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ARTICLE

Rheofermentometer Fermentation and Breadmaking Characteristics of Dough Containing *xylo*-Oligosaccharide Hydrolyzate from Wheat Bran

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The sugar composition of a *xylo*-oligosaccharide enzymolysis solution (XES) hydrolyzed from wheat bran and bread containing XES were studied. The effects of the XES on fermentation properties of dough and on bread crumb and crust color characteristics are reported. The composition of sugars was determined by high performance liquid chromatography (HPLC), and the fermentation properties of dough and bread color were determined by Rheofermentometer F3 and Chroma Meter CR-400, respectively. *xylo*-Oligosaccharides (XOS) (51.3%) and xylose (39.2%) were the major sugars in the XES. XOS remained in the XES-containing bread after breadmaking processes (xylotriose, xylotetraose, and xylopentaose, 21.1%), with xylopentaose accounting for 14.4%. Maximum dough height (*Hm*) was significantly increased by XES, while the maximum gaseous release height (*Hm*′) was not affected. XES increased the brownness index of the crumb and crust of bread when compared to the that of control bread. Consumer acceptability scores of control and XES containing breads were similar.

KEYWORDS: *xylo*-Oligosaccharides; *xylo*-oligosaccharide enzymolysis solution; HPLC; Rheofermentometer F3; xylose; breadmaking

INTRODUCTION

xylo-Oligosaccharides (XOS) are nondigestible sugar oligomers made up of xylose units. XOS have shown prebiotic activity by favoring the growth of *Bifidobacterium* spp. (*1*, *2*). Considered as food ingredients, XOS show favorable technological features including stability in acidic media, resistance to heat, lower available energy, and significant biological effects at low daily intakes (*3*). XOS are noncariogenic, reduce insulin secretion from the pancreas, and stimulate intestinal mineral absorption. Although the maximum permissible dose depends on individual factors, it has been estimated to be about 0.12 g/kg body weight for male Japanese adults (*3*).

XOS are produced from xylan-containing lignocellulosic materials by chemical methods, i.e., autohydrolysis with water or steam or in media catalyzed with externally added mineral acids, direct enzymatic hydrolysis of a susceptible substrate or a combination of chemical and enzymatic treatments (*4*). Studies can be found in the literature on the manufacture of XOS by enzymatic treatments of a variety of feedstocks, including finger millet (*5*), crop residues (*6*), sugar cane bagasse (*7*), hardwoods (*8*), corncobs (*9*), barley hulls (*10*), brewery spent grains (*11*), almond shells (*12*), corn stover and corn fiber (*13*, *14*), rice hulls (*15*), flax shive (*16*), wheat straw (*17*), and bamboo (*18*). However, the hydrolysates from those processes contain a variety of undesirable components, such as soluble lignin, lignin- and sugar-degradation products, organic acids, and ash. In order to produce food-grade and high purity XOS, the enzymatic hydrolysis or autohydrolysis liquors have to be refined by multistage processing for reaction and fractionation (*19*). This complex problem demands high manufacturing costs which limits the market potential of XOS as nutraceuticals in food applications.

Compared with the procedure of producing purified XOS, the preparation of a *xylo*-oligosaccharide enzymolysis solution (XES) is simple and convenient with lower commercial production costs. *Endo*-Xylanases produce mixtures of *xylo*-oligosaccharides upon the hydrolysis of xylans (arabinoglucuronoxylans and glucuronoxylans) and XOS (*20*). There is a lack of information on the fate of XES in the breadmaking process. The objectives of this study were to study the sugar composition of XES and the effects of the XES on dough fermentation and breadmaking characteristics of white bread.

MATERIALS AND METHODS

Raw Materials. Commercial wheat bran (brand name Jiafeng) and bread flour (brand name Jiafeng) were obtained from Eastocean Oils and Grains Industries (ADM joint venture, Zhangjiagang, China). The bread flour moisture, ash, and protein contents were 12.8, 0.58, and 13.5%

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Table 1. No-Time Dough Formula for Pup Loaf Breads and Flour Farinograph Properties[a]

ingredients	amount
flour	100 g
salt	2 g
shortening	4 g
yeast	1.5 g
water[b]	63.4 mL

[a] Control and *xylo*-oligosaccharide enzymolysis solution (XES) bread. Farinograph parameters: development time, 6.3 min; stability, 8.4 min. [b] Optimum Farinograph water absorption. XES dough (170.9 g) contained 597.2 mg of total sugars of which 306.2 mg was XOS.

