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Effect of extrusion processing parameters on structure, texture and dietary fibre composition of directly expanded wholegrain oat-based matrices

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A B S T R A C T

Oat flour mixed with 30 g/100 g rice flour was extruded with a twin-screw extruder using a central composite orthogonal design. Temperatures (120 °C,140 °C, 160 °C) and moisture (14.5 g/100 g, 17.7 g/100 g, 20.6 g/100 g) were adjusted during extrusion, while screw speed was kept constant (400 rpm). Extrudates were analysed for structure (expansion, density, microstructure), texture (hardness), β-glucan (molecular weight and extractability), as well as fibre content. Expansion varied between 250 and 329%, density between 165 and 457 kg/m^3 and hardness between 27 and 64 N. The response surface model showed that more expanded, less dense and less hard extrudates were achieved at low moisture, while high temperature resulted in lower density and hardness. Significant differences in β-glucan extractability were observed depending on extrusion conditions, with values ranging between 0.64 and 1.31 g/100 g. β-glucan extractability correlated with positively with porosity, and negatively with moisture content during extrusion, cell wall thickness and density. The results indicate that conditions that produce a more porous, crispier structure, also increases β-glucan extractability.

1. Introduction

Extrusion cooking is a well-established technology for production of directly expanded crispy snacks or breakfast cereals. The shear energy introduced to the flour mix by rotating twin screws together with barrel heating results in a melt which than expands at the die exit due to instant change in pressure. The thermo-mechanical process results in physical (i.e. water binding, degree of expansion) and chemical (i.e. protein denaturation, starch gelatinization, fibre solubilization, Maillard reactions) changes in the raw material that lead formation of an expanded, crispy texture. From a nutritional perspective, wholegrain oats are an excellent raw material for production of extruded breakfast cereals or snacks, due to the presence of high level of dietary fibres (8.5–13 g/100 g), including cholesterol lowering β-glucan (BG) (1.8–7 g/100 g), healthy lipid profile as well as high protein content (13–20 g/100 g) (Guo et al., 2014; Jokinen et al., 2021; Martínez-Villaluenga & Peñas, 2017). However, the relatively high lipid content in wholegrain oats interferes with the extrusion performance of the material by causing significant reduction in shear rates, dissipating the energy input and eventually limiting degree of expansion which further results in hard, non-crispy textures (Nikinmaa et al., 2023; Singh & Smith, 1997). Also, the presence of high levels of insoluble dietary fibre (DF) content in wholegrain oats hinders the formation of an airy structure (Brennan et al., 2008; Nikinmaa et al., 2017). In addition to this, wholegrain oats are relatively low in starch (ca 63 g/100 g) (Jokinen et al., 2021). In directly expanded extruded cereal products, starch forms the continuous phase in structure building and typically a starch content higher than 70–80 g/100 g is needed for forming an airy, crispy texture. Even defatted endosperm oat flour, where the starch is enriched (73 g/100 g), and lipids and DF reduced (3.9 g/100 g total DF), produced extrudates with low expansion (ca 200%) and hard texture (150 N), indicating that oat starch itself is not an optimal starch for extrusion (Sibakov et al., 2014).

Choosing optimal extrusion conditions may counteract problems with structure and texture to an extent. Optimization of oat extrusion conditions have been performed in a limited number of studies (Liu et al., 2000; Sandrin et al., 2018; Singh & Smith, 1997; Wang et al., 2019). Several studies agree that moisture content is critical for expansion (Liu et al., 2000; Wang et al., 2019), with relatively low moisture leading to improved expansion, and less hard texture (Liu et al., 2000). Furthermore, several studies found that higher screw speeds increased expansion (Brahma et al., 2016; Liu et al., 2000; Wang et al., 2019), although comparing the effect of various speed ranges and screw configurations is difficult. Screw speed would affect a variety of factors, including barrel fill rate, residence time and shear forces on the material. In several articles, oats are mixed with other ingredients, such as corn or rice to improve expansion, while in studies where only oats have been used, even the optimized conditions showed low expansion

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ratios. Optimal conditions for expansion may bring other issues, e.g. lipid oxidation was found to accelerate at extrusion temperatures exceeding 130 °C (Moisio et al., 2015).

Starch is not the only polysaccharide affected by extrusion. In an experiment by Tosh et al. (2010) breakfast cereals consisting of BG rich OatWell oat bran, corn flour, fructose and salt were extruded at four different conditions, namely temperatures ranging from 181 to 237 °C and water addition between 18.7 and 7 g/100 g. Although no depolymerization of the β-glucan was observed at 181 °C, an exponential relationship between the temperature, the standard mechanical energy (SME) and the reduction of the molecular weight could be described. Interestingly, increasing the temperature and SME also led to a significantly higher extractability of the BG under physiological in-vitro conditions. Similarly, Roye et al. (2021) showed that extractability was increased in high-shear and high temperature extrusion conditions, while extract viscosity was reduced. On the other hand, no effect on oat BG Mw was observed when oat flour was extruded at 100°C–130 °C and 15–21 g/100 g moisture, although extractability of oat BG increased from 0.86 g/100 g in the flour to 1.23 g/100 g (Brahma et al., 2016). Reduction of insoluble dietary fibre (IDF) (4.2–3.7 g/100 g) and increase in soluble dietary fibre (SDF) (1.8–3.85 g/100 g) after extrusion has also been observed, probably due solubilization of IDF, as well as formation of resistant starch during extrusion (Sandrin et al., 2019). These previous studies show that dietary fibre fractions, such as BG may be modified by different extrusion conditions, with more severe conditions leading to increased extractability, but also lower molecular weight and viscosity. Higher molecular weight BG (Mäkelä et al., 2021; Wang et al., 2016) has been shown to have greater cholesterol lowering effect, while higher extractability may affect both cholesterol (Mäkelä et al., 2021) and glucose response (Mäkeläinen et al., 2007). Few studies have examined how the processing conditions relate to structure and texture of the oat extrudates. As these characteristics have a great effect on consumer acceptability of extruded food products, they should be weighed against the chemical and nutritional effects of extrusion.

The aim of this research was to understand the effect of extrusion processing conditions on structure (expansion, density, microstructure) and texture (hardness, crispiness) of wholegrain oat extrudates in connection to specific dietary fibre components (IDF, SDF and β-glucan).

