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Full Length Research Paper

Modelling of semisolid natural basis and powder of liquorice as active constituent influence on rheological characteristics of systems

Ada Stelmakiene[1]*, Kristina Ramanauskiene[1], Ernesta Malinauskyte[2] and Daiva Leskauskaite[2]

[1]Department of Pharmaceutical Technology and Social Pharmacy, Lithuanian University of Health Sciences, A. Mickeviciaus Str. 9, 44307, Kaunas, Lithuania.
[2]Department of Food Technology, Kaunas University of Technology, Radvilenu pl. 19, 50254 Kaunas, Lithuania.

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Skin dysfunctions are a common problem. To restore and maintain skin functions, we modeled original semisolid hydrophobic systems from natural crude materials, using the experimental planning technique. The evaluation of the quality of the modeled systems according to the flow characteristics showed that the beeswax content in the systems affected their yield stress, consistency coefficient, and the flow behavior index, while the effect of cholesterol was slight. At temperatures of 40 and 50°C, the systems have pseudoplastic properties, while at 60°C – Newtonian. The evaluation of the viscoelasticity of the systems showed that the systems had elastic properties, and the most stable structure at different temperatures was demonstrated by formula VC6, containing: Beeswax (5.828 g); solid paraffin (5.0 g); white Vaseline (5.0 g); cocoa butter (10.0 g); cholesterol (3.0 g); and olive oil (70.0 g). Introduction of 0.5% liquorice root powder into the modeled base showed that this substance made the system thinner. The evaluation of the sensory properties of the bases showed that the characteristics of the VC6 system were most acceptable to the customer.

Key words: Rheological characteristics, semisolid bases, beeswax.

INTRODUCTION

Ointments are homogeneous, semisolid preparations intended for external application to the skin or mucous membranes. They are used as emollients or for the application of active ingredients to the skin for protective, therapeutic, or prophylactic purposes and where a degree of occlusion is desired (Carter, 2006). Ointments have the following rheological properties – shear stress, viscosity, and elasticity module (Bourne, 2002; Steffe, 1996). Typical texture characteristics of semisolid preparations are softness, strength, viscosity, and elasticity (Gohel and Parikh, 2008). Experimental determination of texture characteristics of the preparations is most commonly performed using rheological examination techniques.

It is also can be regarded as sensitive tools for detecting structural changes in pharmaceutical creams (Korhonen, 2004). To achieve high-quality description of the texture of products, device readings should be associated with consumer's sensory experience (Akiyama et al., 2010) when applying the semisolid preparation.

Since the modern dosage forms are systems consisting of active ingredients and excipients, the selection of the latter is one of the major tasks in drug modeling. Excipients used for the modeling of the semisolid system matrix should ensure the stability, elasticity, and soft texture of the preparation, as well as its acceptability to the customer; in addition to that, the excipients should not alter the effect of the active ingredients and should ensure their stability in the semisolid matrix (Gohel and Parikh, 2008; European Pharmacopeia, 2008).

For the testing, we selected natural excipients to model a stable natural ointment base that would be acceptable for the customer and would have protective, moisturizing, and skin-nourishing properties. For these reasons, the two natural components selected for the modeling of the

*Corresponding author. E-mail: kristinaraman@gmail.com.

Table 1. Amounts (g) of the variable components of the hydrophobic ointment base according to the full composition plan.

Sample No.	Component					
	Yellow beeswax	Cholesterol	Solid paraffin	White vaseline	Cocoa butter	Olive oil
VC1	1.000	1.000	5.000	5.000	10.000	70.000
VC2	1.000	5.000	5.000	5.000	10.000	70.000
VC3	5.000	1.000	5.000	5.000	10.000	70.000
VC4	5.000	5.000	5.000	5.000	10.000	70.000
VC5	0.172	3.000	5.000	5.000	10.000	70.000
VC6	5.828	3.000	5.000	5.000	10.000	70.000
VC7	3.000	0.172	5.000	5.000	10.000	70.000
VC8	3.000	5.828	5.000	5.000	10.000	70.000
VC9	3.000	3.000	5.000	5.000	10.000	70.000

base of the preparation were beeswax and olive oil.

Studies (Al-Waili, 2003; Rit et al., 1999) have proven the anti-inflammatory effect of beeswax, and thus this substance can be used for the treatment of skin diseases and lesions of oral mucosa. Olive oil has an antioxidant, nourishing, protective, antiseptic, and stimulating effect. In addition to that, olive oil protects sensitive and dry skin, restores its protective layer, suppresses water evaporation, improves skin structure, and soothes and nourishes the skin (Visioli et al., 2002; Aburjai and Natsheh, 2003).

