000 r/m by the direct method were not investigated, since the first sample was unstable and the second had the presence of air bubbles inside.

Microscopic studies of the samples, obtained by the phase inversion method at 2000 and 3000 r/m, have shown that they have a different droplet sizes varying

Table 1. Characteristics of test samples of the cream with sapropel extract, n = 5.

Stirring speed / method	Organoleptic properties	Thermal stability	Colloidal stability	Structural viscosity (η), mPas/20 rev/min
2000 direct phase	Creamy consistency with signs of stratification during storage	stable	unstable	–
2000 phase inversion	Creamy consistency	stable	stable	5730 ± 40
3000 direct phase	Creamy consistency	stable	stable	6040 ± 10
3000 phase inversion	Creamy consistency	stable	stable	6700 ± 20
5000 direct phase	Creamy consistency	stable	stable	6100 ± 40
5000 phase inversion	Creamy consistency	stable	stable	6990 ± 30
10000 direct phase	Creamy consistency with the presence of air bubbles	stable	stable	5940 ± 50
10000 phase inversion	Creamy consistency with the presence of air bubbles	stable	stable	6150 ± 20

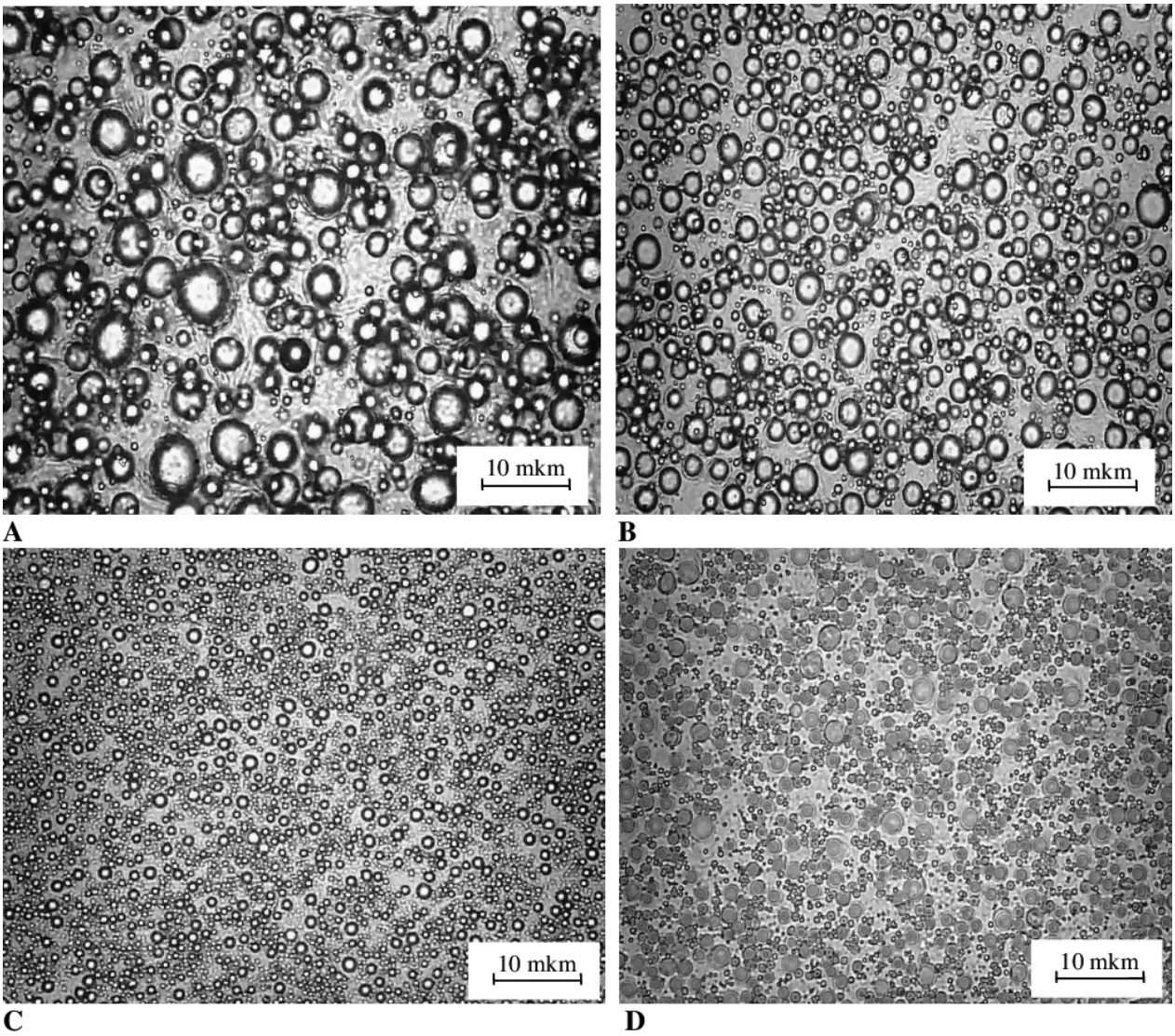

Figure 1. Microscopic photos of the samples of sapropel cream obtained by the phase inversion method depending upon the stirring speed: 2000 r/m (**A**), 3000 r/m (**B**), 5000 r/m (**C**), 10000 r/m (**D**).

