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RHEOLOGICAL PROPERTIES OF FREEZE-DRIED WHEAT STARCH/GALACTOMANNAN GELS

Lesław Juszczak[1], Teresa Fortuna[1], Mariusz Witczak[2], Anna Dymel[1]

[1]Department of Analysis & Evaluation of Food Quality, [2]Department of Engineering and Machinery for Food Industry; University of Agriculture, Cracow

Key words: wheat starch, galactomannans (guar, carob), freeze-drying, rheological properties

The objective of the present work was to determine the effect of galactomannans (guar and carob) on some rheological properties of freeze--dried wheat starch gels. Temperature-dependent apparent viscosity curves, flow curves, shear time-dependent apparent viscosity curves as well as mechanical spectra were plotted. On the basis of the results obtained, it can be said that both freeze-drying and gum addition to the system modify rheological properties of wheat starch pastes/gels, and that these changes depend on the type and concentration of the gum used. In the investigated systems with galactomannans, higher values of apparent viscosity were observed along with increasing temperature as well as higher values of shear stress in the flow time, as compared to native starch. It should be stressed, however, that the guar gum system was characterised by higher viscosity values than that with carob gum. The presence of particular gums in the systems modified also viscoelastic properties of gels obtained, depending on the gum used and its concentration.

INTRODUCTION

Starches of different origin and their modified preparations are the most important polysaccharide hydrocolloids. They are used in the food production as thickening agents, stabilisers, gelling-aid agents, and emulgators. Due to their capability of improving water retention, they are used to improve textural properties, and due to low energy value – in the production of low-caloric foodstuffs [Fortuna, 1995; Golachowski, 1998; Tomasik, 2000]. Apart from the food industry, starch has been applied in other industrial branches [Leszczyński 1998; Tomasik, 2000]. Such a wide application of starch results from its structure and differentiated physico-chemical properties [Leszczyński, 2001; Parker & Ring, 2001]. The use of native starches may be highly inconvenient due to retrogradation and syneresis phenomena as well as low mechanical and thermal stability observed in such material.

Starch is modified in different manner in order to change its physico-chemical properties as well as to obtain desired functional properties. In terms of legal status, chemically-modified starches are food additives and their use is restricted. In their case, the use of interaction between starch polymers and other polysaccharides may be an alternative [Kulicke *et al.*, 1996]. According to Christianson [1982], synergistic interactions in starch-gum systems result from interactions between hydrocolloid and soluble fraction of starch – amylose. Moreover, the addition of non-starch hydrocolloid increases forces acting on swollen starch granules, thus disarranges their structural integrity. An increase in the viscosity and changes in the texture of such systems depend on the starch and gum used, manner of paste/gel preparation, and presence of other substances [Christianson, 1982; Alloncle *et al.*, 1989; Alloncle & Doublier, 1991; Bahnassey & Breene, 1994; Eidam *et al.*, 1995; Sudhakar *et al.*, 1995, 1996; Biliaderis *et al.*, 1997; Tecante & Doublier, 1999; Lo & Ramsden, 2000].

Upon modification, starch is often subjected to the activity of physical factors leading to a partial or total degradation of starch structure, which results mainly from disruption of hydrogen bonds stabilising the conformational structure of macromolecules [Leszczyński, 1992]. In one of the methods of starch modification, starch granules are pasted and the gel obtained is drum-, spray- or freeze--dried. Drum drying is applied on the industrial scale and properties of preparations obtained depend on the type of starch and process conditions used [Doublier *et al.*, 1986; Anastasiades *et al.*, 2002; Kalogianni *et al.*, 2002]. Although not used on the industrial scale for dehydration of starch gels, freeze-drying enables obtaining modified preparations of high solubility as well as porous and crunchy consistency [Hofmann *et al.*, 1998]. According to Yano and Nagai [1989], freeze-dried starch gels are characterised by a greater specific surface area as well as volume and diameter of mesophores. Properties of freeze-dried starch are to a great extent affected by temperature and pressure of the process [Spieles *et al.*, 1995]. Freeze-dried starch gels may as well be used in the production of biodegradable packagings [Hofmann *et al.*, 1998] and in the pharmaceutical industry as a matrix of active substances [Sanchez *et al.*, 1995].

