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Chemical and rheological characterization of arabinoxylan isolates from rye bran

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Abstract

Background: Rye arabinoxylans (AXs) might be used as baking improvers for gluten-free (GF) bread. However, their extraction process still needs to be improved.

Objective and methods: The aim of this study was to simplify AX extraction of rye bran by varying temperature and pH and evaluate its chemical and rheological properties for application in GF bread.

Results and conclusion: The results demonstrated that higher amounts of AX and impurities were extracted with the increasing pH and temperature. AX yield reached values up to 3.3 g AX/100 g bran. Highest ferulic acid (FA) content (117.27 ± 1.46 mg/100 g) was achieved at the mildest extraction conditions (30 °C and 0.17 M NaOH). A/X ratio of isolates ranged between 0.53 and 0.57 and the gluten content between 81.30 and 216.78 ppm. Rheological measurements revealed typical pseudoplastic behavior of isolates. AX extracted at 30 °C and 0.17 M NaOH showed a slightly higher initial viscosity in comparison with the other isolates but was still inferior to carboxylmethylcellulose (CMC) and hydroxypropyl methylcellulose (HPMC).

Keywords: Arabinoxylan, Alkaline extraction, Gelling properties, Rye bran

Background

In wheat bread, the main structure-forming component which gives the dough its viscoelastic properties and its outstanding baking quality is gluten [1]. Therefore, a significant technological challenge arises when this essential structure-building protein has to be replaced. Gluten-free (GF) bread is usually characterized by a poor nutritional and technological quality, generally displaying low volumes and an elevated staling rate [2, 3]. A combination of hydrocolloids, emulsifiers, and proteins has been proven to enhance GF bread's quality as they are able to imitate the three-dimensional gluten network to some extent. Sourdough fermentation has also been used to improve its textural and sensorial properties [4]. Regardless of technological advances, the ability of baking additives to fully match gluten's performance has not been achieved yet [3]. Differences between qualities of gluten-containing and GF breads still remain. A promising alternative to achieve a gluten-like performance in GF bread is by mimicking a rye bread-like structure. This could be accomplished by adding isolated arabinoxylans (AXs) from rye to a GF bread's formulation. It is known that these hydrocolloids can form stable hemicellulose networks by covalent crosslinking between feruloyl-groups with neighborhood AX polymer chains [5]. Moreover, AXs have also the ability to bind proteins by dehydroferulic acid-tyrosine crosslinks. Since strength of the network is highly dependent on intrinsic AX properties, it is believed that by isolating AX with desired characteristics, the potential to imitate a rye dough-like structure could be achieved.

Arabinoxylans are composed of a highly substituted β(1→4)-linked D-xylopyranosyl backbone with single arabinosyl units as main side groups. Ferulic acid is

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additionally attached to arabinosyl units of the polymer chain via ester linkages [6]. Its amount is important for the crosslinking behavior during baking. Depending on their molecular weight, substitution pattern (i.e., A/X ratio), and degree of physical entanglement (e.g., diferulate crosslinking and covalent ester bonds), they are classified into water-soluble AX (WSAX) and water-insoluble AX (WIAX) [7]. Considering that WSAXs have the ability to bind a large amount of water, retain gas in doughs, and slow down staling rate, the importance of AXs in rye bread baking has been proven. Moreover, the molar mass of AXs is determinant to improve bread quality, as chain length and solubility can significantly influence bread properties [8]. It has to be noted that these statements were derived from results conducted with gluten-containing flours. Thus, a similar behavior cannot be transferred directly to GF flours.

Since limited information on the addition of AXs as baking additives is available in GF products, further investigations should be carried out. A recent study showed that the addition of linseed mucilage containing approximately 85% AX to a GF bread formulation led to a significant increase in volume and porosity. The mucilage did not only improve technological quality of GF bread, but proved to be more acceptable in terms of sensory assessment compared with the control [9]. Moreover, Burešová and Kubínek [10] supposed that positive differences among GF doughs made by several GF flours are probably related to the presence and characteristics of AXs.

