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ORIGINAL RESEARCH PAPER

EVALUATION OF THE STRUCTURAL-MECHANICAL PROPERTIES OF COMPOSITE PEAR JELLIES PREPARED WITH DIFFERENT GELLING TIME AND CALCIUM SALTS

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Abstract: The aim was to investigate the influence of different calcium salts and gelation times on the structural-mechanical parameters of a pear composite jellies. Calcium citrate and calcium lactate with a concentration of 0.6 % and 1.2 % were used as sources of calcium ions for the gelling time of 24 and 48 h. The changes in the structural-mechanical properties of the jellies were determined from the obtained typical curves through a penetration test using a texture analyzer. The increase in the concentration of calcium ions led to the increasing values of the structural-mechanical parameters of the fruit jellies, except for adhesiveness and rupture deformation. The shape of the penetration curves was determined to a greater extent by the type and concentration of calcium salts and to a lesser extent by the gelation time. The Pearson correlation coefficients for the compressive stress, deformation force and firmness increased compared to the other structural-mechanical properties with an increase in the gelation time and the concentration of calcium salts.

Keywords: *calcium salts, gelation time, pear jam, structural-mechanical properties*

INTRODUCTION

Water-soluble gums, also called hydrocolloids, are macromolecular proteins or polysaccharides that play a critical role for structure and stability to food [1]. Some hydrocolloids are widely used in the food industry as thickeners and gelling agents, stabilizers of foams and emulsions, inhibition of ice and sugar crystal formation, and controlled release of flavors [2 – 4]. Some gelling agents used alone do not form or make brittle gels, favoring the syneresis process. However, when they are combined with other hydrocolloids, these gelling agents form stable gels [5]. Gelling hydrocolloids form gels by physical crosslinking of their polymer chains, through hydrogen bonds, hydrophobic bonding or cation-mediated crosslinking. Therefore, very often hydrocolloid gels are called - "physical gels" [6]. The most commonly used polysaccharides as gelling agents in the preparation of fruit jellies, jams or gels are alginate, carrageenan and pectin. Alginates are polysaccharides obtained from brown seaweeds and some bacteria [7]. Alginate gelation occurs when sodium alginate in the presence of divalent calcium ions, interact with α-L-guluronic acid from the guluronic blocks of the alginates, resulting in a 3-dimensional network of gelling structures called an 'egg box' [8]. The structural-mechanical properties of alginate gels are related to the number and locations of cross-linked egg-box gelling structures. When their density increases, the higher the compressive stress increases also [9]. Alginates with higher guluronic acid content have a more rigid molecular structure, while those with higher mannuronic acid content have a more flexible structure [10]. Carrageenans are gel-forming or viscous polysaccharides extracted from the cell walls of red algae – *Rhodophyta* [11]. These polysaccharides are the group of sulfated galactans, with high degree of polydispersity and polymerization. They are composed of D-galactose units that are linked by a 1→3 *β*-D-galactose linkage and a 1→4 *α*-D-galactose linkage [12]. Carrageenans are divided into three different types, which are kappa (κ), iota (ι) and lambda (λ) carrageenan [13]. Iota-carrageenan forms soft and elastic gels that are resistant to freezing and thawing [14], and in the presence of calcium ions forms thermoreversible gels. Pectin is a water-soluble polysaccharide that is extracted from plant cell walls [15]. The main component of the building block of pectin is galacturonic acid, which can be partially methoxylated or amidated. According to the degree of methoxylation, pectins can be divided into two main groups - low methoxylated pectin with a degree of methoxylation below 50 % and high methoxylated pectin with a degree of methoxylation above 50 %. Pectins show versatile gelling properties and are capable of forming complexes with other natural compounds for design of food products. The structure-related mechanisms of pectin gelling and linking with cellulose, hemicellulose, ferulic acid, proteins, starch, and chitosan was demonstrated [16]. When highly esterified pectin is deesterified with the help of ammonium ions, a low methoxylated amidated pectin with a degree of esterification below 25 % is obtained [17]. Amidation of pectin is an important chemical modification that can improve the calcium-dependent gelling ability of pectin [18]. Amidated pectin forms thermoreversible gels with high stability and strength, and when the *p*H is low, this facilitates gel formation [19].