In the last 25 years, the world wide production of starch increased *ca.* 4.5-fold [Leszczyński, 2001]. An increased economic demand for new starch materials enforces intensification of studies on their structure, properties, manners of modification, and possible application.

Author's address for correspondence: Lesław Juszczak, Katedra Analizy i Oceny Jakości Żywności, Akademia Rolnicza w Krakowie, ul. Balicka 122, 30-149 Kraków, fax: (48 12) 662 47 46; e-mail: rrjuszcz@cyf-kr.edu.pl

The objective of the present study was to determine the effect of galactomannans (guar and carob gums) on the rheological properties of freeze-dried wheat starch gels, using the starch-gum interactions during pasting/gelling processes and freeze-drying process to dehydrate gel systems.

MATERIALS

The experimental material consisted of: galactomannans - guar (Hortimex, Poland) and carob (Regis, Poland), and wheat starch (Kröner Stärke GmbH, Germany). The investigated starch contained 22.5 g/100 g amylose [Morrison & Laignelet, 1983], 0.34 g/100 g crude fat, and 0.23 g/100 g protein (N x 6.25).

Preparation of freeze-drying gels. Suspensions of starch at the concentration of 10 g/100 g (w/w) or starch with galactomannan addition (guar or carob) at the concentrations of 0.25 g/100 g or 0.50 g/100 g were heated at 95°C for 30 min at continuous stirring (2000 rpm). After cooling, the gels were frozen at -15°C for 24 h. Then, the samples were freeze-dried in a Labor MIM OE-950 freeze-dryer at a temperature range from -25°C to 20°C and pressure of *ca.* 103 Pa. Freeze-drying was continued to the moisture content of the samples reaching *ca.* 3 g/100 g. Next, the samples were comminuted in a laboratory mill and sieved (0.43 mm). The preparations sieved were left at room temperature for 24 h in order to obtain the equilibrium moisture (*ca.* 5 g/100 g).

Preparation of pastes/gels for rheological measurements (flow curves, mechanical spectra). Suspensions of native starch or preparations obtained at the concentration of 5 g/100 g were heated at a temperature of 95°C for 30 min at continuous stirring (250 rpm). Hot paste was put into a measuring system, allowed to relax, and cooled to the measuring temperature (50°C or 25°C) for 15 min.

METHODS

Rheological studies were performed in the following systems: native wheat starch (NS), freeze-dried native starch gel (FDNSG), freeze-dried wheat starch gel with guar gum addition (FDSG/G), freeze-dried wheat starch gel with carob gum addition (FDSG/G).

For the preparations obtained, temperature-dependent apparent viscosity curves (at constant shear stress of 300 s^{-1}) were determined using a rotary viscometer Rheolab MC1 (Physica Messtechnik, Germany) with a system of coaxial cylinders (cup diameter 48.8 mm, bob diameter 45.0 mm). It should be mentioned that the internal cylinder had helical grooves to prevent sedimentation. Suspensions of the preparations obtained were heated and then cooled according to the following programme: temperature increase from 50 to 95°C (for 40 min), constant temperature of 95°C for 20 min, and cooling at a temperature decrease from 95 to 50°C (for 40 min). Due to fast sedimentation of starch granules, this measurement was not performed for native starch.

Flow curves and apparent viscosity time-dependent curves were determined at a temperature of 50°C, using a rotary viscometer Rheolab MC1 (Physica Messtechnik, Germany) with a system of coaxial cylinders (cup diameter 27.12 mm, bob diameter 25.00 mm).

