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The study of structural and mechanical properties and development of technology of the shampoo with antimicrobial complex

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ABSTRACT

Were studied the effects of the active ingredients (D-panthenol, allantoin, antimicrobial complex «JM Acti Care», urea hydroxide, preservative «Rokonsal ND *») on the structural and mechanical (structural viscosity, shear stress, the type of flow) and physical chemical (pH) properties of foam cleaning gel in order to support the optimal composition of baby shampoo. Analysis of the structural and mechanical properties of the developed samples was carried out on a rotary viscometer Brookfield DV-II + PRO (USA) with a spindle SC4-21. By results of the research ascending and descending curves of the hysteresis loops (rheograms) were constructed and was defined structural viscosity - shear rate dependence. Also it was developed and proved rational technology and prepared a draft of technological regulations for the production of shampoo for children aged from 3 to 7 years. This shampoo tested in industrial conditions of pharmaceutical research and development center "Alliance of Beauty" (Kyiv, Ukraine). Thus, developed baby shampoo had satisfactory structural and mechanical, technological and consumer indicators (stability in the process, the extrusion capacity and ease of use)*

Key words: foam cleaning system, detergents, foam-forming indicators, structural viscosity, pH, colloidal stability, thermal stability

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INTRODUCTION

Based on the literature search, it was found that the children hair and scalp have their own characteristics. The scalp of the child is very delicate, thin and vulnerable. Child skin and hair is fully formed only up to 7 years so they require special care [1-3]. Therefore, the development of modern children's shampoos put forward strict requirements, such as: soft washing base; pH-balanced; absence of odor or strong odor; absence of dye and dangerous preservatives, absence of untested active substances; no irritation to the eyes, and others [4-6].

In order to develop the domestic foam cleanser (shampoo) for children that meets modern requirements, we have chosen a range of modern detergents anionic, nonionic and amphoteric nature, namely disodium laureth sulfosuccinate 28 % («Euronaat LS3» Disodium Laureth-3-Sulfosuccinate 28 %, "EOS", Belgium), cocamidopropyl betain 35 % («Cocamidopropyl Betain» «KAO», Japan), coco glucoside and glyceryl oleate («Lamesoft PO 65», «BASF (ex-Cognis)» Germany), glycereth- cocoate («Levenol H & B» «KAO», Yaponiya), PEG-7 glyceryl cocoate and PEG-7 glyceryl palmate («Neopal LIS 80», «Industria Chimica Panzeri», Italy), hydroxypropylmethylcellulose (HPC) («METHOCEL 40-0100» «Dow», Germany) [7-12]. As a regulator of the pH value of the foam detergent lactic acid was used, which is a part of acid mantle of the skin as well as moisturizes and improves the condition and thickness of the epidermis. Lactic acid acts mainly against bacteria, especially anaerobic [4].

On the basis of these substances were prepared foam detergent bases with various concentrations of detergents and selected a rational basis corresponding physical and chemical parameters according to accepted normative documents of Ukraine.

The quality of prepared bases assessed by the following indicators: appearance, organoleptic properties (color, smell), the determination of pH, foaming capacity (the number of foam is not less than – 145,0 mm, the foam stability is not less than – 0,8 - 1, 0 cu). These figures were taken into account for the qualitative assessment of modern foam detergents according to DSTU 4315:2004 «Cosmetics for cleansing the skin and hair» and TU U 24.5-31640335-002:2007 «Products for the care and cleaning the surface of the skin». Based on these data the optimal composition of foam detergent base was chosen: disodium laureth sulfosuccinate 28% – 10,0 %, cocamidopropyl betaine 35 % – 8,0 %, coco glucoside and glyceryl oleate – 1,0 %, glycereth- cocoate – 1,0 %, PEG-7 glyceryl cocoate and PEG-7 glyceryl palmate – 0,5 % HPC – 0,3 % (the number of foam – 151,0 mm, the foam stability is not less than – 0,95 cu) [12, 13].

