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Rheological behaviour of β-glucan preparations from oat products

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Abstract

The cereal dietary fibre β-glucan has outstanding functional and nutritional properties, because of its viscosity in aqueous systems and in the intestinal tract. The rheological behaviour of β-glucan (concentrations: 2% and 4%) isolated from extruded and autoclaved oat meal and from oat bran was evaluated using oscillatory and rheological measurements. In frequency sweep, the storage and loss moduli G′ and G″ of β-glucan preparations from extruded meal and from bran increased continuously with increasing frequency, showing a dominantly viscous behaviour. With increasing frequency, the elastic properties improved. β-Glucan from autoclaved meal also showed elastic behaviour. With the exception of β-glucan from autoclaved meal, G′ and G″ were not influenced by deformation in the amplitude sweep. Complex viscosities decreased with frequency (in all samples) and were independent of deformation (in extruded meal and bran). In shear experiments, β-glucan solutions were structurally viscous non-Newtonian solutions with rheostable behaviour. β-Glucan (2%) from bran had the highest and that from autoclaved meal had the lowest apparent and process viscosities. Fluid dynamic parameters may influence the flow, diffusion or transport behaviour of β-glucan during digestion processes in the small intestine, but the influence of the viscous behaviour is limited.

Keywords: β-Glucan; Rheology; Flow behaviour; Oscillation test; Oat meal; Oat bran

1. Introduction

Oat (*Avena sativa* L.) products are excellent sources of different dietary fibre (DF) components, such as mixed-linkage (1 → 3),(1 → 4)-β-D-glucan (from here on called β-glucan), arabinoxylans and cellulose. The neutral cell wall polysaccharide β-glucan has outstanding functional and nutritional properties. It achieves high viscosities at relatively low concentrations. Autio, Myllymäki, and Mälkki (1987) found that β-glucan solutions (concentration: 1%) have a low flow behaviour index and a high consistency index in the power law model. Viscosity of β-glucan was stable over a wide range of pH (2–10) and decreased with increasing temperature (Dawkins & Nnanna, 1995).

Because of its viscosity, β-glucan may disturb the brewing process (Bamford, 1985) or limit the nutritive value of barley-based feeds for animals (Campbell & Bedford, 1992). On the other hand, β-glucan could be used as a thickening agent in food technology (Wood, 1984), it may influence the sensory quality of beverages (Lyly et al., 2003) and is of particular importance in human

Abbreviations: AU, β-glucan preparation isolated from autoclaved oat meal; BR, β-glucan preparation isolated from oat bran; CA, Casson model; DF, dietary fibre; EX, β-glucan preparation isolated from extruded oat meal; G′, storage modulus; G″, loss modulus; HB, Herschel–Bulkley model; LRW, linear visco-elastic range; OW, Ostwald–de Waele model; RS, resistant starch.

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nutrition (Mälkki & Virtanen, 2001). Different physiological effects of β-glucan in isolated form or as a constituent of oat and barley products, discussed in literature, are related to its viscosity: attenuation of postprandial plasma glucose and insulin responses (Wood et al., 1994), higher transport of bile acids towards lower parts of the intestinal tract and higher excretion of bile acids (Dongowski, Huth, & Gebhardt, 2003; Lia et al., 1995) or lowering of serum cholesterol levels (Braaten et al., 1994; McIntosh, Whyte, McArthur, & Nestel, 1991).

Molecular weight and concentration have a determining influence on the viscosity and the rheological behaviour of β-glucans in aqueous solution or in the intestinal tract. Therefore, the β-glucan content in cereal products, technological effects during processing and conditions in the gastrointestinal tract affect the viscosity. German oat cultivars contain 4.7% β-glucan on average, whereas oat bran contains over 8% β-glucan (Ganßmann, 1998). Differences in rheological properties of β-glucans from several oat varieties were found when measured at the same β-glucan concentration due to the effect of molecular weight (Autio, Myllymäki, Suortti, Saastamoinen, & Poutanen, 1992). Wikström, Lindahl, Andersson, and Westerlund (1994) found higher viscosities in β-glucans extracted from oat bran compared with those from oat endosperm. β-Glucans extracted from enhanced oat lines were more viscous than those of a traditional line when measured at a concentration of 1% (Colleoni-Sirghie, Kovalenko, Briggs, Fulton, & White, 2003). In contrast to β-glucan extracts from untreated barley meal, extracts from extruded meal, and especially from autoclaved meal, had lower molecular weights and viscosities as well as a diminished pseudoplastic behaviour (Huth, Dongowski, Gebhardt, & Flamme, 2000). There are some indications that the molecular weight of β-glucan may partly be reduced during its passage through the upper gastrointestinal tract (Johansen, Bach Knudsen, Wood, & Fulcher, 1997; Robertson, Majsak-Newman, & Ring, 1997). On the other hand, Bach Knudsen, Jensen, and Hansen (1993) found no quantitative losses of β-glucan from oat flour or bran in the stomach and in upper parts of the small intestine in ileum-cannulated pigs.

β-Glucan containing extrudates from oat were able to affect bile acid binding and fermentation in vitro (Drzikova, Dongowski, Gebhardt, & Habel, 2005). Oat products of different composition (e.g. oat meal, oat bran) and after different pre-treatments (extrusion, autoclavation or untreated), used in diets, had several beneficial in nutritional studies with humans and rats (Drzikova, Dongowski, Habel, & Gebhardt, in preparation). During passage through the stomach and small intestine, insoluble β-glucans are partly converted into a soluble form. Therefore, they can form viscous solutions or systems in the gut. There is only limited information on the flow behaviour of β-glucans from different oat sources used in nutrition.

Rheological examinations of suspensions or solutions from macromolecules are powerful tools to characterize material properties as food components (Kunzek, Opel, & Senge, 1997; Senge, Opel, & Kunzek, 1996) and also under the conditions in the intestinal tract.

The objective of this study was to characterize the complex flow behaviour of β-glucan preparations isolated from different oat products using conventional rheological and oscillatory measurements within the linear visco-elastic range. For this purpose, apparent and process viscosities were estimated by conventional stress experiments. Further, dynamic parameters were determined in frequency and amplitude sweep measurements to investigate the structure formation and stability of the β-glucan solutions as gel-like or highly viscous systems.

2. Materials and methods

2.1. Preparation and composition of the oat products

Oat meal (composition: 61.4% starch, 11.6% crude protein, 6.2% total fat, 2.6% soluble DF, 8.4% insoluble DF, 3.5% β-glucan) was prepared from commercial oat kernels by milling. Oat bran (composition: 8.9% β-glucan, 0.5% soluble and 17.8% insoluble DF) was obtained from Peter Kölln Köllnflockenwerke (Elmshorn, Germany).