The aim of the study was to model an original semisolid hydrophobic base (system) from natural crude materials, using the experimental planning technique, and to evaluate the quality of this system on the basis of its rheological characteristics and sensory properties.

MATERIALS AND METHODS

Optimization of the composition of the ointment base

Optimal selection of the amounts of ingredients was performed on the basis of the orthogonal composition plan, using Statistica 6.0 software. The characteristic feature of this plan is that removal of any coefficient of the equation does not affect the values of other coefficients. Experiments were performed at the end-points of the model (+1; -1), the “asterisk” points, and the middle points (mean values) (Shiromani, 2004).

We calculated the amounts of beeswax and cholesterol at the points of the model, and compiled a full orthogonal composition plan. The amounts of solid paraffin, vaseline, cocoa butter, and olive oil remained unaltered. The amounts of the ingredients are presented in Table 1.

The studied sample VC6+S was the one in which the composition of the VC6 base was supplemented with 0.5% liquorice root powder. This sample was used to determine how introduction of suspension substances affected the rheological characteristics of the base.

Rheological flow behavior test

The test was performed using the Carri-med CSL^2 500 rheometer (TA Instruments, Germany), by applying the cone- and- plate geometry system (cone diameter, 60 mm; angle, 2°; sample thickness,150 μm) at temperatures of 40, 50, and 60°C. The shear rate was increased for 2 min from 0 to 500 s^{-1}.

Rheological oscillation test

Viscoelastic properties were tested using the Carri-med CSL^2 500 rheometer (TA Instruments, Germany), by applying the cone-and-plate geometry system (cone diameter, 60 mm; angle, 2°, sample thickness – 1000 μm), with 0.1% strain, 6, 20, and 30°C temperature, and stress rate varying from 0.1 to 100 rad^{-1}. The results were expressed as elasticity G‘, viscosity G‘‘, and complex G* modules (Leskauskaitė et al., 2006; Liutkevičius and Speičienė, 2006).

Evaluation of the sensory characteristics

A trained group of specialists analyzed pre-selected products (samples) and chose terms (compiled the vocabulary) to describe their sensory characteristics. Subsequently, the scales for the evaluation of the intensity of these characteristics were selected and discussed, and the intensity of each characteristic of all products was then marked on separate scales.

Using these data, mathematical statistics techniques were applied to compile a profile of sensory characteristics for each product, indicating the intensity of each characteristic. This profile could then be used to compare products according to their separate properties and their intensity, to determine the relationship between the sensory quality of products and their individual characteristics, etc. A group of 12 evaluators participated in the study (ISO 8586-2:2008).

All experimental measurements were repeated three times, and the results were presented as arithmetic means of these measurements.

RESULTS AND DISCUSSION

According to their composition, the samples were distributed into three groups: Group I – samples VC1,VC2,VC3, and VC4, group II – samples VC5,VC6, VC6+S, and VC9, and group III – samples VC7,VC8, and VC9. In addition to that, ingredients of the systems (white vaseline, cocoa butter, and olive oil) were analyzed as

