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# Effect of inulin on rheological and thermal properties of gluten-free dough

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#### ARTICLE INFO

Article history: Received 19 December 2011 Received in revised form 23 April 2012 Accepted 27 April 2012 Available online 24 May 2012

Keywords: Inulin Starch Dough Bread Rheological behavior Thermal properties

#### ABSTRACT

The aim of the study was to evaluate the influence of inulins with varying degree of polymerization on rheological and thermal properties of gluten-free starch-based dough. The share of inulin reduced the values of consistency coefficient, as well as storage and loss moduli, and increased creep compliance. Inulin preparation with the highest average degree of polymerization had the strongest impact on viscoelastic properties of the obtained dough. The presence of inulin also caused a significant decrease of viscosity upon pasting, and an increase of gelatinization temperatures  $T_{\rm Og}$ ,  $T_{\rm P1g}$ ,  $T_{\rm P2g}$ , and  $T_{\rm Eg}$ . Addition of inulin had no effect on gelatinization enthalpy ( $\Delta H_{\rm g}$ ), while it strongly reduced the enthalpies of retrograded amylopectin after storage. Water binding properties of inulin seem to be the key factor, responsible for modification of dough properties, because they influence solvent availability for other constituents of such system.

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# 1. Introduction

Celiac disease is usually manifested as a digestive malfunction of intestines, caused by the presence of gluten. The trials to reduce gluten toxicity, by modification of its structure have not yet been successful, so strict avoidance of this protein is the only efficient way of celiac treatment (Abdel-Aal, 2009). Technology of gluten free bakery products is based mainly on starches of various botanical origin, as well as corn, rice, soy and buckwheat flours. Such products have significantly lower nutritional value in comparison to conventional bread (Gallagher, Gormley, & Arendt, 2004; Korus et al., 2011; Lazaridou, Duta, Papageorgiou, Belc, & Biliaderis, 2007). The introduction of nutritional or dietary supplements is however difficult, due to its fragile structure that could easily be overloaded with applied additives (Korus, Witczak, Ziobro, & Juszczak, 2009)

Inulin is a polysaccharide with interesting functional properties and pro-health activity (Meyer, Bayarri, Tárrega, & Costell, 2011). It is synthesized as a storage carbohydrate in many cultivated plants (Praznik, Cieślik, & Filipiak-Florkiewicz, 2002). Its chains are composed of fructopyranose residues connected via  $\beta(2,1)$ -glycosydic bonds. Single glucose unit is present on the end of each molecule.

Commercial preparations of inulin contain polymers with varying degree of polymerization (DP) and different amounts of oligosaccharides. Because of its chemical structure inulin could not be digested in alimentary tract, and products with its share are characterized by a reduced energetic value. Moreover inulin, as a fraction of dietary fiber, has a prebiotic function (Praznik et al., 2002). Apart from these health benefits, inulin could play a functional role in food processing (Meyer et al., 2011). It is utilized as a food substitute, bulking and structure forming agent, and as a substance improving rheological properties and taste (Meyer et al., 2011; Praznik et al., 2002; Tárrega & Costell, 2006). Moreover it stabilizes emulsions and foams and regulates water retention in food products. These functional properties are mainly related to its ability to form physically stable gels (Meyer et al., 2011; Tárrega & Costell, 2006).

In earlier studies it was shown, that gluten-free bakery could be enriched with fibers of various origin (Korus & Achremowicz, 2004), resistant starch (Korus et al., 2009), beta-glucans (Hager et al., 2011; Lazaridou et al., 2007) and inulin (Hager et al., 2011; Korus, Grzelak, Achremowicz, & Sabat, 2006; Peressini & Sensidoni, 2009). There are however no reports on the influence of a degree of polymerization on properties of gluten-free dough and bread supplemented with inulin. This is why the aim of this study was to compare the applicability of inulin preparations with varying DP in production of gluten-free bread, by monitoring their impact on rheological and thermal properties of the resulting dough.

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#### 2. Materials and methods

#### 2.1. Materials

The material used in formulations for gluten-free dough consisted of corn starch (Bezgluten, Poland), potato starch (Pepees S.A., Poland), guar gum (Lotus Gums & Chemicals, India), pectin (Pektowin, Poland), freeze dried yeast Saf-instant (S.I. Lesaffre, France), sucrose, salt, plant oil and water. Additionally high soluble inulin HSI (DP < 10), granulated inulin GR (DP  $\geq$  10) and high performance inulin HPX (DP > 23) (BENEO-Orafti, Belgium) were used. According to producer's declaration HPX preparation contains 100% of inulin, while GR and HSI - 92% and 86%, respectively. The remaining 8% (GR) and 14% (HSI) consists of the mixture of glucose, fructose and sucrose.

#### 2.2. Methods

#### 2.2.1. Preparation of gluten free dough

The dough for gluten-free breads was based on the following ingredients: corn starch 480 g, potato starch 120 g, guar gum 8.3 g, pectin 8.3 g, freeze dried yeasts 25 g, sucrose 10 g, salt 8.3 g, plant oil 15 g. Part of starch (4%, 8% or 12% of total starch, i.e. 24 g, 48 g or 72 g) was replaced with the preparation of inulin: HSI, GR or HPX. Due to the important differences in water binding ability of inulin, the content of water was adjusted individually for each modified recipe, with the use of texture analyzer TA-XT2+ equipped with back extrusion rig (A/BE-d 35) (Stable Micro Systems, England), in such a way that the obtained dough systems exhibited comparable values of hardness during extrusion. The amounts of added water equaled 620 g for control sample, and decreased with rising amounts of HSI and GR inulins, reaching 610, 600 and 595 g, for 4%, 8% and 12% addition of inulin, respectively. In the case of HPX inulin, the amount of added water was 630, 640 and 650 g for 4%, 8% and 12% inulin level, respectively. All the ingredients without yeasts and oil were mixed using laboratory stirrer RW20 (IKA Labortechnik, Germany) at ambient temperature for 8 min, at 200 rpm and used for rheological and thermal analyses.