Based on the microbiological, biological and toxic-hygienic researches concentrations of active substances were proved: D-panthenol – 0,5 %, allantoin – 0,05 %, hydroxyethyl urea – 0,5 % preservative «Rokonsal ND» - 0,1 % and antimicrobial complex « JM Acti Care» – 0,6 % [14-18].

It is known that the active components can significantly change the values of the rheological parameters as well as affect other parameters (pH, colloidal stability, thermo stability), what should be considered in the development cosmetics. The study of the structural and mechanical properties is an important step, because it provides the ability to predict the behavior of the shampoo during manufacturing operations, extrusion from the container (bottle), when applied to the surface of the scalp and hair [19, 20].

The aim of this study was to investigate the structural and mechanical properties of the developed one-component foam detergent bases with the active substances (D-panthenol, allantoin, hydroxyl urea, preservative «Rokonsal ND», the antimicrobial complex «JM Acti Care»), as well as ready shampoo for children between the ages of 3 and 7 years.

EXPERIMENTAL SECTION

The objects of the study were one-component samples of shampoos with active substances: D-panthenol, ((Panthenol), «BASF», Germany) - a component that helps to reduce the irritating of surfactants, allantoin («Clariant», Germany) - reparative, anti-inflammatory component, hydroxyethyl urea («Hydrovance», «Akzo Nobel», USA) - a humectant, a preservative «Roconsal ND» (Phenoxyethanol / Benzoic Acid / Dehydroacetic Acid), «ISP», Germany) and antimicrobial complex «JM Acti Care» (Silver Chloride (and) Titanium Dioxide (and) Diethylhexyl Sodium Sulfosuccinate (and) Propylene Glycol), «Clariant», Germany).

During the research technological, structural, mechanical, physical and chemical studies were carried out.

Rheological parameters (structural viscosity, shear stress, etc.) were measured using a viscometer Brookfield DV-II + PRO (USA), spindle SC 4-21. The following technique was used: about 8,0 – 8,5 g of sample of cream was placed in the chamber, after spindle was immersed in the cream and was driven in rotational movement (20, 30, 35, 40, 50, 60, 80 and 100 r/min) from small deformation rates and then in reverse order. At the same time parameters (shear rate (Dr, c-1: 18.6, 27.9, 32.5, 37.2, 46.5, 55.8, 74.4, 93), shear stress (τ, Pa), apparent viscosity (η, mPa·s)) were fixed on the viscometer display [20].

The pH of the samples was determined by potentiometric method using ionometer «pH Meter Metrohm 744» (Germany).

RESULTS AND DISCUSSION

In order to study the type of flow and the thixotropic properties we have constructed reograms of the investigated bases with the active substances (D-panthenol, alontoina, hydroxyethyl urea, preservative «Rokonsal ND », the antimicrobial complex «JM Acti Care») showing the shear stress (τ, Pa) versus the shear rate (Dr, c-1) (Fig. 1).