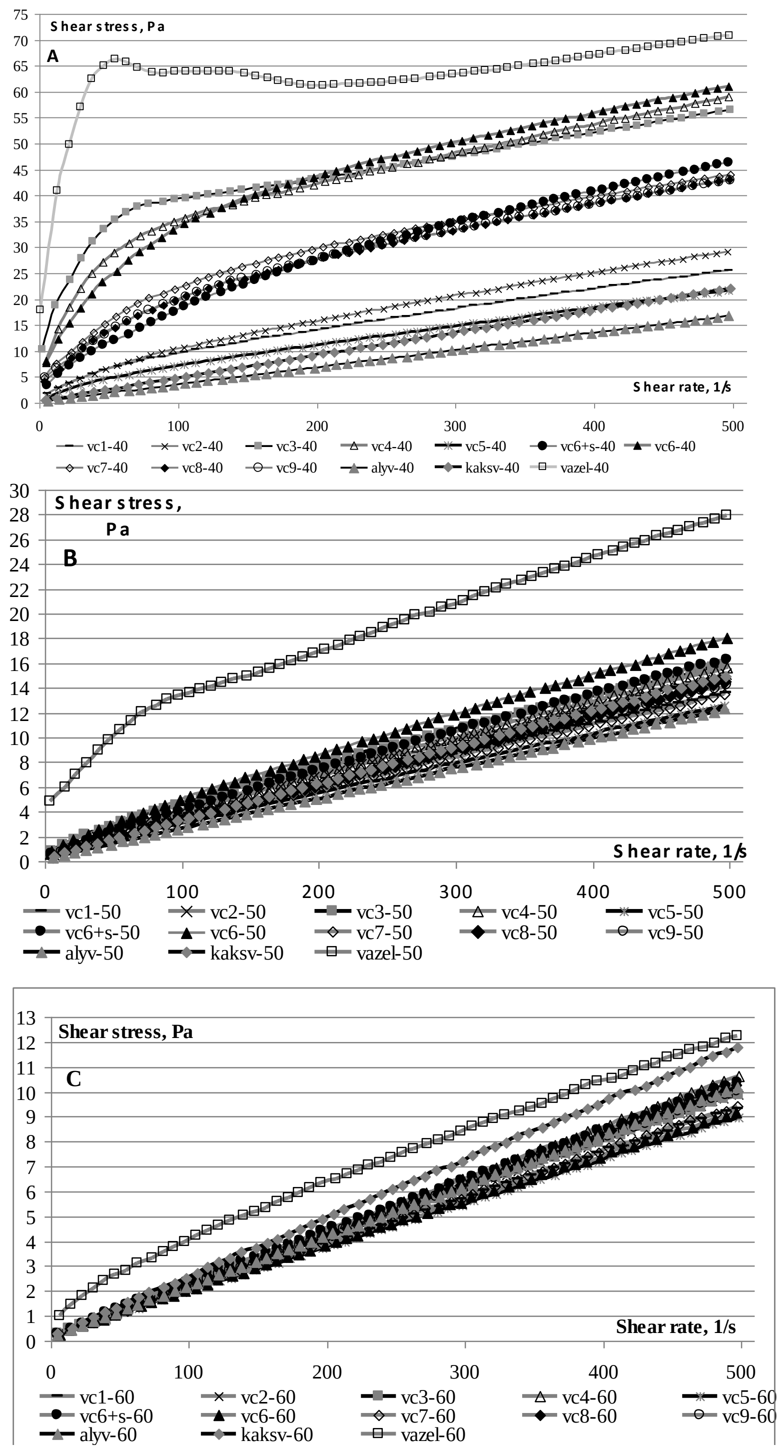

Figure 1. Flow behavior curves of all the studied samples at the temperatures of A) 40, B) 50 and C) 60°C.

well. In order to evaluate the flowability of the optimized compositions and changes in the characteristics of the systems during the technological process, flow behavior testing was performed, which also evaluated the spreadability of the semisolid preparations on the skin. The testing of the flow behavior of the ingredients showed that irrespectively of the temperature, the highest shear stress was observed in vaseline, and the lowest in olive oil (Figure 1).

The results of the testing showed that shear stress

Table 2. Rheological characteristics of ointments tested at the temperature of 40°C.

Sample No.	Herschel-Bulkley		
	σ_o, Pa	K, Pa·s^n	n
VC1	2.845	0.3618	0.6760
VC2	2.804	0.8323	0.6841
VC3	17.62	12.67	0.2333
VC4	13.66	6.8725	0.3436
VC5	1.964	0.1761	0.7668
VC6+S	5.401	1.2455	0.5850
VC6	11.803	5.697	0.3825
VC7	7.494	3.0745	0.43055
VC8	6.492	1.7855	0.5134
VC9	6.765	2.3415	0.4660

directly depended upon beeswax content in the systems; the highest shear stress at the temperature of 40°C was observed in systems VC3, VC4, and VC6 (Figure 1a), and the lowest in systems VC5, VC1, and VC2 (Figure 1a). At temperatures of 50 and 60°C (Figure 1b and c), flowability curves of the majority of the tested systems at low strain coincided, but somewhat differed in higher strain regions. Vaseline was an exception, demonstrating the highest shear stress in all temperature regimens.

The analysis of the relationship between shear stress of the studied systems and their shear rate showed that at the temperature of 40°C, all systems had characteristics of pseudoplastic substances, where increasing shear rate disrupted the structure of the system, resulting in increasing shear stress. At first shear stress increased rather significantly, but later on, at higher shear rates and with a more uniform already disrupted initial structure and lower resistance to deformation, the increase in shear stress became more even.

