

## Textural attributes of mixed whey proteins and carrageenan gels

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The sensory and rheological textural characteristics of heat induced and cold-set mixed whey proteins (WP) – carrageenan (CAR) gels were investigated. Mixed gels containing from 3.0 to 6.0 % WP and from 1.0 to 2.0 % of dry CAR were made. Sensory and instrumental texture characteristics of mixed gels were determined. A list of 15 sensory visual and in-hand attributes of the gels was formed. Panelists perceived textural differences in gels more sensitive by fingers in comparison with those obtained by spoon testing. It was found that sensory differences were in agreement with differences revealed using instrumental techniques.

The overall sensory texture of the mixed gels was highly dependent on the gel structure. Composition of mixed WP-CAR gels was the main factor producing the textural differences between heat induced and cold-set gels, while the method of gels production was a secondary factor.

**Keywords:** whey protein, carrageenan, mixed gels, texture, sensory analysis, instrumental evaluation.

### Introduction

Textural attributes are important factors for quality and consumers acceptance of the product [1]. Understanding the textural properties of a food product can be achieved by examination of its rheological behavior and its microstructure, so long as the instrumental information can be related to the perception of texture by consumers [2].

Mixed whey proteins and polysaccharide gels are of particular interest since synergism between these biopolymers can lead to a microstructure very different from that of pure gels and thus to related texture that can improve product quality. In recent years much work has been done on the gelation of whey proteins (WP) in combination with polysaccharides (PS), focusing mainly on the effect of starch or starch derived gelling polysaccharides [3–5], carrageenans [6–13] and pectin [14, 15]. In these studies heat induced gels were made from the mixed aqueous solutions of WP and PS by heating them to a temperature where the globular protein molecules are partially unfolded and then aggregated to form a three-dimensional network and after that cooling them to the room temperature where PS molecules form a network and entrap water through the capillary forces.

More recently an alternative method for the whey proteins gelation has been introduced that allows production of whey protein gels at low temperatures [16]. Whey proteins solution is heated to a temperature where they partially unfold. However, the pH, mineral content and proteins concentration are carefully controlled so that the proteins do not gel. Under these conditions the proteins undergo a limited extent of aggregation to form thin filamentous structures. Cold gelation is initiated upon reduction of the electrostatic repulsion between the protein molecules, either by increasing the mineral content or by adjusting the pH [17]. The mechanism of cold gelation of whey proteins have been reported and understood quite well. However, we did not succeed in finding reports on the characterisation of mixed gels made from unfolded WP solutions and polysaccharides where only cold gelation is initiated.

The objective of this study was to evaluate the sensory and instrumental textural characteristics of heat induced and cold-set gels made from the blends of whey proteins and carrageenan containing different amounts of biopolymers. Also, the links between the sensory textural characteristics values and instrumental data were investigated.

## Materials and methods

The WPI (Lacprodan DI-9224, Arla Food Ingredients, No. Vium, DK) contained 93.5 % protein, <4.5 % ash, <0.2 % fat, <0.2 % lactose and <6 % moisture (manufacturer's specifications). The CAR (Danisco Cultor, Århus, DK) had a pH value of 7.0–8.0 in a 1 % solution and contained 10 % moisture (manufacturer's specifications).

**WP solution.** Stock solutions of WP (8 %) were prepared in distilled water at room temperature. The pH was adjusted to 6.2 using 1M HCl, 4M HCl or 0.1N NaOH solutions.

**Preheated WP solution.** WP solution (8 %) was heated in an 80 °C water bath for 30 minutes. Immediately after heating the solution was cooled with tap water and stored at 5 °C overnight.

**CAR solution.** Stock solutions containing 3 % (w/w) CAR were prepared separately with distilled water by stirring at 50 °C for 5–6 h, followed by cooling to 20 °C. Adjustment of pH was made as for the WP solutions.

**Gel preparation.** For heat induced gelation WP and CAR stock solutions were mixed in different ratios to obtain WP-CAR solutions with final concentrations from 3.0 to 6.0 % with regards to whey protein and from 1.0 to 2.0 % in terms of dry polysaccharide. Compositions of mixed gels were chosen according to the literature data [6, 7] seeking to obtain mixed gels at the concentration of WP lower than 8 %, i. e. critical concentration of WP gelation [13]. pH of the mixtures were checked and, when necessary, adjusted to pH 6.2. The WP-CAR mixtures were equilibrated at room temperature for 12 h. After that mixtures of WP and CAR were placed into 50 ml beakers and heated in an 80 °C water bath for 30 minutes. Immediately after heating the solution was cooled with tap water and stored at 5 °C overnight.

