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Original article

Textural and Sensory Characterization of Carbomeric Gels with Panthenol

Milica Martinović, Nenad Stojanović, Ivana Nešić

University of Niš, Faculty of Medicine, Department of Pharmacy Niš, Serbia

SUMMARY

Introduction. The aim of this study was to evaluate the influence of the carbomer concentration (used as the gelling agent, in the range 0.5 - 2.0%) on textural and sensory characteristics of gels with 3% panthenol using sensory analysis as subjective and texture analysis as an objective assessment method. An active substance in the gels was panthenol, which also acted as a neutralizing agent of the carbomer.
Participants and methods. Twenty female panelists, aged between 20 and 36, participated in the sensory study and had to fulfill a questionnaire regarding product attributes before, during and after application on the skin. Texture analysis was performed on CT3 Texture Analyzer, after each week for a month, and the following parameters were calculated: hardness cycle 1, hardness cycle 2, cohesiveness, adhesiveness, resilience and springiness.
Results. The results showed that gels were stable over time and the structure of the gels was preserved regardless of compression. An increase in hardness and resilience was followed by an increase in the concentration of carbomer, while adhesiveness, cohesiveness and springiness did not differ much among different gels. The same trend was noticed when gels were compared during time. Sensory analysis confirmed findings of texture analysis and showed that with the increment of carbomer concentration, stickiness and density also enhanced, while absorption rate and spreadability decreased. Gel with 1% carbomer was chosen by the majority of panelists as favorite and as a gel they would regularly use.
Conclusion. The methods of textural and sensory analysis represent valuable approaches in drug/cosmetic product design because they offer insight in the compliance of patients/consumers.

Keywords: **carbomer, panthenol, gel, texture analysis, sensory analysis**

Corresponding author:
Milica Martinović
e-mail: milica.martinovic@medfak.ni.ac.rs

INTRODUCTION

One of the essential phases in the development of drugs is consideration of patient acceptability, which is usually a result of organoleptic, mechanical (textural) and rheological properties of dosage forms. This is very important when it comes to topical dosage forms, such as hydrogels (1). Hydrogels represent three-dimensional networks of hydrophilic polymers that can hold big amount of water (> 10%) within its structure. Due to their structure, flexibility, resemblance to the human tissue (because of the big water content), hydrogels have a very large use (controlled drug delivery, adsorbents for the removal of heavy metals, scaffolds in tissue engineering, contact lenses, pH sensors, biosensors, injectable hydrogels for spinal cord regeneration, etc.) (2).

Carbomer gels are pH-sensitive hydrogels, as a result of ionizable nature of poly(acrylic acid), which is deprotonated in basic media. Deprotonated anions cause electrostatic repulsion between the chains, and the 3D network is formed. Yet, in acidic environment, protonation disrupts the structure of the gel, and hydrogel collapses (3).

Panthenol, as a member of B-complex group, is a normal constituent of the skin. After application, it transforms into pantothenic acid, which has fundamental role in skin epithelial function (4). Panthenol-based formulations can improve skin hydration and decrease transepidermal water loss (5). Therefore, it is widely used in dry and damaged skin treatment. Usually, panthenol can be found in topical preparations in concentrations 2 - 5% (6).

According to ISO 5492:2008, texture represents all of the mechanical, geometrical and surface attributes of a product detectable by receptors. While sensory analysis takes into account interpretation of organoleptic attributes of a tested product perceptible by senses, texture analysis leans on quantitive measurements of product properties, using various devices – texture analyzers (7). A sensory study is a mighty tool for the cosmetic industry since it provides information from the consumers, which is important for further development of the product. Nonetheless, sensory assessment is basically subjective assessment. For objective evaluation, instrumental measurements are needed (8). Originally, texture profile analysis (TPA) was used for solid and semi-solid foods (9, 10). TPA was meant to simulate chewing which leads to deformation of the food (11). Nowadays, TPA is used in many science and industry fields, including cosmetic and pharmacy industry. The instrument, texture analyzer, mimics human finger touching the product (cream, gel, liquid, etc.) surface (12). There are two compression cycles in TPA, when the probe of texture analyzer enters the tested product placed in a sample cup. During the process, a graph containing force-time curve is being formed. As a result, the following TPA parameters can be measured and read from the force-time plot: hardness, fracturability, cohesiveness, adhesiveness, gumminess, chewiness, springiness, stringiness, etc. (13).

AIM

The aim of this study was to determine the influence of the concentration of the gelling agent on textural and sensory characteristics of carbomeric gels with 3% panthenol. For this purpose, texture analysis, as an objective method of structure assessment, was performed *in vitro* using texture analyzer, while sensory analysis (subjective assessment) was performed in an *in vivo* study on healthy volunteers.