### 2.2.2. Rheological properties of dough mixes

Dough rheological properties were characterized at  $25\,^{\circ}\mathrm{C}$  using a rheometer MARS II (Thermo Haake, Germany) equipped with the system of parallel plates (diameter  $35\,\mathrm{mm}$ , gap  $1\,\mathrm{mm}$ ). The samples were placed in the rheometer measuring system and the edges covered with paraffin oil. Dough was left for  $15\,\mathrm{min}$  to relax stress and stabilize the temperature, and then the measurements were performed.

Viscosity curves were obtained under controlled shear rate in the range of  $1-30 \, \mathrm{s}^{-1}$ . Experimental data were described by a power law equation (Korus et al., 2011; Pruska-Kędzior et al., 2008):

$$\eta_{ap} = K \cdot \dot{\gamma}^{n-1} \tag{1}$$

where  $\eta_{ap}$  is apparent viscosity (Pa s), K is the consistency coefficient (Pa s<sup>n</sup>),  $\dot{\gamma}$  is shear rate (s<sup>-1</sup>) and n is the flow behavior index.

The range of linear viscoelasticity was determined by checking the dependence of storage G' and loss G'' moduli on the applied stress in the range of 0.1-100 Pa at a fixed frequency of 1 Hz (Piteira, Maia, Raymundo, & Sousa, 2006). Critical stress values measured for linear viscoelastic range varied from 2.5 Pa to 4.2 Pa.

Mechanical spectra were determined in the range of linear viscoelasticity at constant strain amplitude of 0.05% in the angular frequency of 0.1–100 rad s<sup>-1</sup>. The experimental data were fitted with power-law equations (Georgopoulos, Larsson, & Eliasson,

2004; Sivaramakrishnan, Senge, & Chattopadhyay, 2004; Steffe, 1996):

$$G'(\omega) = K' \cdot \omega^{n'} \tag{2}$$

$$G''(\omega) = K'' \cdot \omega^{n''} \tag{3}$$

where G' is a storage modulus (Pa), G'' is a loss modulus (Pa),  $\omega$  is a angular frequency (rad s<sup>-1</sup>), and K', K'', n', n'' are the experimental constants.

Creep and recovery tests were performed at fixed stress  $\sigma_0$  = 1 Pa in the range of proportionality of strain to stress. The experiments were performed under controlled stress mode. Creep phase continued for 150 s, and recovery 300 s. As a result of these measurements strain values were obtained as a function of time. The experimental data were fitted by Burger's model (a Maxwell body in series with one Kelvin-Voigt body) (Lazaridou et al., 2007; Steffe, 1996; Witczak, Korus, Ziobro, & Juszczak, 2010). This model is described by the equation:

$$J(t) = J_0 + \frac{t}{\eta_0} + J_1 \cdot (1 - \exp^{-t/\lambda_{ret}})$$
 for  $t < t_1$  (4)

$$J(t) = \frac{t_1}{n_0} - J_1 \cdot (1 - \exp^{t_1/\lambda_{ret}}) \cdot \exp^{-t/\lambda_{ret}} \quad \text{for } t > t_1$$
 (5)

where J is a compliance  $(Pa^{-1})$ ,  $J_0$  is an instantaneous compliance,  $J_1$  is a retardation compliance  $(Pa^{-1})$ ,  $\eta_0$  is zero shear viscosity (Pas),  $\lambda_{ret}$  is a retardation time (s), and  $t_1$  is a time after which stress was removed (s).

Calculations were performed using Marquardt–Levenberg method with the use of Statistica 9.0 (StatSoft Inc., USA).

#### 2.2.3. Pasting characteristics of dough mixes

Pasting characteristics were performed using a Micro Visco-Amylo-Graph type 803202 (Brabender, Germany) equipped with a 250 cmg measuring cartridge, run at 75 rpm and operated under Brabender Viscograph ver. 2.4.6 software. Dry blends (5 g) of the ingredients were mixed with 95 g of distilled water, heated/cooled at a rate 6 °C/min according to the following program: rising temperature in the range 25–96 °C, 96 °C constant temperature (10 min), cooling in the range 96–40 °C. Pasting temperature, peak viscosity, and viscosity after 10 min at 96 °C and after cooling were read from Brabender Viscograph – Data Correlation ver. 2.1.6 software

# 2.2.4. Thermal characterization of dough mixes

Thermal properties were characterized using the differential scanning calorimeter DSC 204F1 Phoenix (Netzsch, Niemcy), calibrated with indium. Dough samples (15  $\pm$  1 mg) were hermetically closed in aluminum pans and heated in a calorimeter from 25 to 110 °C at constant rate 10 °C/min. Then, they were kept for 5 min at 110 °C, and cooled back to 25 °C at the same rate 10 °C/min. Empty aluminum pan was used as a reference sample. The samples were stored at 23 °C for 48 h, and reheated in a calorimeter to 110 °C at 10 °C/min. Temperatures ( $T_{\rm O}$  – onset,  $T_{\rm P}$  – peak,  $T_{\rm E}$  – endset) and enthalpy of thermal transitions ( $\Delta H$ ) were determined with the use of instrument's software Proteus Analysis (Netzsch, Germany). Enthalpy values were expressed as J/g starch blend.

