# Investigation of the Influence of Calcium Salts and Gelling Time on the Structural Mechanical Properties of Fruit Jam

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Bulletin UASVM Food Science and Technology 77(2)/2020 ISSN-L 2344-2344; Print ISSN 2344-2344; Electronic ISSN 2344-5300 DOI: 10.15835/buasvmcn-fst: 2020.0010

#### Abstract

Alginate beads attract attention as a encapsulation matrix of bioactive substances in food. However, the stability of beads depends on calcium ion and sodium alginate concentration, gelling time and others factors. The aim of this study is to investigate the influence of different types of calcium salts on the structural and mechanical parameters - the rupture force and rupture deformation at different gelling times of the pear jam prepared with soluble dietary fibers and inulin. The relationships between the rupture force and rupture deformation of the fruit jams were established. By increasing the gelling time from 24 hours to 48 hours, the rupture deformation of jams with 7% calcium lactate were reduced and in those with 7% CaCl<sub>2</sub> the rupture forces increases. Any change in rupture force was observed for the jam with 3.5% CaCl<sub>2</sub>. This study demonstrated the practical application of different calcium salts for preparation of stable pear jam.

Keywords: calcium salts, gel formation time, pear fruit jam.

## Introduction

Alginic acid is found in seaweed in the form of water soluble salts with Na, Ca, K and Mg, called alginate. Among its salts only Ca - alginate is an insoluble one. Sodium alginate is commonly used as a gelling agent in the food industry. Its gelling strength depends on  $\alpha$ -L-guluronic acid and  $\beta$ -D-mannuric acid content. The smaller is the ratio of manuronic acid to guluronic acid (M / G) the greater is the gelling ability (Kloareg and Quatrano, 1988; Perez, 1992). It has been experimentally found that upon the addition of divalent calcium cations in aqueous Na-alginate solution, an instantaneous gelation occurs due to the cooperation of calcium ions with  $\alpha$ -L-guluronic acid from the guluronic blocks of the alginates resulting in a 3-dimentional

network of gelling structures called an 'egg box' (Nussinovitch, 1997; Rousseau, 2004). The best and readily obtainable alginate food jellies are produced with divalent calcium cations, since these ions are non-toxic (Draget, 2006).

It has been found that approximately 7.2% calcium ions is required stoichiometrically based on the weight of sodium alginate for the complete chelation process. Different calcium salts release different amounts of calcium ions into the solution. It is well known that the following factors (temperature, pH and type of calcium) strongly influence the gelling time (Ren, 2008). A series of experiments showed that calcium dilactate, which is well soluble in water, and is not affected by the pH and ambient temperature gels rapidly. It has

also been found that the lower is the pH of the medium, the shorter is the gelling time.

As a result of the experiments, it was found that by increasing the concentration of gelling agent or calcium ions (in the calcium salt type) the strength of the solution of alginate jellies (beads) was also increased. However, the presence of 65% sucrose in gels reduces the rupture and rupture deformation. The following dependence was found in earlier experiments: increasing the concentration of calcium ions in alginate jelly reduces the time to reach the maximum rupture force (Maney, 2013). Moreover, with increasing sodium alginate concentration the diameter of alginate jelly (beads) increases, and with increasing calcium salt concentration (calcium dichloride dihydrate), the size (diameter) of alginate jelly (beads) decreases, respectively (Kaur, 2018). A reduction or minimal improvement in the structural-mechanical parameters were observed with the addition of highly esterified pectin or inulin to pear fruit jam, which has not undergone heat treatment (Petrova, 2019).

The aim of the current study is to investigate the influence of different types of calcium salts on the structural and mechanical parameters - the rupture force and rupture deformation at different gelling times of the pear fruit jam by a texture analyzer.