Extrudate was prepared in the twin-screw extruder ERMAFA DS 6.0 (ERMAFA Kunststofftechnik, Chemnitz, Germany) using following conditions: moisture content 25%, mass temperature 150 °C, screw speed 200 rpm and dosage 100 kg/h. Further, oat meal was treated for 60 min at 140 °C and a moisture content of 25% in the Sanoclav autoclave (Fa. Adolf Wolf, Bad Überkingen-Hausen, Germany) in two cycles. The composition of oat products used is given in Table 1.

2.2. Analytical methods

For determination of β-glucan, sample material was suspended in a phosphate buffer (pH 6.5) and mixed for 5 min at 90 °C. The suspension was hydrolysed with lichenase (Megazyme International, Bray, Ireland) for 60 min at 45 °C (McCleary & Mugford, 1997). After dilution and centrifugation (10 min at 1000*g*), a part of the supernatant was incubated with β-glucosidase (Megazyme) in acetate buffer at pH 4.5 and 40 °C for 15 min. The glucose released was determined with the hexokinase/glucose-6-phosphate dehydrogenase kit from Boehringer (Mannheim, Germany).

Resistant starch (RS) was measured by a modified Englyst-method (Englyst, Klingman, & Cummings, 1992) after hydrolysis of digestible starch and separation of hydrolysis products, dissolution of RS in 1 M NaOH, hydrolysis of RS with amyloglucosidase and enzymatic

Table 1
Composition of the oat products (in % related to dry matter)

Oat product	Extruded oat meal	Autoclaved oat meal	Oat bran
Dietary fibre (DF) component or fraction			
β-Glucan	4.61	4.28	10.37
Resistant starch	0.45	3.95	0.45
Non-digestible oligosaccharides	3.60	1.97	1.47
Soluble DF	4.38	5.15	10.15
Insoluble DF	5.28	3.62	14.54
Total DF[a]	13.26	10.74	26.16
Total fat	6.85	6.80	10.03
Protein	14.49	15.90	21.65
Starch	65.84	58.00	41.60
Ash	2.02	1.97	4.28

[a] Sum of soluble DF, insoluble DF and non-digestible oligosaccharides. Values are means ($n = 2$–6).

determination of the released glucose. Total starch content was analysed enzymatically after solution in 1 M NaOH. Insoluble and soluble DF were analysed by the enzymatic-gravimetric AOAC method (Prosky et al., 1988). Non-digestible oligosaccharides were determined in the supernatant after coagulation of the soluble DF fraction with ethanol using HPLC with RI detection. Total fat was determined by the Weibull–Stoldt method. Protein was estimated by a modified Kjeldahl method ($N \times 6.25$).

Water binding was determined using the capillary suction method in the variant of Heinevetter and Kroll (1982) at 20 °C for 15 min. The water uptake of the samples was expressed as g H_2O/g substance. For evaluation of the molecular weight, the intrinsic viscosity [η] was estimated at 25.0 ± 0.1 °C in an Ubbelohde viscosimeter. The β-glucan preparations were dissolved in water and then centrifuged for 10 min at 2000g. Relative viscosity ($\eta_{rel} = \eta/\eta_0$; where η is the viscosity of the solution and η_0 is that of the solvent) was determined on the basis of flow times at different concentrations. The intrinsic viscosity [η] was determined by extrapolating of the calculated specific viscosity ($\eta_{sp} = \eta_{rel} - 1$) against the concentration of β-glucan using the Huggins equation. The average molecular weight (M_w) was calculated using the Mark–Houwink equation (1):

$$[\eta] = k \times M_w{}^a \quad (1)$$

where k and a are dependent on the nature of the molecule and the solvent. According to Vårum, Martinsen, and Smidsrød (1991), the factor $k = 0.00067$ and the exponent $a = 0.75$ were used for calculations.

2.3. Extraction of β-glucan

A modified procedure of Westerlund, Andersson, and Åman (1993) was used to extract the β-glucans. To inactivate possible present endogenous enzyme activities and for extraction of lipids, 50 g of the oat products were first boiled in 500 ml of 96% ethanol for 5 min under reflux. The residue after centrifugation (20 min at 4 °C and 3800g) was suspended in 600 ml of water and treated for 2 h at 96 °C with 10 ml of Termamyl 120 L (Novo Nordisk A/S, Copenhagen, Denmark) in the presence of 224 mg of $CaCl_2$ (degradation of starch, dissolution of β-glucan). The residue after centrifugation was two times extracted with 200 ml of water. Then the combined supernatants were treated with 200 mg of pancreatin (Merck, Darmstadt, Germany) for 3 h at 40 °C. For separation of low-molecular constituents (e.g. glucose, maltooligosaccharides, amino acids, peptides, salts), 96% ethanol was added slowly under stirring until the final ethanol concentration in the mixture was 50%. After storing the mixture for 16 h at 4 °C, it was centrifuged for 30 min at 4 °C and 3800g. The residue was repeatedly extracted under stirring and centrifugation with 50% to 96% ethanol and then dried in a Speed-Vac.

2.4. Rheological examinations

The β-glucan preparations were dissolved in water under heating two times for 20 min at 90 °C interrupted by Vortex treating. Sodium azide (0.02%) was added to prevent microbial contamination. The concentration of β-glucan was adjusted to 2% and 4% in the aqueous solutions. The solutions were stable until measurements were completed.

Rheological examinations were performed using the Universal Dynamic Spectrometer UDS 200 (Paar Physica, Stuttgart, Germany) with the cylinder-measuring system Z3 DIN and profiled bob (inner cylinder) with a sample volume of 17 cm^3 at 20 ± 0.01 °C.

Standard oscillation experiments were carried out in the strain-controlled mode:

frequency sweep: $0.001 \leqslant f \leqslant 100$ Hz; $\gamma = 0.001$; 30 measuring points;
no pre-shearing;
amplitude sweep: $0.0001 \leqslant \gamma \leqslant 0.1$; $f = 1$ Hz; 30 measuring points.

Storage modulus G′, loss modulus G″, loss factor tan δ and complex viscosity $|\eta^*|$ were calculated in dependence on the frequency f or on the deformation γ, respectively, in frequency sweep and in amplitude sweep, respectively.

Then the shear flow curves $\tau = f(\dot{\gamma})$ were measured in the same system consisting of three phases each of 60 s:

start curve: $0.1 \leqslant \dot{\gamma} \leqslant 10$ s^{-1}; 30 measuring points 60 s;
ramp: $\dot{\gamma} = 10$ s^{-1}; 30 measuring points 60 s;
return curve: $10 \geqslant \dot{\gamma} \geqslant 0.1$ s^{-1}; 30 measuring points 60 s.

Ramp was used to determine the rheodynamic behaviour. The flow curve of the fifth measurement step was

fitted to the Herschel–Bulkley (HB) and Casson (CA) models for plastic systems and to the Ostwald–de Waele (OW) model for non-Newtonian behaviour (structural viscosity). Furthermore, the parameter yield point τ_0, the consistency factor K and the flow exponent n were determined (Lerche, Pisendel, & Senge, 2003).