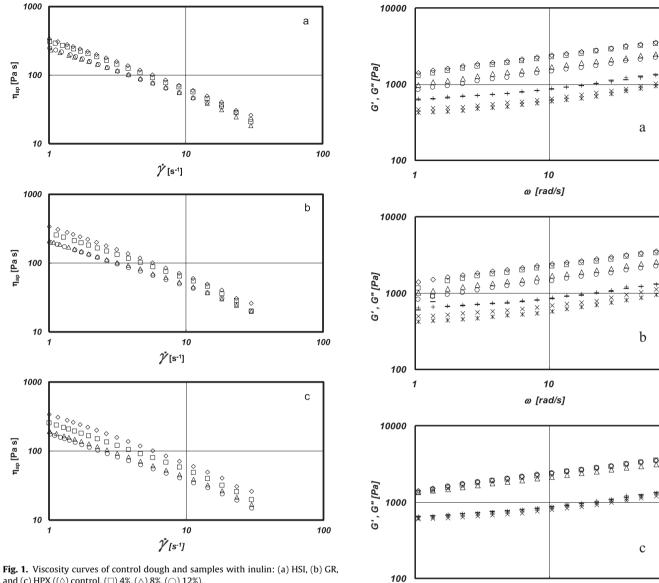
# 2.2.5. Statistical analyses

In order to establish the statistical differences between means the data were treated by one-factor analysis of variance, and the least significant difference (LSD) using Fisher test at significance level 0.05 was calculated. The influence of selected factors was analyzed with the use of two factorial analysis of variance. All calculations were performed with statistical software package Statistica 9.0 (StatSoft Inc., USA).

100

100

100



and (c) HPX (( $\Diamond$ ) control, ( $\Box$ ) 4%, ( $\triangle$ ) 8%, ( $\bigcirc$ ) 12%).

# 3. Results and discussion

# 3.1. Rheological properties of the dough

Fig. 1 demonstrates viscosity curves of the analyzed dough samples. Their shape is typical for shear-thinning substances. In all cases a partial replacement of starch with inulin resulted in a decrease of apparent viscosity. Power law equation was found to adequately describe the experimental data. The differences in flow indices were not statistically significant (Table 1). The obtained values of n were comparable with earlier reports for gluten-free dough (Korus et al., 2011; Mezaize, Chevallier, Le-Bail, & de Lamballerie, 2010; Pruska-Kędzior et al., 2008). The values of consistency coefficient for doughs with inulin were slightly lower than in earlier reports (Korus et al., 2011; Mezaize et al., 2010; Pruska-Kędzior et al., 2008). Two factorial analysis of variance indicates, that equation parameters were only slightly affected by the type of additive (p = 0.059). On the other hand consistency coefficient was visibly influenced by the level of inulin, and decreased with rising share of inulin in the dough. It should be however noticed, that higher doses of inulin do not result in significantly different values of consistency coefficient, which suggests that above a certain level of inulin,

Fig. 2. Mechanical spectra of control dough and samples with inulin: (a) HSI, (b) GR, and (c) HPX (G': ( $\Diamond$ ) control, ( $\Box$ ) 4%, ( $\Delta$ ) 8%, ( $\bigcirc$ ), 12%; G'': (+) control, (-) 4%, ( $\times$ ) 8%, (\*) 12%).

10

ω [rad/s]

there is no further change in the consistency of the dough. It is well known that inulin is highly hygroscopic (Gennaro, Birch, Parke, & Stancher, 2000; Peressini & Sensidoni, 2009) and reduces water availability for other dough constituents. According to Hager et al. (2011) inulin molecules form junction zones and so enclose large amount of water. The replacement of starch with soluble forms of inulin and oligosaccharides leads to a decrease in water absorption and reduction of dough consistency (Peressini & Sensidoni, 2009; Rouillé, Vallea, Lefebvre, Sliwinski, & Vliet, 2005), diluting hydrocolloids and decreasing starch swelling.

Oscillatory studies allow to characterize different materials without damaging their structure. Fig. 2 demonstrates mechanical spectra of the analyzed samples. Values of storage (G') and loss (G'') moduli increased with angular frequency. In all cases G'were larger than  $G''(\tan \delta < 1)$  which implies a prevalence of elastic properties over viscous ones (Table 1). A dependence of G' on angular frequency and the values of phase shift tangent ( $\tan \delta = G''/G'$ )

Table 1
Parameters of the power-law functions describing dependence of storage and loss moduli on angular frequency and flow properties of gluten free dough (mean value of two replication ± standard deviation).

Sample		$G' = K' \cdot \omega^{n'}$		$G'' = K'' \cdot \omega^{n''}$		$ an\delta$	$\eta_{ap} = K \cdot \dot{\gamma}^{n-1}$		
		K' [Pa s <sup>n'</sup> ]	n'	K" [Pa s <sup>n"</sup> ]	n"	at 1 Hz	$K[Pa s^{n'}]$	n	R <sup>2</sup> >
Control		1537.9 ± 142.3 <sup>a</sup>	$0.217 \pm 0.001$	629.9 ± 63.3 <sup>a</sup>	$0.193 \pm 0.004^{a}$	0.37	$346.8 \pm 19.9^{a}$	$0.273 \pm 0.039$	0.994
	4%	$1318.4 \pm 8.7^{b}$	$0.224 \pm 0.005$	$556.6 \pm 11.2^{b}$	$0.203 \pm 0.001^a$	0.39	$296.3 \pm 43.6^{ab}$	$0.281 \pm 0.016$	0.995
HSI	8%	$956.9 \pm 54.8^{cd}$	$0.230 \pm 0.010$	$422.0\pm0.6^{cd}$	$0.216 \pm 0.007^{b}$	0.41	$248.7 \pm 9.7^{bce}$	$0.287 \pm 0.011$	0.994
	12%	$834.9 \pm 7.8^{c}$	$0.236 \pm 0.000$	$374.8 \pm 3.0^{c}$	$0.223 \pm 0.006^{b}$	0.42	$233.3 \pm 29.5^{cde}$	$0.328 \pm 0.034$	0.998
	4%	$1280.0 \pm 112.9^{b}$	$0.216 \pm 0.006$	$534.7 \pm 38.1^{b}$	$0.200 \pm 0.001^a$	0.39	$286.9 \pm 10.5^{bc}$	$\boldsymbol{0.281 \pm 0.004}$	0.992
GR	8%	$1024.4 \pm 31.1^{d}$	$0.226 \pm 0.004$	$447.8 \pm 9.0^{d}$	$0.214 \pm 0.005^{b}$	0.42	$238.3 \pm 30.3^{cde}$	$0.309 \pm 0.029$	0.994
	12%	$887.2 \pm 65.2^{cd}$	$0.224 \pm 0.018$	$384.1 \pm 13.8^{cd}$	$0.216 \pm 0.009^{b}$	0.41	$213.6 \pm 8.8^{de}$	$0.317 \pm 0.031$	0.996
	4%	$1332.6 \pm 52.5^{b}$	$0.226 \pm 0.000$	$572.0 \pm 11.2^{ab}$	$0.195 \pm 0.001^a$	0.39	$259.7 \pm 16.4^{bce}$	$0.273 \pm 0.004$	0.996
HPX	8%	$1226.8 \pm 32.7^{b}$	$0.223 \pm 0.001$	$506.1 \pm 19.3^{b}$	$0.202 \pm 0.002^a$	0.37	$187.8 \pm 17.2^{d}$	$0.319 \pm 0.018$	0.996
	12%	$1354.4 \pm 95.1^b$	$\boldsymbol{0.220 \pm 0.004}$	$545.4\pm36.0^b$	$0.192\pm0.005^a$	0.36	$213.4\pm35.9^{de}$	$\boldsymbol{0.282 \pm 0.006}$	0.998
One-way ANOVA – p		<0.001	0.299	<0.001	<0.001		0.003	0.224	
Two-way	ANOVA – p								
Factor I (type)		< 0.001	0.134	< 0.001	<0.001		0.059	0.709	
Factor II (level)		< 0.001	0.495	< 0.001	0.004		0.003	0.090	
Factor I × Factor II		0.006	0.481	0.020	0.050		0.682	0.281	