One of the most common methods to isolate AXs is through water extraction. As most of the AXs in the grain are crosslinked with other cell-wall components to form a structural network, more severe treatments, such as chemical, enzymatic, or mechanically assisted treatment, are usually applied to increase AX solubilization [11]. In addition, the AX extraction yield highly depends on process parameters, such as temperature, pH, extraction time, type of solvent, and raw material-to-solvent ratio. Earlier investigations reported water extraction yields in the range of 1.9–2.1 g AX/100 g rye flour [12, 13], whereas chemical and enzymatic extractions yielded up to 2.5 g AX/100 g rye bran [14] and 1.08–2.1 g AX/100 g rye flour [15], respectively. For rye, no information about mechanically assisted extractions has been reported yet, in comparison with wheat, where low AX yields were achieved [16].

Overall, elevated extraction yields have almost exclusively been achieved by means of severe, as well as time- and cost-intensive treatments, which consequently lead to the degradation of the extracted compounds [17]. A cost-efficient and environmentally friendly alternative to effectively separate AXs from smaller co-extracted molecules is the use of ultrafiltration. During the separation process, a low molecular cutoff membrane allows smaller molecular weight substances to pass through, while retaining larger molecules. High-purity isolates could therefore be obtained.

Based on a previous study carried out by Mansberger et al. [14], the first aim of this study was to simplify the AX extraction procedure and characterize in more detail the effects of a wider range of temperatures and pH values on the chemical composition of isolated rye AXs. The second aim focused on evaluating the rheological properties of the obtained AX isolates to determine their ability to be used as baking improvers in GF bread.

Methods

Materials

Rye bran flour from Good Mills Austria GmbH (Schwechat, Austria) was used as raw material for AX extraction. For enzymatic treatment, subtilisin (Alcalase 2.4 L), alpha-amylase (Termamyl 120 L), amyloglucosidase (AMG 300 L), and bacillolysin (Neutrase 0.8 L), were purchased from Novozymes Ltd. (Bagsvaerd, Denmark). Corolase®LAP, Corolase®PP, Corolase®7089, Papain®, Veron®P, and Veron Amylofresh® were donated by AB Enzymes GmbH (Darmstadt, Germany). For rheological measurements, hydroxypropyl methylcellulose (HPMC, Methocel™ F450; 320–480 cPs) and carboxymethylcellulose (CMC) E466 were donated by Harke GmbH (Mühlheim an der Ruhr, Germany). All the used reagents were of analytic grade and purchased from Sigma-Aldrich (Steinheim, Germany).

Pilot-scale extraction procedure with temperature and pH variations

Based on a previous study [14], the effects of a broader range of temperatures (up to 70 °C) and pH on the extraction of rye bran AXs were tested. For α-amylase inactivation, the bran (10.8 kg) was dry heated at 130 °C for 90 min in an oven (Memmert GmbH &Co. KG, Schwabach, Germany). Sodium hydroxide was used at two concentrations (0.17 and 0.25 M which correspond to pH of 11.83 and 12.80, respectively) and mixed with rye bran in a 1:10 ratio. The extraction was performed at 30, 50, and 70 °C for 100 min with constant agitation. Afterward, the solids were removed using a horizontal centrifuge decanter (Sharples, Waldkraiburg, Germany), and the pH was reduced to pH 8 using phosphoric acid. The supernatant was preserved with 0.15 g/L of potassium sorbate. Proteases (200 mL Alcalase®; 20 g Corolase® PP and 228 mL Corolase LAP®) were added and stirred at 50 °C for 2 h. Then, the pH was decreased again to 6.5 for 16 h for further enzymatic treatment (17 mL Corolase® 7089, 13 mL Papain®; 15 g Veron Amylofresh®; 15 g Veron®P;

200 mL Neutrase®; 150 mL Thermamyl®; 150 mL AMG®). Enzymes were inactivated by heat treatment at 95 °C for 20 min. Subsequently, the extracted solution was ultra/dia-filtrated using a 8-kDa ceramic membrane (TAMI Industries, Hermsdorf, Germany) at 50 °C, and the remaining solution was then portioned. A representative amount of the extract (20 L) was then freeze dried (FreeZone 6, Fa. Labconco, Outside, USA).