2. Materials and methods

2.1. Raw materials and chemicals

Finnish wholegrain oat flour was extruded with 30 g/100 g (dry matter basis) addition of rice flour (Leipurin Oyj, Finland). The rice flour was added in order to improve the extrusion behaviour, in order to attain clear differences between the different conditions studied. The wholegrain oat flour contained 71 g/100 g starch, 13 g/100 g protein, 11 g/100 g dietary fibre (DF) of which 7 g/100 g soluble dietary fibre (SDF), 5 g/100 g fat and 2 g/100 g ash (Jokinen et al., 2021). The reported composition of the rice flour was 76 g/100 g carbohydrates, of which 0.1 g/100 g was sugars and 0.6 g/100 g dietary fibre, 7 g/100 g protein and 1 g/100 g fat. Rice flour was added to improve degree of expansion and to better be able to observe differences between parameters.

Acetic acid glacial >99.0%, α-amylase from Bacillus licheniformis, (Termamyl 300®L), calcium chloride, calcium chloride dihydrate, pancreatin from porcine pancreas, 2-propanol ≥99.8%, sodium azide ≥99.5%, sodium hydroxide ≥98.0%, sodium hydroxide solution 50–52%, sodium nitrate ≥99.0%, trizma base ≥99.9% and xylanase from Thermomyces lanuginosus (≥2500 U/g) were purchased from Sigma Aldrich (Switzerland). Maleic acid >99% and sodium dihydrogen phosphate anhydrous >99.0% were obtained from Fluka (Germany). The β-Glucan Assay Kit (Mixed Linkage), the Rapid Integrated Total Dietary Fiber Assay Kit, Amberlite® FPA53 (OH-) resin and Ambersep® 200 (H+) resin were purchased from Megazyme, Ireland. The standards for the size exclusion chromatography, PolyCALTM polyethylenoxide (PEO-24 K, Mw = 23′651 g/mol, Mn = 23432 g/mol, dn/dc = 0.132 mL/g, IV = 0.41 dL/g) and PolyCALTM dextran (DEX-70 K; Mw = 70′026 g/mol, Mn = 55411 g/mol, dn/dc = 0.147 mL/g, IV = 0.260 dL/g) were purchased from Malvern Instruments (Malvern Panalytical Ltd, U.K). Hydrochloric acid 37% was obtained from VWR Chemicals and Ethanol abs. from the HCI Shop, Switzerland. Ultrapure (Milli-Q) water was used for all experiments (Merck Millipore, Merck KGaA, Germany).

2.2. Extrusion trials

Extrusion was performed using a co-rotating twin screw extruder (APV Baker Perkins MPF 19/25, UK) with a 19 mm screw diameter and L/D of 25. The screw configuration is presented in Santala et al. (2014). A central composite orthogonal study design with moisture and temperature as variables was used for the extrusion trials (Table 1), as these factors were found to have the greatest effect on the studied variables in preliminary trials (data not shown). The moisture (14–21 g/100 g) and temperature (117–163 °C) range were chosen based on our previous work (Nikinmaa et al., 2023, Alam et al.) values found in the literature (Liu et al., 2000; Sandrin et al., 2019) on oat extrusion. The screw speed and flour feed rate were kept constant at 400 rotations per minute (rpm) and 60 g/min, respectively. Samples were collected as ca 30–40 cm ribbons and dried at 105 °C for 15 min after extrusion.

2.3. Macrostructure and texture

Samples for macrostructure were cut with an electric band saw into 10 cm ribbons. Radial expansion was measured in three spots for each sample using Vernier callipers. Specific length and density were measured based on the dimensions and mass of the ribbons.

Instrumental texture was measured with a Texture Analyser using a uniaxial compression test as described by Alam et al. (2016). Briefly, 1 cm pieces cut with a band saw and stored at 50% relative humidity for 7 days to equilibrate the moisture content in each sample. The samples were compressed at a strain of 70% with a probe speed of at 1 mm/s, using a 25 mm probe and 30 kg load cell. Hardness and crispiness index (CI) were analysed from the force deformation curve as explained in detail by Alam et al., 2016.

X-Ray micro tomography imaging was performed using an RX-solutions Desktom 130 scanner (RX Solutions, France). Triplicate 1 cm samples were cut with an electric band saw. The X-ray detector was run with a 40 kV voltage during the scan. Porosity, cell wall thickness, and air-cell size was analysed with a Python based analytics program.

2.4. β-glucan extraction for molecular weight determination

To determine the effect that extrusion cooking has on the molecular size of BG, the latter was extracted from the extrudates as well as from the native oat-rice flour mixture according to a procedure adapted from Marasca et al. (2020), Rieder et al. (2015) and Lazaridou et al. (2004).

Table 1
Central composite orthogonal study design parameters.

Sample code	Temperature (°C)	Moisture content (%)
N1	120.00	14.50
N2	120.00	20.60
N3	160.00	14.50
N4	160.00	20.60
N5	140.00	14.00
N6	140.00	21.10
N7	117.05	17.70
N8	162.95	17.70
N9	140.00	17.70
N10	140.00	17.70
N11	140.00	17.70

The extrudates were pulverized in a blender (Professional 800, Blendtec, USA). 10 g of sample was placed in 200 mL of 70% ethanol and stirred for 2 h at 80 °C. After cooling to RT, the mixture was centrifuged for 10 min at 4000 rpm (Centrifuge 5810 R, Eppendorf, Germany) and the supernatant discarded. The pellets were resuspended in 95% ethanol, centrifuged and the supernatant discarded. Subsequently, the pellets were resuspended in 400 mL of water and stirred overnight at RT. 2 mL of Termamyl and 20 mg of $CaCl_2$ were added and the bottles were incubated in an oven (FD 115, Binder, Germany) at 96 °C. The mixture was cooled to RT, centrifuged for 10 min at 8000 rpm (Sorvall Lynx 4000 Centrifuge, Thermo Scientific USA) and the supernatant collected. 4 mL of sodium acetate buffer (2.5 M, pH 4.5) and 4 mg of xylanase (2500 U/g, 10 U) were added, the pH was adjusted to 4.5. The mixture was then incubated for 3 h in a shaking water bath (SW22, Julabo, Germany). Subsequently, the pH was adjusted to 7 and 20 mg pancreatin added for another 30 min of incubation in a shaking water bath at 40 °C. After heating the samples to 90 °C for 20 min, they were cooled to RT, centrifuged for 10 min at 8000 rpm and the supernatant collected, precipitated with 2 vol of 95% ethanol, and stored overnight at 4 °C. The samples were centrifuged at 8000 rpm for 10 min, the pellets resuspended in 40 mL of 2-propanol, and centrifuged for 10 min at 4000 rpm. The supernatant was discarded, and the pellet resuspended in 25 mL of 2-propanol and finally dried under nitrogen (Reacti-Therm™ TS-18823, Thermo Scientific) at 60 °C.

