RESEARCH ARTICLE

Experimental Reasoning for the Selection of the Foam-washing Agent Base Carrier at pH 3.3–4.8

I. I. Baranova[1], L. S. Petrovska[2], Yu. O. Bezpala[1], S. M. Kovalenko[1], O. S. Shpychak[3]

[1]Department of Commodity Science, National University of Pharmacy, Kharkiv, Ukraine, [2]Department of Cosmetology and Aromology, National University of Pharmacy, Kharkiv, Ukraine, [3]Department of Pharmaceutical Technology of Medicines named by D. P. Salo, National University of Pharmacy, Kharkiv, Ukraine

Abstract

Aim: the main purpose of our work was to experimentally substantiate and develop the base carrier for a low-level pH foam-washing agent. **Materials and Methods:** The determination of foaming ability was carried out by means of the Ross-Miles Foam Test apparatus as a standard device for measuring the foaming capacity of soaps and synthetic detergents. The structural and mechanical properties were determined using a Brookfield DV-II + PRO viscosimeter (USA) with a rotary adapter with a coaxial cylinder system. The pH value of the investigated foam samples was determined potentiometrically. **Results and Discussion:** Based on the carried out physical, chemical and technological research, the composition of the foam base was substantiated at an acidic pH value: disodium laureth sulfosuccinate - 20.0%, cocamidopropyl betaine - 6.0%, cocoglycoside glyceryl oleate - 1.0%, PEG-7 glyceryl cocoa - 0, 5%, PEG-150 polyglizeril-2 stearate and laureate-3-0.4%, rapeseed oil ethoxylated amide - 1.0%, hydroxypropyl methylcellulose - 0.1%, glycerol - 1.0%, sodium chloride - 1.6, lactic acid with pH values up to 3.5-4.0, purified water up to 100.0%. **Conclusions:** Structural-mechanical studies established that this foam base with a number of modern detergents was characterized by a pseudo-plastic type of flow, the structural viscosity of which was equal to 10,200 mPa • s (at 20 r.p.m.), that corresponds to the regulated requirements in accordance with the current normative documentation.

Key words: Surfactants, foam-washing base, intimate hygiene products, pH (3.5-4.5).

INTRODUCTION

On the modern Ukrainian perfumery and cosmetics market, the manufacturers offer a wide range of products for women's tender and delicate areas: Intimate gels, special soaps, napkins, deodorants, sprays, mousses, etc. Intimate care products make the care of a delicate area pleasant and effective.[1-4]

The mucous intimate area has a number of features: Unique acid–base balance, specific microflora, special activity of secretory glands, etc. As you know, the acid–base balance of the skin is 5.5, and the pH of the vaginal mucous is from 3.5 to 4.5. The acidity within these values is a key to maintaining the balance of healthy microflora. When using soap or shower gel, the intimate area receives an increased dose of alkali, that is, the agents have a pH value >5.5.[5-9] When a pH value changes in female genital organs, the amount of harmful microorganisms increases, which can lead to dysbacteriosis, unpleasant sensations and smell, that is, to upset the physiological balance. The modern manufacturers, when elaborating a set of product ingredients for intimate hygiene, take into account all of the above nuances and try to add natural oils, non-alcoholic herbal extracts, and antiseptic substances to the intimate cosmetics products.[10-13]

However, the producers pay particular attention to the development of a foam-washing base for the upcoming product. At first glance, intimate hygiene products

Address for correspondence:
I. I. Baranova, Department of Commodity Science, National University of Pharmacy, Kharkiv, Ukraine.
E-mail: innabaranovapharm@ukr.net

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consist roughly of the same ingredients as shower gels or liquid soaps: A number of surfactants, cosurfactants, acidity regulators, as well as viscosity modifiers and other components, providing the necessary consumer, physical and chemical, microbiological, and other characteristics.[14-16]

The surfactant system of any foaming agent is intended to cleanse, that is, to remove the remains of dead cells, physiological excretions, impurities, as well as the surplus of residential microflora.

