Carbohydrate Polymers 121 (2015) 254–264

Contents lists available at ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

Thixotropic properties of normal potato starch depending on the degree of the granules pasting

Marek Sikora[a,*], Greta Adamczyk[a], Magdalena Krystyjan[a], Anna Dobosz[a], Piotr Tomasik[b], Wiktor Berski[a], Marcin Lukasiewicz[a], Piotr Izak[c]

[a] University of Agriculture in Krakow, Faculty of Food Technology, Department of Carbohydrates Technology, Balicka Street 122, 30-149 Krakow, Poland
[b] Krakow College of Health Promotion, Krowoderska Street 73, 31-158 Krakow, Poland
[c] University of Science and Technology, Faculty of Materials Science and Ceramics, Mickiewicza Street 30, 30-059 Krakow, Poland

ARTICLE INFO

Article history:
Received 8 October 2013
Received in revised form 5 September 2014
Accepted 22 December 2014
Available online 3 January 2015

Keywords:
Antithixotropy
Degree of structure recovery
Rheological instability
Thixotropy

ABSTRACT

The aim of this paper was the study of the rheological instability (thixotropy and/or antithixotropy) of normal potato starch (NPS) pastes depending on their concentration (2–5%) and degree of pasting. Flow curves with hysteresis loops, apparent viscosity at constant shear rate and in-shear structural recovery tests were carried out. Granule size profiles, the pasting characteristic of corresponding starch suspensions and the transmittance of the pastes, the molecular weights and polydispersity of granular starch and its pastes prepared at 80, 95 and 121 °C were also studied.

The degree of pasting was dependent on the temperature and the concentration and influenced strongly the rheological behavior of the pastes. All pastes belonged to the non-Newtonian liquids thinned by shear and were rheologically unstable to the various extent. Thixotropic properties were connected to the size and the number of the starch granules in the pastes as well as depended on the measuring method used. In the 2 and 3% samples pasted at 80 °C the swelling of the granules prevailed their destruction (thixotropy was observed). In the other samples the destruction predominated the swelling (antithixotropy observed).

1. Introduction

Starch is an abundant, renewable source for the food industry where it is used chiefly as a texture modifier. The properties of starch and its pastes and gels, the latter commonly applicable in practice, depend on several factors, such as the botanical origin of starch, the concentration of its sols and gels, shear rate, the history of shearing applied to samples, and the parameters of gelatinization (Dintzis & Bagley, 1995; Djaković, Soviljii, & Milosevic, 1990; Harrod, 1989; Lagarrigue & Alvarez, 2001). Recognizing the rheological properties of the pastes is necessary for the optimum utilization of starch in practical applications.

Starch pastes belong to non-Newtonian liquids of either thixotropic or antithixotropic properties (Dewar & Joyce, 2006; Nguyen, Jensen, & Kristensen, 1998). Thixotropy is considered as a property associated with the rheoinstability of gels and pastes of starch (Barnes, 1997; Sikora, Adamczyk, & Krystyjan, 2011; Steffe, 1996).

Thus, there is no consistent definition of thixotropy. Initially, it was defined as a phenomenon of the transformation of sol into gel which, on agitation, could return into liquid sol (Barnes, 1997). Currently, thixotropy is identified with any process in which after the destruction of the inner structure of the liquid the inner friction is reduced on shear. The same meaning is attributed to a slow return to the original consistency on rest (Barnes, 1997; Sikora et al., 2011; Steffe, 1996). Antithixotropy is defined as a phenomenon of the return to the original structure, which is faster than destroying the inner structure (Barnes, 1997; Sikora et al., 2011).

The thixotropy and antithixotropy of the pastes can be investigated involving several methods. The most common is the test of the hysteresis loop (Abu-Jdayil, Azzam, & Al-Malah, 2001; Achayuthakan & Suphantharika, 2008; Alloncle, Lefebvre, Llamas, & Doublier, 1989; Al-Malah, Azaam, & Abu-Jdayil, 2000; Djaković et al., 1990; Doublier, 1981; Fortuna & Gałkowska, 2006; Gambuś, Gumul, & Juszczak, 2004; Nguyen et al., 1998; Orczykowska, Dziubiński, & Budzyński, 2010; Tattiyakul & Rao, 2000; Wang et al., 2009; Wang, Li, Wang, & Özkan, 2010; Zhang, Gu, Hong, Li, & Cheng, 2011). The test provides flow curves arranged in the form of a loop, the area of which is considered as a measure of the energy delivered on shearing (Barnes, 1997; Sikora, Tomasik, Krystyjan, &

* Corresponding author. Tel.: +48 12 662 47 74.
E-mail address: rrsikora@cyf-kr.edu.pl (M. Sikora).

http://dx.doi.org/10.1016/j.carbpol.2014.12.059
0144-8617/

Krawontka, 2010; Steffe, 1996). The application of this test meets some limits as the time and shear rate change in a concerted manner (Barnes, 1997; Mewis & Wagner, 2009; Sikora et al., 2010; Wang et al., 2010). Moreover, the appearance of the hysteresis loop is not always associated with thixotropy as it can also be observed in the case of viscoelastic materials exhibiting the properties of rheostable liquids (Bird & Marsh, 1968; Wang et al., 2010). Therefore, an analysis of thixotropy is complemented with such methods as either the determination of the apparent viscosity on shearing at a constant rate either the method of steps of the shear rate (Wang et al., 2010).

There is only limited information on the thixotropic properties of starch gels and pastes. Relevant studies were carried out mainly for corn starch (Achayuthakan & Suphantharika, 2008; Alloncle et al., 1989; Djaković et al., 1990; Nguyen et al., 1998; Tattiyakul & Rao, 2000; Wang et al., 2009, 2010) and wheat starch (Abu-Jdayil et al., 2001; Alloncle et al., 1989; Al-Malah et al., 2000; Doublier, 1981; Gambuś et al., 2004). More detailed studies on the thixotropic properties of potato, cassava and oat starch are lacking. The available data on such properties of potato starch are limited to interpretation of the instability of potato starch pastes from the area of the hysteresis loops (Fortuna & Gałkowska, 2006; Orczykowska et al., 2010; Zhang et al., 2011) and the pattern of relationships of the viscosity changes against the constant shear rate (Fortuna & Gałkowska, 2006).

The aim of this paper was the presentation of the rheological instability (thixotropy and/or antithixotropy) of normal potato starch (NPS) pastes depending on their concentration and degree of pasting.

2. Materials and methods

2.1. Materials

Normal potato starch isolated from potatoes of the Tajfun variety, purchased from the Experimental Station of Variety Evaluation in Węgrzce, Poland (crops of 2011) had the following parameters: dry substance—86.92%, total phosphorus—111.9 mg/100 g d.m., fat—0.13%, amylose—31.99%, aqueous solubility—14.66% and 20.94% at 80 and 95 °C, and water binding capacity—37.34 and 69.34 g/1 g d.m. of starch at 80 and 95 °C, respectively.

2.2. Methods

2.2.1. Sample preparation

Pastes of 1–5% (w/w, d. m.) starch were prepared. The slurries of NPS in distilled water were stirred at 400 rpm for 10 min, at 25 °C, then heated and stirred in three manners (1) at 80 °C for 30 min, (2) at 95 °C for 30 min, (3) at 95 °C for 20 min at 400 rpm, followed by autoclaving (As-2, Spółdzielnia Pracy Mechaników "SMS", Warsaw, Poland) at 121 °C, 0.1 MPa, for 120 min.

2.2.2. Particle size analysis of starch granules

A laser particle size analyzer (Analysette 22, Fritsch GmbH, Idar-Oberstein, Germany) was used for the particle size analysis of the granular NPS. The samples of granular starch were dispersed in deionized water, at room temperature. Determinations were run in duplicate according to the instrument instruction. Particle size distribution (volume based) was calculated using the software provided by Fritsch.

2.2.3. Optical microscopy

Granular starch as well as starch pastes (prepared according to point Section 2.2.1) were analyzed. The samples were stained with iodine solution on the glass plates and submitted to the microscopic observations. The analyses were performed using an optical microscope Nikon Eclipse E200 (Nicon Instruments Inc., Tokyo, Japan) equipped with a digital camera (GKB high resolution ccd camera, CC-8706S, Taichung, Taiwan). The images from the camera were transferred to a computer and subjected to image analysis. The starch granules dimensions were measured using open source software ImageJ (v. 3.17) (Abramoff, Magalhaes, & Ram, 2004). Minimum, maximum and average diameters of the granules were determined. Average number of granules observed in micrograph (objects per picture) and particle size distribution (PSD) were calculated, The minimal number of objects necessary to prepare PSD was set at 500 (Vigneau, Loisel, Devaux, & Cantoni, 2000).