2.5. Molecular weight measurement of soluble BG in extrudates

The weight-average molecular weight (Mw) of the BG in the extracts was determined by size exclusion chromatography (SEC) with triple detection. The system used for this analysis consisted of the OMNISEC resolve and the OMNISEC reveal modules (Malvern Panalytical Ltd, U.K.). This multi-detector module is composed of a differential refractive index (RI), a UV/Vis, and a lightscattering (right-angle 90°(RALS) and low-angle (LALS, 7°)) detector, as well as a viscometer. The column system consisted of a pre-column (Viscotek AGuard Column, 0.50 cm × 6 mm) and two A6000M columns in series (Viscotek, 30 cm × 8 mm, Malvern Panalytical Ltd, U.K), which were kept at 20 °C. The autosampler was kept at 60 °C. The flow rate was set to 0.7 mL/min and the injection volume was 100 μL.

For the analysis, 10 mg of each sample were suspended in 10 mL SEC eluent (0.1 M sodium nitrate and 0.02 g/100 g sodium azide) to a concentration of 0.1 mg/mL. To analyze each sample in triplicates, three times 1.5 mL of the solution was filtered (0.45 μm, nylon) into a HPLC vial immediately before injection. Polyethyleneoxide (PEO-24 K) and dextran (DEX-70 K) solutions were prepared in the SEC eluent as calibration and verification standards.

To calculate the weight-average molecular weight, the obtained data from the RALS/LALS, RI and viscosity detectors was evaluated using the OMNISEC Software Version 10.30. For cereal BG, a dn/dc of 0.145 mL/g was used. A sample chromatogram is included in supplementary files to represent the complexity of the analysis.

2.6. β-glucan extractability

To determine the BG extractability of the extruded samples, a procedure using the β-Glucan Assay Kit (Mixed Linkage) from Megazyme (Ireland) was conducted. Approximately 2.4 g of the pulverized samples were weighted in triplicates into 50 mL falcon tubes, wetted with 0.4 ml ethanol abs. and then mixed with 40 ml of sodium phosphate buffer (20 mM, pH 6.5). The tubes were incubated for 2 h in a shaking (120 rpm) water bath at 38 °C, centrifuged (4000 rpm, 15 min) and a 5 mL aliquot of the supernatant transferred to a new 15 ml falcon tube for the determination of the extractable BG content. For control, 50 mg of oat control flour included in the kit was weighted in triplicates in 15 ml falcon tubes, wetted with 0.4 mL ethanol abs. and dispersed in 5 mL sodium phosphate buffer (20 mM, pH 6.5). The control samples were boiled for 30 min on a stirring plate and let to cool to RT. 0.2 mL lichenase (50 U/mL, 10 U) was added to all tubes, vortexed, and the tubes incubated for 1 h in a shaking water bath at 50 °C. The assay was then continued according to the Megazyme protocol. The absorbance at 510 nm was measured within 50 min with a UV–Vis spectrophotometer (Cary 100 UV–Vis, Agilent Technologies, USA). The BG content was calculated using the "Mega-Calc" excel file provided by Megazyme.

2.7. Dietary fiber content

The content of dietary fibers (DF) of the extrudates was determined in duplicates using the Rapid Integrated Total Dietary Fiber Assay Kit from Megazyme (Ireland) with the same procedure as for AOAC method 2011.25 with some adaptions. The time-consuming steps of filtration were replaced with centrifugation. According to the method description, the AOAC 2017.16 method allows the measurement of insoluble (IDF), high molecular weight soluble dietary fiber (SDFP; soluble dietary fiber which precipitates in 78% ethanol), and low molecular weight soluble dietary fiber (SDFS; soluble dietary fiber that remain soluble in the presence of 78% ethanol, i.e. non-digestible oligosaccharides of DP $\geq$ 3) including resistant starch. The extrudates were pulverized in a kitchen blender (Professional 800, Blendtec, USA) and then analysed according to the assay procedure described by Megazyme. The collected SDFP and IDF residues were dried overnight in an oven set to 105 °C, cooled, and then weighed. The weights were then corrected for ash and protein content. The protein content was determined by total nitrogen measurement using a TOC-L equipped with a TN module (Shimadzu Europe, Germany). 15 mg of the dried sample was dissolved in 15 mL of water and stirred for 2 h at 60 °C. Some of the samples did not dissolve well, therefore they were dried overnight in an oven at 105 °C, dissolved in 2.0 mL of 4 M HCl overnight, neutralized with 420 μL of 50 g/100 g NaOH solution and diluted to 18 mL with 15.58 mL of water. For the determination of the ash content, depending on the available sample amount, 5–90 mg of sample was weighed into crucibles and ashed overnight at approximately 500–600 °C in a muffle furnace (Mod. L 51/S, Naber Industrieofenbau, Switzerland), cooled, and then weighted. The content of SDFS was determined by HPLC-SEC according to the procedure and equipment description of Megazyme. The data was evaluated using Chromeleon software version 7.2.7 (Thermo Scientific, USA).

Starch content was analysed using a Megazyme Total Starch kit (AOAC method 996.11) for selected samples.

2.8. Statistical analysis

The study design for extrusion trials was performed using Modde 13 (Sartorius. Germany) and to create a multiple linear regression (MLR) analysis of extrusion results. MLR was used to investigate the combined effect of process parameters on response variables (expansion, density, hardness).

Statistics of mean and standard deviations (SD) were calculated with Microsoft® Excel® for Microsoft 365 (Microsoft Cooperation, USA).

Multiple linear regression models with the measured variables as response variables and the extrusion conditions temperature and moisture as predictor variables were fitted to explore the effect of the extrusion on the various variables. A principal component analysis (PCA) including correlation analysis was performed to explore the connections between the extrusion conditions and the different outcome variables. These tests were performed in RStudio 2021.09.1 + 372 "Ghost Orchid" Release for Windows (RStudio, PBC, USA). The threshold for indicating a significant difference was set at $p < 0.05$.