The aim of this research is to evaluate the influence of concentration of calcium salts (lactate and citrate) and gelation time on the structural-mechanical properties of composite pear jellies.

MATERIALS AND METHODS

Materials

The pear juice "Greno" was purchased from a local market (Plovdiv, Bulgaria). Sodium alginate (VIVAPUR Alginate FD 120) and low methoxylated amidated citrus pectin (Aglupectin LA-S10) with the degree of amidation 17 % and a degree of methylesterification 34 % were suppled from P.I.C.Co (Bulgaria). Iota-carrageenan and guar gum (used as thickening agent) were kindly delivered by Orion (Karlovo, Bulgaria). Cellulose fibers (Alba-Fibre, Mikro-Technik) with 99 % cellulose were also used to improved the rupture force and compressive strength of pear jams All other reagents and chemicals were supplied from Fillab Ltd. (Plovdiv, Bulgaria).

Methods

Preparation of composite fruit jellies from pears

The ingredients for the preparation of the pear composite jellies were given in Table 1. All the necessary substances (Table 1) were weighed in 50 mL beakers on an analytical balance. After that, they were mixed dry until a homogeneous mechanical mixture was obtained.

Table 1. *Component composition of samples*

Initial component composition (w/w)	**Sample № (Gelling time, [h])**			
	P01 (at 24 h) and P05 (at 48 h)	**P02 (at 24 h) and P06 (at 48 h)**	**P03 (at 24 h) and P07 (at 48 h)**	**P04 (at 24 h) and P08 (at 48 h)**
Sodium alginate, [%]	0.8	0.8	0.8	0.8
Iota-carrageenan, [%]	1.6	1.6	1.6	1.6
Low-esterified amidated pectin, [%]	0.6	0.6	0.6	0.6
Calcium citrate pentahydrate, [%]	0.6	1.2	-	-
Calcium lactate pentahydrate, [%]	-	-	0.6	1.2
Sodium citrate, [%]	1.0	1.0	1.0	1.0
Cellulose fiber, [%]	1.0	1.0	1.0	1.0
Guar gum, [%]	0.4	0.4	0.4	0.4
Pear juice, [%]	94.0	93.4	94.0	93.4

Finally the pear juice was added with up to 100 % (w/w). All the ingredients of the jellies were heated together with occasional stirring. It was boiled to a dry refractometric substance of 18 %, with the loss of water being from 4.5 to 7.4 % depending on the component composition of the samples. The obtained mixture was poured hot into glass Petri dishes with a diameter of 50 mm and a height of 15 mm. The samples were placed in a refrigerator for 1 hour to cool down. After cooling, the composite jellies were removed from the refrigerator and left for 24 or 48 h at room temperature.

Determination of mechanical properties

All mechanical properties were determined by penetration testing with a Stable Microsystems Texture Analyzer (TA.XT. plus Stable Micro Systems, The United Kingdom). The texture analyzer was operated in Y-axis uniaxial strain mode at 80 % compressive stress with a test speed of 2 $mm \cdot s^{-1}$ and a post-test speed of 2 $mm \cdot s^{-1}$ using a 5 mm diameter (P/5) aluminum cylindrical probe with an area of 19.634 mm^2. Each individual sample was measured 7 times. Rupture force and deformation were determined as the maximum value of the penetration curves obtained using the texture analyzer. Compressive stress is calculated by dividing the rupture force by the cross-sectional area of the piston at the rupture point [20]. Firmness is defined as the slope of the force-deformation curve, reported in $N \cdot mm^{-1}$ and reflects the apparent elastic modulus [21]. Rupture energy is calculated from the area under the force-deformation curve to the point of rupture [22]. Toughness is defined as the amount of energy that the material can absorb in a unit volume before breaking [23]. In this study, toughness was determined by a penetration test and is a ratio between the breaking energy and the volume of the product (fruit jelly). The volume of the material was calculated as the product of the pi number, the squared radius of the plunger that penetrates the material (the fruit jelly), and the rupture force, which is the height of the product when the plunger penetrates it before the material to tear. Young's modulus and adhesiveness was determined [24, 25].