Experimental testing showed that the flowability of the modeled systems at the temperature of 40°C corresponded to the Herschel-Bulkley mathematical model and could be described with the following equation:

$$\sigma = \sigma_o + K\gamma^n,$$

where σ is shear stress (Pa), γ is shear (deformation) rate (s^{-1}), σ_o is yield stress indicating the shear stress required to achieve shear rate, K is consistency coefficient, which is the indicator of viscosity, and n is flow behavior index, which is the indicator of deviation from the characteristics typical of Newtonian fluids (Bourne, 2002; Leskauskaitė et al., 2006). The flow behavior index values calculated according to the The flowability of the modeled systems at the temperature of 50°C corresponded to the Casson mathematical model and could be described with the following equation (Steffe, 1996):

Herschel-Bulkley equation (Table 2) in group I ranged from 0.2333 (VC3) to 0.6841 (VC2), in group II - from 0.3825 (VC6) to 0.7668 (VC5), and in group III - from 0.43055 (VC7) to 0.5134 (VC8). The highest consistency coefficient, which may be the indicator of the viscosity of the system, in group I was observed in system VC3 (12.67), in group II – in system VC6 (5.697), and in group III – in system VC7 (3.0745). The highest σ_o, which indicates pressure required to initiate flow and thus the system's initial resistance to deformation in group I was observed in system VC3 (17.62 Pa), in group II in system VC6 (11.803 Pa), and in group III in system VC7 (7.494 Pa). It is noteworthy that the same samples demonstrated the highest yield stress and consistency coefficient.

The comparison of the rheological characteristics with the composition of the tested systems showed that the lowest flow index, the highest consistency coefficient, and the highest yield stress were characteristic of samples that contained more beeswax. In group III, the beeswax content equaled to 3.0 g, while the cholesterol content varied, and thus the effect of cholesterol on the rheological characteristics could be observed.

The test findings presented in Figure 2 and Table 2 indicate that the effect of cholesterol was only slight, yet it may be presumed that this component had a somewhat thinning effect on the systems, because higher amounts of cholesterol (VC8) resulted in lower yield stress and consistency coefficient, compared to systems with lower cholesterol content (VC7).

The relationship between shear stress of the tested systems and the shear rate curve at the temperature of 50°C is presented in Figure 3, indicates that shear stress was lower and similar in all samples. Flow behavior curves in group II samples were somewhat different (Figure 3b).