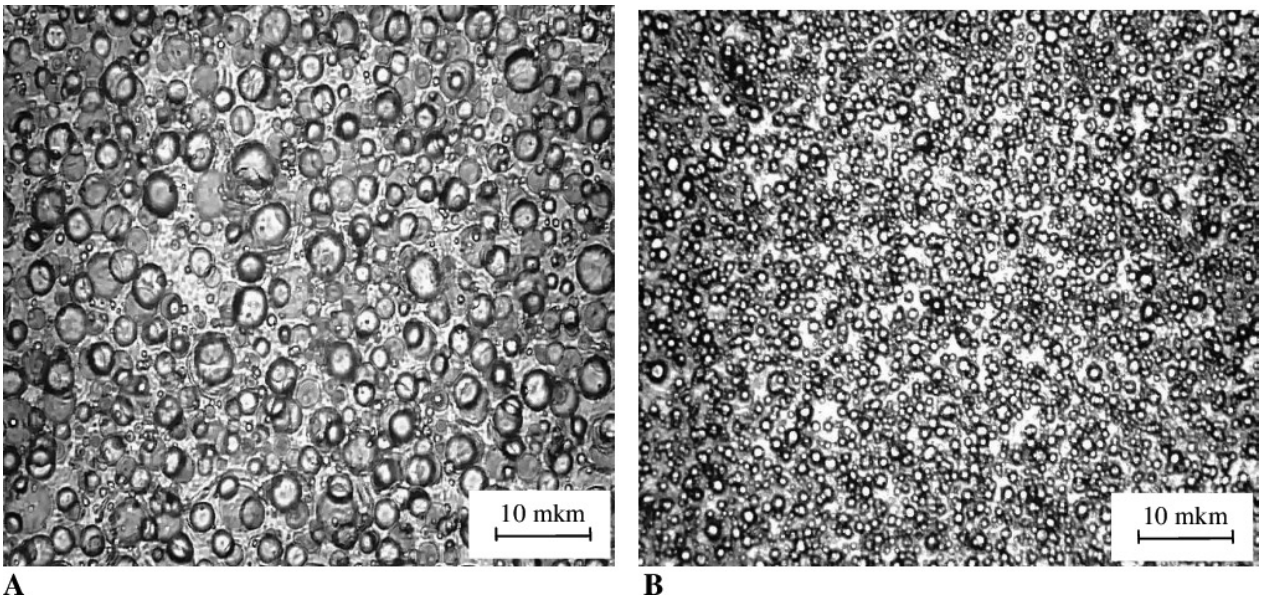

Figure 2. Microscopic photos of the samples of sapropel cream obtained by the direct method depending upon the stirring speed: 3000 r/m (**A**), 5000 r/m (**B**).

from 3 to 10 microns. This indicates a decrease in the degree of dispersion and indirectly displays an aggregative instability of the emulsion system that can lead to its phase separation during long-term storage. (Fig. 1A, B). The size and shape of the sample drops, obtained at 5000 r/m, were monodisperse and more uniform, most of which range from 2 to 3 microns, indicating the stability of the system (Fig. 1C).

The sample obtained at 10000 r/m by the phase inversion method had a significant number of air bubbles that possibly could lead to the phase separation of the emulsion (Fig. 1D).

Microscopic examinations of samples, obtained by the direct method at 3000 r/m, in comparison with the sample, obtained by phase inversion method, showed that they have a different droplet size from 3 microns to 10 microns, with the predominance of a phase that has a larger size of the emulsion phase dispersed particles. Despite the fact that the size and shape of droplets of the sample, obtained by the direct method at 5000 r/m, are more monodispersed, this method does not ensure the uniform distribution of the phase in the dispersion medium (Fig. 2A, B).

Thus, the results of microscopic studies of experimental cream samples indicate that the phase inversion method is more rational when preparing cream with SE. The research of structural-mechanical properties of the test samples obtained under different conditions of emulsification has shown that an increase in the homogenisation stirring speed from 2000 to 3000 r/m leads to an increase in viscosity, that also contributes to the emulsion stability. An increase in the stirring speed with a certain decrease of the cream viscosity is observed (Fig. 3)

Thus, the emulsification should be performed at 5000 r/m.

An important technological parameter is the mixing time, which has an influence on oil particles size of the dispersed phase, the consistency and stability of creams. The optimal oil dispersed phase of the emulsifying creams ranges from 1 to 2 microns.