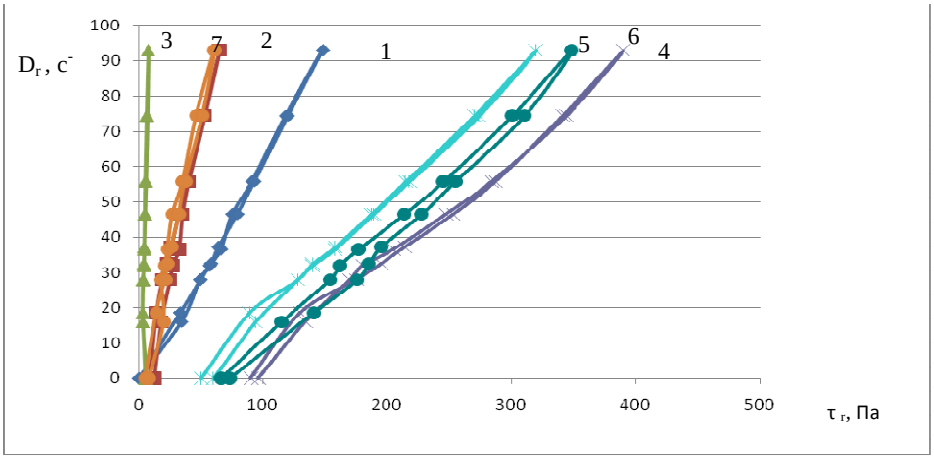

Figure 1. **Rheograms of investigated samples: 1 – the foam washing base of shampoo, 2 – foam washing base with antimicrobial complex «JM Acti Care», 3 – foam washing base with D-panthenol, 4 – foam washing base with allantoin, 5 – foam washing base with preservative «Rokonsal ND», 6 – foam washing base with hydroxyethyl urea, 7 – finished recipe of shampoo**

The data were obtained by continuous ever-increasing destruction of the structure as a function of shear stress. Determinations were carried out by increasing the spindle speed from 20 to 100 rpm, reaching a constant shear stress at maximum speed and then reduce the of number rotations of the spindle.

As can be seen from Fig. 1 samples № 1 (base of shampoo), № 2 (foam washing base with antimicrobial complex «Acti care» – 0,6 %), № 3 (foam washing base with D-panthenol – 0,5 %), and № 7 (ready formulation) characterized by the presence of the lower yield stress and pseudoplastic type of flow (in the investigated range of shear rate).

Studying samples № 4 (foam washing base with allantoin – 0,05 %), № 5 (foam washing base with preservative «Rokonsal ND» – 0,1 %) and № 6 (foam washing base with hydroxyethyl urea – 0,5 %) were observed an increase in the values of rheological parameters (structural viscosity and low yield strength), appearance of plastic properties and minor thixotropic properties, as evidenced by the presence of the hysteresis loop in the rheogram (Fig. 1).

The dependence of structural viscosity of developed samples versus shear rate showed that the structural viscosity of the sample № 4 (foam washing base with allantoin – 0,05 %) № 5 (foam washing base with preservative «Rokonsal ND» - 0,1 %) and № 6 (foam washing base with hydroxyethyl urea – 0,5 %) slowly decreased with increasing of shear rate (Fig. 1) but especially the viscosity decreases rapidly in the range from 20 to 34 s^{-1}. Further structural viscosity does not change so rapidly and at a shear rate of 60 s^{-1} is described by the linear dependence.