Statistical analysis

The obtained data from the conducted studies were processed with MS Excel 2010 software. The obtained results when examining the type and concentration of calcium salts and the gelation time were processed statistically by means of a two-factor variance analysis with replication (ANOVA: Two Factor With Replication) at a level of statistical significance $\alpha = 0.05$, ($p < 0.05$). The correlation dependences between the structural-mechanical properties of the fruit jellies were determined by determining the sample correlation coefficient of Pearson. The statistical significance of the correlation coefficient (Pearson's sample coefficient - r) is determined by comparing the calculated sample coefficient with a critical value of the Pearson's coefficient (rcr.) at a given level (level) of statistical significance $\alpha = 0.05$; ($p < 0.05$) and degrees of freedom - f = n-2, where n - is the number of measurements (number of samples). If the determined Pearson coefficient in absolute value is equal to or greater than the critical value, the coefficient is statistically significant.

RESULTS AND DISCUSSION

Structural-mechanical properties of composite jellies

The data of the structural-mechanical properties of the fruit jellies as a function of the concentration and type of calcium salts and the structuring time were presented in Tables 2 and 3. It was found that when the concentration of the calcium salts (citrate and lactate) increased from 0.6 to 1.2 %, the rupture force of the samples increased to a minimal degree. This positive effect is further supported by the increasing gelling time. Increasing the concentration of calcium ions led to faster gelation and higher strength of calcium alginate and Ca-pectin gels [10].

Table 2. *Structural-mechanical properties of composite fruit jellies at 24 h gelation time*

Structural-mechanical properties	**Type and concentration of calcium salts**			
	Calcium citrate [%]		Calcium lactate [%]	
	0.6	1.2	0.6	1.2
	Sample №			
	P01	**P02**	**P03**	**P04**
Rupture force, [N]	0.18±0.01[a*]	0.21±0.01[b]	0.21±0.01[c]	0.26±0.01[d]
Rupture deformation, [mm]	3.25±0.16[a]	3.18±0.13[a]	2.49±0.11[b]	2.40±0.12[b]
Compressive stress, [kPa]	8.94±0.01[a]	10.62±0.01[b]	10.54±0.01[c]	13.06±0.01[d]
Firmness, [$N\cdot mm^{-1}$]	0.04±0.01[a]	0.05±0.01[b]	0.06±0.01[c]	0.09±0.01[d]
Rupture energy, [mJ]	0.39±0.03[a]	0.44±0.03[b]	0.34±0.02[c]	0.40±0.03[d]
Toughness, [$mJ\cdot cm^{-3}$]	6.20±0.54[a]	6.97±0.23[b]	6.97±0.16[c]	8.43±0.20[d]
Young's modulus, [kPa]	17.14±1.01[a]	19.14±0.74[b]	27.44±0.95[c]	33.16±2.09[d]
Adhesiveness, [$N\cdot s^{-1}$]	-0.16±0.01[a]	-0.13±0.01[b]	-0.23±0.01[c]	-0.21±0.01[d]

*Mean of seven measurements (n = 7) ± standard deviation. Different letters in each row indicate statistically significant differences ($p < 0.05$)

Table 3. *Structural-mechanical properties of composite fruit jellies at 48 h gelation time*