Flow curves were plotted at the shear rate increasing from 1 to 600 s^{-1} for 10 min. The experimental curves obtained were described with a power law model.

Apparent viscosity time-dependent curves were determined at a constant shear rate (100 s^{-1}, for 15 min).

Mechanical spectra were estimated at 25°C, with the use of a rheometer RheoStress RS 150 (Haake, Germany) with cone/plate geometry (cone diameter 35 mm, angle 2°) at the gap width of 0.105 mm. Measurements were performed for linear viscoelasticity at constant amplitude of strain (0.03) and frequency range of 0.1–10 Hz.

RESULTS AND DISCUSSION

Figure 1 shows temperature-dependent apparent viscosity curves for suspensions of freeze-dried gels of native starch and for freeze-dried preparations with galactomannans. In the case of native starch suspension, measurement was not possible due to fast sedimentation of starch granules. Suspensions of freeze-dried gels with galactomannans showed considerably higher values of apparent viscosity than these of the native starch. The viscosity values were higher with a higher concentration of galactomannans in the system (curves with 0.25 g/100 g galactomannans were not shown). In the case of freeze-dried gels with guar and carob gums, a substantial increase in the apparent viscosity value was observed also at cooling. It should be emphasised that freeze-dried gels with guar were characterised by higher viscosity values compared to these with carob. Similar dependency was reported also by Alloncle *et al.* [1989] for corn starch pasted in the presence of galactomannans, which could be in part explained by a lower molecular weight of carob gum. Synergistic increase in the viscosity of wheat starch pasted in the presence of guar gum was noted also by Shi and BeMiller [2002]. On the other hand, Bahnassey and Breene [1994] observed a stronger effect of carob on the changes in the viscosity of native starches pasted with galactomannans. According to Christianson [1982], synergistic increase in the viscosity during pasting of starch-galactomannans systems results from interactions of amylose and low-molecular amylopectin fraction with gum and from the fact that the addition of gum to the suspension increases forces acting on starch granules, which in turn may accelerate release of amylose. When it comes to the freeze-dried gel of native starch, low viscosity of the system may be additionally caused by amylose retrogradation at cooling, and then gel freezing prior to freeze-drying. Wheat starch

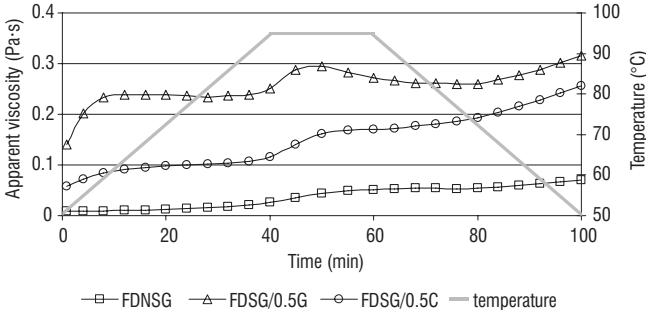

FIGURE 1. Temperature-dependent apparent viscosity curves: □ – freeze-dried wheat starch gel (FDNSG), △ – freeze-dried gel with 0.50 g/100 g addition of guar gum (FDSG/0.5G), ○ – freeze-dried gel with 0.50 g/100 g addition of carob gum (FDSG/0.5C).

is especially susceptible to this process [Błaszczak *et al.*, 2001]. Amylose dendrites formed during the retrogradation process are not easily hydrated, therefore the structure of continuous phase of the paste, formed by amylose, is more susceptible to shearing. Moreover, a part of retrogradaed amylose may not be involved in formation of the paste continuous phase. Since in the course of starch pasting, galactomannans are aggregated in the paste continuous phase, they inhibit the retrogradation process at cooling, which in turn favours rehydration of freeze-dried starch gels with guar addition.