MATERIAL AND METHODS

Materials

Panthenol, Carbopol 940 and Euxyl PE 9100 were purchased from Comcen (Serbia), propylene glycol from Centrohem (Serbia), sodium hydroxide was bought from Zdravlje (Serbia), while purified water originated from the Faculty of Medicine in Niš (Serbia). All chemicals used were of analytical purity (p.a.).

Formulation of gels

For the study, four gels with panthenol as active ingredient and different concentrations of carbomer as gelling agent were prepared (Table 1).

Carbomer was added to water and the mixture was left to swell for one day. After that, panthenol and neutralizing agent were added, as well as propylene glycol and preservative. The gels were agitated for 30 minutes using propeller laboratory stirrer RV16 basic (IKA®VERKE, Germany).

Table 1. *Qualitative and quantitative compositions (%, (w/w)) of gel samples*

Ingredients (Trade name)	Ingredients (INCI)	Function	Sample 1	Sample 2	Sample 3	Sample 4
Panthenol	Panthenol	Active component	3.00	3.00	3.00	3.00
Carbopol 940	Carbomer	Gelling agent	**0.50**	**1.00**	**1.50**	**2.00**
Sodium hydroxide	NaOH, 10%	Neutralizing agent	q.s.	q.s.	q.s.	q.s.
Propylene glycol	Propylene glycol	Humectant	10.00	10.00	10.00	10.00
Euxyl PE 9100	Phenoxyethanol and ethylhexylglycerin	Preservative	1.00	1.00	1.00	1.00
Purified water	Purified water	Water phase	ad 100	ad 100	ad 100	ad 100

Measurement of the pH of the gels

The pH value was determined conductometrically using pH meter (CONSORT multi-parameter analyzer C830, United States) from a 5.00% water solution of prepared gels. All pH values were set to 5.8.

Texture analysis of gels

The texture analysis was performed on CT3 Texture Analyzer (Brookfield, AMETEK Inc., USA) with 10 kg load cell in order to examine the texture analysis parameters, by adjusting the following experimental conditions:

- Test speed: 1.5 mm/s
- Target value: 5 mm
- Trigger load: 5 g

Samples were placed in the sample cups (75% of each cup was filled, avoiding introduction of air) and cone probe TA-STF was used. Texture profile analysis (TPA) test was performed, during which the probe immerged into samples, and hardness cycle 1, hardness cycle 2, cohesiveness, adhesiveness, resilience and springiness were measured. TPA was performed in triplicate and the mean values and

Table 2. *Sensory evaluation questionnaire*

Before application	
Consistency	liquid / semi-solid
Gloss level	matt / pearl gloss / slight gloss / gloss / very glossy
During application	
Spreadability	easy to spread/difficult to spread/ very difficult to spread
Adhesion	not sticky/slightly sticky/ sticky/very sticky
Density	rare/slightly dense/ dense/ very dense
Grease	not greasy/ slightly greasy/ greasy/ very greasy
Gloss	not shiny/slightly shiny/ shiny/ very shiny
Absorption rate	slow/ moderate/ fast
After application	
Residual film	no film/ moderate film/ expressive film
Stickiness	not sticky/slightly sticky/ sticky/very sticky
Grease	not greasy/ slightly greasy/ greasy/ very greasy
Gloss	not shiny/slightly shiny/ shiny/ very shiny
Which sample would you regularly use?	
1/2/3/4	

standard deviations of parameters were calculated.

Sensory analysis of gels

Twenty female panelists who are regular consumers of cosmetic products, aged between 20 and 36, participated in the study. They evaluated the characteristics of the samples before rubbing, during application as well as 10 minutes after application. Sensory analysis was performed 4 weeks after gel production. The participants were explained the purpose and the manner of organoleptic tests and each of them received a questionnaire where they had to choose descriptive terms that best fit the samples (Table 2). Panelists also had to choose which gel they prefer most (which one they would use).

RESULTS

Texture analysis of gels

During the first 7 days of testing, illogical values of the monitored texture analysis parameters were obtained, most likely due to the fact that the carbomer was not structured well enough. As no conclusion could be drawn from these results, they were rejected and not presented in this paper. After this period, the measurement results were repro-