Rheological measurements

Rheological characterization was performed using a Kinexus Rheometer pro+ (KNX 2001, Malvern Instruments GmbH, Herrenberg, Germany) at 25 °C. For comparative means and to avoid broad viscosity ranges, especially for HPMC and CMC, a diluted concentration of 0.01% of hydrocolloid was chosen, which was defined in pre-trials (not presented here). Before measurement, 5 mg of each sample was diluted with 50 ml distilled water and immediately poured on a cone–plate geometry (CP1/60). Flow curves were obtained under continuous shearing over a shear rate ranging from 0.01 to 10 s^{-1}. Measurements were carried out in triplicate, and rheological parameters were evaluated using the manufacturer's supplied computer software (rSpace for Kinexus, Malvern Instruments GmbH, Herrenberg, Germany). Rheological behavior of AXs was described by fitting the experimental data to a power law model:

$$\eta = K\dot{\gamma}^{n-1}$$

where η is apparent viscosity (Pa s), K is the consistency coefficient (Pa s^n), $\dot{\gamma}$ is the shear rate (s^{-1}), and n is flow behavior index.

Gluten quantification

Gluten content of AX isolates was quantified by means of a competitive Enzyme Linked Immunosorbent Assay (ELISA) using the R5 antibody (Ridascreen® Gliadin competitive, R-Biopharm, Darmstadt, Germany) according to the manufacturer's instructions [18]. Gliadin concentrations were calculated based on a (4-PL) nonlinear regression curve-fitting model provided by XLSTAT-Base (Addinsoft, Witzenhausen, Germany) and converted into gluten concentration, by multiplying the gliadin concentration by a factor of two [19].

Analytic methods

AX isolates were analyzed by different methods to evaluate the influence of process conditions on their chemical composition. Dry matter was estimated according to ICC-standard method 110/1 [20]. Protein content was performed by the Bradford assay (Roti-Quant®, Carl Roth GmbH, Karlsruhe, Germany). Soluble and insoluble dietary fibers (SDF and IDF) were determined following the standard method of AACC No. 32-07 [21] (Megazyme test kit, Megazyme International Ireland Ltd., Wicklow, Ireland). Total starch was performed according to the standard method of AACC No. 76-13.01 [21] (Megazyme test kit, Megazyme International Ireland Ltd., Wicklow, Ireland). Extraction of free and bound phenolic compounds of rye bran and AX isolates were analyzed as previously reported by Mattila et al. [22]. The measurement of monosaccharide composition was carried out according to the procedure of Sluiter et al. [23] where hydrolyzation into monosaccharides under the process conditions—72% sulfuric acid (30 min at 30 °C), sterilization (60 min at 121 °C), and centrifugation of the sample—was performed. A HPLC equipped with a refractive index detector (Hitachi LaChrom Elite®, Hitachi Europe GmbH, Düsseldorf, Germany) and a Rezek RPM-Monosaccharide Aminex HPX-87P column (300 × 7.80 mm) attached with ionic form H^+/CO_3 deashing guard column (4 × 3.00 mm) (Phenomenex, Aschaffenburg, Germany) were used. All analyses were performed in triplicate, except for monosaccharide composition and gluten content, which were performed in duplicate. The AX content was calculated as the sum of arabinose and xylose fractions.

Statistical evaluation

Statistical analyses were performed using STATGRAPHICS Centurion XVII, version 17.1.04 (Statpoint Technologies, Inc., Warrenton, USA). Statistical significant differences were determined between the samples using ANOVA (analysis of variance; f-test for multiple samples or two samples with $\alpha = 0.05$); and Fishers least significance tests. A p value <0.05 was considered as statistically significant. Significant differences were indicated by different letters in the rows.

Results and discussion

Influence of temperature and pH on pilot scale AX extraction

As seen in rye dough, covalent crosslinks between AX and proteins, as well as other dietary fiber components contribute to form a hemicellulose network [5]. Since AX may not be the only component to play a significant role in GF dough formation, detailed information about chemical composition of the obtained AX isolates at different extraction conditions was provided (Table 1). In such a way, the purity of AX isolates was also defined. Data on Table 1 show that temperature and pH had significant effects on chemical composition and yield. Overall, all AX isolates had a moderate amount of starch and a high concentration of protein, showing that enzymatic treatment was not able to fully purify AX isolates. Increasing the temperature to 70 °C at a pH of 11.83 increased the extractability of SDF to up to 30.30 g out