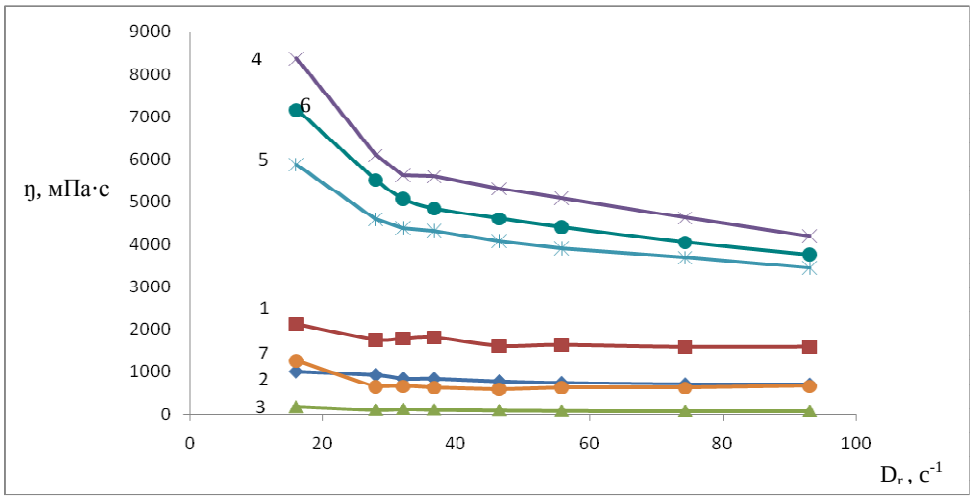

Figure 2. **Structural viscosity-shear rate diagram: 1 – the foam washing base of shampoo, 2 – foam washing base with antimicrobial complex «JM Acti Care», 3 – foam washing base with D-panthenol, 4 – foam washing base with allantoin, 5 – foam washing base with preservative «Rokonsal ND», 6 – foam washing base with hydroxyethyl urea, 7 – finished recipe of shampoo**

Investigation of the dependence structural viscosity versus shear rate of samples № 1 (foam washing base) and № 2 (foam washing base with antimicrobial complex «JM Acti Care» – 0,6 %) and number 3 (foam washing base with D-panthenol – 0,5 %) found that their viscosity have been practically independent of the shear rate.

After the study of structural viscosity versus shear rate of the sample № 7 (ready formulation), it can be concluded that the structural viscosity decreased slightly in the range of deformation from 20 to 30 s^{-1}. Further structural viscosity of shampoo as well as samples №№ 4 and 5 does not change so rapidly and at a shear rate of 60 s^{-1} is described by the linear dependence.

The established dependence of the structural viscosity versus shear rate show more uniform and easy distribution of the shampoo on the scalp and hair**.** Incorporation of active ingredients resulted in an increase of the apparent viscosity, but the character of viscosity shear rate reducing (the form of curves) slightly affected.

It is known, that production technology affects the quality of drug, its therapeutic activity and good consumer properties. Manufacturing process should consist of rational planned system of interrelated processes, where each process step must be justified [6,21,22].

The technological process began with the preparation of foam detergent base on the classical scheme: surface active agents were sequentially dissolved in the totality of purified water: disodium laureth sulfosuccinate 28 %, cocamidopropyl betaine 35 %, cocoglucoside and glyceryl oleate, glyceret- cocoate, PEG-7 glyceryl cocoate and PEG-7 glyceryl palmate (at 35-40 °C, mixing during 20 min, mixer speed 40 rev/min). The water temperature was monitored with a liquid thermometer, which was inserted into the thermowellof apparatus. In parallel glycerine with the powder of HPC were dispersed at room temperature during 10 min. Then, at the temperature of 25 °C-35 °C the following components were successively introduced into the reactor: D-panthenol, allantoin, hydroxyethyl urea, antimicrobial complex «JM Acti Care». The components were homogenized using an anchor mixer during 35 min until a homogeneous clear solution was obtained. Then the sample was taken from apparatus via sampler to determine the pH with a pH meter.

To prevent the formation of foam in the reactor mixer speed was reduced to about 30 ver/ min, and lactic acid solution was introduced to the required pH (5,2-5,8). Then complex preservative «Roconsal ND» was added at mixer speed up to 30 rev / min. Components were mixed during 2-3 min. This preservative has to be introduced only after adjusting the pH level, as at a pH below 6,2 this preservative can be inactivated. As a result transparent gel base was obtained.

The technological process of this product is carried out with the necessary sanitary rules and requirements, set out in the standard sanitary requirements of the enterprise for the production of «Sanitary pre-production of foam detergents».

Production of developed product consists of six basic steps of the technological process and two stages of packaging, which are summarized below.

Step 1.Weighing of components of the developed shampoo.
After passing incoming inspection of raw materials on the scales 200 AD (Germany) in separate labeled clean containers, required number of components, specified in the recipe, which are delivered to the site via transport trolleys, are weighed .