(14% mb), respectively, analyzed by Approved Methods 44-15A, 08-01, and 46-12, respectively (*21*). Standards of xylose, xylobiose, maltose, arabinose, glucose, and oat spelts xylan were purchased from Sigma-Aldrich (USA). Xylanase (Multifect CX 12 L, 60,000 U/g) produced by *Trichoderma reesei* Rut-C30 was obtained from Genencor Bio-Products China Co., Ltd., Wuxi. Protease (150,000 U/g) is a proteolytic enzyme preparation produced by *Bacillus subtilis* fermentation and was obtained from Amano enzyme China Ltd., Shanghai. Thermo-stable α-amylase and pullulanase were both obtained from Novozymes China Co., Ltd., Beijing. Thermo-stable α-amylase (Thermamyl 120 L, 120 KNU/g; KNU, kilo novo units α-amylase is the amount of enzyme which breaks down 5.26 g of starch per hour according to Novozyme's standard method for the determination of α-amylase) is a heat-stable α-amylase produced by *Bacillus licheniformis*. The debranching enzyme, pullulanase (Promozyme 400 L, 400PUN/mL; PUN, pullulanase unit novo is the amount of enzyme which, under standard conditions, hydrolyzes pullulan, liberating reducing carbohydrate with reducing power equivalent to 1 μmol glucose per minute) is produced by *Bacillus acidopullulyticus*.

Standard Analyses. Moisture and ash contents were analyzed using Approved Method 44-15A and 08-01, respectively (*21*). Protein content by the Kjeldahl method was analyzed according to Chinese Approved Method GB5511-85 (*22*). Farinograph parameters including optimum water absorption were determined using Approved Method 54-21 (*21*).

Xylanase Assay. Xylanase activity was analyzed with the 3,5-dinitrosalicylic acid (DNS) method (*23*) by determining the amount of reducing sugars released during 5 min in a reaction mixture containing 1% (w/v) oat spelts xylan at 60 °C and pH 5.0. Citrate sodium−citrate acid buffer (100 mM) was used for the experiments using a pH range of 3.0−7.0. One unit (U) of enzyme activity was defined as the amount of enzyme releasing 1 μmol of reducing sugars in 1 min reaction using D-xylose as standard. Xylanase stability and activity measurements were carried out as described earlier (*24*−*27*).

Wheat Bran Fraction (WBF) Preparation. Wheat bran samples were milled to pass through screen 60 (0.30 mm). Starch and protein of milled wheat bran were hydrolyzed by treatments with thermo-stable α-amylase, β-amylase, and protease, respectively, under optimal conditions of each enzyme. Iodine reagent and ninhydrin assays were used to determine the enzyme reaction completion (*28*). The wheat bran fraction (WBF) was rinsed to remove excess enzymes, filtered, dried at 130 °C, and stored at −4 °C until needed for analysis.

Xylanase Treatment of WBF. The hydrolysis of WBF (50 g/L) with xylanase (800 U/g) was performed for 6 h at 60 °C with constant stirring (*29*). The reaction was stopped by heating the suspension at 100 °C for 10 min. Samples were filtered to remove the solids and the enzymolysis solution containing the XOS was retained. The XOS enzymolysis solution (XES) was used for sugar analysis and breadmaking experiments.

Breadmaking Process. Doughs and breads were obtained using 100 g flour pup loaves and optimized straight-dough breadmaking Approved Method 10-10B (*30*). The bread formula and control flour Farinograph parameters are reported in **Table 1**. Farinograph optimum water absorption was used for control and experimental breads containing XES. All the other ingredients were constant. The baking experiments were performed in triplicate.

High Performance Liquid Chromatography (HPLC) Methods for XOS Analysis in XES and XES-Containing Bread. XOS (comprising xylobiose, xylotriose, xylotetraose, and xylopentaose) and xylose analyses in XES and XES-containing bread were based on the methods described previously (*31*−*33*). Briefly, both samples were freeze-dried. Dried samples were ground to pass through a 40-mesh screen and stored in a double-layered plastic bag at 4 °C until needed for extraction. A 40-g powder was added to 200 mL of water, vortexed, placed in a water bath at 85 °C for 2 h, cooled on ice for 20 min, and centrifuged at 13,500 rpm for 5 min. The supernatant was used for HPLC analysis. XOS analyses were determined by HPLC in a model 600 (Waters Corporation, Milford, MA, USA) equipped with a refractive index detector model 2410 Value addition to corncob: Production and characterization of xylooligosaccharides from an alkali pretreated lignin−saccharide complex using *Aspergillus oryzae* MTCC 5154 and a Sugarpak1 column (6.5 mm × 300 mm), 85 °C, pure water as mobile phase (0.4 mL/min), and injection volume of 10 μL. The sugars were expressed in (a) mg/mL of hydrolyzate or mg/g of bread and (b) as percent of the total concentration (percent distribution). The recovery of XOS was analyzed by spiking actual XES samples with xylobiose, glucose, xylose, and arabinose standards. The spiked concentrations used were 1.5, 1.0, 3.0, and 1.0 mg/mL, respectively. The recovery rates (%) were 96.8 ± 2.1; 93.5 ± 1.6; 95.3 ± 1.3; and 96.1 ± 1.6; respectively. Concentration was calculated by comparing the peak area of XOS to that of standards.

Gas Production and Dough Development Parameters. A Rheo-fermentometer F3 (Chopin, Villeneuve-La-Garenne Cedex, France) was used to measure the volume (mL) of CO_2 production and three parameters of dough development: maximum height of dough (*Hm* (mm)), maximum height of gaseous release (*Hm*′ (mm)), and CO_2 production using the method described by Czuchajowska and Pomeranz (*34*). A dough piece (250 g) was placed in a movable basket of the gas meter with a 2000-g cylindrical weight, and the cover of the vat was fitted with an optical sensor. The test was conducted at 30 °C for 3 h. The analyses were done in triplicate.