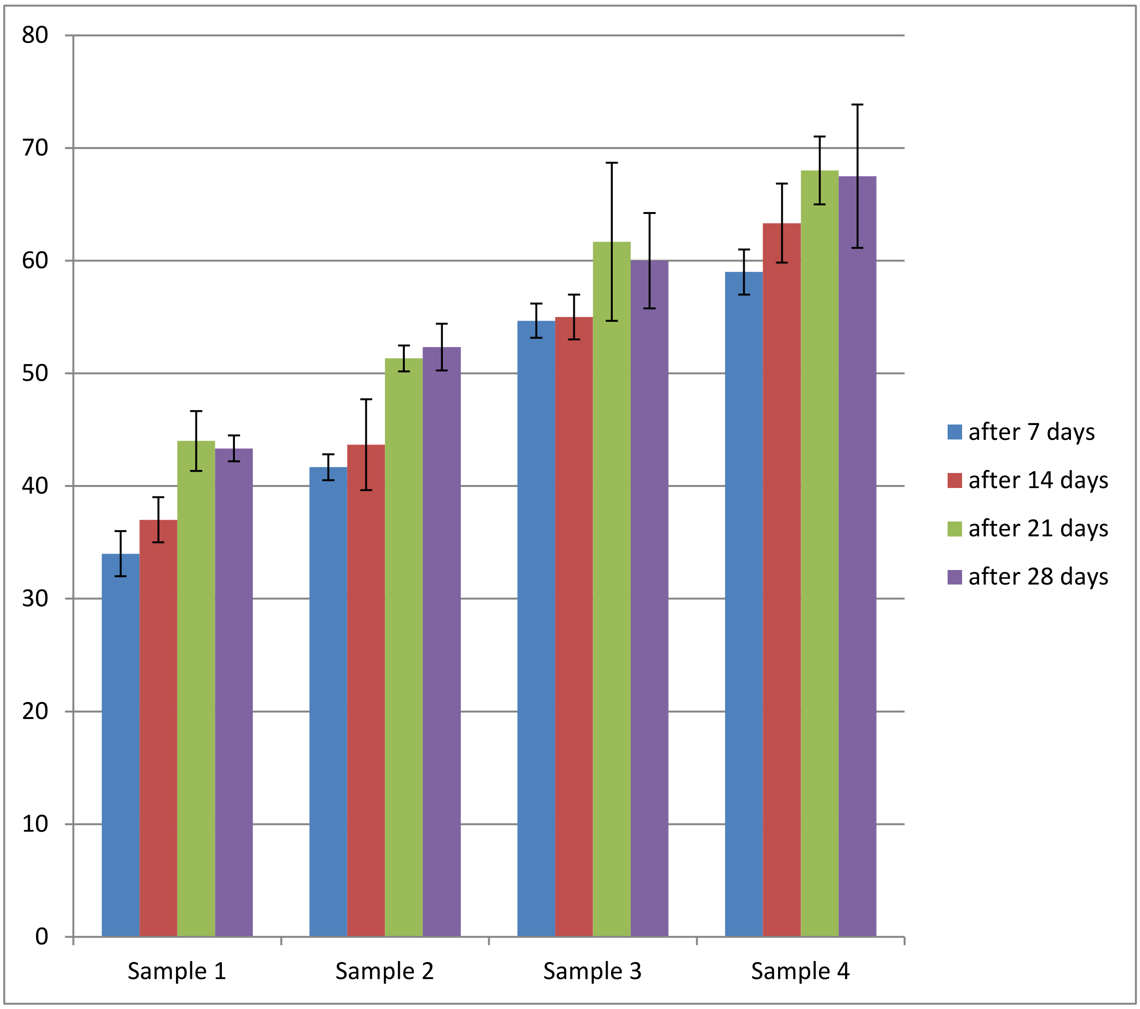

Figure 1. *Chart of hardness cycle 1 of tested panthenol gel samples (Sample 1 – 0.5% carbomer, Sample 2 – 1.0% carbomer, Sample 3 – 1.5% carbomer, Sample 4 – 2.0% carbomer)*