Flow curves of native starch pastes, freeze-dried native starch, and freeze-dried preparation of starch with galactomannans were plotted in Figure 2. All systems examined revealed non-Newtonian, shear thinning behaviour. As in the case of temperature-dependent apparent viscosity curves, the highest values of shear stress were reported for the preparation with guar gum, whereas the lowest ones – for the paste obtained from freeze-dried native starch gel. It was confirmed by parameters of the power law model compiled in Table 1. The lowest consistency coefficient, indicating viscosity, was found for the paste of freeze-dried native wheat starch gel. Pastes of freeze dried preparations with galactomannans were characterised by substantially higher values of consistency coefficients increasing along with higher concentration of the gum, although, guar gum systems showed higher viscosity values compared to the carob systems. Values of flow behaviour index (Table 1) prove considerable shear thinning of the examined systems. It should be stressed, however, that the presence of guar gum in the system substantially lowered the index value. Increased shear thinning behaviour of maize starch pastes with galactomannans was previously observed by Sudhakar *et al.* [1996].

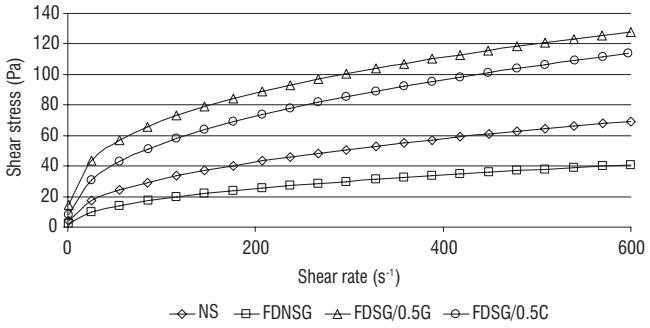

FIGURE 2. Flow curves of pastes: ◇ – native wheat starch (NS), □ – freeze-dried wheat starch gel (FDNSG), △ – freeze-dried gel with 0.50 g/100 g addition of guar gum (FDSG/0.5G), ○ – freeze-dried gel with 0.50 g/100 g addition of carob gum (FDSG/0.5C).

TABLE 1. Rheological parameters of power low model of investigated pastes.

Sample	Consistency coefficient ($Pa \cdot s^n$)	Flow behaviour index (-)	R^2
Native wheat starch	4.15	0.44	0.9989
Freeze-drying wheat starch gel	2.84	0.43	0.9988
Freeze-drying wheat starch gel + 0.25 g/100g guar gum	7.54	0.36	0.9939
Freeze-drying wheat starch gel + 0.50 g/100g guar gum	14.51	0.34	0.9838
Freeze-drying wheat starch gel + 0.25 g/100g carob gum	6.61	0.41	0.9992
Freeze-drying wheat starch gel + 0.50 g/100g carob gum	8.27	0.41	0.9814

Differences in the viscosity values of the investigated pastes are confirmed also by the apparent viscosity time-dependent curves (Figure 3). As in the case of flow curves, pastes of freeze-dried starch/gum preparations revealed considerably higher values of apparent viscosity. While the lowest apparent viscosity values were reported for the paste obtained from freeze-dried native starch preparation. No significant differences were, however, found with reference to apparent viscosity decrease at constant shearing. Therefore it can be assumed that these systems will show similar rheological stability. According to Alloncle *et al.* [1989], starch paste may be described as a two-phase system, wherein continuous phase is formed by soluble starch fraction – amylose, and particles of dispersed phase are formed by amylopectin. Galactomannans present in the system aggregate in the continuous phase and interact with amylose, which consequently evokes viscosity increase. Synergistic increase in the viscosity of freeze-dried wheat starch preparations with galactomannans results from the same interactions. Additional role played by galactomannans in these preparations consists in the inhibition of amylose retrogradation during cooling and then freezing of gels before freeze-drying, which makes further rehydration easier.