For cold-set gelation preheated WP stock solution and CAR stock solution were mixed in the same ratios as in the case of heat induced gelation at 45 °C, incubated at this temperature for 30 minutes and then cooled with tap water and stored at 5 °C overnight.

**Gel hardness.** Gel hardness was determined by the penetrometer. The gels formed in the beaker were penetrated with a cylinder probe of 12 mm diameter. The depth of probe penetration after 10 sec was registered.

**Rheological measurements.** The viscoelastic properties during thermal gelation were monitored using a controlled stress rheometer *CSL 50* (Carri-Med Ltd, UK). The complex modulus,  $G^*$ , defined as  $\sqrt{G'^2 + G''^2}$ , where  $G'$  is the storage modulus

and  $G''$  the loss modulus, was used as an indicator of the total stiffness of the gels and the phase angle,  $\delta$ , where  $\tan(\delta) = G''/G'$ , was used to characterize the balance between viscous and elastic properties of the gel.

The mixed WP-CAR solutions (13 ml) were transferred to the measuring system (Couette geometry, diameter 25 mm). Dynamic oscillatory measurements were performed at a frequency of 1 Hz and a strain of 0.02. The sample was heated from 20 °C to 80 °C with a gradient of 2 °C/min, kept for 30 min at 80 °C, cooled with a gradient of 1 °C/min from 80 °C to 5 °C and held at this temperature for 30 min.

**Sensory evaluation.** The Texture Profile Method described by Lawless and Heymann [18] was used to evaluate the sensory texture attributes of the gels. Sensory evaluation was carried out by a trained panel of 8 judges, aged from 30 to 55, consisting of the staff of Food Institute of Kaunas University of Technology. The panelists were selected and trained according to ISO 8586. All of them had experience in descriptive analysis of different food products. The panelists participated in two orientation and training 2 hours sessions. In session one the list of descriptors only of non-oral textural attributes for the gels was suggested for panelists and they were asked to use them. In case of need, additional descriptors for evaluating textural attributes of presented samples they could use. Evaluation of the samples was conducted by penetrating the gel with plastic teaspoon and by testing the gel by fingers. Samples used for training were prepared in a laboratory in the same manner as for the assessment. During a succeeding round table discussion period the panelists worked as a group to generate definitive attributes to describe the complete texture profile of the WP-CAR gels. In total, a list of 15 textural attributes (Table 1) was developed. In the final training period panellists were familiarised with a line scale usage to quantify tested attributes and practised using the descriptors developed. The unstructured 15 cm line scale, labelled at each end with either “not” or “very” of the attribute was used for evaluating the samples. Each attribute was rated on a separate ballot.

All testing and training sessions were conducted in a climate-controlled sensory analysis laboratory, equipped with individual testing booths. Panelists were provided with water at 30–35 °C temperature and with napkins for cleaning hands and fingers.

Samples were presented to panelists in individual plastic beakers placed in cooled *Petri* plate to keep gel temperature low and uniform during testing. They were randomly presented to the judges

at each session and evaluated over 3 sessions held on separate days. Each judge evaluated each sample 4 times over 3 sessions.

**Data analysis.** Statistical analysis was performed using SPSS software (v. 11.0, SPSS Inc. Chicago, IL). Results were studied using the analysis of variance (ANOVA). A two-way analysis of variance was used for testing possible interactions in the sensory data. Independent variables were concentrations, treatment and panelists; dependent

variables were all sensory attributes [19]. Treatment means were compared using the Duncan's comparison. Differences were considered significant at  $p < 0,05$ .

## Results and discussion

### Sensory evaluation

Definitions of the evaluated attributes are presented in Table 1.