## Materials and methods Materials

The puree from pears "Conference" variety was used as the main raw material for production of fruit jams. The pears were purchased from the local market in Plovdiv, Bulgaria. Food grade water soluble sodium alginate - VIVAPUR Alginate FD 120 (P.I.C.Co, Bulgaria), with the 44 mPa.s viscosity and pH 6 was used for the pear jam preparation. Calcium dichloride dihydrate and calcium lactate pentahydrate were used as the sources of calcium ions with high analytical grade >95 % and they were supplied from Fillab Ltd. (Plovdiv, Bulgaria). Low methoxylated amidated citrus pectin (Aglupectin LA-S10, P.I.C.Co, Bulgaria) was characterized by the degree of esterification - 34% and the degree of amidation - 17% and it was also used as a jellying agent. Inulin Frutafit HD (Sensus, Roosendaal, The Netherlands) with degree of polymerization (DP=9-12) and soluble dietary fibers. Potato starch with pH 6.8 and sulfated ash <0.60% Fluka Analytical (Sigma Aldrich, Germany) was used as thickener agent. Moreover, aspartame and potassium sorbate purchased from Fillab Ltd (Plovdiv, Bulgaria) were used as a sweetener and preservative, respectively. Citric acid (Fillab Ltd. Plovdiv, Bulgaria) was used for correction of pH and as an antioxidant.

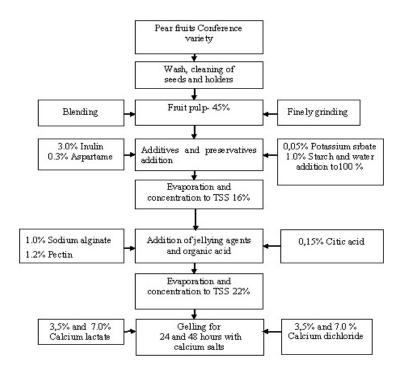


Figure 1. Technological scheme for production of pear jam

#### Methods

The characteristics as the rupture force and rupture deformation of the pear jams were determined by penetration tests using a Stable Microsystems Texture Analyser (SMS) in uniaxial deformation mode at an initial set voltage of 60% with a constant deformation rate 2 mm/s by an aluminum cylindrical piston with 5 mm in diameter. Ten samples were measured for better statistics. The firmness indicated the penetration resistance. It was determined by the slope of the first peak of the force and the depth penetration, expressed as N/mm, (Harnkarnsujarit, 2016). A full experiment factor was applied to determine the rupture force and deformation of the fruit jam. The technological scheme for production of pear fruit jam was presented in Figure 1.

### **Results and discussion**

From the results presented, it can be seen that increasing the calcium lactate concentration from 3.5% to 7% in fruit jams (P01 and P05) results in a twofold increase in tearing strength and in gelling time of 24 hours (Table 1). The same effect was observed at 48 hours of gelling time on the rupture force and firmness, but to a lesser extent (P03 and P07). A similar effect associated with the increase in the strength of the gel when increasing the concentration of calcium ions was found in cubic form alginate gels made by the external gelation method (Ramdhan, 2019). This effect could be explained by the fact that the higher the concentration of the calcium solution added, the more interactions would occur between the calcium ions and the guluronic blocks in the alginate, which leads to increased gel strength (Kaklamani, 2014; Lozano-Vazquez, 2015). A similar increase in the gel strength depending on the type of calcium salt (calcium lactate, calcium dichloride, calcium gluconate, calcium phosphate and calcium propionate) was reported for fruits, vegetables and mushrooms (Martin-Diana, 2007), as well as for restructured strips of pimento (Mousavi, 2019).

From the results (Table 1) and the penetration curves (Figures 2 and 3), it was found that the addition of calcium lactate or calcium dichloride in pear jams slightly affected the rupture deformation. A similar effect was observed with the addition of calcium dichloride in sardine fish (Montero, 2002). The experiments showed that increasing the concentration of calcium salts from 3.5% to 7.0% and gelling time from 24 to 48 hours of fruit jams (P01 and P07; P02 and P08) increase the mechanical parameters - rupture forces and gel strength, while the rupture deformation was minimized. This effect is due to the increasing density of the crosslinked gel as the gelation time and the calcium salt concentration increase (Mammarella, 2002; Saarai, 2013).