Structural-mechanical properties	**Type and concentration of calcium salts**			
	Calcium citrate [%]		Calcium lactate [%]	
	0.6	1.2	0.6	1.2
	Sample №			
	P05	**P06**	**P07**	**P08**
Rupture force, [N]	0.16±0.01[a]	0.19±0.01[b]	0.22±0.01[c]	0.27±0.01[d]
Rupture deformation, [mm]	3.26±0.04[a]	3.56±0.11[b]	2.53±0.09[c]	2.49±0.08[d]
Compressive stress, [kPa]	8.12±0.01[a]	9.52±0.01[b]	11.18±0.01[c]	13.74±0.01[d]
Firmness, [$N\cdot mm^{-1}$]	0.03±0.01[a]	0.04±0.02[b]	0.07±0.01[c]	0.09±0.02[d]
Rupture energy, [mJ]	0.36±0.01[a]	0.45±0.02[b]	0.35±0.02[c]	0.42±0.03[d]
Toughness, [$mJ\cdot cm^{-3}$]	5.63±0.09[a]	6.39±0.33[b]	7.07±0.15[c]	8.55±0.32[d]
Young's modulus, [kPa]	14.80±0.86[a]	17.47±0.62[b]	28.07±0.41[c]	30.80±0.49[d]
Adhesiveness, [$N\cdot s^{-1}$]	-0.16±0.01[a]	-0.15±0.01[b]	-0.22±0.01[c]	-0.19±0.02[d]

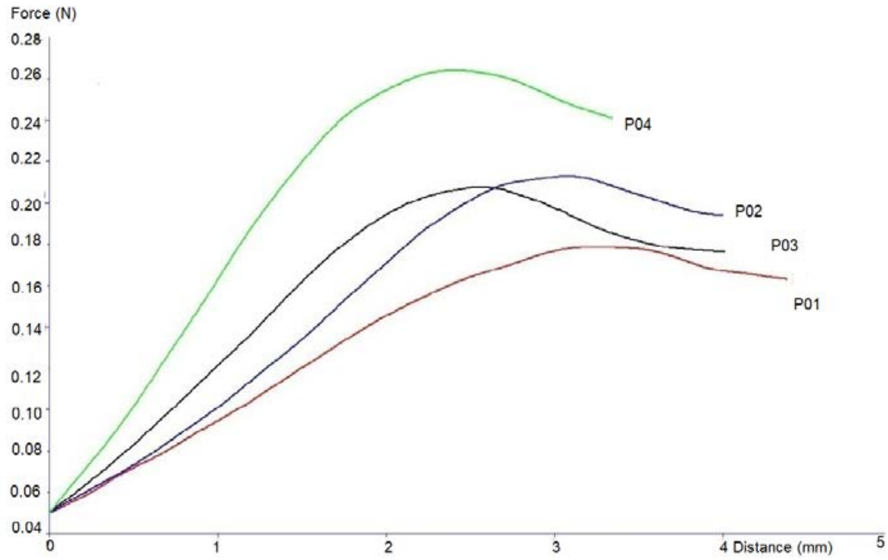
*Mean of seven measurements (n = 7) ± standard deviation. Different letters in each row indicate statistically significant differences ($p < 0.05$)