2.2.4. Molecular weight and molecular mass distribution

Analyses of average molecular weights as well as their distributions were measured by means of GPC (Lukasiewicz & Kowalski, 2012). Three columns: Ultrahydrogel-2000, Ultrahydrogel-500, and Ultrahydrogel-120 (Waters, Milford, USA) connected in a line with a RI detector (Knauer, Berlin, Germany) were used. NPS pastes (3%) were analyzed. The mixture of 0.1 M $NaNO_3$ and 0.02% NaN_3 solution in water was applied as an eluent. The flow rate of a sample (100 μL) was set to 0.6 mL/min. The sample concentration was ca. 5 mg/mL. In the case of granular starch the sample of ca. 30 mg was dissolved in a 3 mL of 1 M NaOH for at least 48 h. After solubilization was completed the sample was neutralized with 1 M HCl against phenolphthalein. Then it was filtered through a 0.45 μm syringe filter and injected into the system. All other samples of starch were pasted as described in Section 2.2.1. A 1 g sample was taken from the cooled paste. It was then dissolved in 3 mL of 1 M NaOH for 48 h at an ambient temperature. After the solubilization was completed the sample was neutralized and filtered as described for the granular starch. The analyses follow the standard calibration that was performed using pullulan standards (Shodex, New York, USA).

2.2.5. Pasting characteristics

A Micro Visco Amylo-Graph of Brabender (Duisburg, Germany) was used. 1–5% (w/w) suspensions of NPS were heated at 7.5 °C/min rate at the following temperature profile: 30–96 °C (maintained constant for 10 min)—50 °C (completed by cooling at the rate of 7.5 °C/min). The pasted samples were agitated at 250 rpm. Measurements were run in duplicate.

2.2.6. Measurements of transmittance

Hot pastes prepared according to Section 2.2.1 were poured into the sealed cells (delivered with the instrument) and cooled to 25 °C for 1 h. Measurements of the transmittance of 2–5% samples were performed using the Turbiscan, (LAB Expert, Formulation SA, L'Union, France) instrument, at 25 °C. Measurements of the parameters of the 1% sample was not carried out because this concentration was not high enough and the obtained trial results were unreliable. Therefore, this concentration was also omitted in the rheological measurements. The reading head of the instrument was equipped with a pulsed near infrared light source (880 nm) and two synchronous detectors. The transmission detector received the transmitted light flux through the paste. The reading head acquired the data every 40 μm while moving along the 55 mm cell height. The acquired data of each measurement were averaged. The measurements were performed in duplicate and the results were presented as an average transmittance of two measurements.

2.2.7. Rheological measurements

A Rheometer RheoStress RS 1 (Gebrüder Haake GmbH, Karlsruhe, Germany) equipped with Z 41 Ti coaxial cylinders was used for the characterization of the features of 2–5% samples. Hot pastes (immediately after their preparation according to Section 2.2.1) were loaded into the measuring system. Measurements were carried out at 50 °C, after equilibration for 5 minutes.

2.2.7.1. Steady flow measurements. The rate of shear was increased linearly from 0 to 300 s^{-1} within 600 s, followed by the constant shear rate of 300 s^{-1} for 120 s, and subsequent decrease (linear) to 0 s^{-1} within 600 s. Resulting flow curves were described by the Ostwald–de Waele rheological model (Steffe, 1996):

$$\tau = k \times \dot{\gamma}^n \quad (1)$$

where τ is the shear stress [Pa s], k is the consistency coefficient [Pa s^n], $\dot{\gamma}$ is the shear rate [s^{-1}], n is the flow behavior index [–].

The areas of thixo- and antithixotropy hysteresis loops (considered as a work executed for the destruction and rebuilding of the inner structure of a sample) were calculated by summing of the areas of particular trapeziums between the curve "up" and "down". The area of the single trapezium was calculated according to the following formula:

$$A_T = \frac{\left(\tau_{U(k-1)} - \tau_{D(k-1)}\right) + \left(\tau_{Uk} - \tau_{Dk}\right)}{2} \times \dot{\gamma}_a [\text{Pa} \ \ s^{-1}] \quad (2)$$

where A_T is the area of the single trapezium at a certain shear rate [Pa s]; $\tau_{D(k-1)}$, τ_{Dk}, are the shear stress values between two neighboring measuring points on the curve "down" [Pa]; $\tau_{U(k-1)}$, τ_{Uk} are the shear stress values between two neighboring measuring points on the curve "up" [Pa]; $\dot{\gamma}_a$ is the average difference of shear rates between neighboring measurement points of the curves "up" and "down" [1/s].

Assuming that the difference of shear rates between two neighboring measurement points of the curve "up" is the same as the difference of shear rates between two neighboring measurement points of the curve "down" $\dot{\gamma}_a$ can be calculated as follows:

$$\dot{\gamma}_a = \dot{\gamma}_k - \dot{\gamma}_{k-1} \quad (3)$$

$\dot{\gamma}_k$, $\dot{\gamma}_{k-1}$ are the values of shear rate (higher and lower, respectively) between two neighboring measuring points on the curves "up" and "down" [1/s].

The total hysteresis loops areas were calculated as the sum of the areas of thixo- and antithixotropy.

The graphs of the power of thixotropy in a volume of the sample (the first derivative of an oriented work executed for destruction and/or rebuilding of the inner structure of a sample over a range of time) were calculated as a function of the shear rates.

If $\Delta\dot{\gamma} \approx \Delta t \to 0$, in the case of the appropriate average shear rate (in the case of a single trapezium) the power of thixotropy for $\Delta t \to 0$ can be calculated according to the formula:

$$P_T = \frac{\left(\tau_{U(k-1)} - \tau_{D(k-1)}\right) + \left(\tau_{Uk} - \tau_{Dk}\right)}{2} \times \dot{\gamma}_p [\text{W} \ \ \text{m}^{-3}] \quad (4)$$

where P_T is the power of thixotropy in a volume of the sample, at a particular shear rate [W/m^3], $\dot{\gamma}_p = \dot{\gamma}_k - \dot{\gamma}_{k-1}/2$ is the average values of the shear rates difference [1/s].

The other symbols are the same as in the formulas (2) and (3).

2.2.7.2. In-shear structural recovery measurements. An in-shear structural recovery test was performed through the modified procedures of Mezger (2002), Achayuthakan and Suphantharika (2008), Wang et al. (2009, 2010). The subsequent steps were as follows: (1) a constant shear rate at 1 s^{-1} for 120 s, (2) a constant shear rate at 300 s^{-1} for 60 s, and (3) a constant shear rate at 1 s^{-1} for 240 s. The results were used to calculate the degree of structure recovery (DSR) of the pastes defined as the ratio of the average apparent viscosity obtained during the first 120 s of the third measurement step and the average apparent viscosity estimated in the first step (Wang et al., 2009, 2010).

2.2.7.3. In-shear structural recovery measurements with pre-shearing. An in-shear structural recovery test was carried out according to the modified procedures of Mezger (2002), Achayuthakan and Suphantharika (2008), Wang et al. (2009, 2010). involving (1) a constant shear rate at 100 s^{-1} for 30 s (pre-shearing), (2) a zero shear rate for 300 s (relaxation), (3) a constant shear rate at 1 s^{-1} for 120 s, (4) a constant shear rate at 300 s^{-1} for 60 s, (5) a constant shear rate at 1 s^{-1} for 240 s. The results provided calculation of DSR of the pastes according to Wang et al. (2009, 2010) taking under consideration the ratio of the average apparent viscosity in the fifth (obtained in the first 120 s) and the third step.

2.2.7.4. Apparent viscosity test. Viscosities at constant rate of shear (50 s^{-1}) taken within 600 s were determined.

2.2.8. Statistics

Duncan's test was used for the analysis of variance at the confidence level of $\alpha = 0.05$, employing Statistica v. 10.0 (Statsoft, Inc., Tulsa, OK., USA) software.

3. Results and discussion

3.1. Particle size, microscopy and molecular weights

The estimated size of NPS granules is highly diverse varying from 5 to 110 μm (Alvani, Tester, & Snape, 2011; Fornal et al., 2012; Singh, Isono, Srichuwong, Noda, & Nishinari, 2008; Swinkels, 1985) as shown in Table 1.