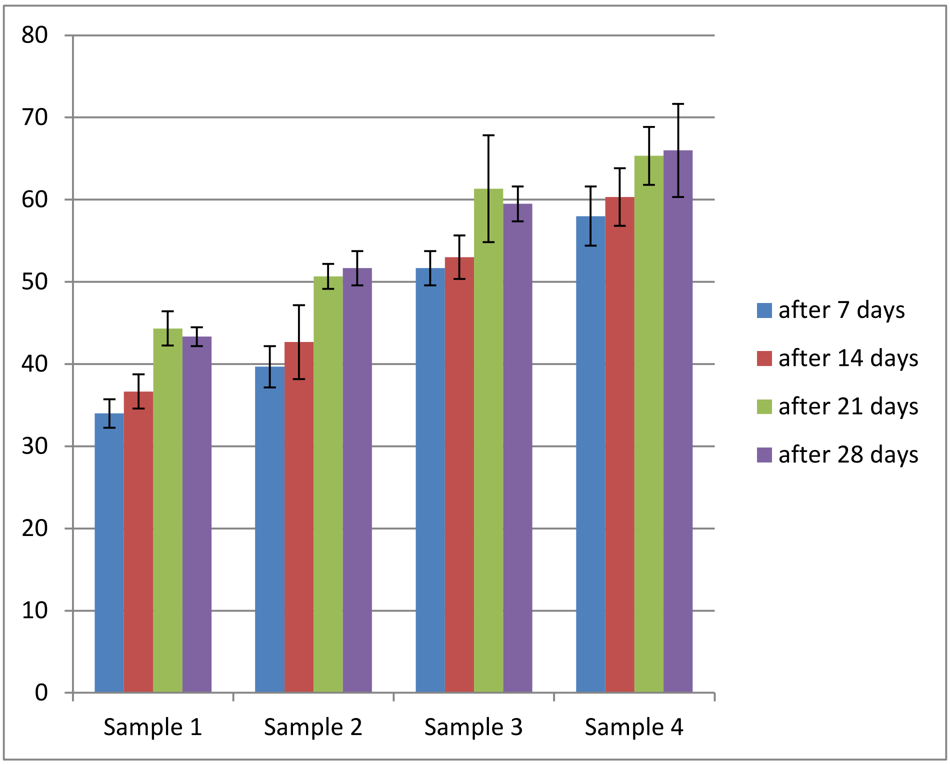

Figure 2. *Chart of hardness cycle 2 of tested panthenol gel samples (Sample 1 – 0.5% carbomer, Sample 2 – 1.0% carbomer, Sample 3 – 1.5% carbomer, Sample 4 – 2.0% carbomer)*

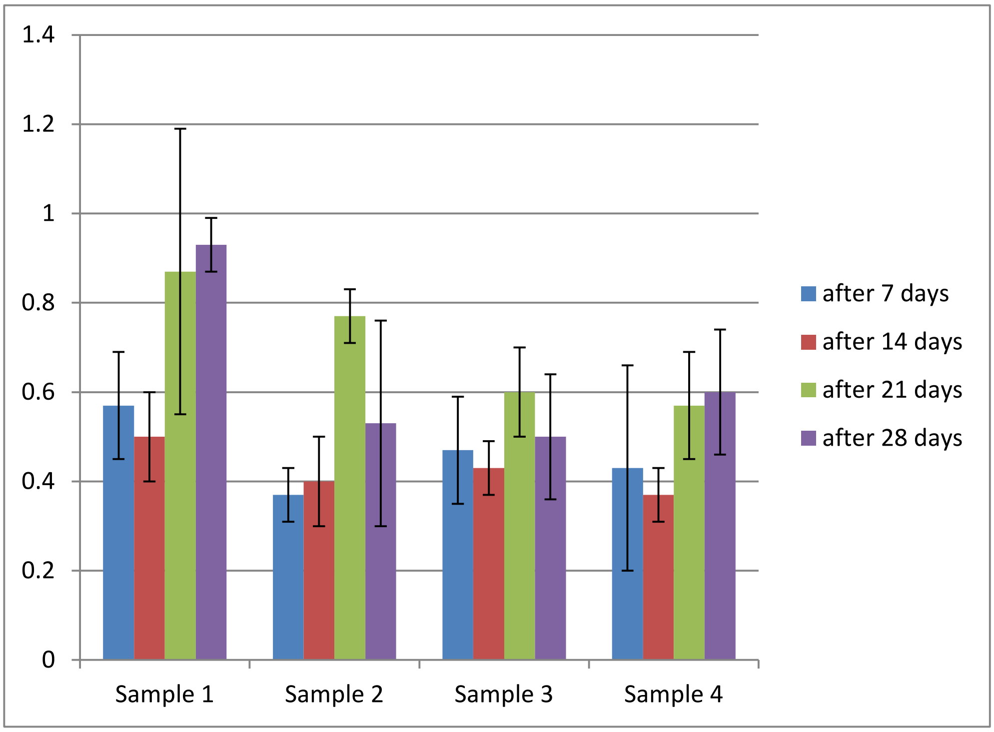

Figure 3. *Chart of adhesiveness of tested panthenol gel samples (Sample 1 – 0.5% carbomer, Sample 2 – 1.0% carbomer, Sample 3 – 1.5% carbomer, Sample 4 – 2.0% carbomer)*