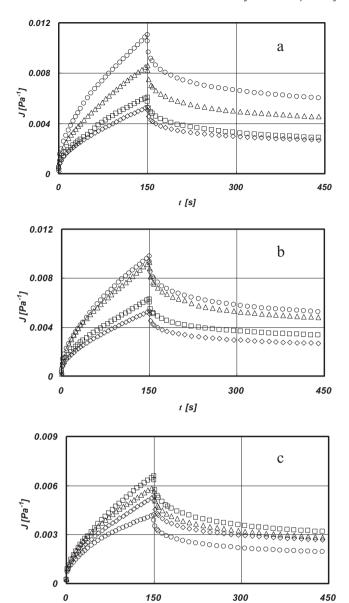
Mean values signed this same letters in particular columns are non-significant different at 0.05 level of confidence.

in the range between 0.26 and 0.52 indicates, that the analyzed samples behave as a weak gel. It confirms earlier results for glutenfree dough based on different raw materials and additives (Korus et al., 2009; Lazaridou et al., 2007; Mezaize et al., 2010; Torbica, Hadnađev, & Dapčević, 2010; Witczak et al., 2010). On the other hand growing values of  $\tan \delta$  for low DP inulin preparations (GR and HSI) and with inulin level, show the shift toward viscous properties. In the case of HPX preparation at low angular frequency (<10 rad s<sup>-1</sup>) the highest values of tan  $\delta$  (0.37–0.49) was observed for sample with 4% HPX, while for the remaining samples were not significantly different. At higher frequency, control sample was more susceptible to deformation, and the values of  $\tan \delta$  decreased with the rising amount of additives, which denotes their more elastic nature. The strongest decrease of moduli was observed after the addition of highly soluble preparation (HSI), and the smallest when HPX inulin was used. It seems to be connected with inulin structure and its ability to bind water. Low average DP and high solubility are accompanied with low values of both loss and storage modulus. The values of moduli gradually decreased with increasing inulin level, which was probably caused by limited water availability. According to Zimeri and Kokini (2003), who examined properties of inulin-waxy maize starch systems in a broad range of concentrations, and applied various rheological methods, viscoelastic behavior of such systems depends on their concentration. Peressini and Sensidoni (2009), studied the influence of inulins with different DP on viscoelastic properties of wheat dough, and divided the effects of inulin addition into direct and indirect. In their opinion direct effects are related to starch/water ratio, and are manifested by high increase in moduli with decreasing water availability. According to Peressini and Sensidoni (2009) inulin with a low DP acts mainly as a diluting substance and does not lead to fundamental changes in dough structure. Similar conclusions could be derived from the above mentioned results. The effects could be caused in a large part by diluting action of oligosaccharides, present in GR and HSI, as well as in the preparation used by Peressini and Sensidoni (2009). In the case of HPX inulin a slight decrease of both moduli, irrespective of addition level, was observed, despite of the increasing amount of added water. The differences between HPX samples containing different levels of inulin were not statistically significant. At constant water addition the presence of this type of inulin leads to the increase of moduli, which is why more water should be added to keep the assumed consistency. It means, that the presence of high DP inulin enhances dough elasticity. Similar effects were observed by Peressini and Sensidoni (2009) for HP

inulin preparation. The authors checked the influence of Raftiline HP and Raftiline HP-gel inulins (Orafti Food Ingredients, Belgium) on dough properties, and stated, that inulin–inulin interactions could lead to a formation of an elastic network, and thus intensify dough elasticity. Moreover, water bound by inulin reduces hydration level of structure forming hydrocolloids, causing an increase in storage modulus.

Table 1 shows parameters of power law equations, describing the dependence of moduli on angular frequency. The values of K' and *K*" decreased with rising levels of applied inulins. The highest values were obtained for control sample, and the lowest for 12% HSI preparation. The exception were the values of K' and K'' observed with increasing content of inulin HPX, where the differences were statistically insignificant. All samples were characterized by similar values of the coefficients n'. The differences were not statistically significant. The values of n' were comparable with those obtained earlier for wheat flour dough (Georgopoulos et al., 2004), dough with added resistant starch (Korus et al., 2009), the systems with the addition of maltodextrins (Witczak et al., 2010) and soft white winter wheat and hard red winter wheat doughs (Schluentz, Steffe, & Ng, 2000). Slightly lower values of n' were reported for gluten-free dough with the addition of defatted fruit seeds (Korus et al., 2011). Taking into account n'' indices, the samples could be classified into two groups. The samples with maximum levels of GR and HSI inulin exhibited higher values of n'' comparing with the other samples. This behavior suggest that the addition of inulin increases viscous character of the samples, however only minor changes could be observed between the samples in the examined range of addition