Confidential statistical parameters of the rheological examinations were given by US 200 software (e.g. SD, correlation coefficient and middle error).

The solid density of the β-glucan solutions was estimated by conventional pycnometry at 20 °C.

3. Results

3.1. Characterization of the oat products and the isolated β-glucan preparations

Oat meal was used in form of an extrudate and an autoclaved product. Further, untreated oat bran was used as a source material. The composition of these oat products is given in Table 1. They contained different proportions of β-glucan and DF fractions. Thus, the β-glucan content differed between 4.3% and 10.4%. The total DF content of the oat products was between 10.7% and 26.2%. As a result of the distinct hydrothermal effects, the autoclaved product contained approximately 4% RS. RS is defined as the starch or starch degradation products, which are not absorbed in the small intestine of healthy individuals, and is classified as a DF (American Association of Cereal Chemists, 2001). The following water uptake values of the oat products were measured (in g H_2O/g dry matter): extruded oat meal = 5.69 ± 0.20; autoclaved oat meal = 2.57 ± 0.16 and oat bran = 2.88 ± 0.29 (mean ± SD).

During extraction of β-glucan from the oat products, digestible starch and protein were enzymatically degraded and removed from the source materials. The isolated β-glucan preparations EX, AU and BR extracted from extruded oat meal, autoclaved oat meal and oat bran, respectively, contained 53.0%, 63.5% and 61.9% β-glucan (related to the original substance), respectively. Approximately 50% of the β-glucan, present in the oat products, was extracted by the applied process.

The following molecular weights of the β-glucan preparations were found, calculated from the intrinsic viscosity using the Mark–Houwink equation (Vårum et al., 1991): EX = 94.8 kDa, AU = 49.1 kDa and BR = 132.4 kDa. Autoclaving of oat meal resulted in a partial depolymerisation of β-glucan. Huth et al. (2000) found molecular weights between 80 and 125 kDa in β-glucan preparations isolated from barley extrudates whereas Woodward, Fincher, and Stone (1983) calculated molecular weights of up to 290 kDa.

3.2. Rheological examination of the β-glucan preparations

The flow behaviour of the β-glucan preparations isolated from different oat products was characterized using rheological and oscillatory measurements within the linear visco-elastic range $\gamma \leqslant \gamma_{crit}$. Apparent and process viscosities were estimated by conventional stress experiments. Further, dynamic moduli such as storage modulus G′, loss modulus G″ and loss angle were determined in frequency and amplitude sweeps.

All samples were homogenous during rheological examination. The solid densities of the β-glucan solutions were in a concentration of 2%: EX2 = 1013.8 kg/m^3, AU2 = 1009.5 kg/m^3 and BR2 = 1008.1 kg/m^3.

3.2.1. Frequency sweep

The structure formation of macromolecules or particles in a disperse system can be described by investigating the visco-elastic properties in terms of oscillation measurements. In contrast to conventional controlled shear rate, the examination retains the native structure at various load levels of strain in the linear visco-elastic range (LVR). The slope and the crossover of the storage modulus G′ (elastic behaviour; reverse storages of energy) and the loss modulus G″ (viscous behaviour; dissipation of flow energy) allow differentiation between a sol/solution, gel-like structure or a dispersion particle system like suspensions, emulsions and foams. A crossover (G′, G″) is for example an indicator of a gel-like structure.

Results were mainly evaluated in the frequency range between 0.01 and 10 Hz. The storage modulus G′ of the β-glucan preparations from extruded meal and from bran increased continuously with increasing frequency (Fig. 1). Higher storage moduli were found when β-glucans were measured in a concentration of 4%. These samples have a similar mechanism of structure formation and a dominant viscous behaviour within the whole frequency range. The β-glucans are very hydrophilic, and electrostatic interactions occur between the functional groups (e.g. OH groups, Keesom- and Debye-energy). In contrast, β-glucan preparation from autoclaved meal showed no influence of frequency on G′. Whereas sample AU2 was more or less instable, behaviour of sample AU4 pointed to a concentration-dependent gel structure (linear visco-elastic range or particle system).

Evaluation of loss modulus G″ showed especially in samples EX2, BR2 and EX4 dominant viscous properties in the measured frequency range. Here the β-glucans form highly viscous systems. With exception of AU4 and BR4, the β-glucan samples had quasi solid-state characteristics (Fig. 2). The increase of G′ and G″ with increasing frequency point to a molecular dependency of the network formation from the measurement conditions.

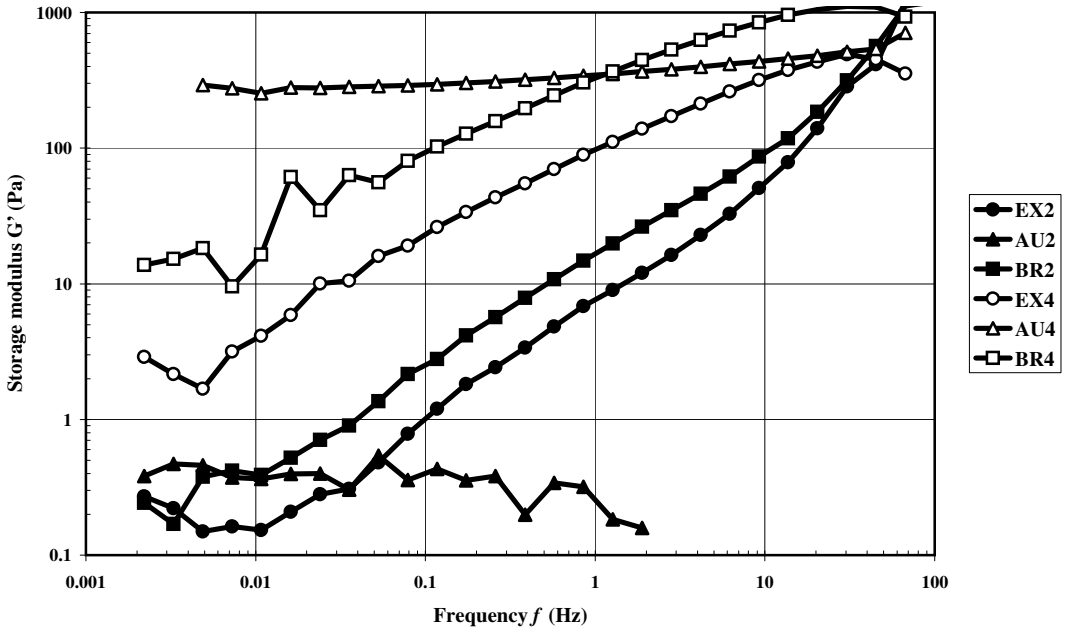

Fig. 1. Storage modulus G′ in the frequency sweep of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%.