The first fundamental requirement when developing compositions for intimate hygiene products is to choose the most delicate combination of surfactants. Typically, in formulations of intimate hygiene products they either combine amphoteric and non-ionic surfactants or combine them with mild anionic detergents. Certainly, traditional sodium or magnesium laureth sulfate (sodium laureth sulfate and magnesium laureth sulfate)[17] is also found in such compositions but only subject to certain conditions: The amount of anionic detergents should be small, and the presence in the formulation of more "soft" surfactants and cosurfactants in a fairly high concentration can significantly reduce the irritating effect of the product as a whole. It is unacceptable to use sodium laureth sulfate, and even more so, lauryl sulfate as a single surfactant in the intimate hygiene agents. It is desirable to introduce "soft" surfactants of different groups and in a variety of combinations in the formulation. Such a method allows not only to reduce the irritant effect on the skin and mucous membranes when applied but also to reduce unwanted skin fluid lipid erosion.[18,19]

Therefore, the main purpose of our work was to experimentally substantiate and develop the base carrier for a low-level pH foam-washing agent.

EXPERIMENTAL SECTION

As a "soft" anionic detergent, we selected disodium laureth-3-sulfosuccinate 40% (Euronaat LS 3, EOS, Belgium), which is most commonly used in the production of foam-washing agents for the cleansing of skin and hair, in particular, for children, as well as in the intimate hygiene products.[2,16,19]

As an additional detergent, we used cocamidopropyl betaine 35% (KAO, Japan), which is a foam stabilizer that can control viscosity and is well tolerated by the skin and mucous membranes, positively affects the dermatological properties of the entire final composition.[12,18,20]

Non-ionic detergent Glucoside/Glyceryl Oleate ("Lamesoft PO 65," manufactured by "BASF," Japan) was chosen for viscosity control.[15,20]

To stabilize the foam base, we used "soft," non-ionic detergents - PEG-7 glyceryl cocoate ("Neopal HE," "BASF," NG Germany), PEG-4 Rapeseed amide ("Amidet N," "BASF" Germany), and PEG-150 Polyglyceryl-2 Stearate and Laureth-3 (Genapol LT, BASF [ex-Cognis] Germany). These substances reduce the irritant action of anionic detergents and regulate the foaming capacity and are recommended for the production of baby shampoos, gels for intimate hygiene, etc.

As gelators we chose:

- Hydroxypropyl methylcellulose ("Methocel 40-0100," "Dow," Germany) - contributes to the thickening of the system and the formation of fine-grained long-term foam. It improves the foaming properties of various cleaning agents, providing dense foam due to its surface activity in conjunction with the properties of the gelator. It is used in such washing agents as shampoos, liquid soap for hands, intimate hygiene products, etc.
- "Salcare SC 80" (Cetacean 10 allyl ether/acrylic copolymer, "Ciba," Switzerland) is a complex acrylic copolymer; gelation is carried out due to the presence of long-chain hydrophobic groups. The dispersion of the gel has a milky color, so when added to the mixture, the water-polymer of the neutralizer (trometamol) instantly creates a gel-like transparent system. The recommended concentration is up to 5%.
- Xanthan is a natural microbial polysaccharide obtained by fermentation of a hydrocarbon by means of the Xanthomonas campestris culture. Xanthan is resistant to high concentrations of salts, pH changes, and high temperatures. The recommended concentration is from 0.5% to 2%.[21,22]

To stabilize and increase the viscosity (due to electrolytic thickening), a solution of sodium chloride was introduced into the foam base.[5]

To regulate the pH value, we used lactic acid (Lactic Acid, Galactic, Belgium) since it is not only physiological (forms part of the acid mantle of the skin) but has some bacteriostatic activity by itself.

The samples of the selected substances were provided by the "Альянс Краси" ("Beauty Alliance") Pharmaceutical Scientific Research Center (Kyiv, Ukraine).