Laser analysis as well as optical microscopy revealed that granules of 30–70 μm dominated. This finding agreed with the data reported by Gomand et al. (2010a) although Alvani et al. (2011) as well as Fornal et al. (2012) found that in their samples there dominated granules of 6.3–8.5 and 9.8 μm, respectively. The number of granules of particular size fractions determined by both methods slightly differed from one another. It is likely that the number of the largest granules found microscopically was lower because the largest granules could quickly sediment on the preparation of the sample. Another possibility is that the results obtained by the laser analyzer tend toward higher values because the large particles account for much more volume.

The number of NPS granules observed under the microscope declined as the temperature of pasting increased. Simultaneously, as a result of swelling, the average diameter of granules increased

Table 1
Granule size distribution profiles.

Sample	Average number of granules[b]	Granule diameter [μm]			Particle size distribution [%]		
		Min.	Max.	Average	<30 μm	30–70 μm	>70 μm
NPS_granules (laser analysis)	–	–	–	–	19.7	58.2	19.6
NPS_granules (microscopy)	7.2	10.9	75.9	32.6	49.0	50.6	0.4
NPS_80[a]	2.5	26.1	316.8	117.4	0.5	10.7	88.8
NPS_95	1.4	28.9	337.0	107.5	0.2	18.3	81.5
NPS_121	0.1	107.2	371.7	158.9	0.0	0.0	100.0

[a] The notation relates to the temperature of pasting applied for the preparation of a given paste.
[b] Granules observed in micrograph.

Table 2
Characteristics of normal potato starch with gel chromatography[a].

Sample	$M_n \times 10^{-6}$ [g/mol]	$M_w \times 10^{-6}$ [g/mol]	P_d
NPS granules	0.37	3.33	9.0
NPS_80[b]	0.18	3.12	16.9
NPS_95	0.15	1.59	10.5
NPS_121	0.11	1.19	11.1

[a] M_n—number average molecular weight, M_w—weight average molecular weight, P_d—polydispersity index.
[b] The notation relates to pasting temperature applied for the preparation of given gel.

together with the contribution from the fraction of medium and large granules. There were more swollen granules in the paste prepared at 80 °C than in the paste made at 95 °C. The latter paste contained less granules above 70 μm in diameter than the paste prepared at 80 °C. Pastes prepared at 121 °C contained few very large granules. Hence one could deduce that on pasting at 80 °C chiefly granule swelling took place whereas on pasting at 95 °C the majority of swollen granules pasted (that is why the percentage of the large granules was smaller) and pasting at 121 °C completed the decomposition of the granules.

As shown by the results of gel permeation analysis, NPS exhibited considerable molecular diversity which depended on the means of sample preparation (Table 2). In the gels a decrease of almost a half in the average weight molecular weight was observed while the degree of the damage of granules increased with pasting temperature.

These results can be explained on the basis of the analysis of physicochemical phenomena occurring during samples preparation. On pasting, apart from destroying granular structures retrogradation is initiated and there is also a share of hydrolysis of starch (BeMiller & Whistler, 2009). Both starch polysaccharides retrograde but in contrast to amylopectin, amylose retrograde essentially faster. The latter constituted 32% (w/w) of the total polysaccharide. Retrogradation produces agglomerates which because of their poor aqueous solubility precipitate (Hoover, 2001; Ottenhof & Farhat, 2004). It is likely that the observed effect results from the partial hydrolysis of polysaccharides.

As seen in Fig. 1, the bimodal molecular weight distribution of the original starch and that pasted at 80 °C resembled one another also in their pattern.

The principal peak at $\log(M_w) = 6.7$ related to the fraction of the branched amylopectin while the peak at $\log(M_w) = 6.0$ related to the amylose fraction. This pattern was typical for normal starches containing 20–30% (w/w) amylose independently of their botanical origin (Lukasiewicz, Bednarz & Ptaszek, 2011). The two other distribution profiles were more complex. The formation of a third fraction at $\log(M_w)$ around 5.0 could be noted. In the diagram for the gel prepared at 95 °C this existed solely as a shoulder located below the peak attained to amylose whereas in the distribution for the gel prepared at 121 °C it could already be clearly seen. That lower molecular weight fraction spoke for the products of hydrolysis of the starch polysaccharides. As the appearance of this fraction was assisted by shifts of the peaks for amylose and amylopectin toward the lower M_w, it is likely that both polysaccharides hydrolyzed. Variation of the average molecular weight is accompanied by changes in the polydispersity. Its index rose in preliminarily pasted samples. This effect bore no relation to the changes in molecular weight at any point of its profile.

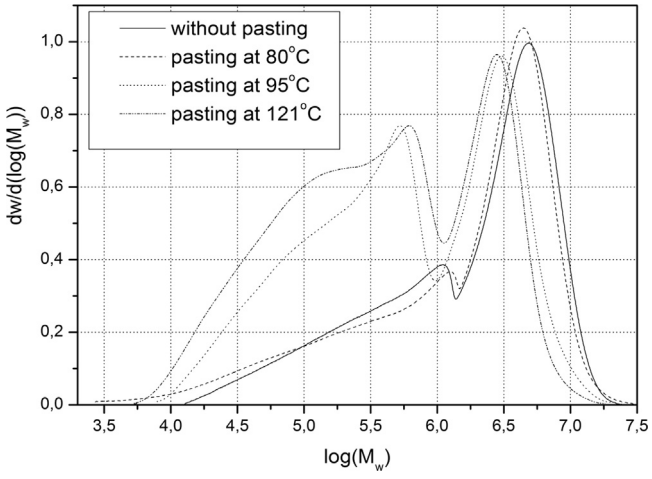

Fig. 1. Molecular weight distribution of NPS.

3.2. Pasting properties

Table 3 presents the parameters of pasting for all the investigated samples i.e. 1–5% (w/w) NPS suspensions.

One could see that these parameters strongly depended on the sample concentration. Particularly, the parameters collected for the 1% sample considerably distinguished themselves from the relevant data for more concentrated samples. That sample began to paste at a higher temperature and its characteristics of pasting exhibited neither any maximum nor minimum viscosity, moreover all other viscosity values were essentially lower than such values for more concentrated samples. Samples of the concentration of 2–5% provided already characteristics of pasting of the pattern common for potato starches (Gomand, Lamberts, Visser, & Delcour, 2010b; Kaur, Singh, Ezekiel, & Guraya, 2007; Kowalski, Sikora, Tomasik, & Krystyjan, 2008; Krystyjan, Adamczyk, Sikora, & Tomasik, 2013; McPherson & Jane, 1999; Sikora, Kowalski, & Tomasik, 2008; Singh et al., 2008; Swinkels, 1985; Zaidul, Nik Norulaini, Omar Mohd, Yamauchi, & Noda, 2007; Zhang et al., 2011).

An increase in the sample concentration resulted in a decrease in the temperature at the beginning of pasting, while in the case of the 3–5% suspensions the differences were not statistically significant. Also it was observed that the increase in the sample concentration caused an increase in the maximum viscosity at 96 °C prior and after a 10-min rest as well as after cooling to 50 °C. Moreover, in more concentrated samples the maximum viscosity was reached faster i.e. for 4 and 5% pastes—before 96 °C.

Parameters which distinguished 1% suspension and its paste from more concentrated samples could likely result from too low a concentration of granules to provide sufficient precision in the measurement of the temperature at the beginning of pasting with the resulting paste being too diluted to form an extended network.

3.3. Transmittance

Transmittance of the gels prepared at 80 °C increased with the sample concentration and no such relation could be observed for gels prepared at 95 and 121 °C. Simultaneously, independently of the concentration, the transmittance of the sample increased with the temperature of the sample preparation (Table 4). Also concerning the samples prepared at 80 °C it was observed that both measured values were much lower than these prepared at 95 and 121 °C.

The results of the transmittance confirm the data obtained in the measurements presented earlier (pasting characteristics, optical microscopy). The gels prepared at 80 °C contained the largest number of non-pasted granules and, eventually, their non-dissolved remains (Kaur, Singh, Ezekiel, & Sodhi, 2009; Kaur, Singh, & Sodhi, 2002; Perera & Hoover, 1999). Gels prepared at a higher temperature contained less non-pasted granules, hence, the transmittance of such samples increased.

Table 3
Characteristics of pasting of NPS.