Step 2. Preparation of surface active agents solution.
In the step of manufacturing of foam detergent base by a centrifugal pump (LEO7752923, Ukraine) into the container (stainless steel, the volume of 1,200 liters, the manufacturer of «YuzhmolProm», Ukraine) the calculated amount of purified water (35 °C-40 °C) is poured. Then, water is pumped through a conduit in the single-walled stainless steel reactor with a steam circuit capacity of 1500 kg, without vacuum equipment (NPP Intermash, Ukraine) with two types of mixers: the anchor (40 rev/min) and propeller (80-100 rev/min) which ensures effective mixing of the components. Manually via the upper loading hatch components are loaded into the reactor (disodium laureth sulfosuccinate 28 %, cocamidopropyl betaine 35 %, cocoglucoside and glyceryl oleate, glyceret cocoate, PEG-7 glyceryl cocoate and PEG-200 glyceryl palmat). Components are mixed until complete dissolution during 20 min with the frame mixer speed 40 rev/min. Then, using the circulation circuit solution, which was obtained, is forced through a propeller mixer during 5-7 min with the rate of turnover of a propeller mixer 80-100 rev/min for averaging mass.

Step 3. Dispersion of HPC in glycerin.
Amount of HPC, weighed in a separate vessel, is mixed with the weighed amount of glycerin. Mixing is during 2-5 min until a uniform translucent mass suspension with no visible clumps will be obtained.

Amount of HPC, weighed in a separate vessel (stainless steel, 25 liters «Ukraine»), is mixed with the weighed amount of glycerin. Mixing occurs by hand using mixer for 2-5 minutes (Stark HM-1350 PRO, Germany) until a uniform translucent mass suspension without visible clumps will be obtained.

Step 4. Dissolution of suspension in a solution of surface active agents.
Glycerin suspension was introduced slowly in the surface active agents solution, which was obtained, through the upper loading hatch in the running frame mixer (30-40 rev/min) and was mixed for 15-20 min, then for 10 min was mixed with a propeller mixer at 80-100 rev/min to obtain a homogeneous transparent mass.

Step 5. Introduction of the active components
With the frame mixer of reactor at 40 rev/min and a temperature of (25-35) °C successively weighed (step 1), the components were introduced into the reactor through the loading hatch: D-panthenol, allantoin, hydroxyethyl urea, antimicrobial complex «JM Acti Care». Then, using the circulation loop with a propeller mixer, components were homogenized for 10 minutes at about 80-100 rev/ min.

Step 6. pH correction and introduction of preservative.
Samples of solution were taken from two zones of reactor (50 ml from the top and the same amount from the bottom), to verify required physical and chemical quality parameters, including pH [7-12].

After receiving the results of laboratory testing of samples, the pH was adjusted with lactic acid. Lactic acid is introduced in the required amounts directly into the reactor and mixed using frame mixer (35-40 rev/min) for 5-7 min. Additional samples were taken for re-checking the pH. In case of obtaining the required pH level between 5,2 and 5,7 we introduced a preservative and homogenized using frame mixer (35-40 rev/min) for 5-7 min. The uniformity of the obtained solution was control. Weight should be transparent homogeneous without extraneous inclusions.

Step 7. Packing in primary packaging.
The obtained product is packed in bottles of PEG («Pharma» v. 250 / 24-410 / art.01) (white) of 250 ml, with dispenser (white IS CG-07-2A 24 | 410). Weight is loaded by pneumatic diaphragm pump (DDA50C, Poland). In the hopper, with the help of semi-automatic dispenser (LLC - Technology-Business, Ukraine), weight is packaged in vials, manually wind the cover, then using the conveyor belt (NPP Intermash, Ukraine) labelis automatically pastedon the vial.

With laser marker (Linx SL501-09-9678, Slovakia) date is automatically stamped on the reverse side of the vial.

Step 8. Packing of vials into boxes and formation of pallets.
Bottles with a product are manually placed in boxes, using weights, completeness of each box is checked. Each box is automatically glued on line (packer boxes SIAT FAMSB-S 53345 -180W, Italy). Sealed boxes are manually placed up to 5 rows on pallets, for group transportation.