Fig. 3 shows the creep and recovery curves of control and dough samples with different types of inulin. All curves have a characteristic course of viscoelastic properties. The phenomenon of creep and recovery is associated with the reorientation of bonds in the viscoelastic material (Onyango, Mutungi, Unbehend, & Lindhauer, 2010). The instantaneous compliance ( $J_0$ ) is related to the elastic stretching energy of bonds, when stress is applied and disappears immediately when the stress disappears. Viscoelastic compliance ( $J_0$ ) is associated with a destruction and transformation of bonds (Onyango, Mutungi, Unbehend, & Lindhauer, 2009). Addition of GR and HSI inulins raised the values of instantaneous compliance ( $J_0$ ), which means that the dough was less resistant to deformation, despite the addition of water content decreased. This behavior may be associated with the presence of low molecular mass sugars, which loosened consistency. In addition, water soluble inulin



**Fig. 3.** Creep and recovery curves of control dough and samples with inulin: (a) HSI, (b) GR, and (c) HPX (( $\Diamond$ ) control, ( $\Box$ ) 4%, ( $\bigtriangleup$ ) 8%, ( $\bigcirc$ ) 12%).

t [s]

forms microcrystals, which interact creating particle gel (Hager et al., 2011; Kaur & Gupta, 2002), and this in turn may result in susceptibility to deformation. In the case of HPX preparation, lower doses (4 and 8%) caused an increase of compliance, while the highest (12%) resulted in its reduction, which was probably forced by the parallel addition of water. High molecular mass inulin restricts water availability and strengthens the structure created by hydrocolloids, depending on inulin level. According to Kim, Faqih, and Wang (2001) formation of a gel structure by inulin is possible only for molecules with appropriate length after adequate thermal treatment. In the case of dough which is not heated, the effect of inulin could only be attributed to amplification of structure forming action of the applied hydrocolloids (pectin and guar gum).

Creep and recovery phenomenon is caused by reorientation of the bonds in viscoelastic material (Onyango et al., 2010). Instantaneous compliance is related to the energy of elastic stretching of the bonds, when stress is applied, and vanishes immediately after its removal. On the other hand viscoelastic compliance is related to the disruption and conversion of the bonds (Onyango et al., 2009). Retardation time is a time characterizing the response of a viscoelastic material to the instantaneous application of a constant stress. Table 2 contains the values of Burger's parameters. The lowest instantaneous compliance was found for control sample and 12% of HPX inulin. The use of HSI and GR preparations resulted in a significant increase of instantaneous compliance, depending on the level of inulin. It could be explained by different degree of polymerization of inulins, the lower DP, and consequently high solubility that makes dough more susceptible to stress. Increase in average DP and decrease in solubility restricted the change in compliance for HPX dough, and at the highest level of its addition caused its decrease.  $I_1$  followed the same tendency as  $I_0$ . Increased with rising levels of HSI and GR and decreased when 12% HPX was added. Smaller quantities of HPX did not give statistically significant effects in comparison to control sample. Lowest zero shear viscosity was observed for 12% additions of HSI and GR dough. For both doughs the values were inversely related to the inulin level, although the differences between 8% and 12% were not statistically significant. Dough samples with HPX did not follow the same trend, and although at the lowest level a drop in viscosity was observed, its value gradually increased with increasing level of inulin. It demonstrates, that inulin with high DP enhances dough structure, causing significant change of zero shear viscosity. This may be explained by interactions between long chains formed by its molecules. Two factorial analysis of variance revealed, that the level of inulin has no significant impact on the values of zero shear viscosity, while there is a clear effect of inulin type. On the other hand retardation time was determined mainly by inulin level and did not depend significantly on its type. All samples with inulin revealed lower retardation times in comparison to control sample. In the case of HSI preparation the retardation time decreased with rising addition level, but no such tendency was observed for GR and HPX samples. It means, that regardless of its degree of polymerization, inulin dilutes the system, causing easier reorientation of structural elements of the dough, which results in the higher rate of recovery after deformation. The obtained times were higher in comparison to those measured after addition of other hydrocolloids, resistant starch and maltodextrins (Korus et al., 2009; Lazaridou et al., 2007; Witczak et al., 2010).

# 3.2. Pasting characteristics of dough mixes

Pasting parameters of gluten-free dough mixes are summarized in Table 3. The compiled data shows that the presence of inulin with different DP in examined systems significantly modified their pasting profiles. According to Tester and Sommerville (2003) nonstarch polysaccharides significantly influence starch gelatinization by limiting the hydration of amorphous regions in starch granule. The presence of inulin in dough mixes additionally reduces the amount of available water, and in this way increases pasting temperature. Such an effect was observed for all analyzed samples, except the lowest level of HSI dough. Two factorial analysis of variance proved, that both type and level of inulin have significant rising effect on pasting temperature, while no such influence was observed for their interaction. Viscosity changes over pasting (Table 3) were also affected by the presence of inulin. Only the lowest level of HSI blend did not cause a significant decrease of viscosity during heating and cooling in comparison to control sample, which is probably due to its low DP. In contrast to large polymer chains, small oligosaccharides, such as maltotriose and maltoheptose, can penetrate amorphous regions of the starch granule (Brown & French, 1977). Thus at their low concentration the competition between swelling starch granules and oligofructans for water may be less important. At higher levels of HSI addition, however, a significant decrease of maximum viscosity, viscosity at 96 °C

**Table 2**Parameters of Burgers model of gluten free dough with inulin (mean value of two replications ± standard deviation).