The sample P08 (Table 3) showed the highest compressive stress due (13.74 kPa) to the highest content of calcium ions (1.2 %) and the longest gelation time (48 h) among the other samples. A similar effect related to the increase in rupture forces a result of the increasing concentration of calcium ions was found in binary composite gels made of sodium alginate and guar gum [26]. The rupture force of fruit jellies was significantly reduced when there was the same concentration of added calcium salt (0.6 % or 1.2 %) but different type (citrate or lactate) at 24 h of gelation time. The same significant reduction in rupture force was observed with increasing calcium lactate concentration (P07 and P08) at 48 h of gelation. This effect is due to the concentration and type of calcium salt and gelling time that led to observation that the fruit jellies become more elastic that is due to the rupture deformation. Ruptue deformation increased statistically significantly only when calcium citrate concentration (P05 and P06) increased at 48 h gel time. The compressive stress increased with increasing concentration of calcium salts (citrate and lactate) both at 24 h and at 48 h gelling time. With increasing concentration of calcium citrate, the highest compressive strength was observed at 24 h of gelation (P02), and with respect to calcium lactate at 48 h (P08). The greatest values of rupture force and toughness are obtained (P02 - Table 2 and P08 - Table 3). The firmness of the fruit jellies increased the most and to the same numerical value ($0.09 N \cdot mm^{-1}$) at the highest concentration of calcium lactate (P04 and P08) regardless of the time of jelly formation. Fruit jellies with added calcium lactate (P03; P04 and P07; P08) always have greater firmness compared to those with added calcium citrate (P01; P02 and P05; P06), regardless of the gelation time. At 48 h of gelling time, it can be seen that with the same concentration of the calcium salt (0.6 % or 1.2 %), but different in type (P05 and P07 or P06 and P08), the firmness of the samples increased slightly more than twice, and at 24 h of gelation (Table 2) no such effect was found. As the concentration of added calcium citrate or calcium lactate increased, the rupture energy was observed to increase slightly at 24 h and 48 h gel time. A longer gelation time (48 h) increased the rupture energy of P07 and P08 with calcium lactate to a slightly greater extent in comparision to the same samples at 24 h gelation time. According to the data shown in Table 2, P06 had the highest rupture energy at 48 h of gelation, and P03 showed the lowest one at 24 h. This fact is due to the higher content of calcium ions in P06 and gelation time compared to P03. At higher calcium salt concentration or gelling time, it is observed that the fracture energy improved to a very small extent. From the data shown in Tables 2 and 3, an increase in the toughness of samples P01; P02 and P05; P06 and P03; P04 and P07; P08 can be observed, which is due to an increase in the content of calcium salts, respectively calcium ions. This observation is explained by the fact that when the content of calcium ions (calcium salts) increases, the rupture force increases, which makes the samples tougher. A similar increase in strength with increasing calcium ion concentration was observed for composite gels of sodium alginate and low-esterified apple pectin [27]. Regarding gelation time, the toughness of P01 and P02 decreased relative to P05 and P06, and that of P07 and P08 increased relative to P03 and P08 as the gelation time increased from 24 to 48 h. This could be explained by the fact that with a longer gelation time the rupture force becomes greater and vice versa. There was a strong correlation between the toughness and the compressive stress of the samples, which was confirmed by the high and statistically significant correlation coefficients ($r = 0.999/24$ h and $r = 0.998/48$ h) between these mechanical properties. The elasticmodulus of the jellies increased with increasing concentration of the added calcium salt. Due to the higher concentration of calcium ions in the calcium lactate-supplemented jellies (P03; P04 and P07; P08)

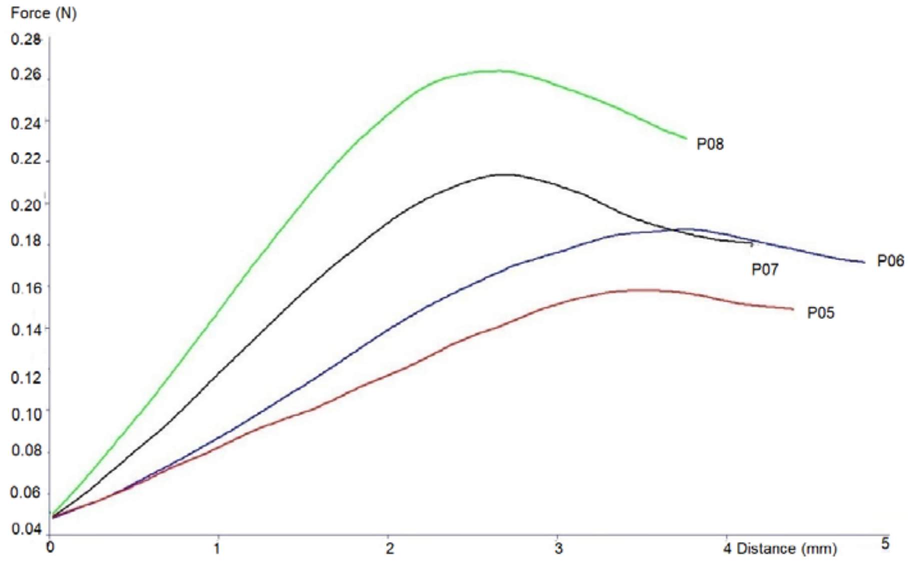
compared to those samples with calcium citrate (P01; P02 and P05; P06), higher values of Young's modulus were observed. A similar increase in elastic modulus with increasing calcium ion concentration was found in pectin-calcium gels [28]. As the gelling time increases from 24 to 48 h, the linear elastic modulus decreases in the gels with added calcium citrate (P01 and P02) and calcium lactate (P04). This reduction in Young's modulus (P01, P02 and P04) with increasing gelation time (from 24 to 48) indicates that these samples become softer, which is due to the higher rupture force. The opposite effect (increase in Young's modulus) was observed only for P03.
A similar effect associated with a decrease in adhesiveness when increasing the amount of calcium ions was found in mixed gels of kappa-carrageenan and cognac gum [29]. As the gelling time increased from 24 to 48 h, it was seen that the adhesiveness of P03 and P04 decreased relative to P07 and P08, and the adhesiveness of P02 increased relative to P06 or remained unchanged (P01 versus P05). The composite fruit jellies with added calcium lactate had the highest adhesiveness at 24 h of gelation time, and the samples with added calcium citrate at 48 h of gelation. The values obtained in this study for adhesiveness are similar or many times higher than the adhesiveness of pectin gel or raspberry jam [30].