$$\sqrt{\sigma} = \sqrt{\sigma_o} + K\sqrt{\gamma}$$

Consistency constants and σ_o calculated according to the

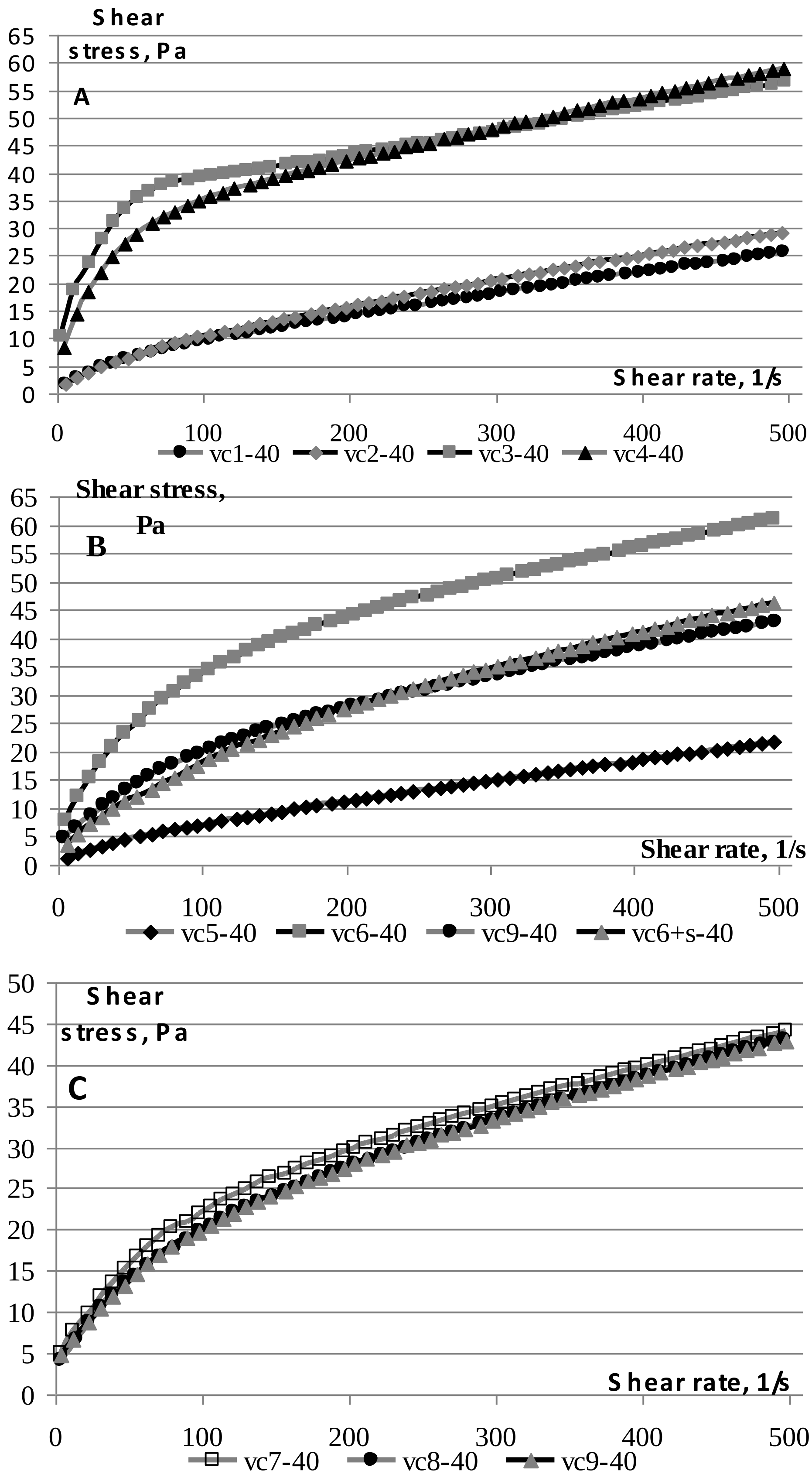

Figure 2. Flow behavior charts at the temperature of 40°C A) group I, B) group II, C) group III.