Sample		$J_0 \times 10^3 \ [Pa^{-1}]$	$J_1 \times 10^3  [Pa^{-1}]$	$\lambda_{ret}$ [s]	$\eta_0  imes 10^{-4} \  ext{[Pa s]}$
Control		$0.97 \pm 0.11^{ab}$	$2.15 \pm 0.22^{a}$	75.6 ± 2.4 <sup>a</sup>	$5.81 \pm 0.19^{a}$
	4%	$1.09 \pm 0.11^{ab}$	$2.56 \pm 0.11^{ac}$	$71.1\pm0.8^{ab}$	$4.82 \pm 0.43^{b}$
HSI	8%	$1.49 \pm 0.01^{c}$	$2.98 \pm 0.13^{bc}$	$61.4 \pm 3.0^{cd}$	$3.12 \pm 0.14^{c}$
	12%	$1.77 \pm 0.08^{d}$	$3.45 \pm 0.18^{b}$	$60.8 \pm 3.2^{cd}$	$2.45 \pm 0.03^{c}$
	4%	$1.10 \pm 0.08^{a}$	$2.20 \pm 0.15^{a}$	$65.5 \pm 1.5^{de}$	$4.28 \pm 0.14^{b}$
GR	8%	$1.42 \pm 0.11^{c}$	$3.02\pm0.46^{bc}$	$63.1 \pm 1.4^{d}$	$3.14 \pm 0.13^{c}$
	12%	$1.49 \pm 0.11^{c}$	$3.21\pm0.24^{b}$	$66.2 \pm 1.4^{\text{bde}}$	$2.96 \pm 0.22^{c}$
	4%	$1.02\pm0.07^{ab}$	$2.40 \pm 0.31^{a}$	$63.6\pm3.0^{\rm d}$	$4.42\pm0.27^{\mathrm{b}}$
HPX	8%	$1.10 \pm 0.04^{a}$	$2.11 \pm 0.23^{ad}$	$70.1 \pm 3.5^{be}$	$5.75 \pm 0.88^{a}$
	12%	$0.88\pm0.09^{\mathrm{b}}$	$1.59\pm0.01^{d}$	$57.3\pm1.3^{c}$	$7.94\pm0.72^{d}$
One-way ANOVA	- p	<0.001	<0.001	0.001	0.001
Two-way ANOVA	– p				
Factor I (type)	•	< 0.001	<0.001	0.647	< 0.001
Factor II (level)		<0.001	0.046	0.009	0.117
Factor I × Factor II		<0.001	0.002	0.002	<0.001

Mean values signed this same letters in particular columns are non-significant different at 0.05 level of confidence.

and final viscosity was observed. HPX and GR inulin blends presented similar effect on viscosity, with a decrease of this parameter with the increase of inulin level. The pattern observed for pasting of gluten-free mixes at large excess of water may be caused by various factors. Inulin is highly hydrophilic (Gennaro et al., 2000; Peressini & Sensidoni, 2009) and reduces water availability for swelling starch granules, as well as for structure forming hydrocolloids present in dough mixes. Limited swelling of starch decreases their volume, and consequently friction, which is manifested by lower maximum viscosity. Additionally oligosaccharides present in inulin samples, and sucrose added to dough mixes dissolved in a continuous phase may play a role of plasticizers for starch granules. Final viscosity, after cooling of starch paste, is related to a formation of spacial network by amylose molecules, which leached from the granules. Its decrease may be expected when leaching of amylose is limited by the presence of oligosaccharides, or when interactions between amylose molecules are disturbed by the presence of fructan chains.

## 3.3. Thermal characterization of dough mixes

Fig. 4a demonstrates a typical DSC curve obtained for starch based dough mix. Similar curves, with two characteristic peaks, were registered for all analyzed samples. It is not strange, taking into account that dough formulations are based on two starches with different gelatinisation temperatures. Potato starch gelatinizes at lower temperatures, in the range 57–78 °C, while corn

starch in slightly higher region, between 62 and 84 °C (Singh, Singh, Kaur, Singh Sodhi, & Singh Gill, 2003), although other authors report more overlapping values: 60-76 °C for potato and 57-84 for corn starch (Abdel-Aal, 2009). Potato starch also exhibits somewhat lower peak temperature than corn starch. According to Singh et al. (2003) the difference is 4°C, while Onyango, Mutungi, Unbehend, and Lindhauer (2011) estimate it to be 7.6 °C. In the case of the examined dough samples onset temperature  $(T_{\text{Og}})$  was in the range 64.2-68.1 (Table 4), which was higher than the values for potato starch. This discrepancy is probably due to the presence of sucrose and hydrocolloids, especially pectins, which could increase onset temperature (Tester & Sommerville, 2003). Addition of inulin resulted in elevation of onset temperature when inulin was added at levels 8 and 12%. Two way analysis of variance proved that type and level of inulin had significant effects on onset gelatinization temperature. The results are in accordance with pasting characteristics (Table 3). Endset temperature ( $T_{\rm Eg}$ ) of the examined systems changed from 94.8 to 99.7 °C (Table 4), and considerably exceeded the values for both potato and corn starch (Abdel-Aal, 2009; Singh et al., 2003). This might be explained by the limited availability of water needed for gelatinization. Potato starch swells and gelatinizes easily thus reducing the amounts of water available for corn starch. Additionally the presence of structure forming hydrocolloids (pectin and guar gum) limits hydration of starch, and inhibits phase transitions. As it was reported by Tester and Sommerville (2003) non-starch hydrocolloids (among others pectin and guar gum) profoundly modify starch gelatinization by prohibiting water

**Table 3**Gelatinization parameters of gluten free dough with inulin (mean value of three replications ± standard deviation).