3. Results

3.1. Effect of extrusion processing parameters on macro-, microstructure and texture

Expansion of extrudates varied between 250% and 329% and density between 165 and 457 kg/m^3 depending on extrusion conditions. Response contour plots for expansion and density were produced based on extrusion results (Figs. 1 and 2). The normalized equation (Eq. 1) for expansion is:

$$Exp = 11.53 + 0.07*T-1.06*MC+0.21*T^2-0.37*MC^2+0.16*MC*T$$

where Exp is the expansion ratio in %, MC is moisture percentage, T is temperature in °C. The corresponding equation (Eq 2.) for density (ρ) was:

$$\rho = 2.95–0.70*T+0.83*MC+0.05*T^2+0.39*MC^2-0.47*T*MC$$

The R^2 value of both models was high, 0.859 and 0.983 for expansion and density. The Q^2 values were 0.079 and 0.876, respectively, which indicates significance for both responses, however the predictivity of the expansion model is low while the model for density has excellent predictivity. The results obtained indicate that moisture is the most important determining factor for radial expansion alone, while temperature has limited effect. Previous studies have indicated that twin-screw extrusion processing under low water addition levels promote higher degree of expansion. Highest degree of expansion was achieved at around 18 g/100 g moisture content when 100% wholegrain oats were used (Brahma et al., 2016; Wang et al., 2019), while with 30 g/100 g wheat starch addition the highest expansion were achieved at the lowest water feeding levels (16 g/100 g moisture) (Yao et al., 2006). In both studies with pure oats, expansion was reduced both when increasing and decreasing water content from 18 g/100 g, possibly due to lower contents leading to high melt viscosity, while higher water amounts caused bubble collapse. Several studies have also found either no effect or a negative effect of higher temperature on radial expansion, which is similar to the results obtained in the current work (Brahma et al., 2016; Yao et al., 2006).

However, both moisture and temperature had a profound effect on density, with the lowest densities obtained at high temperature and low moisture. The high temperature together with sudden pressure release at the die exit leads to expansion in both radial (perpendicular to flow) and longitudinal direction (direction of flow), which together make up volumetric expansion. It has been suggested that radial expansion of starchy foams is dependent on the melt elasticity where higher melt elasticity increased radial expansion and in turn reduced density, while initial bubble growth increases with lower melt viscosity (Willet and Shogren, 2002). Torque values ranged from 23 to 57% of maximum

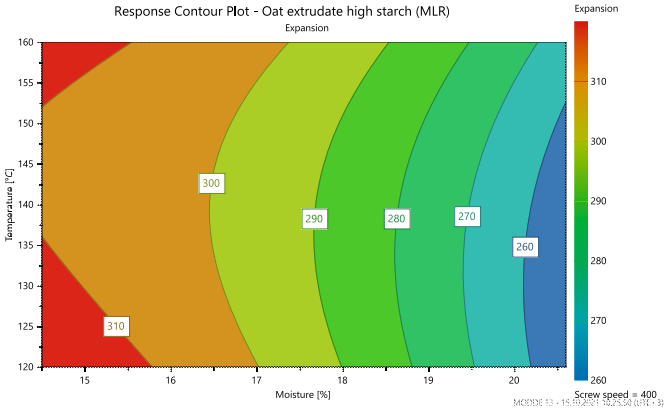

Fig. 1. Effect of moisture and temperature on expansion. R^2 = 0.859, Q^2 = 0.079.

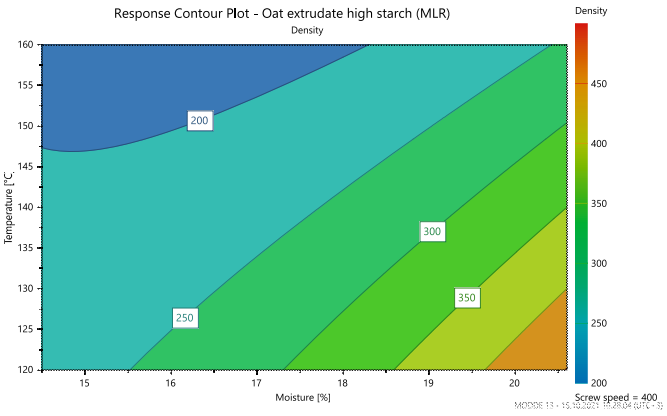

Fig. 2. Effect of moisture and temperature on density R^2 = 0.983, Q^2 = 0.876.

Table 2
Observed torque values during extrusion and porosity, cell wall thickness and pore diameter from X-ray tomography. Letters a-d indicate statistical significance.

Sample	Torque (%)	Porosity (%)	Cell Wall Thickness (μm)	Pore mean diameter (μm)
N1	57	89^{cd}	87^{a}	1107^{ab}
N2	38	80^{ab}	295^{d}	1737^{d}
N3	39	91^{cd}	63^{a}	1267^{abc}
N4	23	85^{bcd}	131^{ab}	1153^{abc}
N5	54	89^{cd}	74^{a}	1062^{a}
N6	29	77^{a}	263^{cd}	1397^{abcd}
N7	50	85^{cde}	199^{bc}	1542^{cd}
N8	27	91^{d}	78^{a}	1317^{abc}
N9	40	87^{cd}	135^{ab}	1394^{abcd}
N10	38	89de	118^{ab}	1541^{cd}
N11	37	87^{cde}	129^{ab}	1492^{cde}

(Table 2). The highest torques were observed at low moisture and temperature (57%, sample N1), whereas both higher temperatures and moistures reduced torque (23%, N4). While viscosity as such was not measured, torque values give an indication of changes in melt viscosity at the different conditions. As the effect of temperature on radial expansion was limited, the reduction in density at higher temperatures is explained by increase in specific length (i.e. longitudinal expansion). Robin et al. (2012) observed a similar effect when extruding whole wheat flour, which similarly to oats is high in DF, i.e. increase in total volumetric expansion due to higher longitudinal expansion at higher temperatures, while higher moisture content reduced volumetric expansion. Higher temperature leads to a reduction in melt viscosity (Della Valle et al., 1997), which favours expansion at the die. However, as discussed by Robin et al. (2012) higher moisture also reduces melt viscosity but may on the other hand increase bubble collapse after expansion.