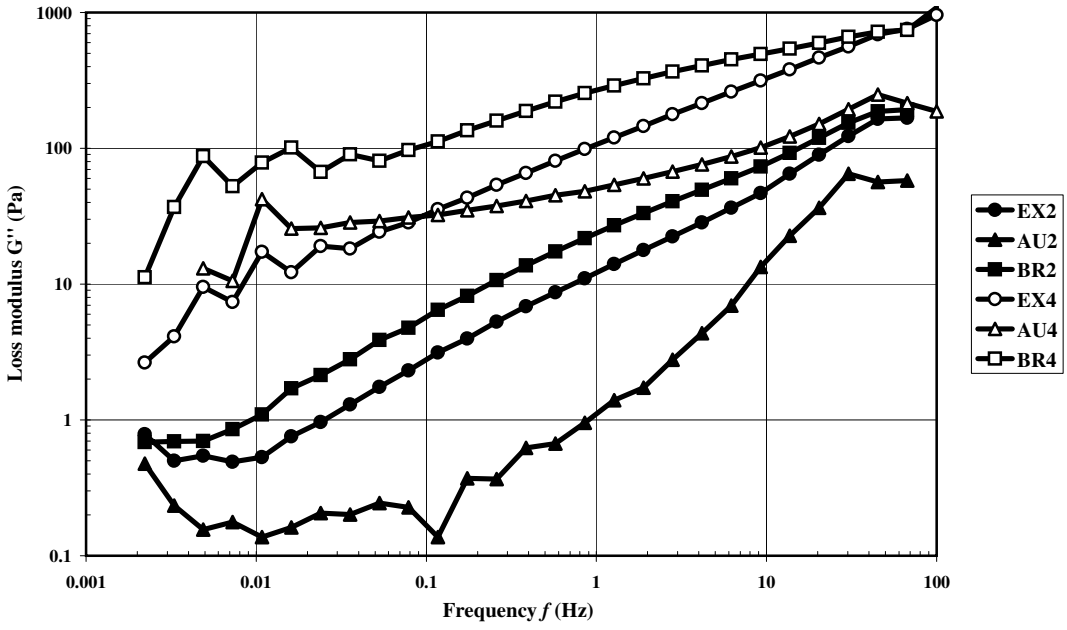

Fig. 2. Loss modulus G″ in the frequency sweep of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%.

The loss factor tan δ is defined as the quotient of G″ and G′. Therefore, the higher the loss factor the higher is the proportion of dissipated energy due to viscous flow under external stress. The loss factor was >1 in samples EX2, BR2, EX4 and BR4 in a wide range of frequency and, therefore, it points to dominant viscous properties in these samples. But with increasing frequency, the elastic properties became greater. In contrast, exclusively elastic behaviour was found in sample AU4 (Fig. 3).

The complex viscosity $|\eta^*|$ of the samples decreased in double logarithmic presentation with increasing frequency (Fig. 4). Sample AU4 was a highly viscous solution. Curves of samples BR4 and EX4 had a more asymptotic course. It is probable that these β-glucan preparations may form gels if their concentration is further increased. At a frequency of 0.01 Hz the following order was found in complex viscosity:

$$AU4 \gg BR4 \gg EX4 \gg BR2 > EX2 > AU2.$$

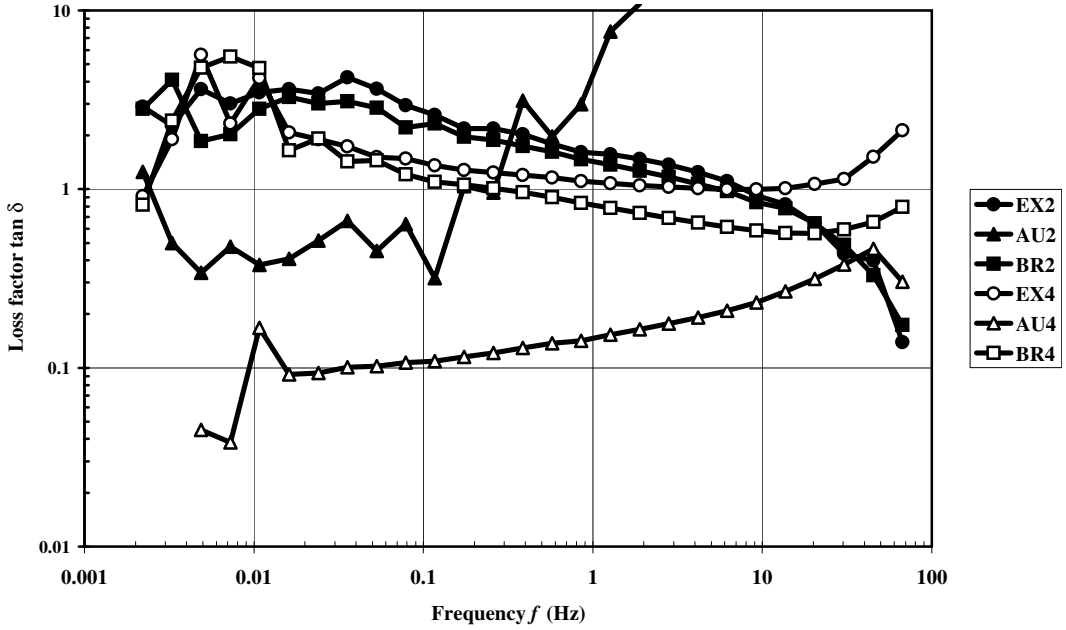

Fig. 3. Loss factor tan δ in the frequency sweep of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%.