Research on the dependence of the average droplet size of the cream oil phase on the time of emulsification (from 5 to 40 minutes) showed that the required dispersion of the oil phase (1–2 microns) is formed during emulsification for 20–25 minutes. Further emulsification does not produce a significant decrease in the diameter of the oil phase drops, however it leads to an increase in energy and product costs (Fig. 4).

Therefore, considering the results of the research on the cream production with the sapropel extract, we suggest, as the most appropriate, the following characteristics: the phase inversion method, optimal stirring speed 5000 r/m, mixing time 20 min.

The outcomes of the investigation were used for development of the scheme for pharmaceutical manufacturing process of the cream with sapropel extract, that is shown in Figure 5 as a schematic block diagram.

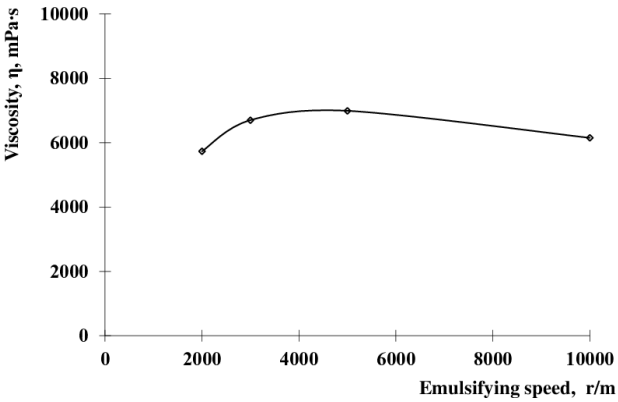

Figure 3. Dependence of a cream structural viscosity from stirring speed.

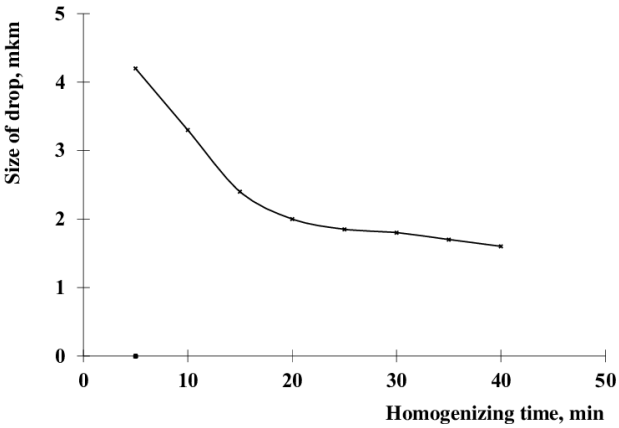

Figure 4. Dependence of the mixing time on the average size of drops of cream oil phase.

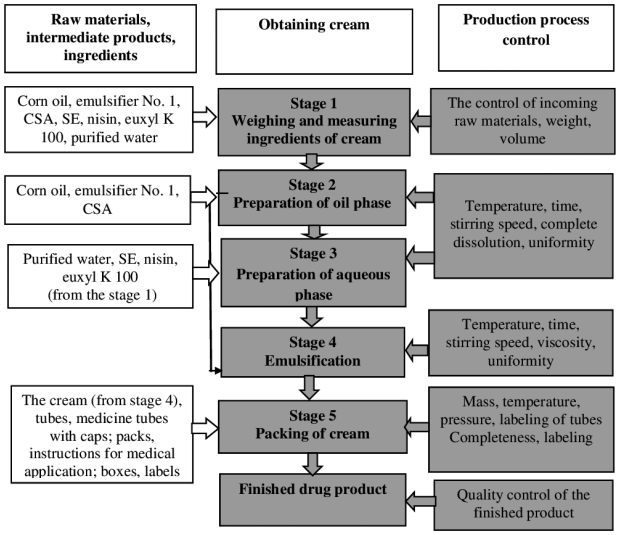

Figure 5. Schematic block-diagram for pharmaceutical manufacturing process of the sapropel cream production.

Conclusions

1. Considering significant natural resources of sapropel in Ukraine as a promising raw material for preparing effective medicines, sapropel was used as an active ingredient for the development of the cream composition.
2. The following parameters of technological process were determined: mixing temperature conditions, speed of homogenisation and mixing time. It was proved experimentally that, for production of the sapropel cream, it is appropriate to use the phase inversion method and homogenisation should be carried out at 5000 r/m for 20 min.
3. The technological process of the cream production includes the following stages: the preparation of aqueous and oil phases, emulsification and packaging.
4. The sequence of the technological stages, temperature and mixing time and speed of homogenisation all correspond to the technological requirements of the production of semi-solid preparations.

Acknowledgements

We are thankful to "Zander – Ukraine" LTD for providing free samples of sapropel.

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