One of the main physical and chemical parameters of any foam-washing agents is the foaming capacity, namely, the foam number (mm) and the foam resistance (conventional units). According to the current normative documents, namely, the State Standard of Ukraine - DSTU "cosmetic products for the cleansing of skin and hair" and the Technical Specifications of Ukraine - TU U 24.5-31640335-002: 2007 "agents for care and cleaning of the skin surface" foam number should be not <145.0 mm, and foam resistance - 0.8–1.0 conventional units.[23,24]

The determination of foaming ability was carried out by means of the Ross–Miles foam test apparatus as a standard device for measuring the foaming capacity of soaps and synthetic detergents. The foam-forming capacity of the test specimens was determined according to the method described in DSTU ISO 696: 2005 "determination of foam forming capacity by a modified Ross–Miles method" and GOST 22567.1-77 "synthetic detergents method for determination of foaming capacity." For this test, the Ross–Miles foam test apparatus, UT-15 ultra-thermostat, a stopwatch, an enema syringe, 3rd accuracy class general purpose laboratory scales, pipettes 1-2-50, pipettes 1-2-1-2 (10), flasks 1-1000-2, glasses B-1-100 (500) (1000) TC[9] were used. The thermostat was connected to the water jacket, switched on, and the temperature of the liquid in the water jacket was increased to (37 ± 2)°C. At the same time, 300 cm^3 of the solution of the tested surfactant was brought to the same temperature. 50 cm^3 of the solution were taken out from this amount, poured into a measuring cylinder trickling on its wall to prevent foam formation. 10 min after that, using an enema syringe or pump, the test solution of surfactant of 200 cm^3 in volume was introduced into the pipette in such a way that no foam was formed. The pipette with solution was fixed on a tripod so that its outlet was at a distance of 900 mm from the level of liquid in the cylinder and provided a jet hit in the center of the liquid. Then the tap of the pipette was opened. When the solution completed its streaming down from the pipette the stopwatch was switched on, the height of the formed pillar of foam was measured in millimeters (measurement was carried out 30 s later). Then, 5-min later, the height of the formed pillar of the foam was measured in millimeters.[25]

The structural and mechanical properties were determined using a Brookfield DV-II+PRO viscosimeter (USA) with a rotary adapter with a coaxial cylinder system. The coaxial geometry of the viscometer is a cylindrical spindle and a cylindrical chamber, which ensures precise control of the measurement of the rheological parameters of the non-Newtonian fluids. With this instrument, the following parameters were measured.

The pH value of the investigated foam samples was determined potentiometrically (DFU 1.2, 2.2.3) using the "pH meter Metrohm 744" device (Germany).[26]

The present studies were carried out on the premises of the scientific laboratory of the Department of Commodity Science at the National University of Pharmacy. All experimental samples were prepared taking into account the following technological parameters:

- Dissolution of detergents was carried out in the temperature range from 35 to 45°C for 10–20 min;
- Revolutions of the mixer - up to 40 rpm (to prevent formation of a foam array);
- pH values were adjusted by means of lactic acid up to 3.5–4.5.

RESULTS AND DISCUSSIONS

The first stage of the study was presented to substantiate the concentration of disulfide laurence sulfosuccinate. For this experiment, we prepared aqueous solutions at concentrations of 15.0, 20.0, and 25.0%. As a result, transparent homogeneous, liquid solutions, and odorless ones were obtained. The reasoning for the concentrations of this detergent was carried out by determining the foam-forming ability and foam stability [Table 1].

As it can be seen from the results in Table 1 at a concentration of detergent of 15.0% the foam number was only 130.0 mm, while the index of foam stability was high −0.81 conventional units. In the 20.0% solution of surfactant both foam number and foam stability increased. When the concentration was increased to 25.0%, the foam number reached the maximum value and was 141.0 mm, and the foam stability was 0.84 conventional units. Therefore, we can conclude that this detergent should be used at a concentration of 20.0%, since the solution had a high enough foam number and the highest value of the foam stability index. This component itself was inoperative because it did not provide the necessary foam values, whereas the foam number was too low, that is, other detergents needed to be added.

We chose cocamidopropyl betaine as another detergent. Therefore, at the next stage, the 20.0% solution of disodium laureth sulfosuccinate was prepared according to the abovementioned technology, and cocamidopropyl betaine was added at the following concentrations: 3.0, 6.0, and 9.0%.