Sample		Pasting temperature [°C]	Maximum viscosity [BU]	Viscosity after 10 min in 96 °C [BU]	Final viscosity [BU]	
Control		76.3 ± 2.3	81.3 ± 5.8 <sup>a</sup>	$62.0\pm4.6^{ab}$	115.3 ± 6.5 <sup>ab</sup>	
	4%	$74.8 \pm 1.4$	$79.7 \pm 4.0^a$	$64.7 \pm 4.0^{a}$	$116.3 \pm 2.5^{a}$	
HSI	8%	$77.5 \pm 1.3$	$68.0\pm2.6^{bd}$	$55.0 \pm 2.6^{be}$	$98.3 \pm 1.5^{def}$	
	12%	$78.0 \pm 5.3$	$60.7\pm10.3^{cde}$	$53.0 \pm 11.8^{cde}$	$90.7 \pm 15.3^{cf}$	
	4%	$78.3 \pm 1.1$	$71.0\pm2.0^{\rm b}$	$57.0\pm1.0^{abe}$	$105.0 \pm 2.0^{bd}$	
GR	8%	$79.6 \pm 1.0$	$62.7 \pm 3.2^{de}$	$50.3 \pm 1.2^{cde}$	$94.0\pm3.0^{ef}$	
	12%	$80.3\pm0.8$	$55.7 \pm 2.5^{ce}$	$46.0 \pm 3.6^{d}$	$82.7 \pm 6.7^{c}$	
	4%	$76.9 \pm 1.7$	$71.0\pm1.7^{\mathrm{b}}$	$54.0 \pm 2.0^{ce}$	$103.3 \pm 5.1^{de}$	
HPX	8%	$78.9 \pm 1.3$	$62.7 \pm 1.5^{de}$	$51.3 \pm 0.6^{cde}$	$94.7 \pm 1.2^{\text{def}}$	
	12%	$80.2\pm1.3$	$53.7 \pm 1.2^{c}$	$46.3\pm1.5^{cd}$	$82.7\pm1.2^{c}$	
One-way ANOVA – p		0.082	<0.001	<0.001	<0.001	
Two-way A	NOVA – p					
Factor I (type)		0.03	0.000	0.000	0.000	
Factor II (level)		0.04	0.000	0.006	0.015	
Factor I × Factor II		0.967	0.926	0.755	0.752	

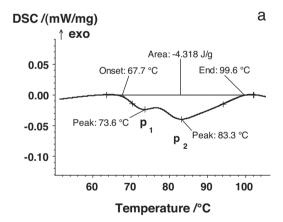
Mean values signed this same letters in particular columns are non-significant different at 0.05 level of confidence.

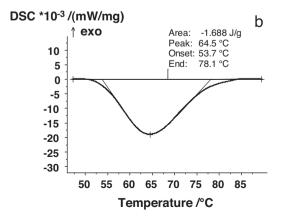
**Table 4**Thermal characteristics of gluten free dough with inulin (mean value of three replications ± standard deviation).

Sample		Starch gelatinization					Melting of starch after retrogradation			
		T <sub>Og</sub> [°C]	$T_{\rm P1g} \ [^{\circ} \rm C]$	T <sub>P2g</sub> [°C]	T <sub>Eg</sub> [°C]	$-\Delta H_{ m g}$ [J/g starch]	T <sub>Or</sub> [°C]	$T_{\mathrm{Pr}} \ [^{\circ}\mathrm{C}]$	T <sub>Er</sub> [°C]	-ΔH <sub>r</sub> [J/g starch]
Control		$64.2 \pm 0.06^{\circ}$	$70.1 \pm 0.17^{c}$	$79.0 \pm 0.10^{e}$	$94.8 \pm 0.31^{a}$	11.0 ± 0.24	51.9 ± 0.56 <sup>cde</sup>	$62.3\pm0.20^a$	$76.8 \pm 0.91^{ac}$	$5.06 \pm 0.11^{a}$
	4%	$65.7\pm0.06^a$	$71.2\pm0.06^a$	$80.6\pm0.25^a$	$98.1 \pm 1.97^{b}$	$10.6\pm0.52$	$52.9\pm0.64^{ab}$	$64.5\pm0.10^{de}$	$78.6 \pm 0.80^{b}$	$3.81 \pm 0.11^{e}$
HSI	8%	$66.9 \pm 0.26^{b}$	$72.6 \pm 0.06^{b}$	$82.2\pm0.15^{d}$	$99.5 \pm 0.36^{b}$	$11.1 \pm 0.37$	$54.1 \pm 1.01^{f}$	$64.7\pm0.25^{de}$	$76.5\pm1.06^a$	$3.93 \pm 0.26^{bce}$
	12%	$68.1 \pm 0.17^{d}$	$73.8 \pm 0.15^{\rm f}$	$83.6 \pm 0.12^{g}$	$99.3 \pm 0.95^{b}$	$11.2 \pm 0.57$	$52.4\pm0.46^{ade}$	$64.5\pm0.38^{de}$	$78.5\pm0.64^{\rm b}$	$3.75 \pm 0.20^{e}$
	4%	$65.7\pm0.12^a$	$71.5\pm0.15^a$	$80.7\pm0.42^{ac}$	$98.5 \pm 2.15^{b}$	$11.1\pm0.41$	$53.1\pm0.20^{abg}$	$64.3\pm0.06^{be}$	$76.8\pm0.45^{ac}$	$3.91 \pm 0.08^{bce}$
GR	8%	$66.6 \pm 0.01^{e}$	$72.7\pm0.17^{b}$	$81.9 \pm 0.15^{d}$	$99.1\pm0.36^b$	$10.7\pm0.29$	$53.3\pm0.26^{bfg}$	$64.8\pm0.75^{d}$	$76.8\pm0.15^{ac}$	$4.14 \pm 0.18^{bcd}$
	12%	$67.6 \pm 0.15^{f}$	$73.4 \pm 0.20^{e}$	$83.1 \pm 0.20^{\mathrm{f}}$	$99.6\pm0.68^{b}$	$10.8\pm0.38$	$53.8\pm0.32^{\mathrm{fg}}$	$64.4\pm0.17^{de}$	$77.7\pm0.56^{bc}$	$4.24\pm0.16^{d}$
	4%	$65.5\pm0.21^a$	$71.2\pm0.10^a$	$80.1\pm0.49^{b}$	$98.3\pm1.94^{b}$	$11.7 \pm 0.19$	$51.3 \pm 0.15^{c}$	$64.5\pm0.25^{de}$	$78.4 \pm 0.10^{b}$	$4.27\pm0.07^{\rm d}$
HPX	8%	$66.3 \pm 0.15^{g}$	$71.9 \pm 0.40^{d}$	$80.4\pm0.06^{ab}$	$99.2 \pm 0.51^{b}$	$11.2 \pm 1.11$	$51.8 \pm 0.45^{cd}$	$63.6 \pm 0.21^{c}$	$78.7 \pm 0.17^{b}$	$4.17 \pm 0.21^{cd}$
	12%	$67.1\pm0.06^{b}$	$72.9\pm0.15^b$	$81.0\pm0.15^{c}$	$99.7\pm0.71^{b}$	$10.9 \pm 0.36$	$52.7 \pm 0.26^{abe}$	$63.8\pm0.06^{bc}$	$76.1\pm0.49^a$	$3.88\pm0.04^{\text{be}}$
One-way ANOVA – p < 0.001		p <0.001	<0.001	<0.001	0.004	0.379	<0.001	<0.001	<0.001	<0.001
Two-wa	y ANOVA -	- р								
Factor I (type)		≤0.001	≤0.001	≤0.001	0.991	0.281	≤0.001	≤0.001	0.032	0.002
Factor II (level)		_ ≤0.001	_ ≤0.001	_ ≤0.001	0.100	0.762	0.040	0.449	0.135	0.259
Factor I × Factor II		0.004	0.001	≤0.001	0.974	0.177	0.002	0.013	≤0.001	0.015