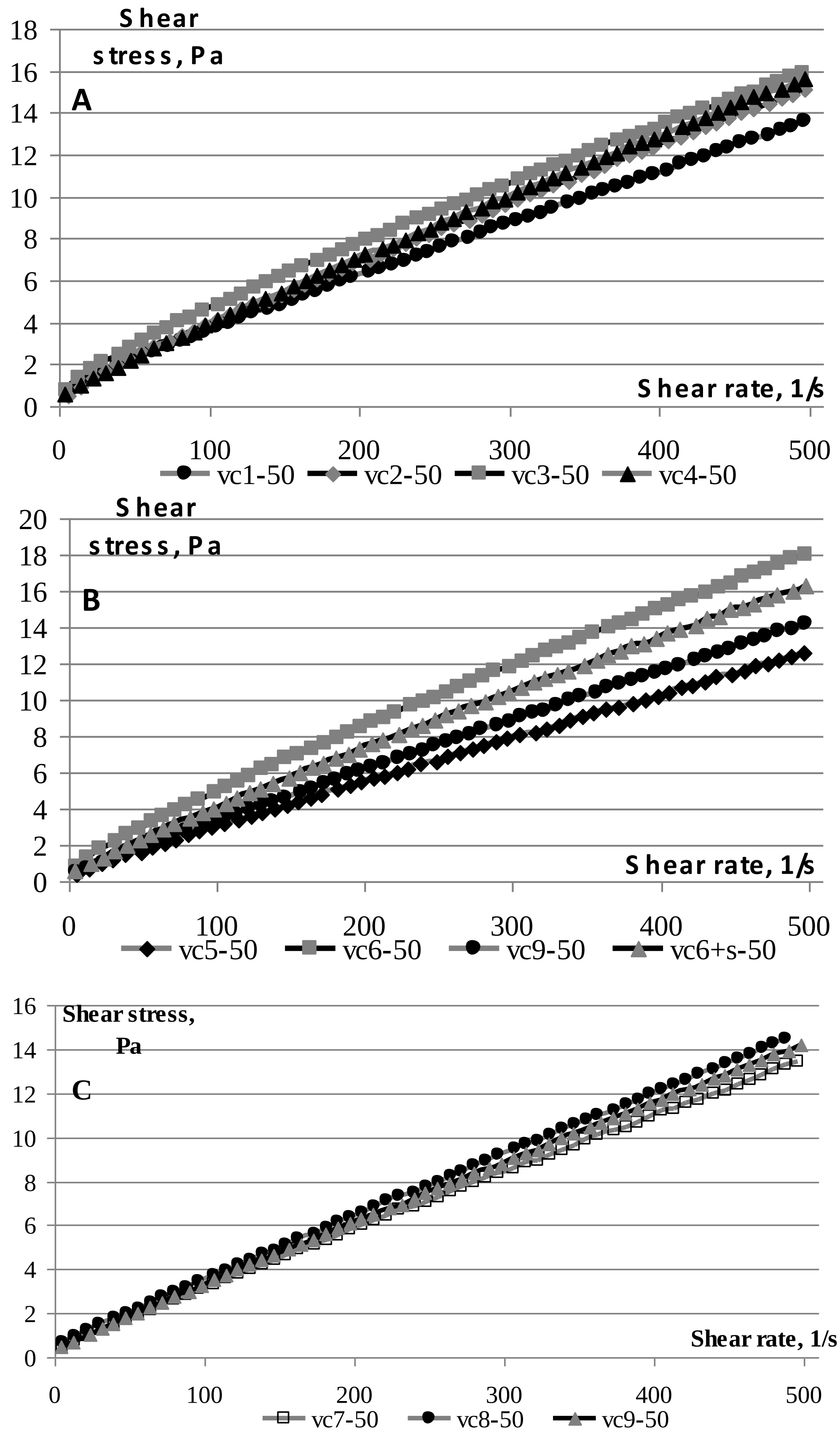

Figure 3. Flow behavior curves at the temperature of 50^{o}C A) group I, B) group II, C) group III.

Table 3. Rheological characteristics of ointments tested at the temperature of 50°C.

Sample No.	Casson	
	σ_o, Pa	K Pa s
VC1	0.2081	0.0249
VC2	0.1875	0.0239
VC3	0.4248	0.0223
VC4	0.1608	0.0258
VC5	0.0749	0.0216
VC6+S	0.1742	0.0268
VC6	0.3650	0.0266
VC7	0.0686	0.0269
VC8	0.0499	0.0260
VC9	0.0605	0.0249

Table 4. Rheological characteristics of ointments tested at the temperature of 60°C.

Sample No.	Newtonian
	η Pa s
VC1	0.0192-
VC2	0.0204
VC3	0.0200
VC4	0.0216
VC5	0.0183
VC6+S	0.0216
VC6	0.0188
VC7	0.0190
VC8	0.0208
VC9	0.0204

Casson equation (Table 3) indicated that in all groups the consistency coefficient dropped below one and was similar in all samples, between 0.0216 (VC5) and 0.0269 (VC7). σ_o was more varied from 0.0499 (VC8) to 0.4248 (VC3), which indicates that quite different initial stress was required to initiate flow in the tested systems despite their similar viscosity. This shows that even at higher temperatures the structure stability of the systems remained different.

The flowability of the modeled systems at the temperature of 60°C corresponded to the Newtonian model described with the following equation (Bourne, 2002; Steffe, 1996):

$$\sigma = \eta \cdot \gamma,$$

Where η – dynamic viscosity. The dynamic viscosity was low and similar in all systems; from 0.0183 to 0.0216 (Table 4). This shows that at the temperature of 60°C, the studied semisolid systems transformed into Newtonian fluids.

Semisolid preparations should be of suitable consistency (neither too hard nor too soft, and should not crack when being spread on the skin) and should retain the stability of their structure at changing temperatures. Oscillation testing was performed in order to evaluate the viscoelastic characteristics of the optimized compositions and the stability of their structure at temperatures of 6, 20, and 30°C.

The evaluation of the viscoelastic characteristics at temperatures of 6, 20, and 30°C showed a higher elasticity module G‘ than the viscosity module G‘‘ in the majority of the samples, which indicates that the elastic characteristics predominated over the flowability properties in these systems (Table 5). Data presented in Table 5 show that in many samples G* quite significantly differed at different temperatures; from 178.113 to 22.353 Pa (VC4) or from 69.857 to 4.289 Pa (VC9). Testing results showed that the most stable structure at various temperatures was observed in the system VC6, whose complex module (G*) varied the least, compared to other systems (from 28.777 to 32.650 Pa).

Table 5. Viscoelastic characteristics at 55.18 rad/s frequency and 6, 20, and 30°C temperature.

Sample No.	G', Pa			G", Pa			G*, Pa		
	6°C	20°C	30°C	6°C	20°C	30°C	6°C	20°C	30°C
VC1	9.891	41.246	2.752	9.149	38.498	725	18.577	56.747	2.849
VC2	-	41.181	2.048	4.479	28.152	525	17.824	49.917	2.125
VC3	29.617	47.793	21.260	43.924	12.643	10.677	54.957	50.867	28.890
VC4	28.370	20.055	14.360	12.259	1.471	12.360	178.113	33.320	22.353
VC5	31.497	7.322	538	4.693	2.930	225	44.487	8.177	584
VC6+S	18.797	10.554	971	1.200	4.341	451	37.977	11.743	1.071
VC6	24.640	29.250	27.113	6.775	24.760	4.716	31.700	32.650	28.777
VC7	29.883	18.680	46.364	45.500	19.395	4.609	81.447	63.890	96.344
VC8	35.520	12.050	4.564	5.620	2.541	1.536	38.977	62.993	4.822
VC9	21.050	55.640	3.650	-	53.597	2.210	41.313	69.857	4.289

*Note – negative value.