The changes in micro-structure of extrudates with changing processing parameters can be seen in selected cross-section images obtained from XRT images (Fig. 3) as well as sample photos provided in supplementary files. Porosity ranged from 77% to 91%, cell wall thickness from 63 μm to 295 μm and mean pore diameter from 1062 to 1737 μm, depending on extrusion conditions. In particular, samples with low moisture and high temperature during extrusion (e.g. N3 showed high porosity. XRT results also show that while the difference in radial expansion was limited during extrusion, the microstructure of the extrudates is profoundly different depending on the conditions. Samples with high moisture (e.g. N2 or N6) had thick cell walls and few large air bubbles, while less dense samples (e.g. N3) had many air bubbles with thin cell walls. High moisture content may lead to bubble collapse, due to the lower glass transition temperature at higher moisture, causing

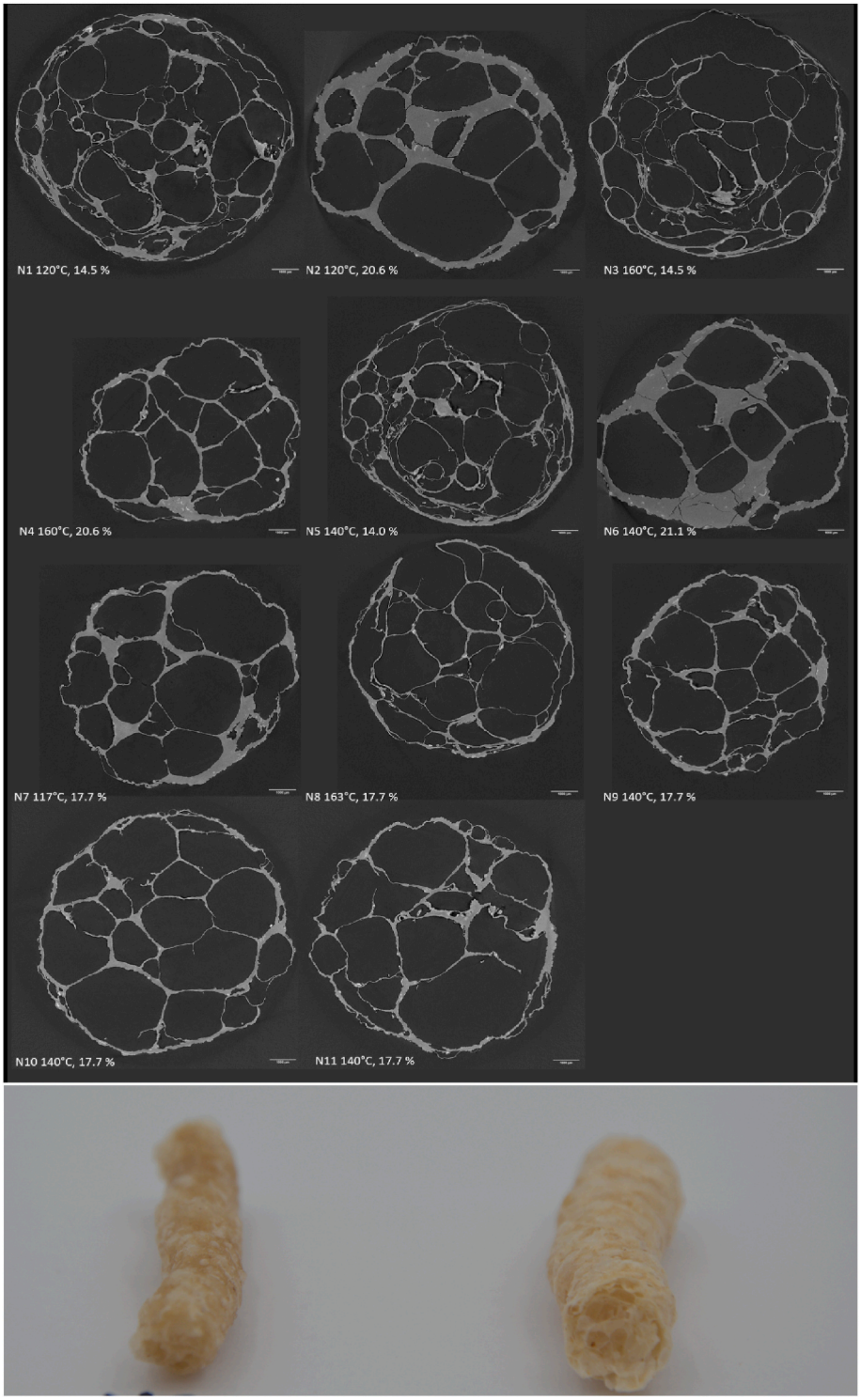

Fig. 3. Above: XRT cross-section images of extrudates with extrusion temperature and moisture. Scale bars indicate 1000 μm. Below: Picture of a sample with low expansion (N2) and high expansion (N3).

thicker cell walls. On the other hand, larger bubbles may be formed due to increased bubble coalescence favoured at high moisture contents (Kristiawan et al., 2016).

Hardness of extrudates varied between 27 and 64 N depending on the extrusion conditions. In previous studies, oat extrudates containing 30% other starch sources had hardness of ca 75 N with added corn flour (Liu et al., 2000) and wheat starch (Yao et al., 2006). The oat flour in this study was chosen based on its good extrusion behaviour in our previous paper on oat extrusion (Nikinmaa et al., 2023). These results highlight the importance of both flour composition and extrusion parameters on the final product during extrusion. The following response contour was achieved for hardness (Eq 3, Fig. 4), with an R^2 of 0.941 and a Q^2 of 0.628, which indicates a good fit and predictivity:

$$\text{Hardness} = 4.13 - 1.06*T + 0.47*MC - 0.20*T^2 + 0.17*MC^2 + 0.34*T*MC$$

Extrusion temperature had the highest effect on hardness, with

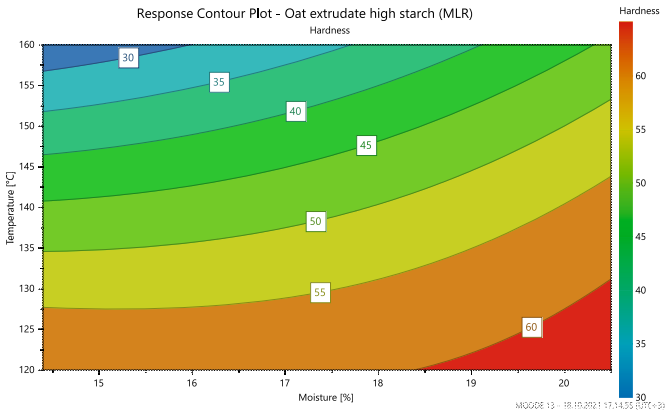

Fig. 4. Effect of temperature and moisture on hardness of extrudates.

higher temperatures leading to lower hardness values. According to state-diagrams, bubble expansion during extrusion can occur at a temperature above the boiling line. Thus, increasing the extrusion temperature can both effect melt rheology as well as pressure build-up and vaporization (at the die exit). Therefore, starchy foams extruded at higher temperatures (T > 120 °C) exhibit coarser cellular structures with larger cell sizes and thicker cell walls, along with reduced cell number density. Both longitudinal and radial expansions contribute to these structural changes which further effects the textural attributes (Kristiawan et al., 2016). In this study the reduction in density (Fig. 2) caused by the increase in temperature – a less dense, more porous structure with thinner cell walls will also break more easily when compressed, thus leading to a lower hardness value.