The obtained data indicate that the foam number in the experimental samples increased. The foam stability in samples with cocamidopropyl betaine concentration of 3.0% (0.92 conventional units) and 9.0% (0.93 conventional units) was lower compared to a sample of cocamidopropyl betaine

Table 1: Foaming ability of the base with disodium laureth sulfosuccinate

Concentration of disodium laureth sulfosuccinate,%	Foaming ability	
	Foam number, mm	Foam stability, conventional units
15.0	130.0	0.81
20.0	138.0	0.86
25.0	141.0	0.84

at a concentration of 6.0% (0.95 conventional units). That is, the effective concentration of cocamidopropyl betaine was 6.0% because this sample had a high value of the foam stability and foam number [Table 2].

The next stage was devoted precisely to the stabilization of foam level for the future foam-washing agent. As a foam stabilizer and a superfatted agent, we selected coco-glucoside glycerol oleate ("Lamesoft PO 65"), the recommended concentration of which for the development of foam-washing agents is 1.0–5.0%.

Initially, we prepared a 20.0% solution of sodium laureth sulfosuccinate disodium, where we added 6.0% of cocamidopropyl betaine according to the abovementioned technology. After the complete dissolution of the latter, we added the required amount of coco-glucoside glycerol oleate to the solution. As a result of the complete dissolution, a liquid, transparent, odorless light yellow solution was formed. The samples were prepared with a concentration of coco-glucoside glycerol oleate at the following concentrations: 1.0, 2.0, 3.0, 4.0, and 5.0%. The obtained experimental data indicate that this component contributes to the reduction of the foam level and its stability in the system [Table 3].

However, from the obtained data presented in Table 3, it can be concluded that precisely at the concentration of 1.0% the selected detergent had higher foam-forming ability as compared to other samples.

To achieve a stable foam-forming system, it was decided to introduce non-inogenic detergent ethoxylated amide of rapeseed oil ("Amidet N"), which further thickens the system and stabilizes the foaming capacity of the agent being developed. The recommended concentration is from 0.5% to 1.5%.

To determine the rational concentration, the samples with ethoxylated amide of rapeseed oil were prepared at various concentrations: 0.5, 1.0, and 1.5% [Table 4] using the following technology: First, a 20.0% solution of disodium laureth sulfosuccinate was prepared, to which 6.0% of cocamidopropyl betaine and 1.0% of coco-glucoside glyceryl oleate were added, and then, the required amount of ethoxylated amide of rapeseed oil was added to the solution. After the complete dissolution of all detergents, a liquid solution, odorless, and colorless, was formed.

Analyzing the obtained data [Table 4], it can be stated that when introducing this surfactant at a concentration of 1.0%; there is an increase in the level of foam stability. At

Table 2: Foaming ability of the base with disodium laureth sulfosuccinate (20.0%) and cocamidopropyl betaine

Concentration of cocamidopropyl betaine,%	Foaming ability	
	Foam number, mm	Foam stability, conventional units
3.0	166.0	0.92
6.0	175.0	0.95
9.0	170.0	0.93

Table 3: Foaming ability of the base of disodium laureth sulfosuccinate (20.0%), cocamidopropyl betaine (6.0%) and coco-glucoside glycerin oleate

Concentration of coco-glucoside glycerol oleate,%	Foaming ability	
	Foam number, mm	Foam stability, conventional units
1.0	173.0	0.91
2.0	164.0	0.90
3.0	166.0	0.92
4.0	152.0	0.90
5.0	166.0	0.90

Table 4: Foaming ability of the base of disodium laureth sulfosuccinate (20.0%), cocamidopropyl betaine (6.0%), and coco-glucoside glycerol oleate (1.0%) and ethoxylated amide of rapeseed oil

Concentration of Ethoxylated Amide of rapeseed oil,%	Foaming ability	
	Foam number, mm	Foam stability, conventional units
0.5	162.0	0.92
1.0	174.0	0.96
1.5	167.0	0.94

concentrations of 0.5–1.5%, the foam level was reduced because the foam became fine-grained and as a result of that, it lost its volume.