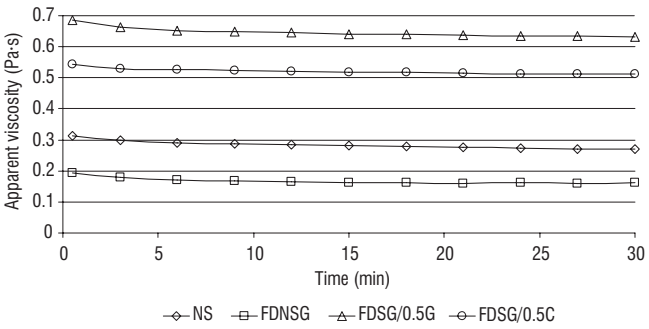

FIGURE 3. Apparent viscosity time-dependent curves of pastes: ◇ – native wheat starch (NS), □ – freeze-dried wheat starch gel (FDNSG), △ – freeze-dried gel with 0.50 g/100 g addition of guar gum (FDSG/0.5G), ○ – freeze-dried gel with 0.50 g/100 g addition of carob gum (FDSG/0.5C).

Mechanical spectra of the investigated systems were shown in Figures 4 and 5. The systems showed behaviour typical of gels (G' > G"). Significantly lower values of both moduli were observed for the gel obtained from freeze-dried starch preparation compared to the native starch gel. The addition of galactomannans considerably modified viscoelastic properties of the systems under study. In the case of both guar (Figure 4) and carob (Figure 5) addition, a substantial increase in the loss modulus value was observed compared to that reported for the gel from native starch and freeze-dried native starch. An increase in the storage modulus value was higher along with higher concentration of galactomannans in the system. It should be emphasised, though, that the storage modulus values were higher in gels with carob (Figure 5) than in these with guar gum (Figure

4). Different changes were observed for the values of storage modulus which when noted for the systems with guar gum (Figure 4) were considerably higher compared to these noted for the gel of freeze-dried starch preparation and lower compared to the native starch gel. When it comes to the carob systems, changes in the values of the storage modulus were insignificant especially in a higher frequency range, compared to the native starch gel. On the other hand, the values of storage modulus of both the native starch gel and freeze-dried preparations with carob were considerably higher than these of the gel obtained from the freeze-dried native starch.

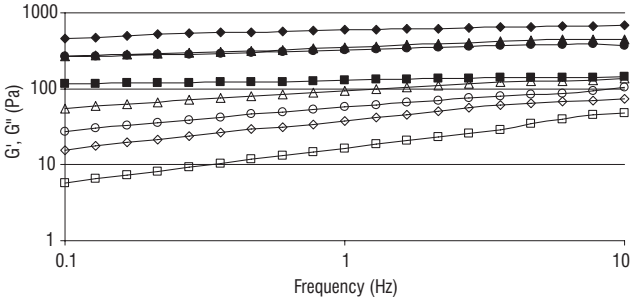

FIGURE 4. Mechanical spectra (G' – filled markers; G" – empty markers): a) native wheat starch gel (diamonds), b) freeze-dried wheat starch gel (squares), c) freeze-dried gels with 0.25 g/100 g (circles) or 0.25 g/100 g (triangles) addition of guar gum.

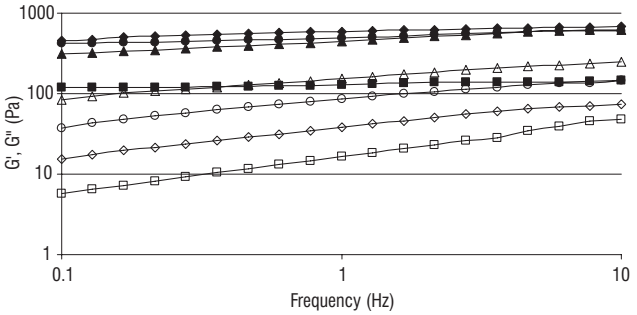

FIGURE 5. Mechanical spectra (G' – filled markers; G" – empty markers) a) native wheat starch gel (diamonds), b) freeze-dried wheat starch gel (squares), c) freeze-dried gels with 0.25 g/100 g (circles) or 0.25 g/100 g (triangles) addition of carob gum.