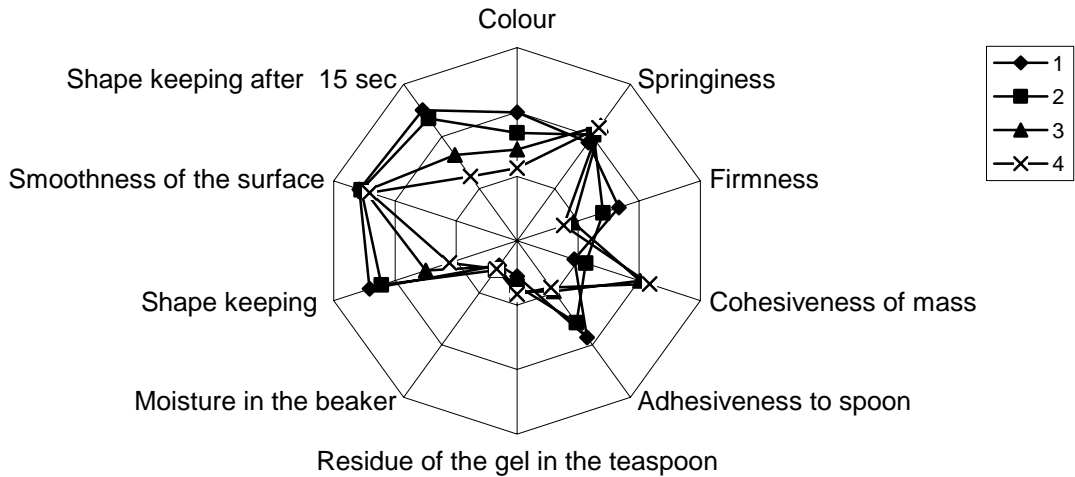
**Table 1.** Texture attributes and definitions of WP-CAR gels

Attribute	Definition
<i>Texture attributes, perceived by penetrating the gel with a plastic spoon</i>	
Springiness	Degree to which the sample returns to the original shape after partial compression with spoon
Firmness	Force required to fracture the sample in the beaker with spoon
Cohesiveness of mass	Degree to which the sample mass stays together as appropriate amount of gel is taken out from beaker by spoon
Adhesiveness to spoon	Degree to which the sample moves (or falls down) in teaspoon by tilting spoon 45°
Residue of the gel in the spoon	Amount of the gel in the spoon after it fall down from the spoon
Moisture in the beaker	Amount of the liquid exuded in the beaker after part of the gel is removed by spoon
Shape keeping	Degree to which the sample retains its initial shape after putting it in the plate
Smoothness of the surface	Visual perceiving of the shape and size of the particles on the surface of the gel
Shape keeping after 15 sec	Degree to which the sample keeps its initial shape in the plate after 15 sec
<i>Texture attributes, evaluated by fingers</i>	
Smoothness of the surface	Degree to which the surface of the gel feels smooth after putting it between thumb, first and middle fingers
Springiness	Degree to which the sample returns to the original shape after partial compression with fingers
Firmness	Force required to compress the sample using thumb and middle finger
Smoothness of the mass	Degree to which the mass of the gel feels smooth after compressing it between thumb, first and middle fingers
Adhesiveness on the fingers	Amount of the residual gel stock onto the fingers after evaluation of the smoothness
Moisture in the hand	Amount of the liquid observed around the gel when piece of the gel is placed in the hand

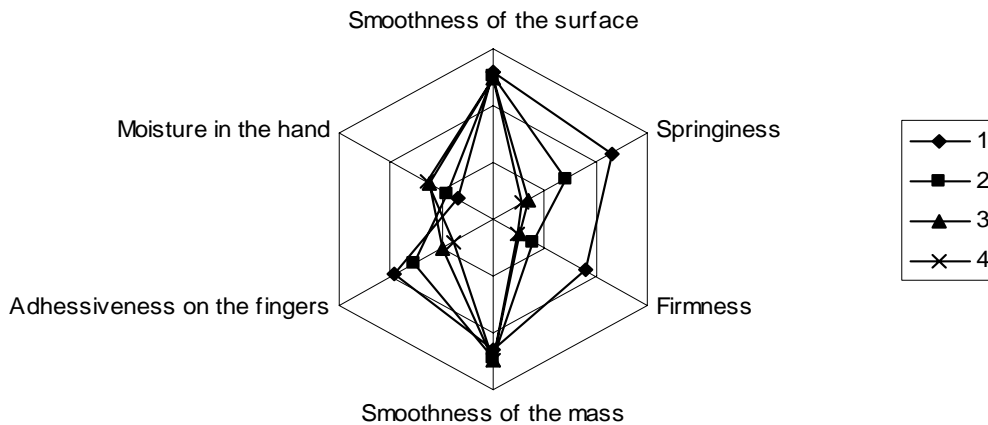
The mapping of the mean values of visual and tactile textural attributes evaluated for mixed WP-CAR gels containing different amount of WP and produced by heating is summarized in the radar plots in Figures 1 and 2. In general, increase in WP content of mixed gels caused an increase in shape keeping offhand and after 15 sec, adhesiveness to spoon, firmness and springiness by fingers, adhesiveness on the finger and decrease of the amount of the moisture in the hand. From the sensory maps it is noticeable that gels made with 5 and 6 % of WP were fairly similar when the overall