As with density and expansion, low moisture gave the most favourable hardness results, which has been observed previously when extruded at similar moisture ranges (Yao et al., 2006). As with higher temperature, extrusion at lower moisture also led to reduced density (Fig. 2). Thus, as textural characteristics of the extrudates are a direct result of their structure, it is expected that the conditions that lead to a less dense, more porous structure with thinner cell walls, also decreases the hardness.

3.2. Effect of extrusion on dietary fibers

The effect of extrusion processing condition on oat BG, its extractability, as well as its weight average molecular weight, were specifically evaluated. The determined content of extractable BG (EBG) was between 0.60 and 1.31 g/100 g which is approximately 20–50% of the total BG content in the extrudates (Table 3). While it seems that the temperature during extrusion did not significantly influence the amount of EBG, the moisture content did have a significant impact ($p < 0.001$). The highest BG extraction rate was obtained for extrudates that were produced at a low moisture content (14–14.5 g/100 g), irrespective of the temperature. Our linear model indicates that increasing the moisture content by 1 g/100 g leads to 0.089 g/100 g less extractable BG. There was no combined effect of the two extrusion conditions temperature and moisture on the extractability of BG. Roye et al. (2021) also observed that extruding oat bran at a lower moisture content (17 g/100 g) leads to increased BG extractability as opposed to bran extruded at a higher moisture content (23 and 27 g/100 g). Additionally, in their experiments the BG extractability was also influenced by the last-barrel set temperature, where low temperature (120 °C) led to a higher extractability than high temperature (150 °C). While the content of EBG showed consistent results with acceptable variability (coefficient of variation ca. 5%, indicating that the sampling procedure was reproducible), the results for the Mw distribution of BG remain more challenging to interpret. The weight average molecular weight of BG was around 1000 kg/mol for all samples and did not seem to be influenced by the varying process

Table 3
Extractable β-glucan content (EBG), weight average molecular weight (Mw) of β-glucan and the dietary fiber composition of the extruded samples.

Sample identifier	EBG[a] [g/100 g dwb]	Mw[a] [kg/mol]	IDF[b] (% dwb)	SDFP[c] (% dwb)	SDFS[d] (% dwb)	TDF[e] (% dwb)
N1	1.20 ± 0.10	957 ± 228	3.8	4.4	0.072	8.3
N2	0.60 ± 0.005	958 ± 338	3.7	5.1	0.098	8.9
N3	1.14 ± 0.02	1243 ± 9	3.8	4.8	0.050	8.7
N4	0.73 ± 0.03	1259 ± 39	3.9	3.6	na	na
N5	1.31 ± 0.03	1208 ± 15	4.6	5.6	0.052	10.3
N6	0.64 ± 0.03	1038 ± 192	5.7	4.3	0.047	10.1
N7	0.73 ± 0.01	951 ± 188	7.1	3.3	0.045	10.4
N8	0.77 ± 0.001	1009 ± 162	6.1	5.7	0.048	11.8
N9	0.99 ± 0.02	752 ± 12	4.6	4.3	0.059	8.9
N10	0.76 ± 0.02	1176 ± 268	4.3	4.2	0.044	8.6
N11	0.68 ± 0.02	1615 ± 41	4.5	3.9	0.044	8.4

[a] Values are means (±SD) of triplicate measurements.
[b] Insoluble dietary fiber. Values are means of duplicate measurements.
[c] Soluble dietary fiber that precipitate in 78% ethanol. Values are means of duplicate measurements.
[d] Soluble dietary fiber that remain soluble in 78% ethanol. Values are means of duplicate measurements.
[e] Values are the sum of IDF, SDFP, and SDFS.

parameters (Table 3), which is in line with the results from Tosh et al. (2010) where they observed minimal depolymerization of the BG in the cereal premix when extruded at typical commercial processing conditions (181 °C, 18.7 g/100 g moisture content). Nevertheless, one must keep in mind that the variability of Mw distributions between the analyses of replicate samples were in some cases broad. Therefore, it may be difficult to see a clear effect on and differences between the extrusion conditions on the Mw of BG. This variability could stem from the extraction procedure of BG, or from aggregations and other interactions of the BG chains in solution, as well as the various filter steps in the SEC analysis as is generally seen molecular weight measurements of BG extracts (Mäkelä et al., 2015; Ulmius et al., 2012). As the content of extractable BG was reproducible one can conclude that the variability stems from aggregation of BG in solution rather than extraction.

IDF, SDFP, and SDFS were quantified to evaluate whether different process parameters lead to a shift in the proportions of DF (Table 3). In the SDFS chromatogram of sample N4, no area could be integrated. The ratio between IDF and SDFP was roughly 1 in most samples, except for sample N7 and N10 where it was 2 and 1.5, respectively. SDFS contributed only about 0.5–1g/100 g to the TDF content. No significant effect of the extrusion conditions on the different DF components could be observed.