According to Alloncle and Doublier [1990], galactomannans exert a considerable effect on retrogradation of amylose and kinetics of starch gel formation. The same authors noted also that in the corn starch-guar gum systems the values of the loss modulus are substantially higher than these of the storage modulus both in hot starch paste and in fully-formed gel [Alloncle & Doublier, 1991]. Considerably higher increase in the loss modulus values, than in values of the storage modulus, were reported also by Eidam *et al.* [1995] in the corn starch-galactomannan systems compared to native starch. Although the increase, especially in the storage modulus, was to a low degree affected by the fact whether the guar gum was added to the systems as polysaccharide or substituted a part of starch. Acceleration of starch gel formation by gums may result from the thickening effect. In hot starch paste, particles of gum limit mobility of amylose particles, therefore local association of helical components and formation of junction zones proceed easier and faster [Eidam *et al.*, 1995]. On the other hand, waxy starches are affected by galactomannans in a different manner. Kulicke *et al.* [1996] found out that galactomannan addition to the systems with waxy rice starch causes disappearing of platou region of the storage modulus, and difference in the values of both moduli decreases to a high extent.

CONCLUSIONS

On the basis of the study conducted, it was stated that both freeze-drying and gum addition to systems modify rheological properties of wheat starch pastes/gels, and the consequent changes depend on the gum used and its concentration. In the investigated systems with galactomannan addition, higher values of apparent viscosity (depending on temperature), higher values of shear stress (depending on shear rate) and higher values of apparent viscosity (depending of shear time) were observed compared to native starch, although systems with guar gum revealed higher viscosity than these with carob gum. The addition of the mentioned gums to the systems modified also viscoelastic properties of gels obtained, depending on the type and concentration of gum. The mechanism of modification of freeze-dried wheat starch gels by galactomannans is analogous to other cereal starches. In this case, inhibition of retrogradation process by galactomannans is of special importance. The use of synergistic interactions in the starch-gum systems seems to be an alternative for chemically-modified starch on the one hand, and a helpful tool in creation of rheological and functional properties of modified starch preparations on the other.

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WŁAŚCIWOŚCI REOLOGICZNE LIOFILIZOWANYCH ŻELI SKROBI PSZENNEJ Z DODATKIEM GALAKTOMANNANÓW

Lesław Juszczak[1], Teresa Fortuna[1], Mariusz Witczak[2], Anna Dymel[1]

[1]Katedra Analizy i Oceny Jakości Żywności, [2]Katedra Inżnierii i Aparatury Przemysłu Spożywczego; Akademia Rolnicza w Krakowie, Kraków

Celem pracy było określenie wpływu galaktomannanów (guaru i carobu) na niektóre właściwości reologiczne liofilizowanych żeli skrobi pszennej. Wykreślono krzywe zależności lepkości pozornej od temperatury, krzywe płynięcia oraz krzywe zależności lepkości pozornej od czasu ścinania, a także mechaniczne spektra. Na podstawie przeprowadzonych badań stwierdzono, że zarówno proces liofilizacji jak również udział gum w układzie modyfikują właściwości reologiczne kleików/żeli skrobi pszennej, a zmiany te zależą do rodzaju i stężenia zastosowanej gumy. Dla badanych układów z udziałem galaktomannanów zaobserwowano większe wartości lepkości pozornej wraz ze wzrostem temperatury oraz wzrost wartości naprężeń stycznych w czasie płynięcia w stosunku do skrobi natywnej. Przy czym układy z gumą guar charakteryzowały się większą lepkością niż z gumą carob. Udział poszczególnych gum w układach modyfikował również właściwości lepkosprężyste otrzymanych żeli, w zależności od zastosowanej gumy oraz jej stężenia.