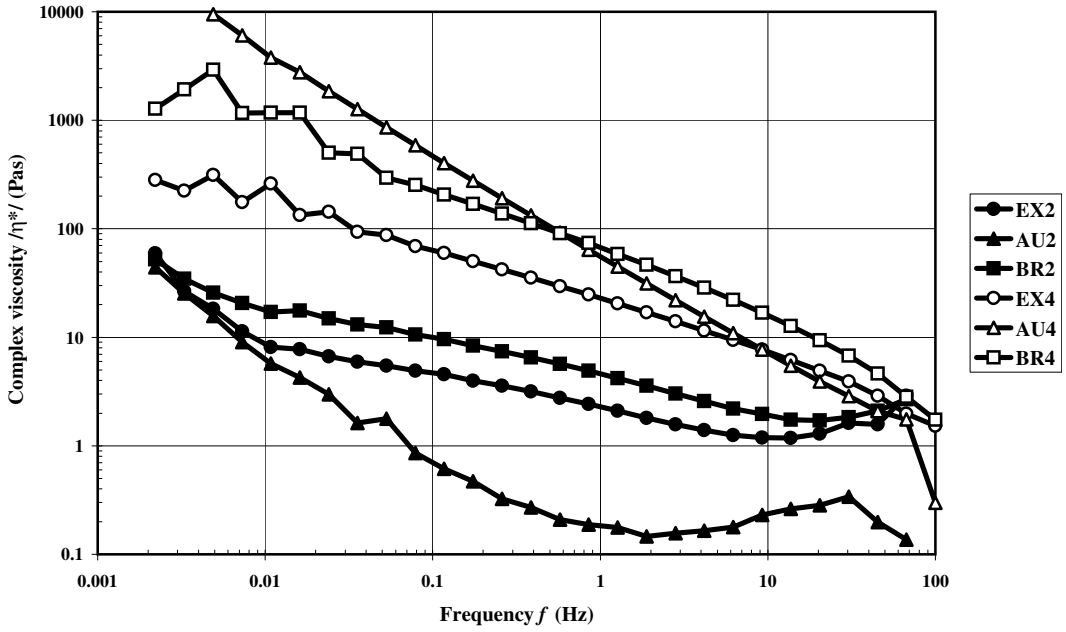

Fig. 4. Complex viscosity $|\eta^*|$ in the frequency sweep of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%.

A similar order was estimated for the storage modulus G′.

3.2.2. Amplitude sweep

Executing the amplitude sweep is normally the first step in oscillation tests. At an angular frequency of 1 Hz, the LVR must be determined with $\gamma \leqslant \gamma_{crit}$ as critical deformation. For all samples it was found that $\gamma = 0.001$ assured the native structure. For this reason, all frequency sweeps were carried out using this parameter. The amplitude sweeps of these materials and their dependence on concentration are shown in Figs. 5 and 6.

The dominant viscous behaviour of this solutions was derived from the fact that the storage modulus was lower than the loss modulus. The structural stability of the solutions can also be determined. In such highly

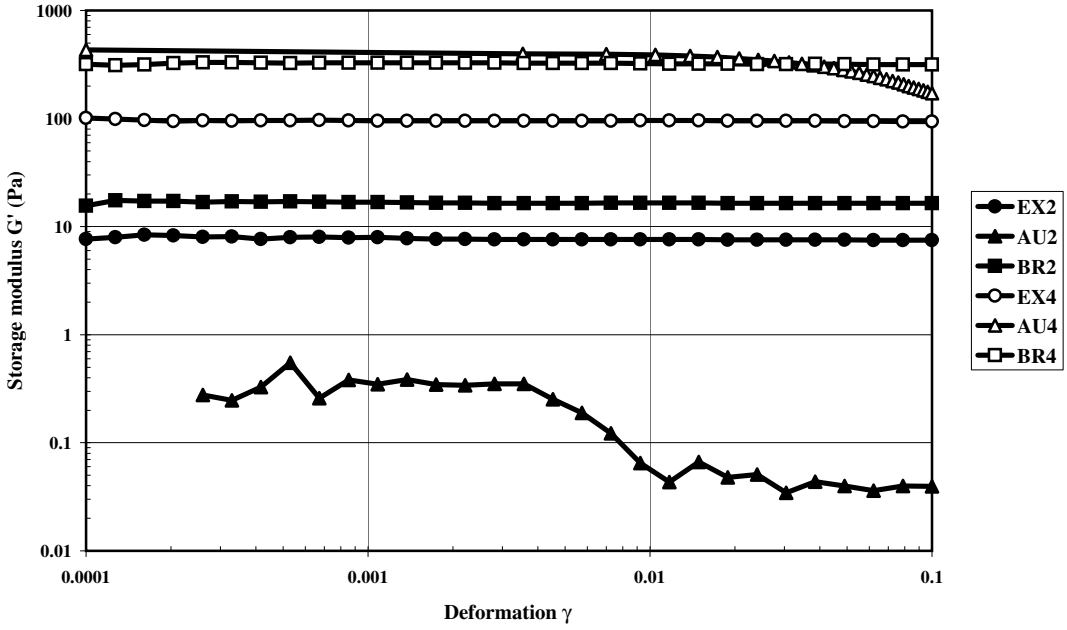

Fig. 5. Storage modulus G′ in the amplitude sweep of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%.

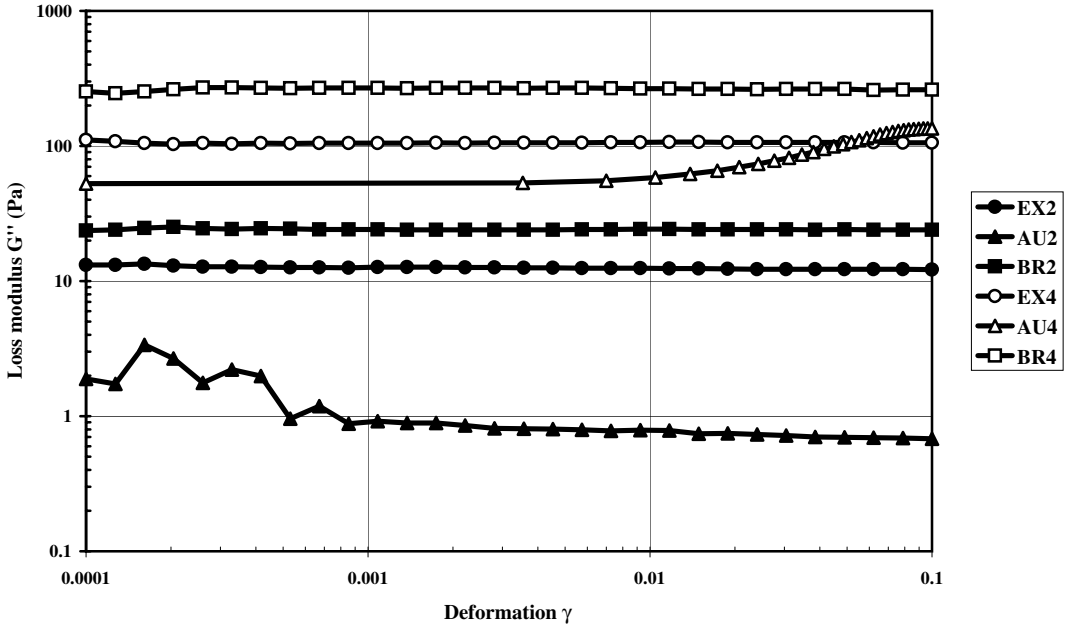

Fig. 6. Loss modulus G″ in the amplitude sweep of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%.

viscous solutions, the fluid properties are more important. This means that complex viscosity $|\eta^*|$ is the best parameter for comparing the fluidity of the materials.