To evaluate how the rheological characteristics of a system were affected by the introduction of suspension substances into the ointment base, we studied a sample containing 0.5% liquorice root powder (VC6+S). Upon introduction of liquorice root powder into this system, the complex module at different temperatures ranged between 37.977 and 1.071 Pa, which indicates that this structure did not remain stable when affected by temperature and stress (Table 5). The testing has shown that as the result of the introduction of suspension substances, the consistency coefficient dropped from 5.697 (VC6) to 1.2455 (VC6+S), and yield stress from 11.803 (VC6) to 5.401 (VC6+S), while the flow behavior index increased from 0.3825 (VC6) to 0.5850(VC6+S) (Table 2) at the temperature of 40°C, which shows that liquorice root powder had a thinning effect on the ointment.

To improve the sensory characteristics of the ointment that is, to achieve semisolid consistency, acceptable spreadability, and elasticity, the following substances were added to the ointment base: Cocoa butter, vaseline, and paraffin. All the tested preparations had light yellow color and acceptable mild scent. The following sensory criteria were evaluated: Color, scent, spreadability, lubricity, and consistency. The results of the evaluation are presented in Figure 4.

Findings presented in Figure 4 indicate that the system VC6 demonstrated the most acceptable spreadability, while the spreadability of the system VC5 was least acceptable. The ointment base VC6 also had the most acceptable consistency, while the consistency of VC7 was the poorest. Even though the preparations did not contain any scent-enhancing substances, ointment bases VC6, VC 4, and VC5 demonstrated the most intensive scent.

The evaluation of the color ranged between 6 and 8 points. The color of the base VC6 was considered to be most acceptable to the customers. The results of the evaluation showed that the content of beeswax and cholesterol affected the sensory characteristics of the modeled ointment bases. These results corroborated literature data (Aulton, 1998) indicating that excipients affect the sensory characteristics of the preparations.

The evaluation of the lubricity and the moisturizing ability of the preparations showed that the base VC6 was most acceptable, while the base VC3 was the least acceptable.

The results of the evaluation of the sensory characteristics showed that the base VC6 was most suitable for further testing.

Conclusions

The study showed that beeswax had the greatest effect on the decrease in the flow behavior index and the increase in the consistency coefficient and the yield stress parameters of semisolid system bases. Evaluation of the rheological characteristics of the semisolid systems showed that temperature may affect the characteristics of the modeled system.

At the temperature of 40 to 50°C, the systems started demonstrating characteristics of pseudoplastic substances and at the temperature of 60°C, the characteristics of Newtonian substances. Using the experimental planning technique and results of the evaluation of sensory and rheological characteristics of the samples, we designed an original semisolid hydrophobic ointment base from the following natural crude materials: Beeswax, 5.828 g; solid paraffin, 5.0 g; white vaseline, 5.0 g; cocoa butter, 10.0 g; cholesterol, 3.0 g; olive oil, 70.0 g.

It has been found that the introduction of liquorice root powder into the selected optimal-composition base entailed changes in the structure of the semisolid system, resulting in its instability when affected by temperature