Table 1 Chemical composition of AX isolates at different extraction conditions

Extraction conditions	Protein (g/100 g)	Starch (g/100 g)	β-Glucan (g/100 g)	IDF (g/100 g)	SDF (g/100 g)
Effect of temperature					
30 °C–0.17 M	19.70 ± 0.13	9.03 ± 0.52[a]	2.59 ± 0.03	1.41 ± 0.24	22.64 ± 0.34[a]
50 °C–0.17 M	20.77 ± 0.68	15.65 ± 0.51[c]	2.54 ± 0.72	0.84 ± 0.11	26.13 ± 0.11[b]
70 °C–0.17 M	22.26 ± 1.03	12.05 ± 0.15[b]	2.35 ± 0.18	1.19 ± 0.03	30.29 ± 0.01[c]
p value[1]	*0.0815*	*0.0014*	*0.8487*	*0.0749*	*0.0001*
Effect of pH[2]					
30 °C–0.25 M	16.68 ± 0.24	14.69 ± 0.01	2.21 ± 0.06	0.75 ± 0.16	25.24 ± 2.40
p value[3]	*0.0041*	*0.0042*	*0.0136*	*0.0835*	*0.2691*

Mean value of triplicate determinations ± standard deviation

IDF Insoluble dietary fiber; *SDF* Soluble dietary fiber

[1] Comparison of isolates extracted at 30, 50, and 70 °C at a fixed pH (0.17 M NaOH: values associated with different lower case letters at the same pH denote significant differences ($p < 0.05$)

[2] 0.17 M NaOH corresponds to a pH of 11.83, while 0.25 M NaOH corresponds to a pH of 12.80

[3] Comparison of isolates extracted at 0.17 and 0.25 M at a fixed temperature (30 °C)

of 100 g rye bran. Due to the strong alkaline solvent, water-insoluble fibers were also extracted. The high pH has been shown to generate oxyanions and improve solubility [24]. Regardless of the extraction conditions, about 2.2–2.6% of β-glucans were recovered.

A similar outcome could be observed in the investigation of Mansberger et al. [14], where only low temperatures of 20 and 30 °C were tested. Nonetheless, the previous investigation was not able to clearly state the individual effects of pH and temperature on yield and physiology of the AX isolates, as both parameters were simultaneously varied (i.e., 20 °C and 0.25 M NaOH; 30 °C and 0.17 M NaOH). Statistical analyses from the current study revealed that an increase of temperature led to significantly higher starch and SDF yields, and starch was further increased with higher pH. On the contrary, the extraction of protein and β-glucan decreased with the increasing pH. IDF content was not affected by either factor.

Monosaccharide composition of isolates is presented in Table 2. As the temperature increased, higher shares of AXs were obtained (not significant). In comparison with Table 1, all the AX contents of the isolates exceeded the amount of total dietary fiber. This discrepancy was attributed to the complexity of the dietary fiber method, in which precise handling skills of more than 30 steps are required. This consequently caused major fiber losses in contrast to the HPLC-RI method. Among the analyzed sugars, only the glucose content was influenced by the altered process conditions. In all processing conditions except for 70 °C, an increase in temperature as well as in pH resulted in significantly higher concentrations of glucose, which was favored by thermal degradation of starch. In contrast, extraction at 70 °C generated

Table 2 Monosaccharide composition of AX isolates

Extraction conditions	Glucose (g/100 g)	Galactose (g/100 g)	Xylose (g/100 g)	Arabinose (g/100 g)	AX content[1] (g/100 g)	A/X ratio
Effect of temperature						
30 °C– 0.17 M	20.45 ± 3.95[a]	5.11 ± 1.45	18.17 ± 3.33	10.36 ± 2.55	28.52	0.57 ± 0.04
50 °C– 0.17 M	32.21 ± 0.10[b]	4.37 ± 0.42	23.46 ± 0.06	12.52 ± 0.56	35.97	0.53 ± 0.02
70 °C– 0.17 M	23.79 ± 0.82[a]	2.68 ± 0.25	22.56 ± 1.05	12.52 ± 0.67	35.07	0.55 ± 0.01
p value[2]	*0.0314*	*0.1440*	*0.1449*	*0.3959*	*0.2200*	*0.5267*
Effect of pH[3]						
30 °C– 0.25 M	33.19 ± 1.28	3.94 ± 0.89	24.05 ± 0.10	13.54 ± 1.29	37.59	0.56 ± 0.04
p value[4]	*0.0492*	*0.4350*	*0.1298*	*0.2565*	*0.1680*	*1.0000*