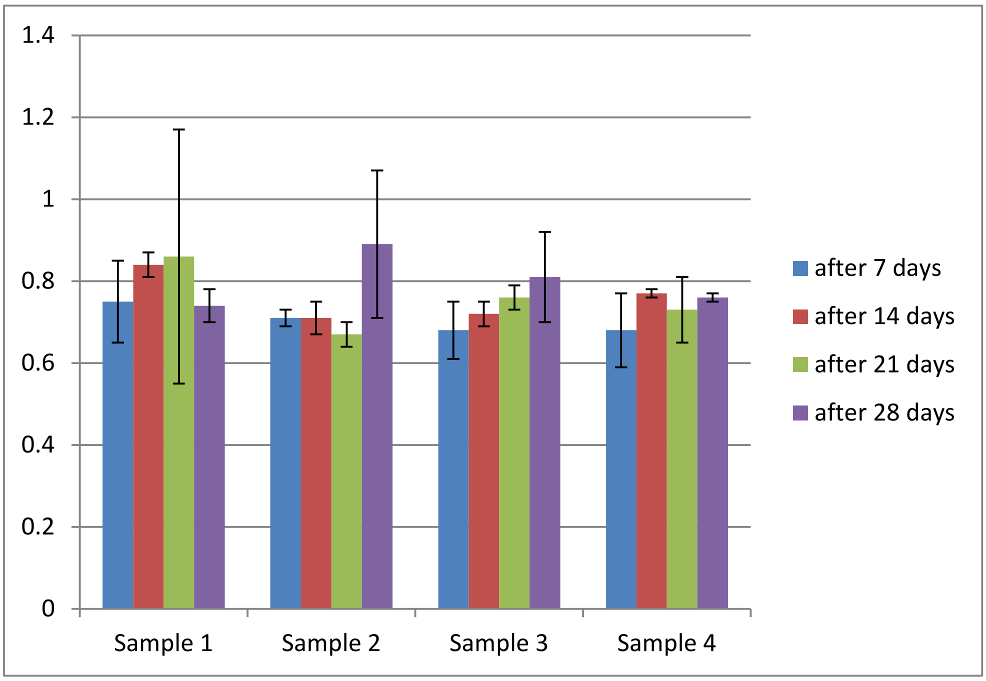

Figure 4. *Chart of cohesiveness of tested panthenol gel samples (Sample 1 – 0.5% carbomer, Sample 2 – 1.0% carbomer, Sample 3 – 1.5% carbomer, Sample 4 – 2.0% carbomer)*

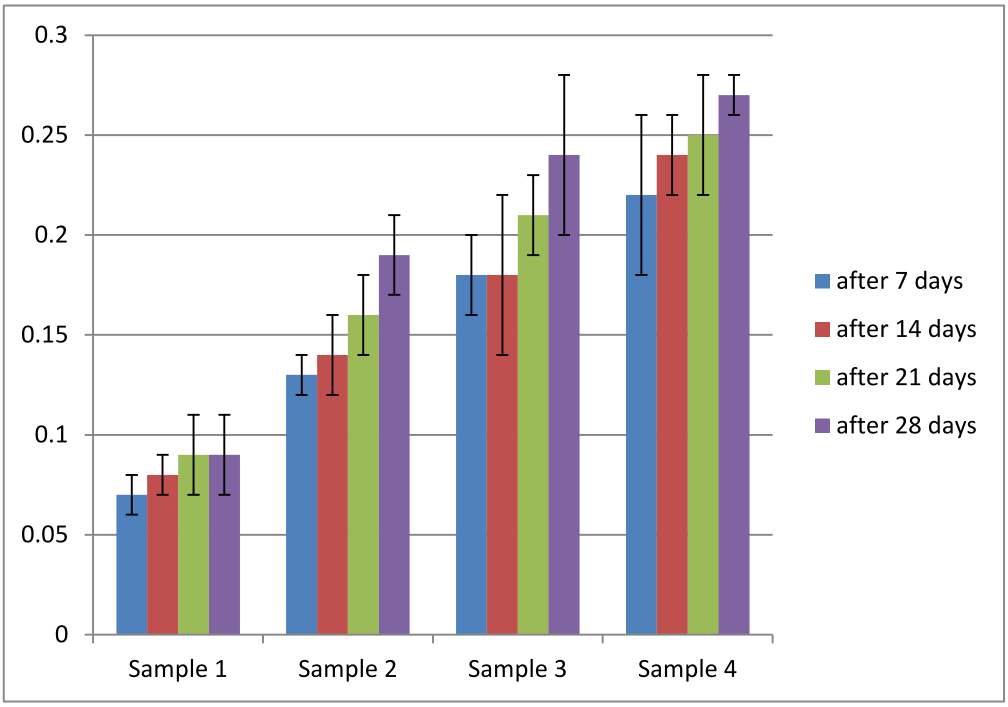

Figure 5. *Chart of reselience of tested panthenol gel samples (Sample 1 – 0.5% carbomer, Sample 2 – 1.0% carbomer, Sample 3 – 1.5% carbomer, Sample 4 – 2.0% carbomer)*