Surprisingly in the amplitude sweep, the storage modulus G′ was not influenced by the deformation (Fig. 5). The structure-forming β-glucan molecules are so strongly involved in a viscous matrix of permanently reproducing electrostatic and hydrophilic interactions, that the structure was not destroyed within the tested deformation range. Sample AU2 exhibited an exception to this behaviour. In a similar manner, the loss modulus G″ of the β-glucan samples, especially from extruded meal and from oat bran, were independent of the used deformation γ (Fig. 6). Both moduli were relatively low in sample AU2. This points to the existence of a low-viscous solution.

With exception of sample AU2, the loss factor tan δ was independent of the deformation in the range $\gamma < 0.01$ (Fig. 7). The loss factors of the samples BR4 and AU4 were below 1, indicating that the viscous and

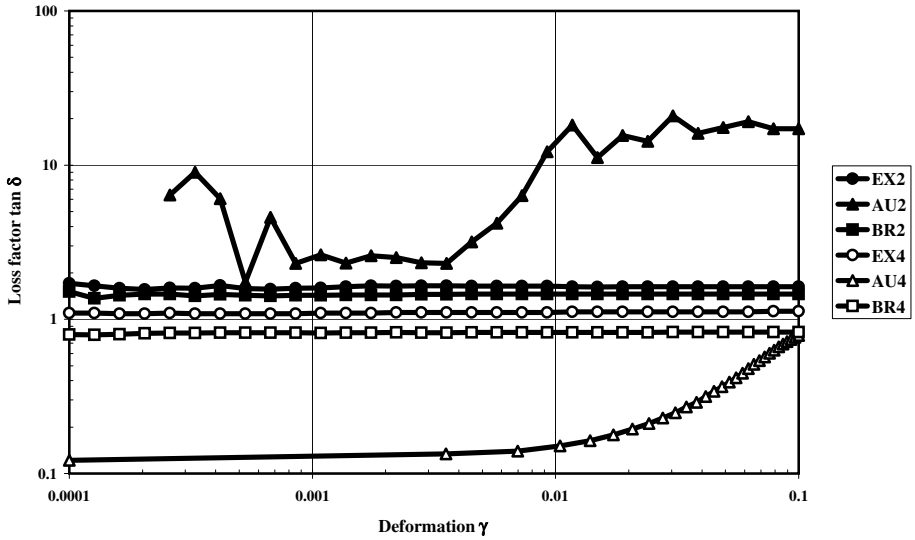

Fig. 7. Loss factor tan δ in the amplitude sweep of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%.

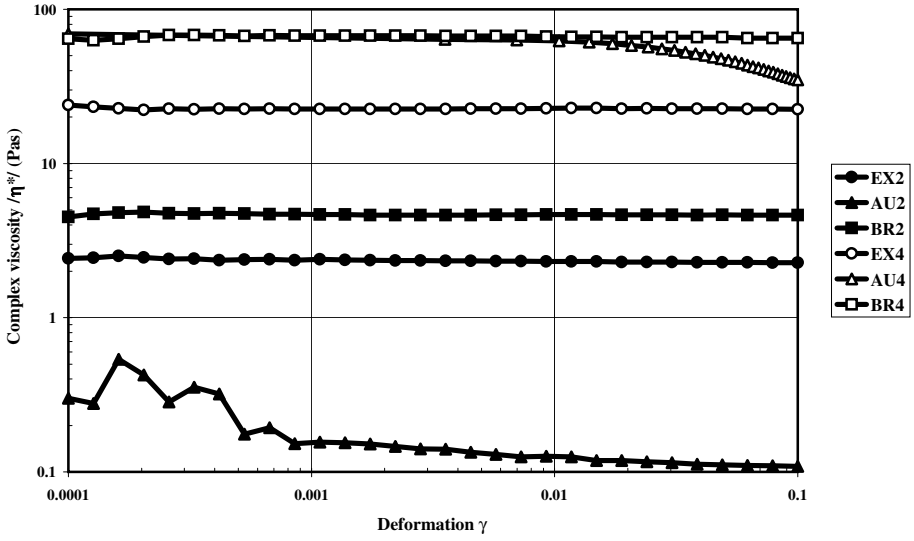

Fig. 8. Complex viscosity $|\eta^*|$ in the amplitude sweep of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%.

elastic properties are on same level. The complex viscosity $|\eta^*|$ of all samples in the amplitude sweep was independent of the deformation (Fig. 8). The following order was found:

$$\mathrm{BR4} = \mathrm{AU4} \gg \mathrm{EX4} \gg \mathrm{BR2} > \mathrm{EX2} \gg \mathrm{AU2}.$$

3.2.3. Shear experiments

The tested β-glucan solutions are structural viscous non-Newtonian systems with a rheostable behaviour. Rheodynamic dependencies were only found in sample AU4.

A useful model to describe of the measured solutions was the power law equation of Ostwald–de Waele (Eq. (2))

$$\tau = K \cdot \dot{\gamma}^n \quad \text{in Pa}, \tag{2}$$

where K is the consistency factor and the exponent n is the flow index.

Models with yield points (Herschel–Bulkley and Casson) gave flow yields in the range of the error of measurements or negative values. Correlation coefficients were >0.999 when the OW model was used. The data obtained are summarized in Table 2. The mechanically most stable samples were BR4 and EX4 followed by sample AU4 (Fig. 9). Samples containing 2% β-glucan had a lower shear stress in the measured range. The lowest values were found in sample AU2.

Table 2
Rheological parameters of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4% from shear experiments

Preparation	Model	Yield point τ_0 (Pa)	Consistency factor K; Casson viscosity η_{CA}; (kg/ms^{2-n}; Pas^n)	Flow index n	Correlation coefficient R	SD s (Pa)	Thixotropy area A_{TH} (Pa/s)
EX2	HB[a]	−0.086	4.472	0.698	0.999	0.19	1.6
	CA	0.334	1.914	2	0.981	1.10	1.6
	OW	–	4.339	0.713	0.999	0.25	1.6
AU2	HB[a]	−0.021	0.117	0.947	0.999	0.01	0.1
	CA[a]	(not evaluable)					0.1
	OW	–	0.099	1.019	0.999	0.01	0.1
BR2	HB[a]	−0.215	9.640	0.662	0.999	0.43	1.5
	CA	0.940	3.615	2	0.978	2.36	1.5
	OW	–	9.324	0.677	0.999	0.56	1.5
EX4	HB	0.531	51.92	0.561	0.999	1.18	33.8
	CA	11.18	12.18	2	0.980	9.21	33.8
	OW	–	52.64	0.555	0.999	1.03	33.8
AU4	HB	2.102	41.51	0.364	0.994	2.11	150.9
	CA	20.84	3.308	2	0.959	5.60	150.9
	OW	–	43.91	0.348	0.995	2.02	150.9
BR4	HB[a]	−30.54	194.74	0.407	0.999	3.13	2.7
	CA[a]	(not evaluable)					2.7
	OW	–	160.90	0.470	0.998	6.91	2.7

HB: Herschel–Bulkley; CA: Casson; OW: Ostwald–de Waele.
[a] Not applicable, negative yield points.