Mean value of duplicate determinations ± standard deviation

[1] AX was calculated as the sum of arabinose and xylose fractions

[2] Comparison of isolates extracted at 30, 50, and 70 °C at a fixed pH (0.17 M NaOH): values associated with different lower case letters at the same pH denote significant differences ($p < 0.05$)

[3] 0.17 M NaOH corresponds to a pH of 11.83 while 0.25 M NaOH corresponds to a pH of 12.80

[4] Comparison of isolates extracted at 0.17 and 0.25 M at a fixed temperature (30 °C)

isolates with lowest glucose content. It was assumed that a more extensive depolymerization took place, which was produced by higher solubilization and hydrolysis of the starch. Hence, efficient purification of the isolates was achieved. Galactose, xylose, and arabinose contents were not affected by the process parameters. Similarity in A/X ratios for all isolates was visible, lying between 0.53 and 0.57. In comparison, Hell et al. [17] were able to extract around 30% of xylose-based polysaccharides from wheat bran using 1 M sodium hydroxide. They also reported that alkaline (sodium hydroxide) extraction increased the solubilization of solids, especially glucose (~61%), and favored AXs with small ramifications.

FA is the key component of AX structure, due to its ability to connect one AX to another AX-chain or even to other structural polymers [25]. Therefore, it plays an essential role when forming a hemicellulose network. Bound FA and gluten content of the isolates, as well as the extraction yield, are presented in Table 3. The values for yield were calculated and present an approximate estimation of the real AX extraction yield, since not the whole extracted suspension (which amounts were at least 60 L), but only a portion of the isolates were dried due to the limited capacity of the freeze drier. Compared with the investigation of Mansberger et al. [14], the final yields were much lower because the dead volume of ultra/dia-filtration unit was not considered. At higher temperatures, higher amounts of FA crosslinks were broken down, reducing the amount of bound FA and increasing the concentration of free FA, which was removed during ultra/dia-filtration. Determination of gluten content of the AX isolates showed that in parallel to the high protein content of the isolates, the amount of CD toxic gluten remained above 20 ppm, which is the threshold for gluten-free products.

In comparison with the previous study by Mansberger et al. [14] the AX isolates in this present study showed a greater gluten contamination. These differences were mainly attributed to the used quantification method. In contrast to this study, Mansberger, et al. [14] did not use the competitive ELISA test suitable for hydrolyzed samples. Therefore, an underestimation of the gluten content could have been possible. Moreover, the previous study applied longer purification times, in which more extensive gluten depolymerization could be achieved. Gluten concentration was not affected by pH or temperature, except at 70 °C, where it was significantly lower. An explanation for this phenomenon could be that an increase of temperature led to higher solubilization of gluten from rye bran, but simultaneously higher temperatures also increased gluten hydrolysis which was removed at higher rates during purification.

Most appropriate conditions for the extraction of AX were determined according to AX yield and FA content. Even though the lowest temperature showed isolates with the most favorable FA content, a poor extraction yield was achieved. An opposite behavior could be observed at 70 °C. Because the amount of FA has been well correlated with the degree of molecular weight [12], isolates with high FA concentrations were desired. Therefore, intermediate extraction conditions (50 °C and 0.17 M NaOH) were considered as the best compromise. At these conditions, an approximated yield of 3.13 g out of 100 g bran was possible which was even higher compared with several reported studies [12–14]. However, most of the available AXs still remained bound to other polymers in the rye bran matrix or their high molecular weight inhibited solubilization.