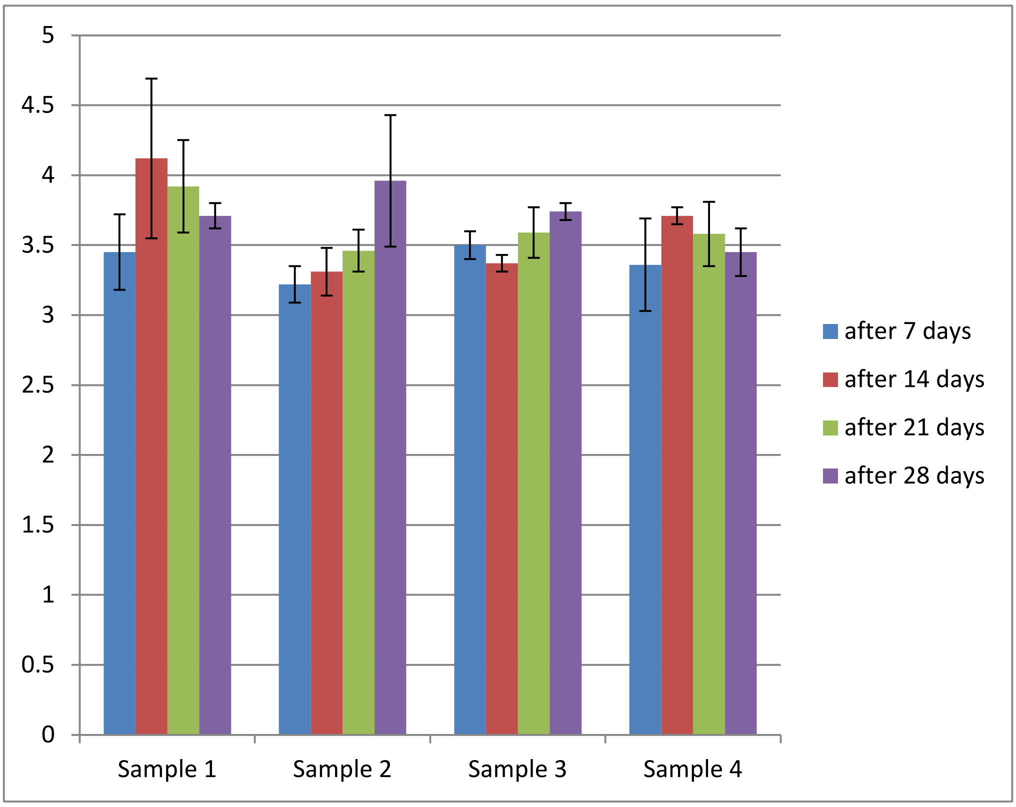

Figure 6. *Chart of springiness of tested panthenol gel samples (Sample 1 – 0.5% carbomer, Sample 2 – 1.0% carbomer, Sample 3 – 1.5% carbomer, Sample 4 – 2.0% carbomer)*

ductive and completely acceptable.

From the graph, the following parameters could be read: hardness cycle 1, hardness cycle 2, adhesiveness, cohesiveness, resilience and springiness.

Hardness was measured during both cycles (hardness cycle 1 and hardness cycle 2) and the results are shown in Figures 1 and 2. Figure 3 presents adhesiveness of the samples, as a measure of stickiness of the sample and probe, while Figure 4 shows cohesiveness of the tested gels, which indicates strength of internal bonds in product. Resilience of the tested carbomer gels is portrayed in Figure 5, while springiness of the tested products was presented in Figure 6. All graphs (Figures 1 - 6) show the measured values for gels with different gelling agent concentration and their changes during time (1 week after gel formulation, after the period of 2 weeks, after 3 weeks, and after 1 month of product formulation).

Sensory analysis of gels

Most of the panelists (56.25%) have chosen Sample 2 as favorite and as a gel they could regularly use. A second favorite was Sample 1 (31.25%). No one chose Sample 4, the gel with the highest carbomer concentration.

Table 3 shows the most common answers of the questionnaire with the percentage of respondents who rated the gels (sample 1-4) in this manner. All gels were rated as semi-solid, shiny, easy to spread and not greasy. Other characteristics differed among the samples. The gel with the highest gelling agent concentration showed the slowest absorption rate, while density increased with the increase in carbomer concentration. Also, the stickiness increased with the concentration of a gelling agent.