3.3. Correlation analysis of structure, texture, and DF analysis

Correlation analysis (Fig. 5) showed significant correlation between structural and textural factors, as well as extractability of BG. As in section 3.1, moisture content correlated positively with density, and cell wall thickness, while it correlated negatively with expansion and porosity. Similarly, extrusion temperature correlated negatively with hardness, density and cell wall thickness. BG extractability showed positive correlation with expansion and porosity, and on the other hand correlated negatively with moisture content during extrusion, density

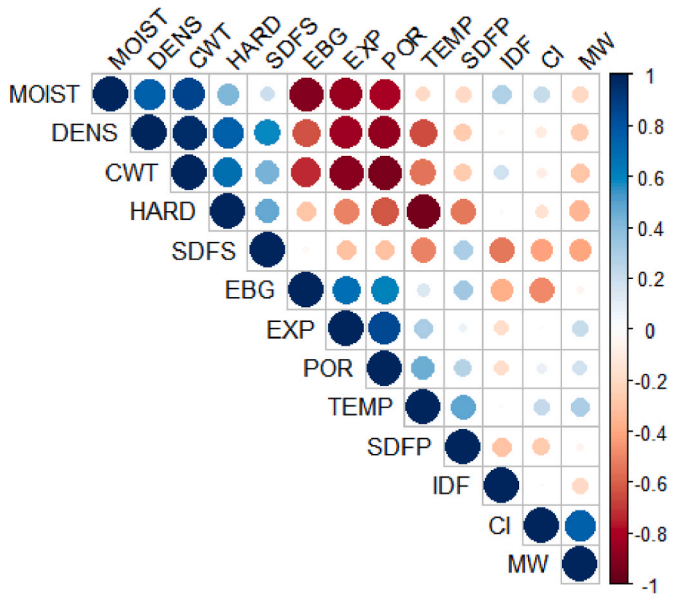

Fig. 5. Correlations between the extrusion conditions, moisture content (MOIST) and temperature (TEMP), and the various characterization measurements of the extrudates: density (DENS), cell wall thickness (CWT), expansion (EXP), porosity (POR), hardness (HARD), crispiness index (CI), extractable β glucan content (BG), soluble DF that stay soluble in 78% ethanol (SDFS), soluble DF that precipitates (SDFP), insoluble dietary fibre (IDF) and molecular weight (MW).

and cell wall thickness. Roye et al. (2021) attributed higher extractability of beta glucan to increased specific mechanical energy (SME) and degradation of BG. Based on our results, it is also likely that the more open structure of the more expanded extrudates improves extractability. In other words, the conditions that favour expansion, lead to a more open structure with thinner cell walls, from which BG is more readily extracted. No clear correlation between extrusion parameters and DF components or Mw were shown. Thus, from a DF perspective, conditions that favour expansion may also have a positive effect on the DF components in oats. On the other hand, increased expansion may lead to higher starch hydrolysis index, which means that more expanded products may cause more rapid glucose response (Alam et al., 2016). On the other hand, Brahma et al. (2016) found that extrusion at low moisture (15 g/100 g, similar to the most expanded samples in the current study) increased content of resistant starch.

3.4. Conclusions

Large differences in structure and texture were achieved by varying extrusion conditions, with low moisture and high temperature leading to more expanded, crispy extrudates. Increased BG extractability was achieved at conditions that favour expansion, while no clear trend was observed for DF content or BG Mw. These results indicate that the health promoting effects of BG or dietary fibre are unlikely to be negatively affected by extrusion. Conversely, the higher extractability of BG may even increase its benefits in the digestive system. The results of this indicate that choosing the extrusion conditions that favour texture may have favourable effects on both palatability, thus potentially increasing the consumption of wholegrain oat extrudates, as well as having health benefits. However, the effect on starch digestibility and thus glucose response is still unclear. Thus, the effect of the extrusion parameters and changes to oat extrudate structure and DF on starch hydrolysis could be a topic for further study.

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CRediT authorship contribution statement

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2023.114972.

References

Alam, S. A., Järvinen, J., Kokkonen, H., Jurvelin, J., Poutanen, K., & Sozer, N. (2016). Factors affecting structural properties and in vitro starch digestibility of extruded starchy foams containing bran. *Journal of Cereal Science, 71*, 190–197. https://doi.org/10.1016/j.jcs.2016.08.018

Brahma, S., Weier, S. A., & Rose, D. J. (2016). Effects of selected extrusion parameters on physicochemical properties and in vitro starch digestibility and β-glucan extractability of whole grain oats. *Journal of Cereal Science, 70*, 85–90. https://doi.org/10.1016/j.jcs.2016.05.001

Brennan, M. A., Monro, J. A., & Brennan, C. S. (2008). Effect of inclusion of soluble and insoluble fibres into extruded breakfast cereal products made with reverse screw configuration. *International Journal of Food Science and Technology, 43*(12), 2278–2288. https://doi.org/10.1111/j.1365-2621.2008.01867.x

Della Valle, G., Vergnes, B., Colonna, P., & Patria, A. (1997). Relations between rheological properties of molten starches and their expansion behaviour in extrusion. *Journal of Food Engineering, 31*(3), 277–295. https://doi.org/10.1016/s0260-8774(96)00080-5

Guo, L., Tong, L. T., Liu, L., Zhong, K., Qiu, J., & Zhou, S. (2014). The cholesterol-lowering effects of oat varieties based on their difference in the composition of proteins and lipids. *Lipids in Health and Disease, 13*(1), 1–10. https://doi.org/10.1186/1476-511X-13-182

Jokinen, I., Pihlava, J., Puganen, A., Sontag-strohm, T., Linderborg, K. M., Holopainen-mantila, U., Hietaniemi, V., & Nordlund, E. (2021). Predicting the properties of industrially produced oat flours by the characteristics of native oat grains or. *Foods, 10*(1552). https://doi.org/10.3390/foods10071552

Kristiawan, M., Chaunier, L., Della Valle, G., Ndiaye, A., & Vergnes, B. (2016). Modeling of starchy melts expansion by extrusion. *Trends in Food Science & Technology, 48*, 13–26. https://doi.org/10.1016/j.tifs.2015.11.004

Lazaridou, A., Biliaderis, C. G., Micha-Screttas, M., & Steele, B. R. (2004). A comparative study on structure-function relations of mixed-linkage (1→3), (1→4) linear β-D-glucans. *Food Hydrocolloids, 18*(5), 837–855. https://doi.org/10.1016/j.foodhyd.2004.01.002

Liu, Y., Hsieh, F., Heymann, H., & Huff, H. E. (2000). Effect of process conditions on the physical and sensory properties of extruded oat-corn puff. *Journal of Food Science, 65*(7), 1253–1259. https://doi.org/10.1111/j.1365-2621.2000.tb10274.x

Mäkeläinen, H., Anttila, H., Sihvonen, J., Hietanen, R.-M., Tahvonen, R., Salminen, E., Mikola, M., & Sontag-Strohm, T. (2007). The effect of β-glucan on the glycemic and insulin index. *European Journal of Clinical Nutrition, 61*, 779–785. https://doi.org/10.1038/sj.ejcn.1602561

Mäkelä, N., Rosa-Sibakov, N., Wang, Y.-J., Mattila, O., Nordlund, E., & Sontag-Strohm, T. (2021). Role of β-glucan content, molecular weight and phytate in the bile acid binding of oat β-glucan. *Food Chemistry, 358*, Article 129917. https://doi-org.libproxy.helsinki.fi/10.1016/j.foodchem.2021.129917.