The improved isolation procedure has shown superior economic feasibility than traditional alkaline extraction,

Table 3 Influence of process parameters on extraction yield, bound ferulic acid and gluten content of AX-isolates

Extraction conditions	Ferulic acid(mg/100 g), $n = 3$	Gluten content (mg/kg), $n = 4$	Yield[1]
Effect of temperature			
30 °C–0.17 M	117.26 ± 2.06^{c}	216.20 ± 29.01^{b}	1.18
50 °C–0.17 M	38.20 ± 4.30^{b}	216.78 ± 0.77^{b}	3.13
70 °C–0.17 M	15.23 ± 5.72^{a}	81.30 ± 9.42^{a}	3.3
p value[1]	*0.0003*	*0.0049*	
Effect of pH[2]			
30 °C–0.25 M	N.D.	197.88 ± 1.25	1.7
p value[3]		*0.0733*	

Mean value of duplicate or triplicate determinations ± standard deviation

Approximation of yield is expressed as g AX from 100 g rye bran

N.D. Not determined

[1] Comparison of isolates extracted at 30, 50 and 70 °C at a fixed pH (0.17 M NaOH): values associated with different lower case letters at the same pH denote significant differences ($p < 0.05$)

[2] 0.17 M NaOH corresponds to a pH of 11.83 while 0.25 M NaOH corresponds to a pH of 12.80

[3] Comparison of isolates extracted at 0.17 and 0.25 M at a fixed temperature (30 °C)

as costs have been partially reduced. Although industrial scale isolation is somewhat limited by process safety requirements and equipment costs, a sensitive purification procedure has been adapted in order to avoid expensive alcoholic precipitation of AX and to reduce environmental impact. Even with enzymatic purification treatments, the extraction procedure would still cause lower costs compared with traditional purification by precipitation with organic solvents.

Appearance of AX isolates extracted at different pH can be contemplated in Fig. 1. A darkening effect can be seen by increasing the pH, which can mainly be attributed to caramelization and Maillard reaction of co-extracted glucose and probably dextrins.

Viscosity

Knowledge about rheological behavior of hydrocolloids is essential for providing information for appropriate application. Therefore, steady-state shear properties of AX isolates extracted at different process conditions were examined over a specific (0.1–10 s^{-1}) range of shear rates. Steady-state flow curves of AX solutions in comparison with CMC and HPMC are shown in Fig. 2. All samples showed a typical pseudoplastic or shear-thinning behavior, which was also displayed by the flow behavior index (n) < 1 (Table 4). Viscosity drastically decreased with increasing shear rate. This was mainly caused by the faster disruption of polymer network due to external forces, which exceeded the formation rate of new entanglements within the polymer. Cui and Mazza [26] showed similar shear-thinning flow behavior of AX extracted from flaxseed flour in aqueous solutions above 1% (w/w).

HPMC and CMC displayed highest initial viscosities followed by the AX isolated at 30 °C and 0.17 M NaOH. Considering that mildest extraction conditions allowed extraction of AXs with the highest FA content and thus the greatest molecular weight, a slightly higher initial viscosity could be achieved. At this stage of the investigation, viscosity differences between AX isolates were minor, which suggests that shear-rate dependence of flow behavior was not only influenced by molecular weight, but could also be affected by other properties such as fine structure and conformation of the polymer [27]. Also other polymeric impurities such as starch and proteins have to be considered.

Flow curves of hydrocolloids within the linear region were fitted to the Power-Law model in order to describe the apparent viscosity as a function of shear rate (Table 4). All experimental data fitted well with the model, since R^2 values remained above 0.946. Overall, consistency index (K) did not vary at different extraction temperatures and alkalinity. Flow behavior index (n) was estimated to determine pseudoplasticity. This property decreased as pseudoplastic behavior increased [28]. HPMC and AX extracted at 30 °C and 0.17 M displayed the highest pseudoplastic behaviors.

Buksa et al. [29] studied the rheological properties of crosslinked and hydrolyzed AX samples from rye flour. They reported that the highest consistency index and the lowest flow behavior index were found for crosslinked

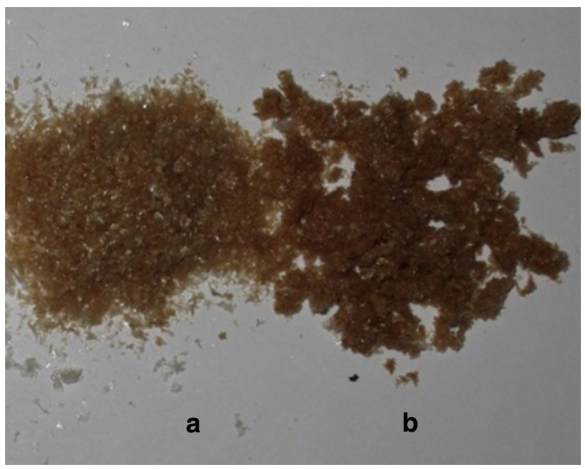

Fig. 1 Appearance of isolated AX at different sodium hydroxide concentrations: **a** 30 °C and 0.17 M NaOH; **b** 30 °C and 0.25 M NaOH