Texture profile obtained by penetration test

The results (Figure 1 a and b) show the type and slope of the curves obtained from the penetration test through the texture analyzer of the composite jellies. Each of the obtained curves was characterized by a clearly expressed linear section, which corresponds to the elastic deformation, Young's modulus and the firmness of the samples.
None of the obtained penetration curves (Figure 1a,b) show a non-linear section, that is, plastic deformation before the maximum rupture force, which means that the jellies do not exhibit plastic properties, only elastic ones. As the concentration of a given type of calcium salt increases from 0.6 to 1.2 %, regardless of the gelling time, it can be seen that the slope of the curves for all composite gels increases, which means an increase in Young's modulus (elasticity) and firmness. The elastic modulus is a parameter that serves to determine the stiffness of the material [30], so samples with higher elastic modulus are stiffer.
From the data shown in Figure 1 a and b, it can be seen that at the same concentration of the calcium salts (P01; P03 or P02; P04) and (P05; P07 or P06; P08) a reduction in the length of the linear segment before the inflection is observed point (the rupture force) of all permeation curves regardless of gelation time. This fact is due to a reduction in the rupture force, which on the other hand results in a harder jelly-like structure. The rupture force increased in proportion to the increasing concentration of the calcium salts - citrate or lactate, i.e. when the concentration of calcium ions increased. A similar increase in gel strength was observed in alginate gels made by external gelation [31].



Variant A *- 0.6 % Ca-citrate (P01); 1.2 % Ca-citrate (P02); 0.6 % Ca-lactate (P03) and 1.2 % Ca-lactate (P04); at 24 h gelation time*



Variant B *- 0.6 % Ca-citrate (P05); 1.2 % Ca-citrate (P06); 0.6 % Ca-lactate (P07) and 1.2 % Ca-lactate (P08); at 48 h gelation time*

Figure 1. *Typical force versus distance curves obtained from a penetration test of composite fruit jellies as a function of calcium salt concentration and gelling time*

Correlation analysis

Pearson correlation coefficients between the structural-mechanical properties and their statistical significance at 24 and 48 h of gelation time were presented.

From these data (Tables 4 and 5), it can be seen that the correlation coefficients of the rupture force at 48 h of gelation time in comparision to the other structural-mechanical properties were slightly higher compared to the same dependences at 24 h of gelation time. The correlation between compressive stress and rupture force was the strongest, positive and statistically significant regardless of gelation time (r = 0.999).