Suspension concentration [%]	T_0 * [°C]	η_{max} [B.U.]	T_{max} [°C]	$\eta_{96\,°C}$ [B.U.]	$\eta_{96\,°C\ after\ 10\ min}$ [B.U.]	η_{min} [B.U.]	T_{min} [°C]	$\eta_{50\,°C}$ [B.U.]
1	93.5 ± 2.5^{a}**	–	–	12 ± 1^{e}	33 ± 1^{e}	–	–	57 ± 1^{e}
2	72.2 ± 0.0^{b}	128 ± 6^{d}	96.0 ± 0.0^{a}	97 ± 4^{d}	123 ± 2^{d}	123 ± 2^{d}	96.0 ± 0.0^{a}	241 ± 4^{d}
3	67.5 ± 0.1^{c}	286 ± 8^{c}	96.0 ± 0.0^{a}	280 ± 7^{c}	206 ± 4^{c}	206 ± 4^{c}	94.8 ± 0.0^{b}	371 ± 6^{c}
4	66.2 ± 0.3^{c}	501 ± 7^{b}	89.0 ± 0.0^{b}	472 ± 6^{b}	289 ± 6^{b}	288 ± 6^{b}	94.1 ± 0.0^{b}	527 ± 16^{b}
5	66.0 ± 0.1^{c}	824 ± 11^{a}	74.5 ± 0.6^{c}	672 ± 12^{a}	369 ± 6^{a}	368 ± 6^{a}	96.0 ± 0.1^{b}	695 ± 19^{a}

* T_0—the temperature at the beginning of pasting, η_{max}—maximum viscosity, T_{max}—temperature at maximum viscosity, $\eta_{96\,°C}$—viscosity at 96 °C, $\eta_{96\,°C\ after\ 10\ min}$—viscosity at 96 °C after 10 min, η_{min}—minimum viscosity, T_{min}—temperature at minimum viscosity, $\eta_{50\,°C}$—viscosity after cooling to 50 °C.
** Parameters in columns denoted with the same letters do not differ statistically at the level of confidence $\alpha = 0.05$.

The effect of temperature of the gel preparation was obvious whereas the effect of the concentration upon the transmittance of the gels needs additional discussion. It is worthful to note that an increase of transparency of the systems prepared at 80 °C with an increase of concentration was confirmed by the results of pasting characteristics (Table 3). The higher was the concentration of starch the lower was the temperature at the beginning of pasting and at the maximum of the viscosity thus, less granules were present in the system, because the granule disruption exceeded swelling. An application of higher pasting temperatures resulted in very small quantities of granules (compare the results in Table 1) and the concentration did not influence the results of the transmittance. The differences in the transmittance results between the samples obtained at 95 °C were very small and could be the consequence of the presence of a small number of starch granules with differentiated diameters. According to Kaur et al. (2002, 2009) and Perera and Hoover (1999) the bigger share of smaller granules results in more opaque pastes of lower transmittance. The transmittance of the gels prepared at 121 °C varied with concentration in an apparently strange manner, probably due to the presence of single extremely swollen granules. Anyway, a decreasing tendency could be observed, because with an increase of the concentration of starch the amylose and amylopectin content as well as remains of the granules in the samples increased.

3.4. Rheological tests

3.4.1. Steady flow measurements

The pattern of the flow curves (Fig. 2) and the parameters of the Ostwald–de Waele rheological model (Table 5) showed that all the investigated pastes could be accounted for by non-Newtonian liquids thinned by shear, that is, their behavior was common for such type systems (Fortuna & Gałkowska, 2006; Sikora et al., 2008, 2010; Zhang et al., 2011). In such systems, the destruction of their network by shear was faster than the structure recovery and, hence, their apparent viscosity declined.

Table 4
Transmittance of 2–5% starch gels.

Temperature of pasting [°C]	Concentration [%]	Transmittance [%]
80	2	2.79 ± 0.11^{h}*
	3	5.95 ± 0.71^{h}
	4	13.19 ± 0.04^{g}
	5	27.36 ± 1.49^{f}
95	2	57.26 ± 0.73^{de}
	3	62.25 ± 5.50^{c}
	4	56.56 ± 1.41^{e}
	5	60.99 ± 0.12^{cd}
121	2	75.34 ± 0.94^{a}
	3	70.36 ± 0.01^{b}
	4	66.69 ± 0.85^{b}
	5	69.74 ± 2.57^{b}

* Parameters in columns denoted with the same letters do not differ statistically at the level of confidence $\alpha = 0.05$.

As seen in Fig. 2, at each temperature shear stress increased with the concentration of the pastes. At a given concentration the shear stress tended to decrease with the temperature at which the paste was prepared, with the lowest being for the pastes prepared at 121 °C. The decrease of the shear stress with increasing temperature of pasting was connected to the decrease of an apparent

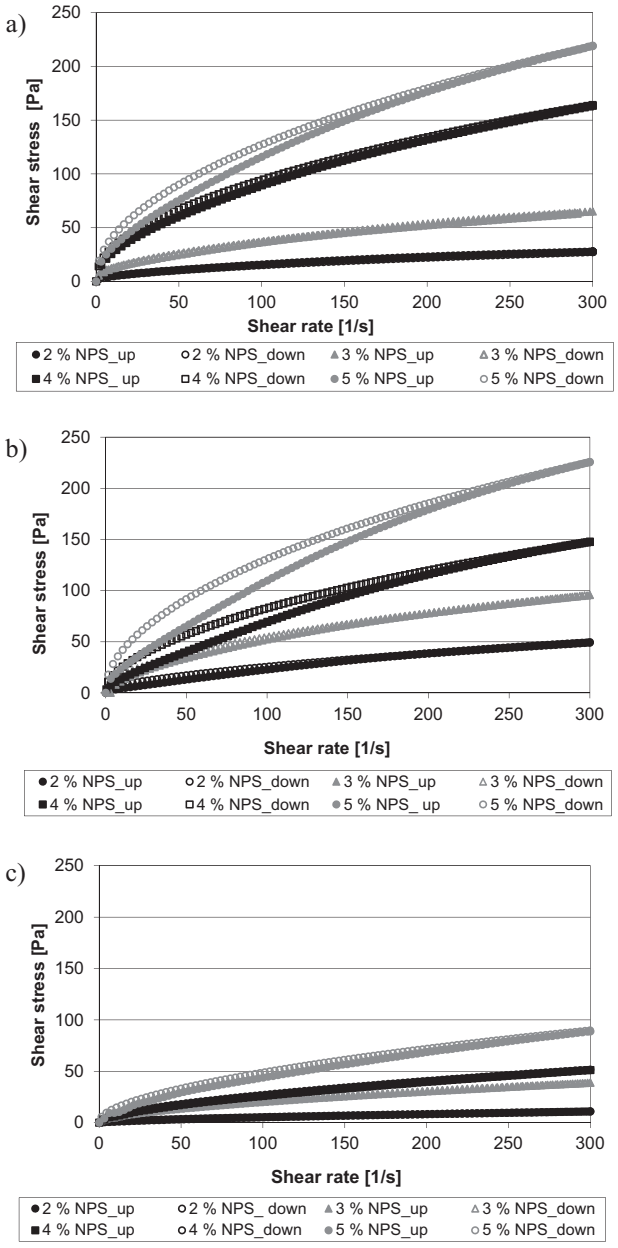

Fig. 2. Flow curves for 2–5% (w/w) pastes prepared at: (a) 80 °C, (b) 95 °C, (c) 121 °C.

Table 5
Parameters of the Ostwald–de Waele rheological model and areas of the hysteresis loops of starch pastes.

Temperature [°C]	Concentration [%]	Ostwald–de Waele model			Area of hysteresis loop [Pa s]		
		k^* [Pa s^n]	n [–]	R^2	Thixotropy	Antithixotropy	Total area
80	2	$1.42 \pm 0.17^{ef**}$	0.525 ± 0.015^{f}	0.999 ± 0.001	90 ± 17^{a}	0 ± 0^{f}	90 ± 17^{ef}
	3	3.22 ± 0.10^{c}	0.529 ± 0.016^{f}	0.999 ± 0.001	93 ± 71^{a}	53 ± 8^{ef}	146 ± 79^{def}
	4	7.13 ± 0.74^{a}	0.550 ± 0.016^{ef}	0.999 ± 0.000	0 ± 0^{c}	508 ± 24^{c}	508 ± 24^{c}
	5	7.79 ± 0.36^{a}	0.587 ± 0.006^{def}	0.999 ± 0.000	1 ± 0^{c}	983 ± 76^{b}	983 ± 76^{b}
95	2	0.82 ± 0.05^{fg}	0.723 ± 0.013^{a}	0.999 ± 0.000	22 ± 8^{bc}	224 ± 40^{d}	246 ± 48^{d}
	3	4.71 ± 0.26^{b}	0.530 ± 0.015^{f}	0.999 ± 0.001	33 ± 8^{bc}	158 ± 101^{de}	191 ± 110^{de}
	4	2.71 ± 0.37^{cd}	0.705 ± 0.021^{ab}	0.999 ± 0.000	2 ± 1^{c}	1086 ± 79^{b}	1088 ± 80^{b}
	5	4.92 ± 0.27^{b}	0.675 ± 0.009^{abc}	0.999 ± 0.000	3 ± 5^{c}	1719 ± 128^{a}	1723 ± 133^{a}
121	2	0.23 ± 0.25^{g}	0.730 ± 0.102^{a}	0.999 ± 0.000	4 ± 6^{c}	28 ± 1^{ef}	32 ± 6^{f}
	3	1.66 ± 0.38^{e}	0.555 ± 0.007^{ef}	0.999 ± 0.000	66 ± 8^{ab}	4 ± 1^{f}	70 ± 9^{ef}
	4	1.57 ± 0.13^{e}	0.611 ± 0.012^{cde}	0.999 ± 0.000	0 ± 0^{c}	128 ± 9^{def}	128 ± 9^{def}
	5	2.38 ± 0.15^{d}	0.634 ± 0.009^{bcd}	0.999 ± 0.000	$0 \pm 0c$	486 ± 27^{c}	486 ± 27^{c}