Mäkelä, N., Sontag-Strohm, T., & Maina, N. H. (2015). The oxidative degradation of barley β-glucan in the presence of ascorbic acid or hydrogen peroxide. *Carbohydrate Polymers, 123*, 390–395. https://doi.org/10.1016/j.carbpol.2015.01.037

Marasca, E., Boulos, S., & Nyström, L. (2020). Bile acid-retention by native and modified oat and barley β-glucan. *Carbohydrate Polymers, 236*(January), Article 116034. https://doi.org/10.1016/j.carbpol.2020.116034

Martínez-Villaluenga, C., & Peñas, E. (2017). Health benefits of oat: Current evidence and molecular mechanisms. *Current Opinion in Food Science, 14*, 26–31. https://doi.org/10.1016/j.cofs.2017.01.004

Moisio, T., Forssell, P., Partanen, R., Damerau, A., & Hill, S. E. (2015). Reorganisation of starch, proteins and lipids in extrusion of oats. *Journal of Cereal Science*. https://doi.org/10.1016/j.jcs.2015.04.001

Nikinmaa, M., Alam, S. A., Raulio, M., Katina, K., Kajala, I., Nordlund, E., & Sozer, N. (2017). Bioprocessing of bran with exopolysaccharide producing microorganisms as a tool to improve expansion and textural properties of extruded cereal foams with high dietary fibre content. *LWT - Food Science and Technology, 77*, 170–177. https://doi.org/10.1016/j.lwt.2016.11.041

Nikinmaa, M., Mustonen, S. A., Huitula, L., Laaksonen, O., Linderborg, K. M., Nordlund, E., & Sozer, N. (2023). Wholegrain oat quality indicators for production of extruded snacks. *LWT - Food Science and Technology, 174*, Article 114457. https://doi.org/10.1016/j.lwt.2023.114457

Rieder, A., Knutsen, S. H., Ulset, A. S. T., Christensen, B. E., Andersson, R., Mikkelson, A., Tuomainen, P., Maina, N., & Ballance, S. (2015). Inter-laboratory evaluation of SEC-post-column calcofluor for determination of the weight-average molar mass of cereal β-glucan. *Carbohydrate Polymers, 124*, 254–264. https://doi.org/10.1016/j.carbpol.2015.02.019

Robin, F., Dubois, C., Pineau, N., Labat, E., Théoduloz, C., & Curti, D. (2012). Process, structure and texture of extruded whole wheat. *Journal of Cereal Science, 56*(2), 358–366. https://doi.org/10.1016/j.jcs.2012.02.014

Roye, C., Van Wayenbergh, E., Henrion, M., De Bondt, Y., Chanvrier, H., King, R., Lamothe, L. M., & Courtin, C. M. (2021). Extrusion-cooking affects oat bran physicochemical and nutrition-related properties and increases its β-glucan extractability. *Journal of Cereal Science, 102*(July). https://doi.org/10.1016/j.jcs.2021.103360

Sandrin, R., Caon, T., Zibetti, A. W., & de Francisco, A. (2018). Effect of extrusion temperature and screw speed on properties of oat and rice flour extrudates. *Journal of the Science of Food and Agriculture, 98*(9), 3427–3436. https://doi.org/10.1002/jsfa.8855

Sandrin, R., Mejía, S. M. V., Caon, T., & de Francisco, A. (2019). Optimization of extrusion process parameters for preparing fiber-rich oat flour. *Journal of Food Process Engineering, 42*(1), 1–10. https://doi.org/10.1111/jfpe.12943

Sibakov, J. K., Kirjoranta, S. J., Alam, S. A., Kokkonen, H., Jurvelin, J. S., Jouppila, K., Poutanen, K. S., & Sozer, N. (2014). Effect of oat bran fractions on extrudates made of defatted oats. *Food and Bioprocess Technology, 8*, 445–458. https://doi.org/10.1007/s11947-014-1425-4

Singh, N., & Smith, A. C. (1997). A comparison of wheat starch, whole wheat meal and oat flour in the extrusion cooking process. *Journal of Food Engineering*. https://doi.org/10.1016/S0260-8774(97)00069-1

Tosh, S. M., Brummer, Y., Miller, S. S., Regand, A., Defelice, C., Duss, R., Wolever, T. M. S., & Wood, P. J. (2010). Processing affects the physicochemical properties of β-glucan in oat bran cereal. *Journal of Agricultural and Food Chemistry, 58*(13), 7723–7730. https://doi.org/10.1021/jf904553u

Ulmius, M., Onning, G., & Nilsson, L. (2012). Solution behavior of barley beta-glucan as studied with asymmetrical flow field-flow fractionation. *Food Hydrocolloids, 26*(1), 175–180. https://doi.org/10.1016/j.foodhyd.2011.05.004

Wang, Y., Harding, S. V., Eck, P., Thandapilly, S. J., Gamel, T. H., Abdel-Aal, E.-S. M., Crow, G. H., Tosh, S. M., Jones, P. J. H., & Ames, N. P. (2016). High-molecular-weight β-glucan decreases serum cholesterol differentially based on the CYP7A1 rs3808607 polymorphism in mildly hypercholesterolemic adults. *The Journal of Nutrition and Disease, 146*(4), 720–727. https://doi.org/10.3945/jn.115.223206

Wang, J., Xia, S., Wang, B., Ali, F., & Li, X. (2019). Effect of twin-screw extrusion on gelatinization characteristics of oat powder. *Journal of Food Process Engineering, 42*(3), 1–9. https://doi.org/10.1111/jfpe.13014

Willett, J. L., Shogren, R. L., Processing and properties of extruded starch/polymer foams. Polymer, 43, 5935-5947. https://doi.org/10.1016/S0032-3861(02)00497-4.

Yao, N., Jannink, J. L., Alavi, S., & White, P. J. (2006). Physical and sensory characteristics of extruded products made from two oat lines with different β-glucan concentrations. *Cereal Chemistry, 83*(6), 692–699. https://doi.org/10.1094/CC-83-0692