Table 4. *Pearson correlation coefficients between structural-mechanical properties of composite fruit jellies at 24 h jellying time*

Type MP	**RF**	**RD**	**CS**	**FI**	**RE**	**TO**	**MY**	**AD**
RF	1							
RD	-0.766	1						
CS	0.999*	-0.757	1					
FI	0.977*	-0.856	0.969*	1				
RE	0.109	0.547	0.127	-	1			
TO	0.999*	-0.761	0.997*	0.980*	0.111	1		
MY	0.890	-0.970*	0.881	0.954	-0.354	0.889	1	
AD	-0.450	0.909	-0.433	-0.607	0.837	-0.449	-0.807	1

Notes: Pearson's coefficient is statistically significant at the α = 0.05 significance level; ($p < 0.05$) and critical value of the Pearson correlation coefficient - r = 0.950

Table 5. *Pearson correlation coefficients between structural-mechanical properties of composite fruit jellies at 48 h jellying time*

Type MP	**RF**	**RD**	**CS**	**FI**	**RE**	**TO**	**MY**	**AD**
RF	1							
RD	-0.791	1						
CS	0.999*	-0.805	1					
FI	0.980*	-0.892	0.984*	1				
RE	0.251	0.391	0.229	-	1			
TO	0.998*	-0.771	0.997*	0.971*	0.279	1		
MY	0.942	-0.925	0.947	0.987	-	0.924	1	
AD	-0.593	0.866	-0.602	-0.726	0.471	-0.549	-0.825	1

Notes: Pearson's coefficient is statistically significant at the α = 0.05 significance level; ($p < 0.05$) and critical value of the Pearson correlation coefficient - r = 0.950

All correlation dependences according to the determined Pearson coefficients between the rupture deformation and the other mechanical properties (tables 4 and 5) are relatively low and statistically insignificant. The only significant correlation dependence of the rupture force was that with the Young's modulus (r = -0.970) at 24 h of gelation time. The Pearson correlation coefficient between compressive stress and toughness was very high, significant and the same in numerical value (r = 0.997) regardless of gelation time. Another positive and strong correlation was found between compressive strength and firmness, and with increasing gelation time from 24 to 48 h. This relationship increased (from r = 0.969 to r = 0.984). As the gelation time increased from 24 to 48 h, all the correlation coefficients of firmness with respect to the other mechanical properties increased, except for the dependence between firmness and toughness, where the opposite effect was observed. Regardless of the gelling time (24 or 48 h), no correlation was found between firmness and rupture energy. All the correlation dependences between the rupture energy and the other mechanical properties are relatively weak and statistically insignificant. As the gelation time increased from 24 to 48 h, it was observed that the correlation coefficient of the dependence between rupture energy and adhesiveness decreased almost by two from r = 0.837 (Table 4) to r = 0.471 (Table 5). Moreover, the toughness exhibited very strong, positive and significant correlations with only some of the structural-mechanical properties (rupture force, compressive strength and firmness), and relative to the other properties, the dependencies are weaker and statistically insignificant. The correlation coefficient of the Young's modulus decreased in absolute value and became statistically insignificant. The presented correlation coefficients of adhesiveness with respect to the other mechanical properties were negative and statistically insignificant regardless of the increase in gelation time.

CONCLUSIONS

From the conducted research it was found that the values of almost all structural-mechanical parameters increased with an increase in the concentration of calcium citrate or calcium lactate. The exception of this trend was observed for adhesiveness and rupture deformation, where the opposite effect appeared. The type and concentration of added calcium salts had a more substantial and significant influence on the type of penetration curves compared to the gelation time. An increase in the gelation time and the concentration of calcium salts leads to an increase in the correlation coefficients of the rupture force, compressive strength and firmness in relation to the other structural-mechanical properties that results in the good quality of the final jelly product. From all concentrations and jellying time it was found that the better results and quality of final product can be obtained when 1.2 % calcium lactate was used for jellation time 48 h.

LIST OF NOTATIONS AND SYMBOLS

AD – adhesiveness
RE – rupture energy
FI – firmness
MP – mechanical properties
MY– Young's modulus
RD – rupture deformation
RF – rupture force
TO – toughness
CS – compressive stress

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