* k—Consistency coefficient [Pa s^n], n—flow behavior index [–], R^2—determination parameter.
** Parameters in columns denoted with the same letters do not differ statistically at the level of confidence $\alpha = 0.05$.

viscosity due to the hydrolysis of both amylose and amylopectin (compare the results of the gel chromatography).

The Ostwald–de Waele model very well fitted the flow curves for all pastes at an increasing shear rate (see R^2 in Table 5). For pastes prepared at 80 °C the consistency coefficient, k, increased with the paste concentration but such a relation could not be observed for pastes prepared at higher temperatures. However, for pastes of a given concentration there was a relationship between the k-coefficient and the temperature of the preparation of the pastes. That prepared at 121 °C exhibited the lowest k-value. Although for all pastes flow index, $n < 1$ pointing to their shear thinning character, there was no regularity in the course of the change of n against pasting temperature as well as paste concentration.

The pattern of the flow curves (Fig. 2) revealed that the pastes were either thixotropic, antithixotropic (either a clockwise or anticlockwise course of the changes of the curves, respectively), or mixed thixotropic/antithixotropic. All 4 and 5% pastes were antithixotropic and all 2 and 3% pastes were either thixotropic or mixed thixotropic/antithixotropic in their character, while a small thixotropy was observed merely at the highest shear rates. Summarizing the rheological behavior (Table 5) in each sample the thixo- or antithixotropic properties dominated. One can state that almost all studied pastes were antithixotropic. The exception were the 2 and 3% samples pasted at 80 °C as well as the 3% paste prepared at 121 °C, in which the dominating thixotropy was detected. Knowing that only the latter sample was thixotropic (as the only one prepared at 121 °C) it can be supposed that the behavior of this sample could be disregarded (from the point of view of this measuring method).

It was observed the tendency to increased total areas of hysteresis loops with an increasing concentration, which confirms the lowering of rheological stability. Pastes prepared at 95 °C were regularly characterized by the largest total area of the hysteresis loops (the lowest rheological stability), and the greatest stability (the smallest areas of hysteresis loops) had the samples prepared at 121 °C.

Basing oneself on the flow curves (Fig. 2, Table 5) and the degree of pasting determinations (molecular masses, optical microscopy, pasting characteristics, transmittance) one can say that thixo- and antithixotropic behavior takes place mainly when the pasting of starch is partial, something which was also observed by Abderahim, Ramaswamy, and van de Voort (1995). Likely, the time dependent rheological behavior is connected to the degree of pasting and starch concentration, and more precisely to the size (swelling) of the starch granules and their number. The thixotropic behavior was observed when the starch concentration was lower (2 and 3%) and the temperature of pasting was lowest (80 °C). It is worthful to note that in these pastes the degree of swelling of the granules was the lowest (optical microscopy) and the swelling of the granules exceeded their degradation (pasting characteristics). An antithixotropic character was more likely if the number of swollen granules was higher, and parallely more leached amylose and amylopectin was in the paste. Possibly, starch granules dispersed in the aqueous phase containing both amylose and amylopectin under shear reorganized themselves (Nayouf, Loyser, & Doublier, 2003) lowering resistance to shear and in this way making flow easier. The latter upon a diminishing shear suspense in the net formed of amylose and amylopectin molecules leached into the aqueous phase. Thus, the new structure formed is stronger.

The power of thixotropy in a volume of the sample, at a defined shear rate against shear rate was presented in Fig. 3. The presented graphs aimed to assess the speed of initial structure recovery in the whole range of shear rate. The highest absolute values of the power of thixotropy in a volume of the sample (the rate of execution of the work for the destruction and rebuilding of the inner structure) were noted at lower shear rates. Negative and positive values reflected the antithixotropic and thixotropic character of the pastes, respectively. This meant that at a low shear rate there was the strongest competition between the destruction and recovery of the inner structure caused by friction in a gradually more thickened (more concentrated) system of swollen starch granules. Because the values of the power of thixotropy varied uniformly in the whole range of the shear rate, the system seemed to be uniform and only this single mechanism of the action was involved.

3.4.2. In-shear structural recovery

The in-shear structural recovery test provided determination of the ability of the pastes to recover their original structure ruined at a high shear rate during the application of a low shear rate (Fig. 4) (Achayuthakan & Suphantharika, 2008; Mezger, 2002; Wang et al., 2009, 2010). It should be pointed out that the results of the in-shear structural recovery tests with and without pre-shearing were similar, so that only the results (charts) of the latter are presented (Fig. 4).

As one can see from Fig. 4 the thixotropic behavior was observed in the samples of the concentration 2 and 3%, pasted at 80 °C. Both the other samples pasted at this temperature (80 °C) and those pasted at 95 and 121 °C showed the antithixotropic behavior, independently on the concentration.

In Table 6 the values of the degree of structure recovery (DSR) calculated according to the points Sections 2.2.7.2 and 2.2.7.3 are presented. If the degree of structure recovery (DRS) exceeded 1, one might assume that the recovered structure was more shear resistant than that of the original. This means that the paste was