The next stage of our work was devoted to the choice and reasoning for the choice of thickeners. As detergents that are able to thicken the foam system, we selected PEG-150 polyglyceryl-2 stearate and laureate-3 (Genapol LT) and PEG-7 glyceryl cocoate (Neopal HE). Both are "soft" non-ionic detergents that have a satisfactory cleansing ability and significantly reduce the irritating effect of anionic detergents. According to the literature sources and the manufacturer's advice, the recommended concentration for intimate hygiene foam-washing agents is "Genapol LT" - 0.2–0.8%, "Neopal HE" - 0.25–1.0%.

To determine the concentration of "Genapol LT," we prepared experimental specimens at concentrations of 0.2, 0.4, 0.6, and 0.8% using the following technology: First, a 20.0% solution of disodium laureth sulfosuccinate was prepared, to which 6.0% of cocamidopropyl betaine and 1.0% of coco-glucoside glyceryl oleate were added, then 1.0% of ethoxylated amide of rapeseed oil and the required amount of "Genapol LT" were added. On complete dissolution of all detergents, a liquid solution, without odor and color, was formed.

Analyzing the data from Table 5, we found that when adding "Genapol LT" at different concentrations, the foam number was not satisfactory; however, with an increase in concentration up to 0.6 and 0.8%, we observed a decrease in foam stability.

Also from the data in Table 5, it is obvious that the structural viscosity of samples at a concentration of "Genapol LT" of 0.2 and 0.4% is insufficient (the recommended value of structural viscosity should be in the range of 2000–12000 mPas • s).

It was also noted that, at a concentration of "Genapol LT" of 0.6 and 0.8%, the structural viscosity values were much higher than in the samples at concentrations of 0.2 and 0.4%, but in their turn, the increased concentrations of "Genapol LT" affected the value of the foaming ability, namely, the value of foam number and foam stability were lower compared to the first two samples. This is due to the overfitting of the foam base.

At the concentration of "Genapol LT" of 0.4%, the structural viscosity had rather satisfactory performance but had high foam-forming capacity compared to other samples, therefore, further increase in the concentration of this component was not feasible.

To stabilize and increase the structural viscosity of the foam base, we additionally introduced PEG-7 glyceryl cocoate ("Neopal HE") as a component that thickens the system and stabilizes the foam level. This was because in the future the introduction of a complex of active substances, which may affect the viscosity value, is planned for developing the foam base. The recommended concentration of "Neopal HE" is from 0.25% to 1.0%. It was noted that the system thickening occurred only at a concentration of "Neopal HE" from 0.5% to 1.0% [Table 6].

Therefore, samples of this non-ionic surfactant were prepared at a concentration of 0.25, 0.5, 0.75, and 1.0%. As it can be seen from the data in Table 6, the structural viscosity of the samples with a concentration of "Neopal HE" was 0.25 and 0.5% lower than in the two following samples. However, when comparing the values of foaming capacity, it was found that in the sample with concentration of 0.5% they

Table 5: Foaming ability of the base of disodium laureth sulfosuccinate (20.0%), cocamidopropyl betaine (6.0%), and coco-glucoside glycerol oleate (1.0%), ethoxylated amide of rapeseed oil (1.0%), and PEG-150 polyglyceryl-2 stearate and laureate-3

Concentration of PEG-150 polyglyceryl-2-stearate and laureate-3,%	Structural viscosity, mPa • *P* (20 rpm)	Foaming ability	
		Foam number, mm	Foam stability, conventional units
0.2	1500	182.0	0.92
0.4	2700	194.0	0.96
0.6	5300	187.0	0.93
0.8	6800	178.0	0.92

Table 6: Foaming ability and structural viscosity of investigated samples

Concentration of PEG-7 glyceryl cocoate foam base, %	Structural viscosity, mPa • *P* (20 rpm)	Foam number, mm	Foam stability, conventional units
0.25	3400	183	0.93
0.5	4700	219	0.94
0.75	7800	205	0.95
1.0	9100	180	0.91

were slightly higher than in samples with concentration of 0.25%.

It was noted that at a concentration of “Neopal HE” of 0.75 and of 1.0% the values of structural viscosity of the samples were significant, the system formed a dense mass, that later might not correspond to the extrusion properties of the agent. However, comparing the values of the foaming capacity of the experimental samples, it was found that with an increase in the concentration of “Neopal HE” from 0.75% to 1.0% [Table5], they decreased, indicating that the foam of the washing system was overfitted. Therefore, for further research, we selected a sample with a concentration of 0.5%, which had the best value of foaming capacity compared to other samples.