CONCLUSION

Thus, studies have shown that selected active ingredients of shampoo for children aged 3 to 7 years, can significantly affect the basic characteristics of foam washing base. With addition of D-panthenol and antimicrobial complex «JM Acti Care» viscosity reduction of developed bases of shampoo was observed (Newtonian fluids). With addition of a allantoin preservative «Rokonsal ND» and hydroxyethyl urea on the contrary there was an increase in viscosity, indicating the presence of plastic properties.

A rational technology of baby shampoo was developed and justified. Also on the basis of this project technological regulations for production of shampoo is composed. This shampoo is tested under industrial conditions on the basis of pharmaceutical research and development center «Alliance of Beauty» (Kyiv, Ukraine).

Thus, we have developed baby shampoo, which has a satisfactory structural-mechanical, technological and consumer indicators (stability in the process, extrusion capacity, and ease of use).

REFERENCES

[1**]** ES Keshishian. *J. of Med. Council.*, **2008**, 1, 57-60.
[2**]** RM Trüeb. *J. Dtsch. Dermatol. Ges.*, **2007**, 5, 356-365.
[3**]** Z Matthew; M Jessica. *Dermatitis.*, **2009**, 20(2), 106-110.
[4] MF Reis; AM de Almeida; PM Rezende. *Int. J. Trichology*, **2014**, 6(3), 95-99.
[5**]** L Ho Tan Tai. Formulating Detergents and Personal Care Products. A Guide to Product Development, New York, **2000**, 3141-3247.
[6**]** AG Bashura; NP Polovko; EV Gladuh. Rezende. Technology of cosmetics and perfumery means, Golden Pages, Kharkiv, **2002,** 272**.**
[7**]** KR Lange. Surfactants: Synthesis, Properties, Analysis, application, Profession, Saint Petersburg, **2007**, 240**.**
[8**]** MYu Pletneva. Surface-active substances and compositions. Handbook, «Firm Clavel», Moscow, **2002**, 768**.**
[9**]** AA Abramson; VV Bocharov; DA Gaevoi. Surfactants Handbook, Chemistry, Lviv, **1979**, 376**.**
[10] RL Philip; NR Palme; CA Noam. *Am. J. Rhinol. Allergy*, **2013**, 27(3), 177–180.
[11**]** EV Zhuk; II Baranova. *News of the Tajik Nation. Univ.*, **2015**, 1/1(156), 263-266.
[12**]** CR Robbins. The physical properties and cosmetic behavior of hair, 5th Edition, Springer-Verlag, New York, **2012**, 158-199.
[13**]** VJ Hamme; A Singh; OP Ward. *J. Biotechnol.*, **2006**, 24, 604–620.
[14**]** P Romanowski. *J. of the Univ. of Chem. Technology and Metallurgy*, **2011**, 3, 54-56.
[15**]** A Hollis. *J. of the Univ. of Chem. Technology and Metallurgy*, **2010**, 42(2), 39-44.
[16**]** C Phyllis; SA Flawn; CS Woodroffe. *J. Soc. Cosmetc. Hem*, **1999**, 42(2), 187-194.
[17**]** AV Hudz. *News of Vinnytsia Nation. Univ.*, **2004,** 8(2), 409-413.
[18**]** OV Zhuk; LP Petrovskaya; II Baranova. *News of Pharmacy*, **2014**, 1(77), 12–16.
[19**]** DR Picout; SB Ross-Murphy. *The Scientific World J.*, **2003**, 24(3), 105–121.
[20**]** AYa Malkin. Rheology Concepts, Methods, and Applications, Applied Science Publishers, London, **2006**, 474.
[21**]** OV Zhuk; II Baranova. *Pharmaceutical J.*, **2014**, 1, 49-55.
[22**]** J Swarbrick; JC Boylan. Encyclopedia of Pharmaceutical Technology, Marcel Dekker, New York, **2002**, 3005 – 3019.