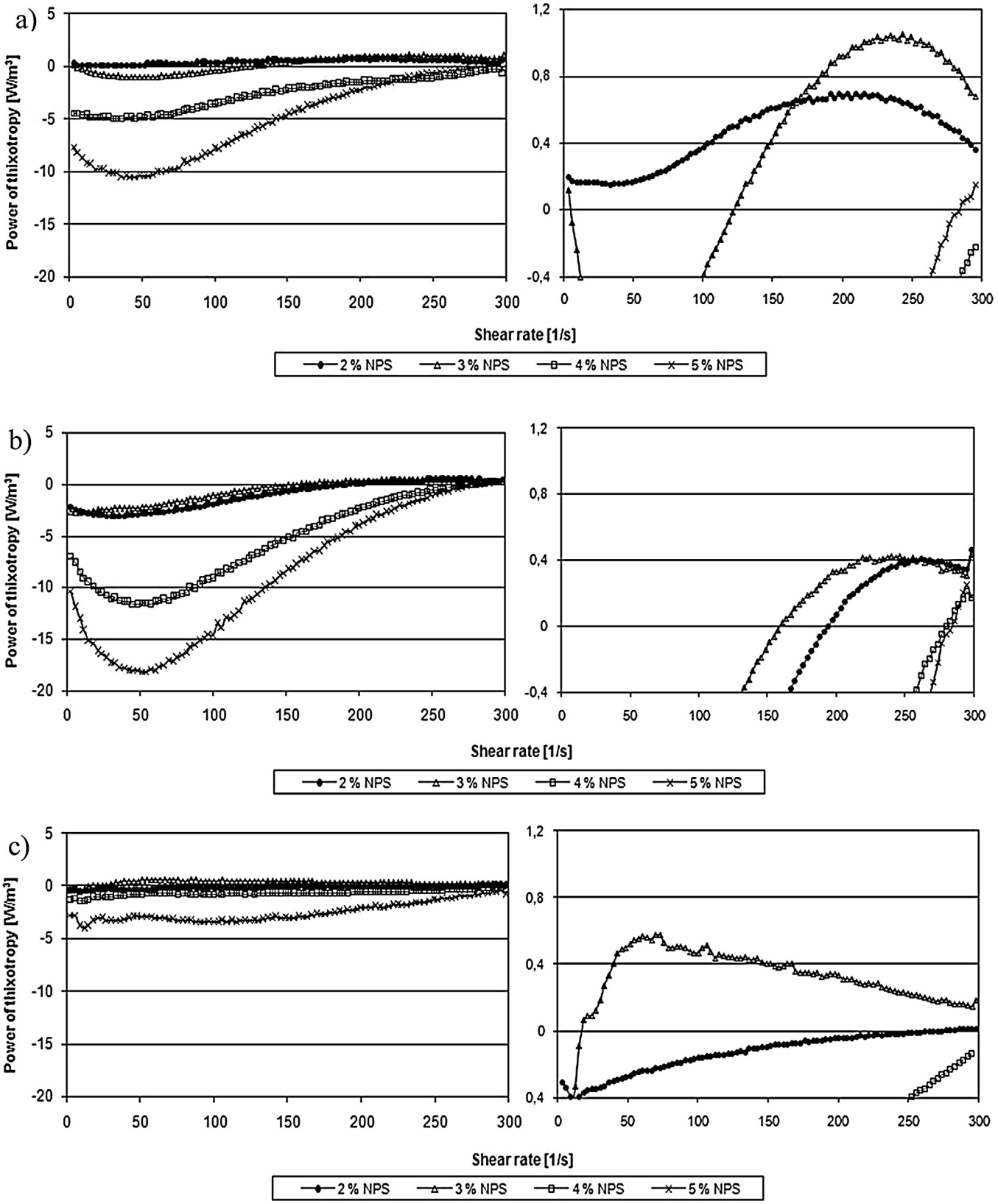

Fig. 3. Relationship between the power of thixotropy in a volume of the sample, at a defined shear rate and the shear rate in 2–5% (w/w) pastes prepared at: (a) 80 °C, (b) 95 °C, (c) 121 °C.

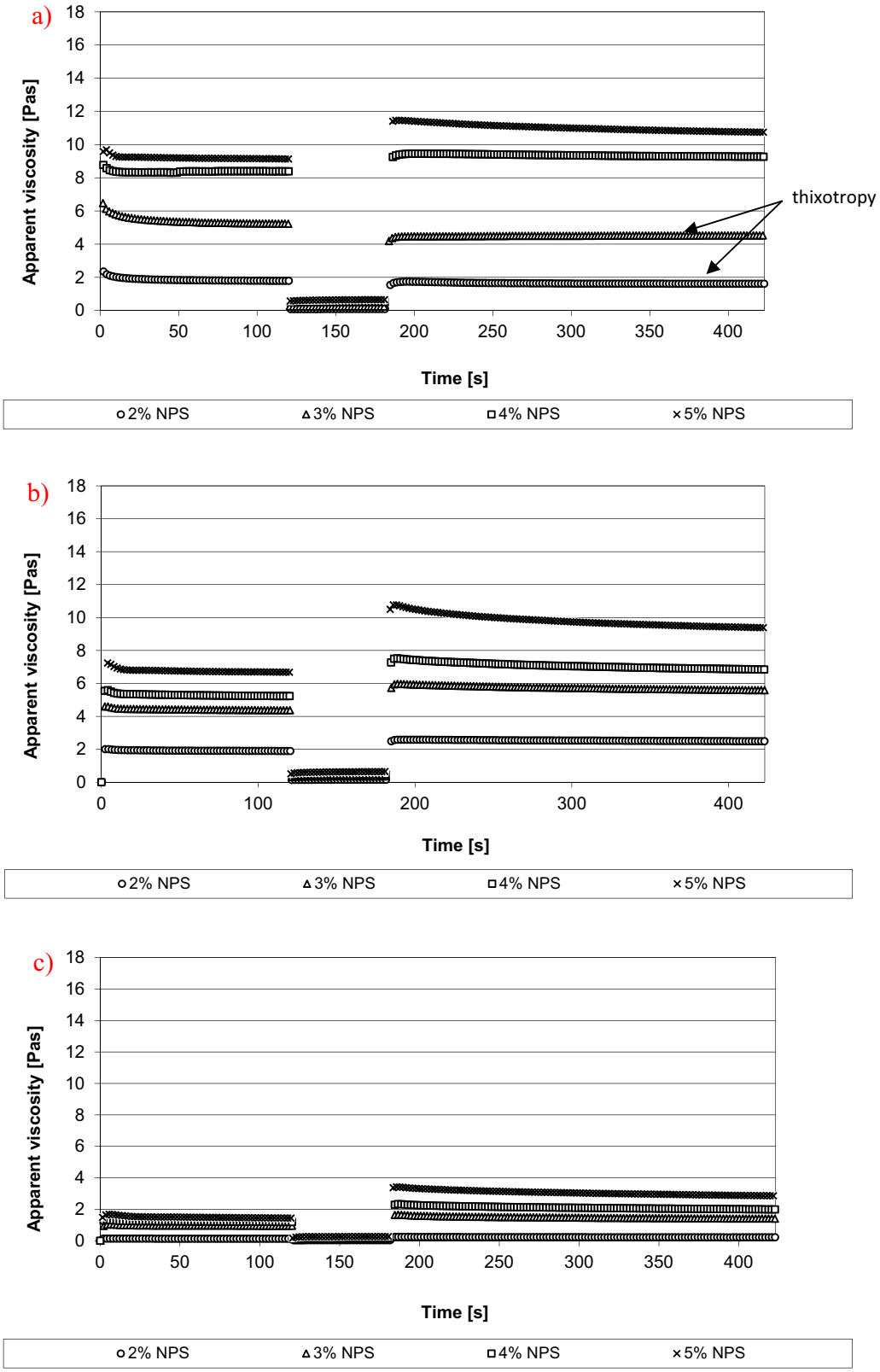

Fig. 4. Results of the in-shear structural recovery test with pre-shearing of the samples pasted at: (a) 80 °C, (b) 95 °C, (c) 121 °C.

antithixotropic. In turn, DSR < 1 revealed that the recovered paste was less resistant to shear and its recovery was slower i.e. it was thixotropic (Barnes, 1997; Sikora et al., 2011; Wang et al., 2009).

The DSR values (Table 6) allowed one to state that in a test performed either with or without preliminary shear only 2 and 3% pastes prepared at 80 °C were thixotropic. The other pastes estimated at DSR > 1 were antithixotropic. The rheological behavior of the samples in this measurement was confirmed by the hysteresis loop test. The samples in which the hysteresis loop test showed superiority of the thixo- or antithixotropic behavior had the same

Table 6
Degree of structure recovery (DSR).

Temperature [°C]	Concentration [%]	DSR without pre-shearing [–]	DSR with pre-shearing [–]
80	2	$0.89 \pm 0.04^{e*}$	0.91 ± 0.03^{g}
	3	0.87 ± 0.04^{e}	0.84 ± 0.08^{g}
	4	1.14 ± 0.05^{de}	1.12 ± 0.01^{f}
	5	1.12 ± 0.01^{de}	1.23 ± 0.08^{ef}
95	2	1.63 ± 0.25^{bc}	1.33 ± 0.03^{e}
	3	1.30 ± 0.17^{cd}	1.32 ± 0.04^{e}
	4	1.40 ± 0.02^{bcd}	1.37 ± 0.02^{de}
	5	1.32 ± 0.15^{cd}	1.51 ± 0.18^{cd}
121	2	1.72 ± 0.29^{b}	1.79 ± 0.01^{b}
	3	2.06 ± 0.12^{a}	1.59 ± 0.06^{c}
	4	1.38 ± 0.06^{bcd}	1.78 ± 0.12^{b}
	5	1.25 ± 0.21^{d}	2.13 ± 0.01^{a}

* Parameters in columns denoted with the same letters do not differ statistically at the level of confidence $\alpha = 0.05$.

properties in the in-shear structural recovery test. This confirmed that starch pastes had thixotropic behavior when the paste concentration was relatively low and the pasting temperature was not high enough in order to the destruction of granules could exceed their swelling. The antithixotropic behavior occurred when the degree of pasting was higher. On the other hand the rheounstable properties of the starch pastes could be connected to the close packing concentration C^* (the concentration at which the swollen granules fill up all the available space in the slurry), which is a very important parameter that allows one to explain the rheological properties of the samples (Earlingen, Jacobs, Block & Delcour, 1997; Gomand et al., 2010b). The approximate values of the C^*, calculated based on the results of the water binding capacity, were 2.7 and 1.4% at 80 and 95 °C, respectively. Comparing these values with the results of DSR (Table 6) one might assume that when the concentration of the starch was smaller or close to C^*, the samples were thixotropic. Whereas the pastes, the concentration of which was higher than C^*, were characterized by antithixotropic properties.

The DSR of 2 and 3% pastes investigated with in-shear structural recovery test without preliminary shear increased with the temperature at which the pastes were prepared. The DSR of more concentrated pastes reached the highest value for those prepared at 95 °C. When the preliminary shear was applied, the DSR regularly increased with the temperature of paste preparation regardless of their concentration. Perhaps, preliminary shearing partly destroyed the original structure and formed in the second step (without shearing) more stable structures of the samples.