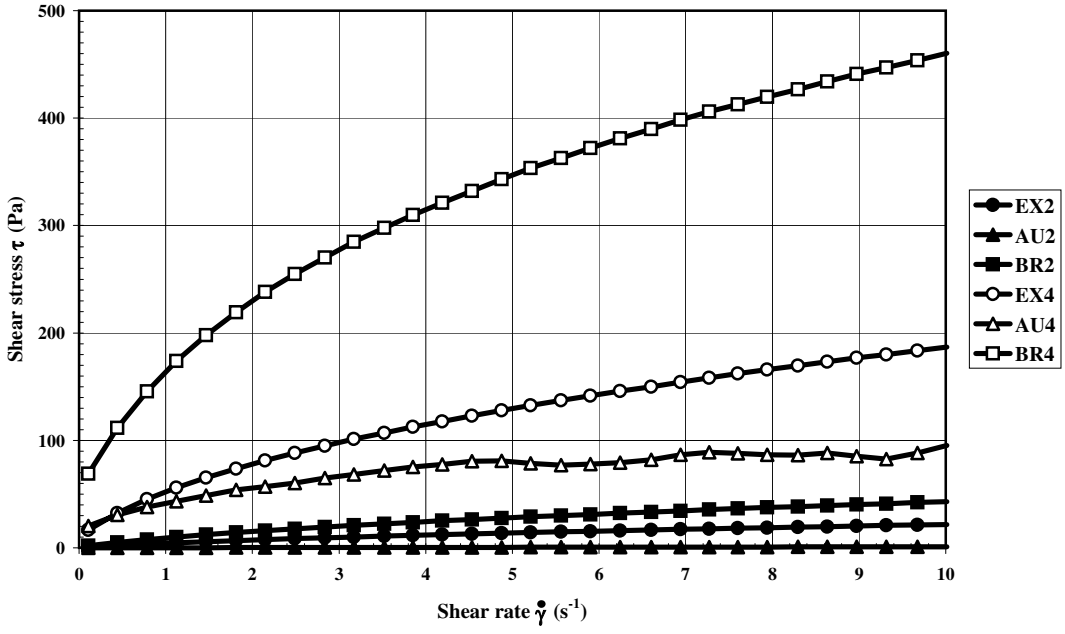

Fig. 9. Rheogram of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%.

The highest levels of effective viscosity $\eta(\dot{\gamma})$ were measured in sample BR4 and lowest in sample AU2 (Fig. 10).

The process viscosity $\eta_{process}$ (Eq. (3)) can be differentiated from Eq. (2). Under the applied rheological conditions, the process viscosity seems to be better suitable than the apparent viscosity $\eta_{apparent}$ or $\eta_{effective}$ (Eq. (4)).

$$\eta_{process} = d\tau/d\dot{\gamma} = n \cdot K \cdot \dot{\gamma}^{n-1} \quad \text{in kg/ms } (= \text{Pas}), \tag{3}$$

$$\eta_{apparent} = \frac{\tau}{\dot{\gamma}} = K \cdot \dot{\gamma}^{n-1} \quad \text{in kg/ms } (= \text{Pas}). \tag{4}$$

Table 3 shows the viscosities calculated using Eqs. (3) and (4). Because of the high flow indices n (Table 2),

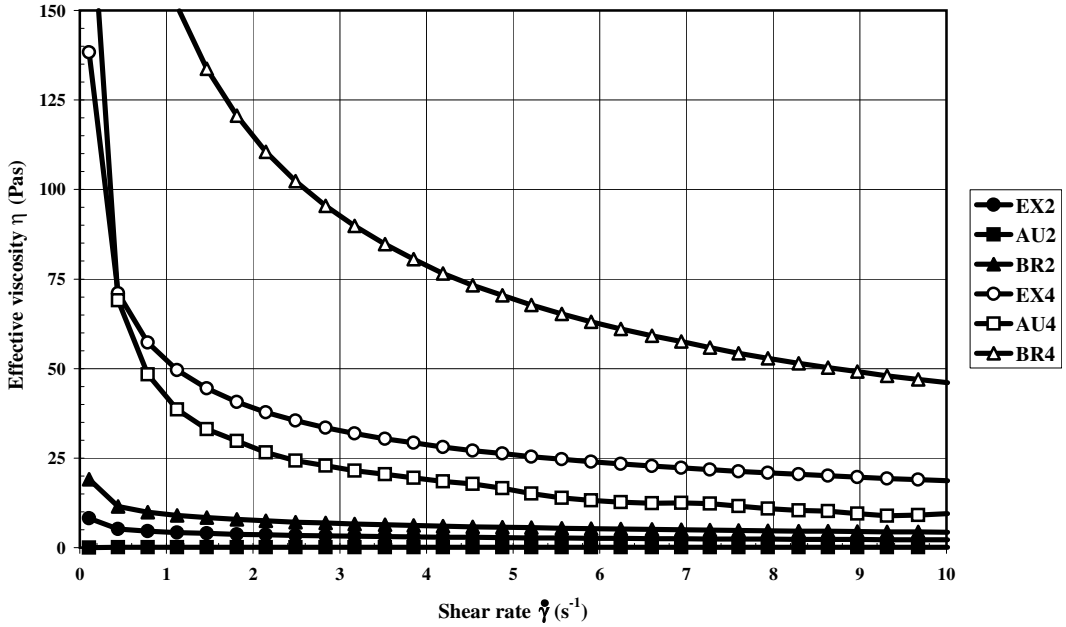

Fig. 10. Effect of shear rate $\dot{\gamma}$ on the shear viscosity η of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%.

Table 3
Apparent viscosity and process viscosity of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4% at a shear rate of 0.01 s^{-1} (simulated small intestine conditions)

Preparation	Apparent viscosity $\eta_{apparent}$ (0.01 s^{-1}) in Pas	Process viscosity $\eta_{process}$ (0.01 s^{-1}) in Pas
EX2	16.27	11.60
AU2	0.09	0.09
BR2	41.08	27.81
EX4	408.6	226.8
AU4	884.3	307.7
BR4	1847.4	868.3

Table 4
Complex viscosity $|\eta^*|$ (from oscillation experiments in the linear visco-elastic range) of the isolated β-glucan preparations EX, AU and BR extracted from extruded and autoclaved oat meal as well as from oat bran in concentrations of 2% and 4%

Preparation	Complex viscosity in Pas (amplitude sweep at 1 Hz)	Complex viscosity in Pas (frequency sweep at 0.01 Hz)	Storage modulus in Pa (LVR)
EX2	2.3	ca. 9	No
AU2	0.12	ca. 7	ca. 0.6
BR2	4.8	ca. 20	No
EX4	22.6	ca. 250	No
AU4	66.0	ca. 3000	ca. 300
BR4	65.0	ca. 1100	No

sample AU4 was the most structurally stable material against mechanical pressure. Table 4 summarizes the results of oscillation experiments.