sensory attributes are considered. Even so these two samples were significantly different ( $p < 0,05$ ) in all attributes evaluated by fingers (springiness, firmness, adhesiveness). Gels with 4 and 3 % of WP were also quite similar. Meanwhile, the shape keeping was significantly higher with increasing of WP content.

There were no differences in springiness evaluated by spoon for samples with different WP content, but panel found significant differences between gels in springiness and in firmness by evaluation by fingers.



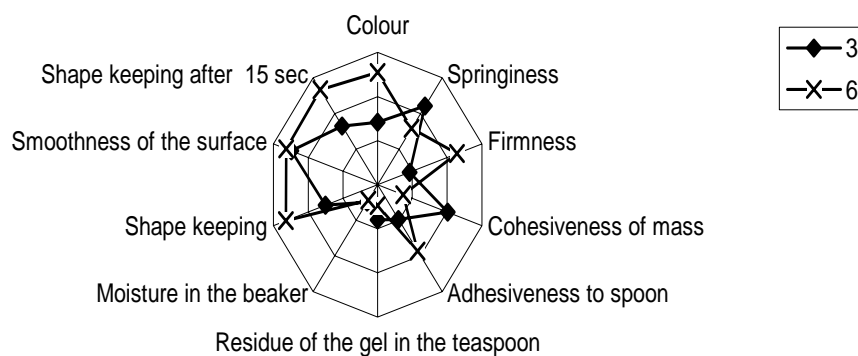
**Fig. 1.** Sensory maps (visual attributes) for heat induced mixed WP-CAR gels with 1 % of CAR and different WP content: 1 – 6 % WP; 2 – 5 % WP, 3 – 4 % WP, 4 – 3 % WP



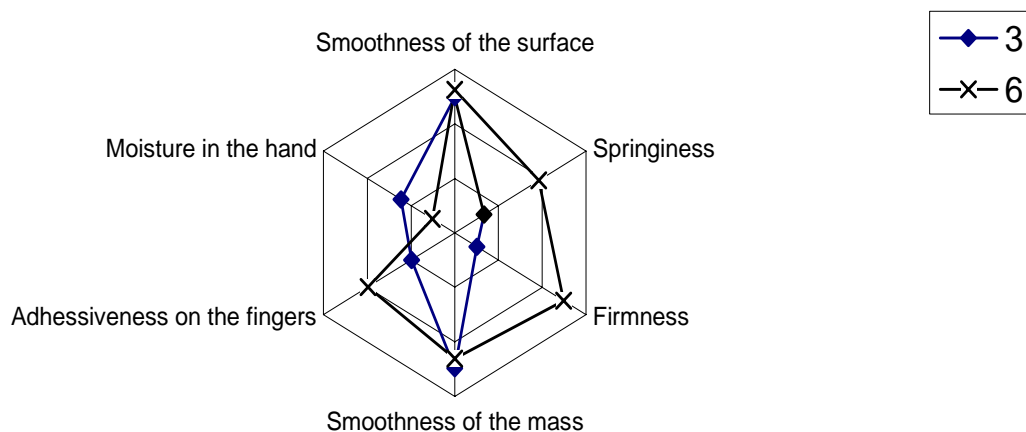
**Fig. 2.** Sensory maps (tactile attributes) for heat induced mixed WP-CAR gels with 1 % of CAR and different WP content: 1 – 6 % WP; 2 – 5 % WP, 3 – 4 % WP, 4 – 3 % WP

For gels made with constant content (3 %) of WP and increasing CAR content from 1 to 2 % (Figures 3 and 4) significant differences ( $p < 0,05$ ) between the gels were visible for almost all attributes except syneresis in beaker, smoothness by fingers and cohesiveness of the mass. The increasing in CAR content caused high values of shape keeping offhand and after 15 sec, adhesiveness to spoon, firmness by fingers, springiness by fingers, adhesiveness in hand and small values of moisture in hand.