Table 3. *The results of sensory analysis of investigated gels*

	Sample 1	Sample 2	Sample 3	Sample 4
Before rubbing				
Consistency	semi-solid	semi-solid	semi-solid	semi-solid
	100%	100%	100%	100%
Gloss level	gloss	gloss	gloss	gloss
	68.80%	43.80%	43.80%	62.50%
During application				
Spreadability	easy to spread	easy to spread	easy to spread	easy to spread
	100%	100%	81.30%	68.80%
Adhesion	not sticky	not sticky	slightly sticky	slightly sticky
	87.50%	62.50%	68.80%	68.80%
Density	slightly dense	slightly dense	dense	dense
	62.50%	62.50%	68.80%	56.30%
Grease	not greasy	not greasy	not greasy	not greasy
	93.80%	56.30%	50.00%	50.00%
Gloss	shiny	slightly shiny	slightly shiny	slightly shiny
	50%	56.30%	62.50%	62.50%
Absorption rate	fast	fast	moderate	slow
	50%	50.00%	50.00%	37.50%
After application				
Residual film	no film	no film	moderate film	moderate film
	68.80%	56.30%	68.80%	68.80%
Stickiness	not sticky	not sticky	slightly sticky	slightly sticky
	87.50%	68.80%	66.70%	56.30%
Gloss	not shiny	not shiny	slightly shiny	slightly shiny
	56.30%	50%	62.50%	62.50%

DISCUSSION

Hydrogels are widely used semi-solid preparations. As 3D networks of water-soluble polymers, they can bind big amount of water and are biocompatible. Thus, due to their positive characteristics, they are commonly used in skin care cosmetic formulations (14). Carbomer, polymer of acrylic acid, is one of the most popular gelling agents, because it can be used at low concentrations for preparing bioadhesive and thermostable hydrogels, compatible with many active ingredients. However, for making carbomer gels, appropriate pH needs to be set, in order to enable polyacrylic acid to reach the anionic form (15). For that purpose, 10% NaOH was used. Moreover, the active substance itself – panthenol, showed basic properties and acted as a carbomer neutralizing agent.

Texture analysis provides instrumental information on some structure properties of products. Results of texture analysis are important for the development of topical dosage forms, because the patient acceptability of the product depends mostly on its application and organoleptic attributes. Whether the product can be easily removed from the container or can be easily spread on the skin can significantly affect compliance and the choice of preparation (1). These parameters can also be expressed in sensory analysis, where a group of people evaluate organoleptic and tactile characteristics of the product while answering the questions in a specially designed questionnaire (16).

Hardness is defined as a force required for deformation, and is measured as the maximum force on the texturometer force vs. time curve (13). Hardness cycle 1 represents the peak force during the first compression cycle, while hardness cycle 2 corresponds to the second peak force. In the Figures 1 and 2, it could be seen that the hardness of the gels was increasing with the carbomer concentration, which is consistent with data from literature (17). Also, there was an increase in the hardness over time, so it could be said that the strength of gel structure increased over time. Moreover, the gel network structure did not weaken between two compression cycles, which can be seen after comparison between hardness cycle 1 and hardness cycle 2 values. Hardness represents the force required to fully compress a product between the thumb and forefinger and indicates the applicability of the gels to the skin (18). Gels with higher gelling agent content are therefore harder to rub on the skin. Sensory analysis confirmed this finding – while 100% of panelists said that samples 1 (0.5% of carbomer) and 2 (1.0% of carbomer) were easy to spread, only 81.30% and 68.80% of panelists described sample 3 (1.5% of carbomer) and sample 4 (2.0% of carbomer), respectively, as easy to spread.

Adhesiveness is calculated as a negative force area between the first and second compression cycles. It is an indicator of the work required for overcoming attractive forces between the surface of the probe and the product; therefore, it signalizes the stickiness of the gel to the skin or the degree to which the skin is held back when fingers are separated during rubbing the product between them (18, 19). Adhesiveness of our gels ranged from 0.37 to 0.93 mJ and it did not differ much among different samples and over the time. Figure 3 shows that adhesiveness of the sample 3 and sample 4 was very much alike, which was also confirmed by panelists – 68.8% of them described both of these gels as slightly sticky. However, the adhesiveness of sample 1 and sample 2 did not correlate with panelists' opinion.

On the other hand, cohesiveness is a parameter that points out the stamina of internal bonds in the product (13). It is the work required to obtain deformation of the product when the probe is moved down (1). In the TPA, cohesiveness is calculated as quotient of areas under the curve in the second and first compression cycle (13). Cohesiveness of our gels did not demonstrate changes dependent neither on gelling agent concentration nor time (Figure 4). All the calculated values were close, in the range 0.67 - 0.89. This shows that the gel network was not broken during both compression cycles. These 'strongly structured gels' are thought to be easy for administration. Also, the absence of a change in cohesiveness over time indicates the stability of the gels (17). Other authors reported different observations considering cohesiveness of carbomer gels – in some cases cohesiveness decreased with increasing in carbomer concentration (20 - 22), in others, the situation was the opposite (22, 23), while Calixto et al. reported that cohesiveness did not change significantly with carbomer concentration (17).