There was no quantitative relation between the DSR values (Table 6) and the areas of the hysteresis loops (Table 5), but there was qualitative relation between the DSR and the rheounstable properties expressed in terms of flow curves. The lack of the quantitative connection between the results of the above mentioned methods was a result of the fact, that the in-shear structural recovery test did not provide information which shear rate this recovery was the most efficient at, and whether the process proceeded successively, and whether it was dependent on the inner structure of the paste.

3.4.3. Apparent viscosity test

The apparent viscosity of the pastes sheared at 50 s^{-1} was dependent both on the concentration and on the temperature of their preparation. It rose with the paste concentration (Fig. 5a–c). It was found that at the given concentration the pastes prepared at 121 °C exhibited the lowest apparent viscosities, which could be explained by the largest hydrolysis of these samples.

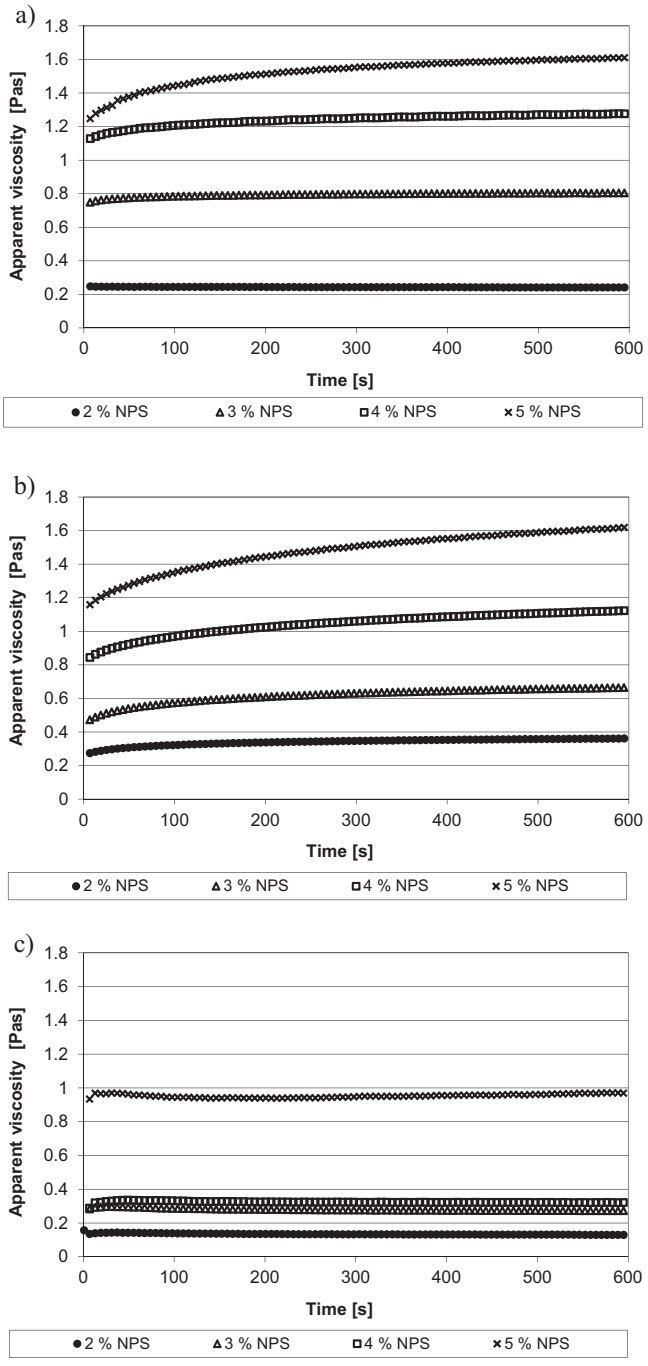

Fig. 5. Apparent viscosity vs. time at a shearing rate of 50 s^{-1}, at: (a) 80 °C, (b) 95 °C, (c) 121 °C.

The pattern of the changes of apparent viscosity in time (Fig. 5a–c) showed that 2% pastes prepared at 80 and 121 °C had decreasing courses, indicating thixotropic properties. Since the apparent viscosity of the other pastes increased the latter accounted for antithixotropic liquids. One can state that the results obtained for the samples pasted at 80 and 95 °C confirm the results obtained in the hysteresis loop test. In this test at the rate of shear of 50 s^{-1} the thixotropic behavior was found only in the 2% paste prepared at 80 °C and the remaining samples were antithixotropic (Figs. 2 and 3). Concerning the samples pasted at 121 °C the similar effect between the results of the hysteresis loop test and the viscosity at constant shear rate of 50 s^{-1} was not observed in an each instance. The small differences observed in the samples pasted at

121 °C (Fig. 5c), could be a result of the presence of single, extremely swollen granules in the pastes. It is worthful to underline that these samples showed small almost negligible changes of the viscosity during shearing at 50 s^{-1}, which could prove their big rheological stability. This stability was also confirmed by the flow curves, i.e. the smallest hysteresis loops areas (Fig. 2, Table 5) as well as the smallest power of the thixotropy in the unit of the volume (Fig. 3).

Generally, the values of the DSR obtained with and without pre-shearing allow to confirm the rheological character of the samples, which dominated during flow curves determination. On the basis of the apparent viscosity results one can state to which extent the sample was rheologically unstable at the given rate of shear.

4. Concluding remarks

The pasting of NPS at 80 °C resulted mainly in granule swelling whereas the process at 95 °C caused the further swelling of granules and their incomplete deterioration. The latter was practically completed just on pasting at 121 °C. However, under such conditions pasting was assisted by considerable hydrolysis of amylose and amylopectin. In the 2 and 3% samples pasted at 80 °C the swelling of the granules prevailed their destruction. In the other cases the inverse phenomenon was observed–the destruction predominated the swelling.

All pastes were rheologically unstable in time and shear thinned. The samples pasted at 121 °C were the most stable and those pasted at 95 °C—the most unstable.

Rheologically unstable (thixo- or antithixotropic) behavior took place mainly when the pasting of starch was partial. It was connected to the size (swelling) of the starch granules and their number. The thixotropic properties were observed when the degree of swelling of the granules was the lowest and the swelling of the granules prevailed their degradation (samples 2 and 3%, pasted at 80 °C). The pastes with more numerous and simultaneously more swollen (disrupted) granules had more antithixotropic character (the other samples).

Rheologically unstable properties depended on the measurement method used. The values of the DSR obtained with and without pre-shearing allow to confirm the rheological character of the samples, which dominated during flow curves determination. On the basis of the apparent viscosity results one can state to which extent the sample was rheologically unstable at the given rate of shear. Thus, in order to characterize fully the rheological behavior of the samples the results of all the measuring methods should be taken into account.

A differentiated regime of pasting allows one to receive pastes of a various degree of rheological stability. As was shown in this work all the studied pastes were rheologically unstable while, rather stable pastes have an applicative meaning in food processing. Considering the above, and taking into account, for example, the DSR as a measure of instability one can try to create in the future stable systems by e.g. the blending of starch systems (of an appropriate degree of pasting) with non-starchy polysaccharides.

Acknowledgment

This project was financed from funds provided by the National Science Centre of Poland awarded on the basis of decision number DEC-2011/03/B/NZ9/00152.

References

Abderahim, K. A., Ramaswamy, H. S., & van de Voort, F. R. (1995). Rheological properties of starch solutions underaseptic processing temperatures. *Food Research International*, *28*, 473–480.

Abramoff, M. D., Magalhaes, P. J., & Ram, S. J. (2004). Image processing with ImageJ. *Biophotonics International*, *11*(7), 36–42.

Abu-Jdayil, B., Azzam, M. O. J., & Al-Malah, K. I. M. (2001). Effect of glucose and storage time on the viscosity of wheat. *Carbohydrate Polymers*, *46*, 207–215.

Achayuthakan, P., & Suphantharika, M. (2008). Pasting and rheological properties of waxy corn starch as affected by guar gum and xanthan gum. *Carbohydrate Polymers*, *71*, 9–17.

Alloncle, M., Lefebvre, J., Llamas, G., & Doublier, J. (1989). A rheological characterization of cereal starch-galactomannan mixtures. *Cereal Chemistry*, *66*, 90–93.

Al-Malah, K. I., Azaam, M. O. J., & Abu-Jdayil, B. (2000). Effect of glucose concentration on the rheological properties of wheat-starch dispersions. *Food Hydrocolloids*, *14*, 491–496.