Mean values signed this same letters in particular columns are non-significant different at 0.05 level of confidence.

access to amorphous parts of the granules. Presence of inulin and oligosaccharides additionally stabilized crystalline regions of starch causing an increase of endset temperature. Based on two factorial analysis of variance it was concluded, that both type and level of inulin had significant impact on gelatinization temperatures of both peaks (Table 4). The rise in level of inulin preparations caused significant increase of temperatures of the first peak ( $T_{\rm P1g}$ ), and of the second one ( $T_{\rm P2g}$ ). All the average values of peak temperatures





**Fig. 4.** Typical DSC curves of gluten free dough with addition of 12% GR inulin: (a) starch gelatinization, (b) melting of amylopectin after retrogradation.

were significantly higher than for control sample without inulin. It demonstrates that various inulin preparations have different influence of thermal transitions of starch. According to the producer information the content of low molecular weight sugars is higher in HSI than in GR. Their presence could increase all characteristic temperatures of starch gelatinization (Perry & Donald, 2002). In the case of HPX inulin additional influence could be exerted by inulin–inulin interactions, which are suggested by Peressini and Sensidoni (2009). No significant influence of inulin addition was observed for gelatinization enthalpies (Table 4). The values are however lower than reported earlier by Singh et al. (2003) for potato starches (12.5–17.9J/g) and corn starches (12–14J/g), possibly due to the presence of hydrocolloids, which are known to reduce gelatinization enthalpy in systems with limited water availability (Tester & Sommerville, 2003).

Table 4 demonstrates values describing thermal transitions of samples after starch retrogradation, while Fig. 4b shows a corresponding DSC curve. Two factorial analysis of variance proved that the change in temperatures and enthalpy of the transition was mainly caused by the type of added inulin. Its level was important only in the case of onset temperature, and the influence of this factor was only slight (p = 0.04). It could be noticed that thermal transitions temperatures are higher after inulin incorporation, which might be caused by the presence of gel structure formed by this polymer. Another factor which could impact the values of transition temperatures could be limited water availability for crystals of retrograded amylopectin. Lower temperatures related to retrogradation are caused by the formation of smaller and/or less regular crystalline regions (Ronda & Roos, 2008). The degree of amylopectin recrystallization, manifested by enthalpy values, was in all cases diminished by the addition of inulin. At lower addition levels, the effect was more pronounced for soluble inulin (HSI and GR). However an increase in addition level did not result in further changes. On the other hand gradual slowdown of recrystallization degree with addition level was observed for HPX inulin. Therefore it might be expected that retrogradation was inhibited mainly by the oligosaccharides, present in HSI and GR preparations, and additionally by gel network formed by inulin. According to Kim et al. (2001) the increase in heating temperature and concentration of inulin results in its partial hydrolysis. The occurrence of low molecular weight sugars leads to a decrease in starch recrystallization (Aee, Hie, & Nishinari, 1998; Babić et al., 2009) with effects similar to those found after addition of HSI and GR samples.

#### 4. Conclusions

Basing on the obtained results it could be concluded that the presence of inulin significantly modifies rheological and thermal properties of gluten free dough. The changes depend on the type of inulin, i.e. the degree of polymerization and the presence of low molecular weight sugars in the formulation and the level of its contribution in the recipe. Modified properties included lower values of consistency, namely apparent viscosity, storage and loss moduli, retardation time and zero shear viscosity, and higher values of compliance. Presence of inulin in dough formulations resulted also in a significant decrease of paste viscosity, and increase in gelatinization temperatures  $T_{\text{Og}}$ ,  $T_{\text{P1g}}$ ,  $T_{\text{P2g}}$ ,  $T_{\text{Eg}}$ . In the case of inulins with lower degree of polymerization (HSI and GR) thermal parameters could be significantly modified by the presence of low molecular weight sugars, while inulin-inulin interactions could play a role for inulin with high DP (HPX). No effect was observed for gelatinization enthalpy ( $\Delta H_g$ ). It was however stated, that the addition of inulin significantly reduces enthalpy of retrograded amylopectin, which may suggest its role in bread staling retardation. Water binding ability of inulin seems to be the key factor in modifying rheological and thermal properties of the system, because its limited availability for other dough constituents results in restrained starch swelling and lower hydration of structure forming hydrocolloids, and consequently dough weakening. In the design phase of gluten-free products with added inulin it is then especially important to choose a preparation with appropriate DP and properly adjust amounts of water used in the recipe.

## Acknowledgement

The authors want to thank Biesterfeld Chemia Specjalna, Polska for donating inulin preparations.

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