## Conclusion

As a result of the experimental work and the results obtained, the influence of the type and concentration of calcium salts, as well as the gelling time on the rupture force, deformation and firmness of the fruit jams obtained was evaluated. Increasing the gelling time from 24 to 48 hours in

Table 1. Tec	hnologica	l and rheo	logical (	characteristics of	pear fruit	iams

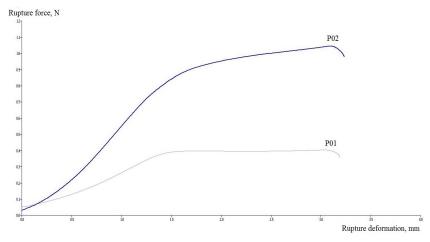
Sample	Gelling time (h)	Type and concentration of calcium salt (%)	Rupture forceª (N)	Rupture deformationª (mm)	Jam firmnessª (N.mm <sup>-1</sup> )
P01	24	3.5 lactate	$0.409 \pm 0.057^{\rm b}$	$3.054 \pm 0.112^{b}$	$0.123 \pm 0.020^{b}$
P02	24	3.5 dichloride	1.046±0.085	3.082±0.108	0.325±0.043
P03	48	3.5 lactate	0.557±0.035	2.617±0.296	0.198±0.036
P04	48	3.5 dichloride	$1.048 \pm 0.024$	2.972±0.038	0.338±0.013
P05	24	7.0 lactate	0.841±0.025	3.286±0.061	0.246±0.009
P06	24	7.0 dichloride	1.083±0.023	2.897±0.081	0.356±0.018
P07	48	7.0 lactate	0.653±0.050	2.712±0.164	0.230±0.027
P08	48	7.0 dichloride	1.133±0.067	2.901±0.111	0.376±0.024

<sup>a</sup>Mean value (n=10)

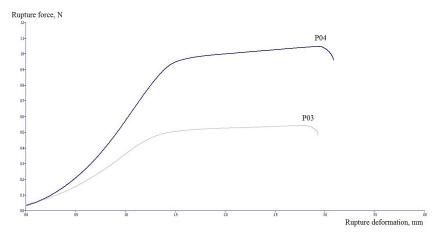
<sup>b</sup>SD- standard deviation

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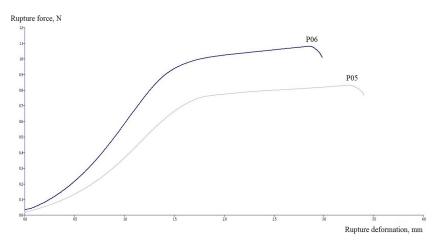
jams prepared with 3.5% calcium lactate increases the rupture force and firmness, while the rupture deformation values were minimized. In fruit jams jellied with 7.0% calcium lactate, a decrease in the values of all structural characteristics was observed with an increase in the gelling time from 24 to 48 hours. Under the same other conditions of gelation and increasing the gelling time from 24 to 48 hours of jams obtained with calcium dichloride at the concentrations 3.5% and 7.0%, it



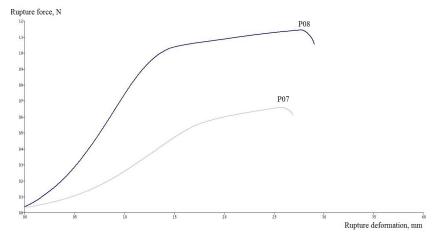
a) 3,5% Ca-lactate (P01) and 3,5% Ca-dichloride (P02) at 24 hours gelling time



b) 3,5% Ca-lactate (P03) and 3,5% Ca-dichloride (P04) at 48 hours gelling time **Figure 2.** Typical curves of the penetration for pear jams at different gellying time



a) 7,0% Ca-lactate (P05) and 7,0% Ca-dichloride (P06) at 24 hours gelling time



b) 7,0% Ca-lactate (P07) and 7,0% Ca-dichloride (P08) at 48 hours gelling time

Figure 3. Typical curves of the penetration for pear jams at different structuration time (gellying time)

is observed that the rapture force and the firmness increase to a minimum and the deforming forces changed negligibly.

*Acknowledgements.* This experimental work was supported by the Bulgarian Ministry of Education and Science for the funding of basic scientific research for young scientists and postdoctoral students – 2018 with a research project topic "Production of a low-calorie pear fruit jam enriched with fiber" and a contract number KΠ-06-M27 / 8 from 08.12.2018.

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