For additional thickening, improvement of extrusion and consumer properties, we introduced the selected gelators, namely, hydroxypropyl methylcellulose (HPMC), xanthan, and “Salcare SC 80.” The selected gelators are recommended to be used precisely for the development of foaming agents with acidic pH values. The recommended concentrations of gelators in the development of foaming agents with acidic pH values range from 0.1% to 0.5%.

We made experimental samples at room temperature as shown in Table 7 according to the following technology: The foam base was prepared separately based on the technology given above, and the gelators were introduced. For rapid swelling of xanthan and HPMC, dispersion of the latter with 1.0% glycerin was initially carried out, and then purified water (1/4 of the total concentration) was added at a temperature of 80–90°C and stirred for 5–10 min.

The gel base of the “Salcare SC 80” was prepared using the following technology: This gelator was injected into the required portion of water purified from the total concentration, and then, it was neutralized by means of trometamol in a ratio of 1:1. After stirring, a transparent gel base was obtained. The prepared gel bases were added to the foam bases as well at room temperature and the pH values were corrected.

In the study of the samples, we found that the samples with xanthan became stratified during storage. Analyzing the samples with “Salcare SC 80,” it was found that even at a concentration of 0.1%, they were too thick and had poor extrusion properties.

In the course of this experiment, it was noted that the specimens with a concentration of HPMC from 0.3% to 0.5% had too thickened systems and failed to comply with the established norms of the current normative documentation. Analyzing the results of the experiment with a sample at a concentration of 0.1 and 0.2% of HPMC, it was found that they had satisfactory consumer and extrusion properties. Therefore, from an economic point of view, we selected the concentration of HPMC equal to 0.1%.

Table 7: Physical and chemical properties of experimental samples with selected gelators

Concentration of gelator,%	Structural viscosity, mPa s (20 r.p.m.)	Foam number, mm	Foam stability, conventional units
HPMC			
0.1	11200	227	0.94
0.2	11210	226	0.93
0.3	12300	231	0.93
0.4	14200	230	0.94
0.5	16450	229	0.92
Xanthan			
0.1	12100	224	0.92
0.2	13200	228	0.94
0.3	13450	236	0.95
0.4	14280	243	0.95
0.5	17360	232	0.94
“Salcare SC 80”			
0.1	12340	223	0.93
0.2	13410	235	0.97
0.3	15320	239	0.96
0.4	16410	241	0.96
0.5	17650	251	0.97

HPMC: Hydroxypropyl methylcellulose

To stabilize and increase the viscosity (due to electrolytic thickening), we introduced a solution of sodium chloride to the foam base. It was experimentally established that at a concentration of 1.6%, the viscosity of the developed foam base was 10.200 mPa s (20 r.p.m.). The resulting foam base had satisfactory consumer, technological, physical, and chemical properties. The value of the foaming ability (foam number not <−145.0, foam stability of not <−0.8–1.0) was in accordance with the requirements of the current normative documentation, namely, DSTU 4315: 2004 "cosmetic products for the cleansing of skin and hair."

CONCLUSIONS

Based on the carried out physical, chemical, and technological research, the composition of the foam base was substantiated at an acidic pH value: Disodium laureth sulfosuccinate - 20.0%, cocamidopropyl betaine - 6.0%, coco-glucoside glyceryl oleate - 1.0%, PEG-7 glyceryl cocoa - 0, 5%, PEG-150 polyglyceryl-2 stearate and laureate-3 - 0.4%, rapeseed oil ethoxylated amide - 1.0%, hydroxypropyl methylcellulose - 0.1%, glycerol - 1.0%, sodium chloride - 1.6, lactic acid with pH values up to 3.5–4.0, purified water up to 100.0%.

Structural-mechanical studies established that this foam base with a number of modern detergents was characterized by a pseudoplastic type of flow, the structural viscosity of which was equal to 10.200 mPa s (at 20 r.p.m.), that corresponds to the regulated requirements in accordance with the current normative documentation.

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