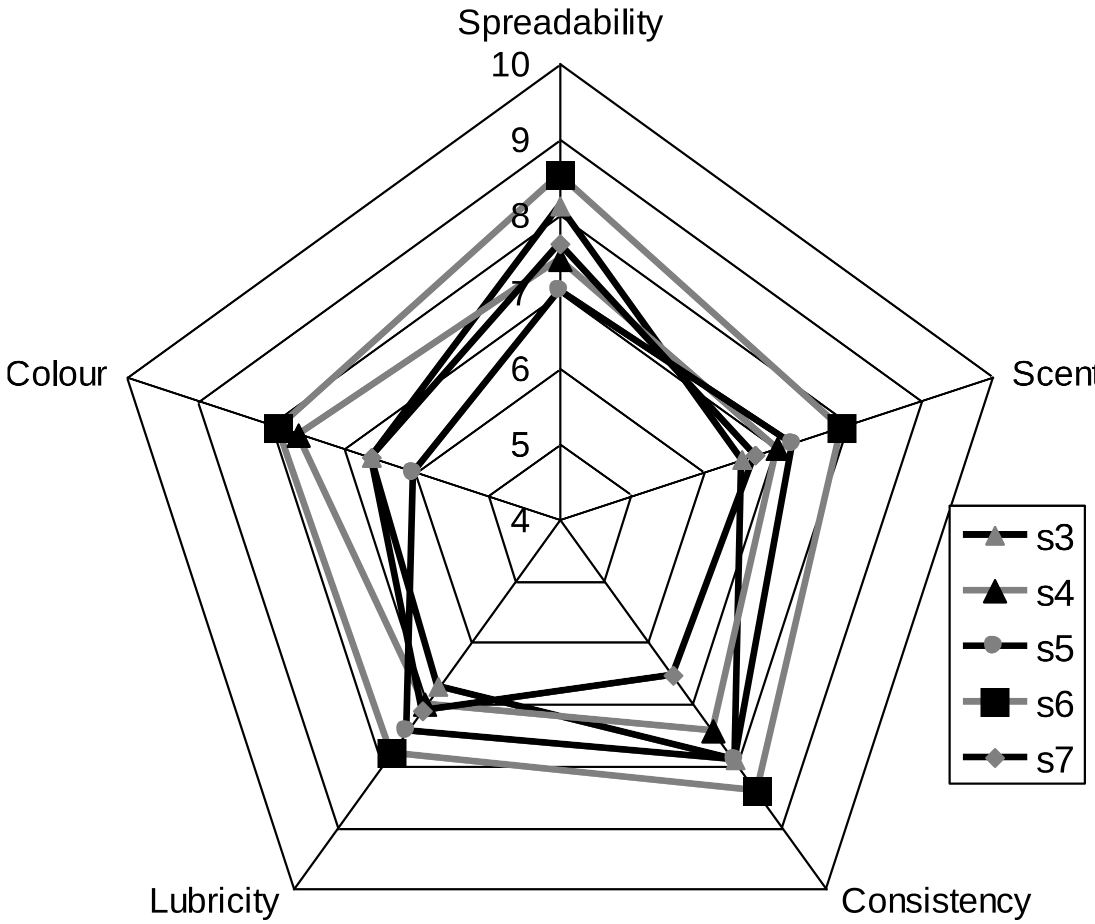

Figure 4. Results of the evaluation of the sensory characteristics.

and stress.

REFERENCES

Aburjai T, Natsheh FM (2003). Plants used in cosmetics. Phytother. Res., 17(9): 987–1000.

Akiyama Y, Izumi Y, Nishijima S (2010). Evaluation of tactile sensation of cosmetics by tactile movement — Application movement of skin care products. Int. J. Cos. Sci., 32(2): 164–165.

Al-Waili NS (2003). Topical application of natural honey, beeswax and olive oil mixture for atopic dermatitis or psoriasis: Partially controlled, single-blinded study. Compl. Ther. Med., 11(4): 226-234.

Aulton ME (1998). Pharmaceutics: The Science of Dosage Form Design, Churchill Livingstone, pp. 257-258.

Bourne MC (2002). Food texture and viscosity: Concept and measurement, 2nd ed., Academic Press, pp. 77-99.

Carter SJ (2006). Cooper and Gunn: Dispensing for Pharmaceutical Students, 12Th Ed,CBS Publishers, Delhi, pp.192-193.

European Pharmacopeia (2008) 6th Ed. Vol 1, Council of Europe, Strasburg, pp. 24: 27- 746

Gohel M, Parikh R (2008). Novel semisolid dosage forms. Available from URL: http://www.pharmainfo.net/

Sensory analysis (2008). General guidance for the selection, training and monitoring of assessors -- Part 2: Expert sensory assessors (ISO 8586-2:2008).

Korhonen M (2004). Rheological properties of pharmaceutical creams containing sorbitan fatty acid ester surfactants. PhD dissertation, University of Helsinki, Helsinki, Finland.

Leskauskaitė Dk, Malinauskytė E, Ramoškienė S (2006). Jelly-forming additives on the rheological properties of dairydesserts. Food Chem. Technol., 40(1): 39-46.

Liutkevičius A, Speičienė V (2006). Functional foodsubstances influence of ice characteristics. Food Chem. Technol., 40(1): 47-54.

Rit TP, Keunen K, Behrer R (1999). Beeswax through the ages. Available from URL: http://www.kosterkeunen.com/News/customerfiles/Beeswax%20Through%20The%20Ages.pdf.

Shiromani PK (2004). Statistical optimization of pharmaceutical formulations. Pharm&BioIngridients, 1,1. Available from URL: http://www.pharmabioingredients.com.

Steffe JF (1996). Rheological methods in food process engineering 2nd ed., Freeman press, USA, pp. 21-27.

Visioli F, Poli A, Gall C (2002). Antioxidant and other biological activities of phenols from olives and olive oil. Med. Res. Rev., 22(1): 65–75.