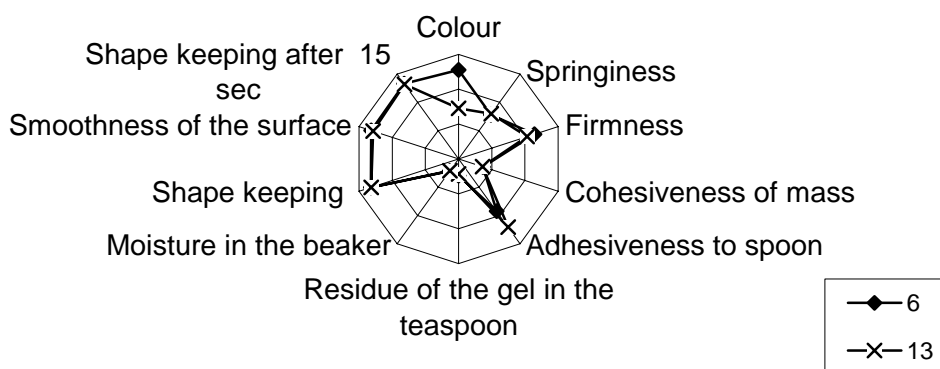
No significant difference ( $p > 0,05$ ) between texture attributes for cold-set gels and gels induced by heat was detected. Figures 5 and 6 shows the mean values of textural attributes of mixed WP-CAR gels, containing 3 % WP and 2 % CAR and different methods of preparation. Similar results were obtained for the gels with other concentrations of WP and CAR, except those containing 1 % of CAR and 5 or 6 % of WP. The mean values of firmness, springiness and shape keeping of cold-set gels were higher than for these attributes of gels induced by heat.



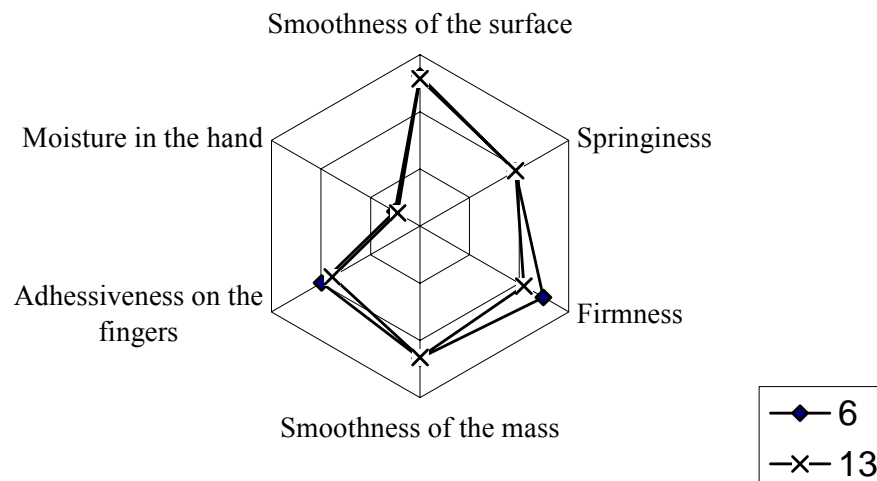
**Fig. 3.** Sensory maps (visual attributes) for heat induced mixed WP-CAR gels with 3 % of WP and different CAR contents: 3 – 1 % CAR; 6 – 2 % CAR



**Fig. 4.** Sensory maps (tactile attributes) for heat induced mixed WP-CAR gels with 3 % of WP and different CAR contents: 3 – 1 % CAR; 6 – 2 % CAR



**Fig. 5.** Sensory maps (visual attributes) of mixed WP-CAR gels containing 3 % of WP and 2 % CAR: 6 – heat induced; 13 – cold-set



**Fig. 6.** Sensory maps (tactile attributes) of mixed WP-CAR gels containing 3 % of WP and 2 % CAR: 6 – heat induced; 13 – cold-set

All tested gels were characterized as being opaque and having good water holding properties. These observations are consistent with other reports on physical properties of whey proteins gels [20].