The highest elastic resistance to deformation refers to the product with the highest consistency (24). Resilience and springiness are resembling but not the same parameters. Resilience indicates product's tendency to return to the initial position after deformation and is calculated as ratio of upstroke and downstroke energies of the first compression cycle. On the other hand, springiness is estimated after the second compression cycle and represents the quotient of a product's height after second cycle compared to the initial height. While resilience refers to the energy needed for elastic recovery, springiness denotes the height of the product before and after deformation (25). Sensory estimation of our gels showed that samples 1 and 2 were described as slightly dense, while samples 3 and 4, with the highest concentration of carbomer, were described as dense. All samples were characterized as semi-solid. In figure 5, it can be seen that resilience was the concentration dependent and that it grew over time. It means that more energy was needed e.g. for sample 4 with 2% of carbomer for elastic recovery after compression, than for sample 1 with 0.5% of the thickening agent. In addition, as far as all samples are concerned, it was more difficult to return to the original state over time. Figure 6 shows that regardless of the concentration of the gelling agent, no significant changes in springiness occurred, i.e., after deformation, all gels returned to approximately the same height in the cup, compared to the initial one.

According to the majority of panelists, sample 2, with 1% carbomer gel, was the most preferred one. This gel was marked with the following attributes: semi-solid, shiny, easy to spread, not sticky, not greasy, and slightly dense during application, with fast absorption rate and with no residual film after rubbing (Table 3).

CONCLUSION

Texture analysis, along with sensory evaluation, is a powerful tool in product design. Textural and sensory analysis of carbomer gels (carbomer concentration 0.5-2%) with 3% panthenol as active substance showed that the best properties are prescribed to the gel with 1% carbomer. In our study, hardness and resilience showed to be concentration-dependent texture parameters, while adhesiveness, springiness and cohesiveness were almost unchanged during the 4-week period, which indicates long-term stability of the gels. Both texture and sensory parameters are important from the aspect of consumer compliance. The next step would be to evaluate bioadhesiveness of the formulations and to perform *in vivo* skin bioengineering experiment.

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Teksturna i senzorna karakterizacija karbomernih gelova sa pantenolom

Milica Martinović, Nenad Stojanović, Ivana Nešić

Univerzitet u Nišu, Medicinski fakultet, Katedra za farmaciju, Niš, Srbija

SAŽETAK

Uvod. Cilj ove studije bio je da se proceni uticaj koncentracije karbomera, gelirajućeg sredstva (primenjenog u koncentracijskom rasponu 0,5% – 2,0%) na teksturne i senzorne karakteristike gelova sa 3% pantenola, pomoću senzorne analize, kao subjektivne i teksturne analize, odnosno kao objektivne metode procene. Aktivna supstanca u gelovima bio je pantenol, koji je takođe delovao kao sredstvo za neutralizaciju karbomera.

Materjal i metode. Dvadeset panelista ženskog pola, starosti između 20 i 36 godina, učestvovalo je u senzornoj studiji, koja je podrazumevala popunjavanje upitnika u kojima su se nalazila pitanja u vezi sa aplikativnim svojstvima gelova pre, tokom i nakon nanošenja na kožu. Analiza teksture izvršena je na CT3 ***Texture Analyzeru,*** **nakon svake nedelje tokom mesec dana. Izmereni su sledeći parametri: čvrstoća nakon prvog ciklusa, čvrstoća nakon drugog ciklusa, kohezivnost, adhezivnost, rezilijentnost i elastičnost.**

Rezultati. Rezultati su pokazali da su tokom trajanja studije gelovi bili stabilni i da je struktura gela ostala očuvana bez obzira na kompresiju. Povećanje tvrdoće i rezilijentnosti pratilo je povećanje koncentracije karbomera, dok se adhezivnost, kohezivnost i elastičnost nisu mnogo razlikovale među ispitivanim gelovima. Isti trend primećen je i kada su gelovi upoređivani tokom vremena. Senzorna analiza potvrdila je nalaze teksturne analize i pokazala da se sa porastom koncentracije karbomera, lepljivost i gustina takođe povećavaju, dok se brzina apsorpcije i razmazivost smanjuju. Većina panelista odabrala je gel sa 1% karbomera kao gel za redovnu upotrebu.

Zaključak. Teksturne i senzorne metode predstavljaju dragoceni pristup prilikom dizajniranja lekova/kozmetičkih proizvoda, jer pružaju uvid u komplijansu pacijenata/korisnika.

Ključne reči: **karbomer, pantenol, gel, analiza teksture, senzorna analiza**