3.2.4. Shear rate calculation and resulting viscosity effects under simulated conditions during passage of the small intestine

The shear rates (non-Newtonian flow) during digestion in the small intestine were calculated according to the technical conditions in a cylinder tube. The small intestine of humans is approximately 3 m long, with a diameter of approximately 4 cm. This corresponds to a surface of approximately 0.33 m^2. But it should be taken into consideration that the real lumen-limited surface of the small intestine is approximately 200 m^2 because of the complex architecture and morphology of the mucosa. The daily input of liquids into the gastrointestinal tract is approximately 9 l (~2 l from food and drinks; ~7 l from secretions of salivary gland, stomach, pancreas, bile, jejunum and ileum). Of these, approximately 7.9 l is absorbed in the jejunum and ileum (Thews, Mutschler, & Vaupel, 1991).

The shear rate in the small intestine was calculated using Eq. (5) as a basis:

$$\dot{\gamma} = \frac{4 \cdot \dot{V}}{3.14 \cdot R^3} \quad \text{in } s^{-1}, \tag{5}$$

where the length of small intestine was defined as 3 m and the diameter as 40 mm and the volume throughput was 0.005 m^3/d (5 l/24 h) in form of a continuous transport. The shear rate $\dot{\gamma}$ was estimated:

$$\dot{\gamma} = \frac{4 \cdot 0.005}{3.14 \cdot 3600 \cdot 24 \cdot 0.02^3} = 0.01 \ s^{-1}.$$

This means that the transport of food is in the range of glide flow. The limit to the classic shear flow with determined hydrodynamic conditions is at a shear rate of $\geqslant 0.1\ s^{-1}$. Because of the lower shear rates by factor of ten, an increase of the viscosity level occurs according to Eqs. (3) and (4). The resulting effective viscosity increased enormous and is obtained in Table 3.

4. Discussion

The cereal DF component β-glucan plays a role in food science and in nutrition, because of its viscosity in aqueous systems and in the intestinal tract. The rheological behaviour of β-glucan preparations isolated from extruded and autoclaved oat meal as well as from oat bran was evaluated using oscillatory and rheological measurements within the linear visco-elastic region in concentrations of 2% and 4% in aqueous solution. Fluid dynamic parameters of structure-viscous behaviour (e.g. apparent/effective viscosity or process viscosity of non-Newtonian systems) may influence the flow, diffusion or transport behaviour during digestion processes in small intestine.

The high levels of apparent and process viscosities found for BR4, AU4 and EX4 (Table 3) under small intestine conditions are a chance to limit processes of mass transport and/or to reduce food intake using β-glucan preparations from oat products.

The solubility, extractability and yield of β-glucan are influenced by the particle size and pre-treatment of the cereal materials as well as by the extraction conditions (e.g. temperature, pH, ionic strength) (Zhang, Doehlert, & Moore, 1998). During extraction of β-glucan, partial depolymerisation may occur because of action of β-glucan degrading enzymes or hydrothermal effects. Physiological effects of the β-glucans in the small intestine are connected with their macromolecular state and properties (e.g. viscosity, flow behaviour) of its soluble part. Therefore, it is not important to extract completely β-glucan from oat or barley products when their rheological properties are evaluated for physiological aspects. Like in our experiments, Westerlund et al. (1993) isolated approximately 50% of the β-glucan from oat bran and endosperm after inactivating of endogenous β-glucanases, extraction of lipids and enzymatic starch and protein degradation with water (3–4 h at 70–80 °C). This procedure was modified by several groups (Colleoni-Sirghie et al., 2003; Gómez, Navarro, Manzanares, Horta, & Carbonell, 1997a; Gómez, Navarro, Garnier, Horta, & Carbonell, 1997b). Further, β-glucan can be extracted under alkaline (Wood, Siddiqui, & Paton, 1978) or acidic conditions (Bhatty, MacGregor, & Rossnagel, 1991). Often a higher yield was connected with a higher degradation of β-glucan (Beer, Wood, & Weisz, 1997). Also removal of protein in isolated β-glucan preparations using trypsin may decrease the viscosity (Autio et al., 1992).

The shear rate–shear stress relation data of β-glucan solutions followed the power law and conditional for higher concentrations of ß-glucan the Herschel–Bulkley model. The flow behaviour index varied from 0.97 and 0.31, indicating mild to highly pseudoplastic solutions, respectively, where the pseudoplasticity increased with the concentration (Tejinder, Bhupinder, & Harinder, 2000). Solutions of β-glucans isolated from steamed oat groats were very viscous and highly pseudoplastic (power law equation) and more visco-elastic (higher G′ and G″) than those from raw or roasted samples (Zhang et al., 1998). Autio (1988) found that the shear, dynamic and complex viscosities of β-glucans from oat bran were similar at low shear rates. In oscillation tests, G′ was higher than G″ at low frequencies but G″ was higher at high frequencies where both moduli increased with increasing frequency. Böhm and Kulicke (1999a) reported that β-glucan exists in concentrated solution in two states: molecular disperse with normal visco-elastic flow behaviour in freshly prepared aqueous solution and as an infinite gel-like network structure. The gelation rate raised with decreasing molar mass and increasing β-glucan concentration as shown by oscillatory time experiments (Böhm & Kulicke, 1999b). Cooked oat bran exhibited shear thinning behaviour during a thixotropic test experiment, whereas an Oatrim-10 designed β-glucan preparation exhibited an unexpected region of shear-thickening behaviour. Both preparations differed in their oscillation shear data (Carriere & Inglett, 1998). Doublier and Wood (1995) found in flow and oscillatory measurements of aqueous solutions from partially hydrolysed β-glucans in contrast to original samples a more gel-like behaviour with a tendency to aggregate and to form a three-dimensional macromolecular network. β-Glucans showed a typical shear-thinning flow behaviour. This points to the existence of a structure formation in the solution which would be progressively disrupted by increasing shearing forces (Gómez et al., 1997b). At 25 °C, storage modulus G′ was higher than lost G″ if a high-molecular β-glucan was measured. In case of β-glucan having a lower molecular weight, higher G′ was found only at high frequency. The shear thinning and visco-elastic behaviour of fresh β-glucan solutions were typical of a random coil type polysaccharide and dependent on molecular size and concentration. With decreasing molecular weight of β-glucans, the gelation time increased and the gelation rate decreased (Lazaridou, Biliaderis, & Izydorczyk, 2003).

Our results show that the β-glucans isolated from oat products had a complex rheological behaviour depending on the source material, the technological pre-treatments and the used concentration. This behaviour may influence the effects of β-glucans in the intestinal tract.

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