All sensory attributes could be evaluated both by spoon and by fingers. However, it can be predicated that by fingers it is possible to perceive more exactly textural differences in gels than with spoon.

#### *Rheological evaluation*

The rheological parameters obtained by large and small deformation tests are shown in Table 2. It

was observed that WP concentration in mixed gels had effect on the hardness, expressed as complex modulus  $G^*$ , of heat induced and cold-set gels. Absolute values of this parameter seem to suggest that mixed gels made with higher content of WP (6 and 5 %) when the content of CAR was 1 %, were harder than those with WP content 4 and 3 %. However, mixed gels made from 4 % of WP and 2 % of CAR showed the highest values of this parameter. Results also showed that  $G^*$  of cold-set gels was higher than that of heat induced gels at the same concentration of WP.

**Table 2.** Rheological characteristics of heat induced and cold-set gels with different contents of WP and CAR

Composition of gels: WP/CAR	Heat induced gels				Cold-set gels			
	$G^*$ , Pa	$G'$ , Pa	P, 1/mm	Syneresis, %	$G^*$ , Pa	$G'$ , Pa	P, 1/mm	Syneresis, %
6/1	1789	52,0	0,0128	35,8	1918	59,5	0,0130	31,8
5/1	1611	37,2	0,0120	32,9	1770	52,0	0,0125	35,4
4/1	1137	29,7	0,0112	38,4	1310	37,2	0,0120	38,1
3/1	869	22,3	0,0101	48,5	1135	29,7	0,0106	42,8
3/2	2847	74,3	0,0192	30,1	3014	81,7	0,0220	29,8
4/2	3281	89,1	0,0208	24,2	3536	96,6	0,0240	20,4

The results of large deformation test indicated the same tendencies as in the case of small deformation tests, i. e. the sample resistance to penetration increased as the WP concentration in

mixed gels increased and was higher for the cold-set gels.

The increase in CAR content from 1 to 2 % in mixed gels containing WP content constant 3 or 4 %

caused the increase in  $G^*$  value and resistance to penetration.

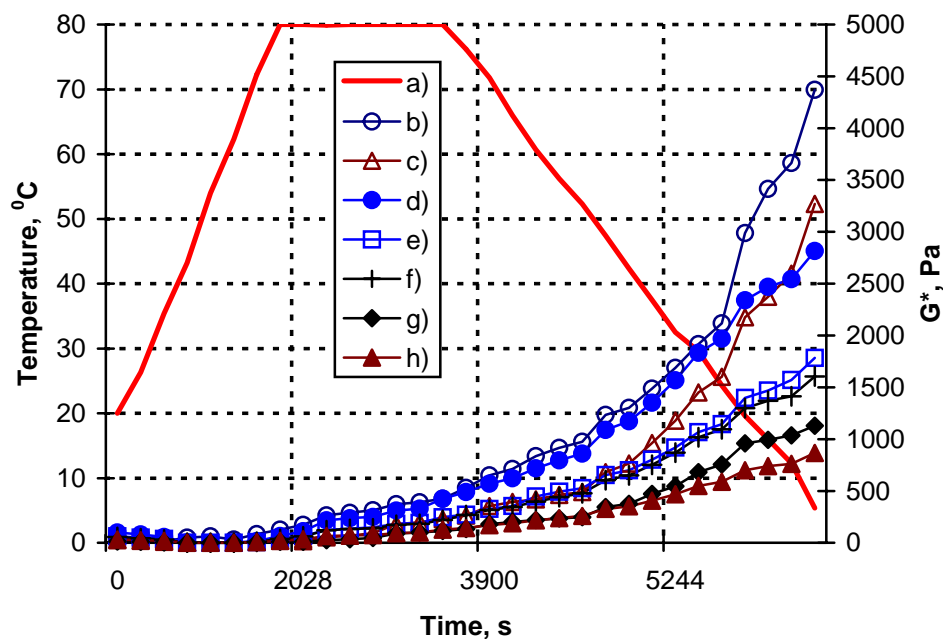
The hardness of mixed gels measured instrumentally and assessed by the sensory panel (firmness by spoon and firmness by fingers) showed similar trends for gels made with different content of WP and CAR. However, because of the similarity of the sensory evaluation results between two types of mixed gels produced by heat and cold-set gels, a new procedure should be investigated.