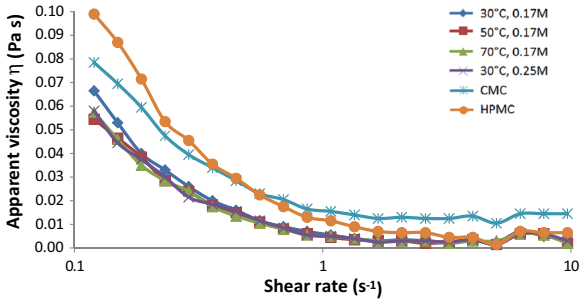

Fig. 2 Shear rate dependence of viscosity for AX isolated at different temperatures and NaOH concentrations in comparison to CMC and HPMC

Table 4 Power law model parameters of AX-solutions prepared at 0.01% in comparison to CMC and HPMC

Hydrocolloid	Model parameters		
	K (Pa s^n)	n	R^2
AX 30 °C, 0.17 M	0.007	0.025	0.996
AX 50 °C, 0.17 M	0.009	0.033	0.977
AX 70 °C, 0.17 M	0.007	0.046	0.995
AX 30 °C, 0.25 M	0.007	0.055	0.946
CMC	0.016	0.266	0.990
HPMC	0.013	0.024	0.991

AX, while the hydrolyzed samples displayed contrary values. These findings would support the results in this investigation. It is believed that AX extracted at the mildest conditions would possess a higher crosslinking ability, since their complex structure would be less damaged. Therefore, further characterization of AX crosslinking ability should be carried out.

One possibility to estimate the degree of crosslinking of AX isolates by rheological methods would be to monitor the viscosity from higher to lower shear rates. The solution would not be able to return to its original viscosity, if crosslinking takes place. Alternatively, gelling ability of AX isolates could also be tested by adding crosslinking enzymes. In this way, functionality between AX isolates would be more visible, as seen by Carvajal-Millan et al. [30] where a dependency between gelling ability and FA content was rheologically confirmed.

Furthermore, future studies should not only focus on optimizing AX extraction yields but also preserve AX functionality. As a next phase, effect of AX isolates in GF bread formulations will be persecuted for improving GF dough stability.

Conclusion

This study could successfully evaluate the influences of various conditions on the AX extraction from rye bran. Among the tested processing conditions, temperature has proven to have a greater influence on the extraction of SDF, which was mainly composed of AXs. On the other hand, the protein solubility was significantly affected by the pH. Overall, high extraction yields could be achieved, even though AX isolates were not fully purified. Since AXs are meant to be used as additives for GF products, gluten detoxification by hydrolysis must be improved before application.

Despite the fact that AX viscosity was still inferior to CMC and HPMC, isolates could have the potential to imitate a rye bread-like structure by the AX crosslinking. The use of higher AX concentrations could provide clearer information about the effect of the extraction conditions on crosslinking properties, although still more research has to be performed in this respect. Hence, knowledge about gelling properties of AX in dough matrices should be the focus of future study.

Abbreviations

A/X ratio: arabinose to xylose ratio; AX: arabinoxylan; CD: celiac disease; CMC: carboxymethylcellulose; FA: ferulic acid; GF: gluten-free; HPMC: hydroxypropyl methylcellulose; WIAX: water-insoluble arabinoxylans; WSAX: water-soluble arabinoxylans.

Authors' contributions

DB performed the statistical analysis of the data and drafted the manuscript. MS carried out AX extractions and chemical characterization of the isolates; SN participated in the AX purification process; RN carried out the rheological characterization of all AX isolates; ST contributed toward the design and coordination of the study; KT performed the monosaccharide compositional analysis and gluten quantification; RS participated in the design of the study and performed the critical revision of the manuscript; SD contributed to the manuscript by way of the interpretation of data and help in drafting the manuscript. All the authors read and approved the final manuscript.

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Competing interests

The authors declare that they have no competing interests.

Availability of data and materials

All data are presented in the MS.

Consent for publication

We confirm that all authors have approved the manuscript for submission.

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