Alvani, K., Qi, X., Tester, R. F., & Snape, C. E. (2011). Physico-chemical properties of potato starches. *Food Chemistry*, *125*, 958–965.

Barnes, H. A. (1997). Thixotropy—A review. *Journal of Non-Newtonian Fluid Mechanics*, *70*, 1–33.

BeMiller, J. N., & Whistler, R. L. (2009). *Starch chemistry and technology*. London: Academic.

Bird, R. B., & Marsh, B. D. (1968). Viscoelastic hysteresis. Part I. Model predictions. *Journal of Rheology*, *12*, 479–489.

Dewar, R., & Joyce, M. J. (2006). The thixotropic and rheopectic behavior of maize starch and maltodextrin thickeners used in dysphagia therapy. *Carbohydrate Polymers*, *65*, 296–305.

Dintzis, F. R., & Bagley, E. B. (1995). Shear-thickening and flow-induced structure in a system of DMSO containing waxy maize starch. *Journal of Rheology*, *39*, 1399–1409.

Djaković, L. J., Soviljii, V., & Milosevic, S. (1990). Rheological behavior of thixotropic starch and gelatin gels. *Starch/Stärke*, *42*, 380–385.

Doublier, J. L. (1981). Rheological studies on starch—Flow behaviour of wheat starch pastes. *Starch/Stärke*, *33*, 415–420.

Earlingen, R. C., Jacobs, H., Block, K., & Delcour, J. A. (1997). Effects of hydrothermal treatments on the rheological properties of potato starch. *Carbohydrate Research*, *297*, 347–356.

Fornal, J., Sadowska, J., Błaszczak, W., Jeliński, T., Stasiak, M., Molenda, M., et al. (2012). Influence of some chemical modifications on the characteristics of potato starch powders. *Journal of Food Engineering*, *108*, 515–522.

Fortuna, T., & Gałkowska, D. (2006). Influence of the saccharide addition upon rheological properties of modified starches (in Polish). *Żywność, Nauk, Technologia, Jakość*, *13*(4), 5–17.

Gambuś, H., Gumul, D., & Juszczak, L. (2004). Rheological properties of pastes obtained from starches derived from immature cereal Kernels. *Starch/Stärke*, *56*, 225–231.

Gomand, S. V., Lamberts, L., Derde, L. J., Goesaert, H., Vandeputte, G. E., Goderis, B., et al. (2010 a). Structural properties and gelatinisation characteristics of potato and cassava starches and mutants thereof. *Food Hydrocolloids*, *24*, 307–317.

Gomand, S. V., Lamberts, L., Visser, R. G. F., & Delcour, J. A. (2010 b). Physicochemical properties of potato and cassava starches and their mutants in relation to their structural properties. *Food Hydrocolloids*, *24*, 424–433.

Harrod, M. (1989). Time-dependent flow behavior of starch pastes, with food process applications. *Journal of Food Process Engineering*, *11*, 297–309.

Hoover, R. (2001). Composition, molecular structure, and physicochemical properties of tuber and root starches: A review. *Carbohydrate Polymers*, *45*, 253–267.

Kaur, A., Singh, N., Ezekiel, R., & Guraya, H. S. (2007). Physicochemical, thermal and pasting properties of starches separated from different potato cultivars grown at different locations. *Food Chemistry*, *101*, 643–651.

Kaur, A., Singh, N., Ezekiel, R., & Sodhi, N. S. (2009). Properties of starches separated from potatoes stored under different conditions. *Food Chemistry*, *114*, 1396–1404.

Kaur, L., Singh, N., & Sodhi, N. S. (2002). Some properties of potatoes and their starches II. Morphological, thermal and rheological properties of starches. *Food Chemistry*, *79*, 183–192.

Kowalski, S., Sikora, M., Tomasik, P., & Krystyjan, M. (2008). Starch polysaccharide hydrocolloid gels. *Polimery*, *53*, 457–464.

Krystyjan, M., Adamczyk, G., Sikora, M., & Tomasik, P. (2013). Long-term storage stability of selected potato starch—Non-starchy hydrocolloid binary gels. *Food Hydrocolloids*, *31*, 270–276.

Lagarrigue, S., & Alvarez, G. (2001). The rheology of starch dispersions at high temperatures and high shear rates: A review. *Journal of Food Engineering*, *50*, 189–202.

Lukasiewicz, M., & Kowalski, S. (2012). Low power microwave-assisted enzymatic esterification of starch. *Starch/Stärke*, *64*, 188–197.

Lukasiewicz, M., Bednarz, S., & Ptaszek, A. (2011). Environmental friendly polysaccharide modification—Microwave-assisted oxidation of starch. *Starch/Stärke*, *63*, 268–273.

McPherson, A. E., & Jane, J. (1999). Comparison of waxy potato with other root and tuber starches. *Carbohydrate Polymers*, *40*, 57–70.

Mewis, J., & Wagner, N. J. (2009). Thixotropy. *Advances in Colloid and Interface Science*, *147–148*, 214–227.

Mezger, T. G. (2002). Rotational tests. In U. Zorll (Ed.), *The rheology handbook: For users of rotational and oscillatory rheometers* (pp. 55–68). Hannover: Vincentz Verlag.

Nayouf, M., Loisel, C., & Doublier, J. L. (2003). Effect of thermomechanical treatment on the rheological properties of crosslinked waxy corn starch. *Journal of Food Engineering*, *59*, 209–219.

Nguyen, Q. D., Jensen, C. T. B., & Kristensen, P. G. (1998). Experimental and modeling studies of the flow properties of maize and waxy maize starch pastes. *Chemical Engineering Journal*, *70*, 165–171.

Orczykowska, M., Dziubiński, M., & Budzyński, B. (2010). Effect of temperature upon stability of thixotropic starch gels in the presence of a thickener (in Polish). *Acta Agrophysica*, *16*, 401–411.
Ottenhof, M.-A., & Farhat, I. A. (2004). Starch retrogradation. *Biotechnology & Genetic Engineering Reviews*, *21*(1), 215–228.
Perera, C., & Hoover, R. (1999). Influence of hydroxypropylation on retrogradation properties of native, defatted and heat-moisture treated potato starches. *Food Chemistry*, *64*, 361–375.
Sikora, M., Kowalski, S., & Tomasik, P. (2008). Binary hydrocolloids from starches and xanthan gum. *Food Hydrocolloids*, *22*, 943–952.
Sikora, M., Adamczyk, G., & Krystyjan, M. (2011). Thixotropy as a measure of instability of liquid foodstuffs (in Polish). *Żywność, Nauka, Technologia, Jakość*, *18*(1), 5–14.
Sikora, M., Tomasik, P., Krystyjan, M., & Krawontka, J. (2010). Mixed pastes of starches with guar gum. *Polimery*, *55*, 582–590.
Singh, N., Isono, N., Srichuwong, S., Noda, T., & Nishinari, K. (2008). Structural, thermal and viscoelastic properties of potato starches. *Food Hydrocolloids*, *22*, 979–988.
Steffe, J. F. (1996). *Rheological methods in food process engineering*. USA: Freeman Press.
Swinkels, J. J. M. (1985). Composition and properties of commercial native starches. *Starch/Stärke*, *37*, 1–5.
Tattiyakul, J., & Rao, M. A. (2000). Rheological behaviour of cross-linked waxy maize starch dispersions during and after heating. *Carbohydrate Polymers*, *43*, 215–222.
Vigneau, E., Loisel, C., Devaux, M. F., & Cantoni, P. (2000). Number of particles for the determination of size distribution from microscopic images. *Powder Technology*, *107*, 243–250.
Wang, B., Li, D., Wang, L.-J., & Özkan, N. (2010). Anti-thixotropic properties of waxy maize starch dispersions with different pasting conditions. *Carbohydrate Polymers*, *79*, 1130–1139.
Wang, B., Wang, L.-J., Li, D., Özkan, N., Li, S.-J., & Mao, Z.-H. (2009). Rheological properties of waxy maize starch and xanthan gum mixtures in the presence of sucrose. *Carbohydrate Polymers*, *77*, 472–481.
Zaidul, I. S. M., Nik Norulaini, N. A., Omar Mohd, A. K., Yamauchi, H., & Noda, T. (2007). RVA analysis of mixtures of wheat flour and potato, sweet potato, yam, and cassava starches. *Carbohydrate Polymers*, *69*, 784–791.
Zhang, Y., Gu, Z., Hong, Y., Li, Z., & Cheng, L. (2011). Pasting and rheological properties of potato starch and maize starch mixtures. *Starch/Stärke*, *63*, 11–16.