Among the instrumental parameters chosen for the characterisation of the gels in this study  $G'$  was suggested as parameter characterizing elasticity of gels. Results showed that gels became considerable less elastic with the increase of WP in the gels and more elastic when the content of CAR was higher. The cold-set gels in all cases were more elastic in comparison with heat induced gels containing the same amounts of WP and CAR. These results can be related with the springiness of gels observed by sensory assessment. The springiness of gels measured by fingers showed the same tendencies,

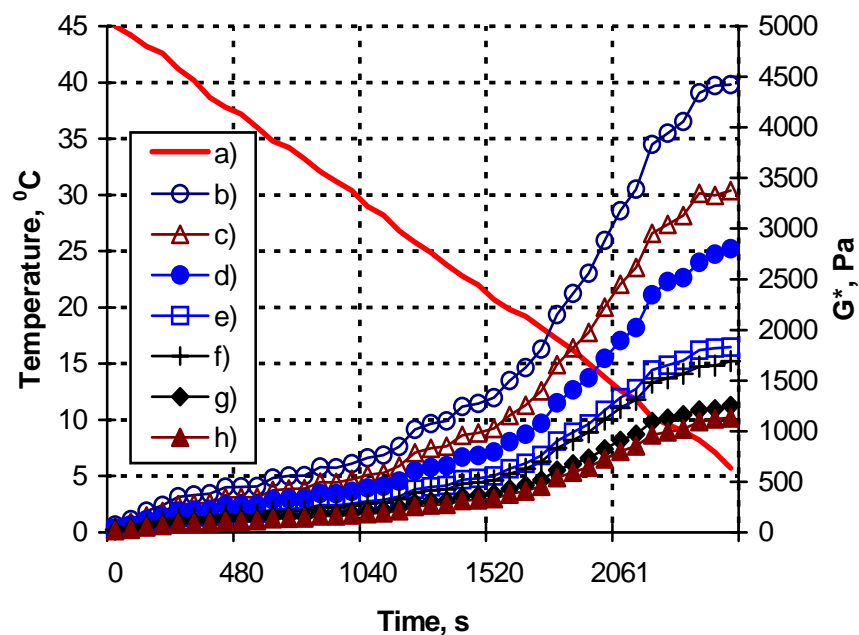
however no differences were observed among the results of springiness by spoon.

Syneresis of both kinds of mixed gels (heat induced and cold-set) decreased with the increasing WP content in gels. Likewise, for heat induced gels whey separation was higher in comparison with cold-set gels. Results of syneresis measured instrumentally are in agreement with those obtained in the sensory evaluation of gels (moisture in the hand).

The gelation kinetics (Fig. 7 and 8) of mixed WP-CAR gels in the presence of various WP and CAR concentrations were studied through the evaluation of  $G^*$ . In the case of heat induced gels, the sample was heated from 20 to 80 °C with a gradient of 2 °C/min, kept for 30 min at 80 °C, cooled with a gradient of 1 °C/min from 80 °C to 5 °C and held at this temperature for 30 min. The kinetics obtained for formation of mixed gels induced displayed a lag phase, as exponential growth phase and a not significant plateau region during cooling stage.



**Fig. 7.** Evaluation of the complex modulus  $G^*$ (Pa) as a function of time for heat induced mixed WP-CAR gels containing different concentrations of WP and CAR: a) temperature, °C; b) WP 2 % and CAR 3 %; c) WP 4 % and CAR 2 %; d) WP 3 % and CAR 2 %; e) WP 6 % and CAR 1 %; f) WP 5 % and CAR 1 %; g) WP 4 % and CAR 1 %; h) WP 3 % and CAR 1 %



**Fig. 8.** Evaluation of the complex modulus  $G^*$ (Pa) as a function of time for cold-set mixed WP-CAR gels, containing different concentrations of WP and CAR: a) temperature, °C; b) WP 2 % and CAR 3 %; c) WP 4 % and CAR 2 %; d) WP 3 % and CAR 2 %; e) WP 6 % and CAR 1 %; f) WP 5 % and CAR 1 %; g) WP 4 % and CAR 1 %; h) WP 3 % and CAR 1 %

Fig. 8 described  $G^*$  values measured versus cooling time from 45 to 5 °C for cold-set mixed gels. The absence of plateau phase and the slow increase of exponential growth were observed. The rheological parameters obtained by large and small deformation tests are shown in Table 2. It was observed that WP concentration in mixed gels had effect on the hardness, expressed as complex modulus  $G^*$ , of heat induced and cold-set gels. Absolute values of this parameter seem to suggest that mixed gels made with higher content of WP (6 and 5 %) when the content of CAR was 1 %, were harder than those with WP content 4 and 3 %. However, mixed gels made from 4 % of WP and 2 % of CAR showed the highest values of this parameter. Results also showed that  $G^*$  of cold-set gels was higher than that of heat induced gels at the same concentration of WP.

### Conclusions

1. Heat induced and cold-set mixed WP-CAR gels at different WP and CAR concentrations can be texturally discriminated using a trained sensory panel and visual and in-hand attributes.
2. Panel perceived textural differences in gels more sensitive by fingers in comparison with those obtained by spoon testing. Sensory differences were in agreement with differences revealed using instrumental techniques.

3. Composition of mixed WP-CAR gels is the main factor producing the textural differences between heat induced and cold-set gels with method of production of gels as a secondary factor.

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## ISRŪGŲ BALTVMŲ IR KARAGENINO MIŠRIŲJŲ GELIŲ TEKSTŪROS SAVYBIŲ TYRIMAI

### Santrauka

Šio darbo tikslas buvo įvertinti išrūgų baltymų ir karagenino mišriųjų gelių tekstūros savybes juslinės analizės bei instrumentiniais metodais. Tirti geliai, pagaminti iš išrūgų baltymų ir karagenino tirpalų mišinių, kai baltymų koncentracija gelyje svyravo nuo 3 iki 6 %, o karagenino koncentracija keitėsi nuo 1 iki 2 %. Geliai gaminti „karštuuju“ ir „šaltuoju“ būdu. Mišriųjų gelių tekstūros savybės nustatytos jusliškai ir instrumentiniais metodais. Jusliškai gelių tekstūra tirta vizualiai ir rankomis, naudojant 15 deskriptorių. Nustatyta, kad tiek gelių sudėtis, tiek jų gamybos būdas turėjo įtakos mišriųjų gelių tekstūros savybėms. Pirštais vertintojai gebėjo jautriau įvertinti atskirų tekstūros savybių pokyčius, negu vertindami gelius šaukšteliu. Nustatyta, kad juslinės analizės rezultatai sutapo su instrumentinio vertinimo rezultatais analizuojant mišriųjų gelių stiprumą, elastingumą bei sinerezę.

**Raktažodžiai:** išrūgų baltymai, karageninas, mišrūs geliai, tekstūra, juslinė analizė, reologinis vertinimas.

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## ИССЛЕДОВАНИЕ ТЕКСТУРЫ СМЕШАННЫХ ГЕЛЕЙ ИЗ СЫВОРОТОЧНЫХ БЕЛКОВ И КАРАГЕНАНА

### Резюме

В статье представлены данные сенсорных и реологических исследований текстуры смешанных гелей, выработанных из сывороточных белков (СБ) и карагенана (САР). В ходе эксперимента концентрации СБ меняли от 3,0 до 6,0 %, САР – от 1,0 до 2,0 %. Гели выработывали холодным и горячим способами. Группа дегустаторов исследовала текстуру гелей визуально и пальцами, применяя 15 дескрипторов.

Установлено, что состав гелей, как и способ приготовления, оказали влияние на текстурные показатели смешанных гелей. При сенсорном анализе установлено, что пальцами дегустаторы могут более точно установить изменения текстурных показателей гелей по сравнению с исследованием гелей ложкой. Результаты сенсорного анализа соответствовали результатам реологических исследований при установлении твердости, эластичности и синерезиса смешанных гелей.