Bread Crumb and Crust Color Measurements. The crumb and crust color of bread samples was measured using a Chromameter (CR-400, Konica Minolta Holdings Inc., Japan) portable spectrophotometer using the CIE *L*, *a*, *b* system. The bread loaves were cooled for 2 h to reach room temperature (22 ± 1 °C) before color measurement. Under this tristimulus color coordinate system, the *L* value is a measure of lightness, and varies from 0 (black) to 100 (white); the chromaticity coordinates of *a* and *b* values vary for *a* from −100 (green) to +100 (red) and *b* from −100 (blue) to +100 (yellow). Top crust color of the loaf was divided into three regions, while the tristimulus color parameters *L*, *a*, and *b* were determined at each point in duplicate (*35*). The loaf breads were sliced into 1.0 cm, and two center slices were used for crumb color analysis. The brownness index (*BI*) was calculated according to Maskan (*36*) using the following equation:

$$BI = \frac{100 \times [x - 0.31]}{0.17}$$

where

$$x = \frac{(a + 1.75 \times L)}{(5.645 \times L + a - 3.012 \times b)}$$

Sensory Analysis. Sensory analysis was carried out on the bread samples within 3−5 h of baking. The samples served were sliced (1.0 cm thick) and evaluated by 11 nontrained panelists. The panelists were 4 males and 7 females ranging from 22 and 34 years old with a mean of 27 years. Sensory attributes of bread, including appearance, color, flavor, mouth feel, and overall acceptability, were measured using a seven-point hedonic scale with 1, 4, and 7 representing extremely dislike, neither like nor dislike, and extremely like, respectively. Means and standard error of scale scores were reported.

Statistical Analyses. All analyses were performed in triplicate. A general linear model (ANOVA) followed by pairwise comparison using Tukey's method were performed using SAS and *P*-values of 0.05.

RESULTS AND DISCUSSION

Wheat Bran Analyses. The wheat bran moisture, ash, protein, and starch contents were 13.2, 5.13, 14.5, and 18.8%, respectively.

Characterization of Xylanase. The *endo*-1,4-β-xylanase used in this study was from *Trichoderma reesei* Rut-C30. The optimal enzyme activity was at 60 °C and pH 5.0. The activity of the xylanase

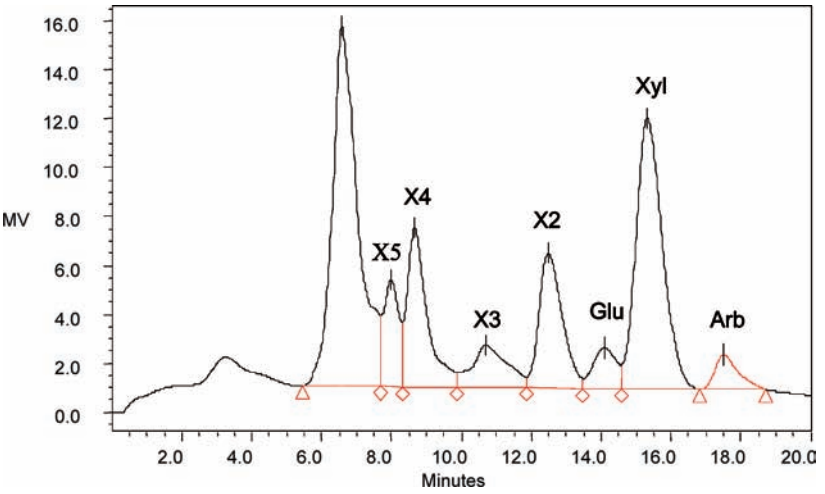

Figure 1. HPLC chromatogram of oligosaccharides of a *xylo*-oligosaccharide enzymolysis solution from wheat bran fraction. Sugarpak1 column, 300 × 6.5 mm i.d.; flow rate, 0.4 mL/min; temperature, 85 °C; pure water as mobile phase; and injection volume, 10 μL. Arb, arabinose; Xyl, xylose; Glu, glucose; X2, xylobiose; X3, xylotriose; X4, xylotetraose; X5, xylopentaose.

Table 2. Retention Time, Relative Area, and Concentration of Sugars from *xylo*-Oligosaccharide Enzymolysis Solution (XES) of Wheat Bran Fraction by HPLC Analysis

sugars	retention time (min)	relative area[a] (%)	concentration[b] (mg/mL)	concentration[b] percentage (%)
xylopentaose	8.0	5.7	0.80 ± 0.03	8.5 ± 0.87
xylotetraose	8.7	11.7	1.65 ± 0.04	17.5 ± 1.33
xylotriose	10.7	5.4	0.77 ± 0.01	8.2 ± 0.64
xylobiose	12.5	11.4	1.61 ± 0.03	17.1 ± 1.21
glucose	14.1	3.4	0.48 ± 0.02	5.1 ± 0.69
xylose	15.3	26.1	3.69 ± 0.02	39.2 ± 0.92
arabinose	17.5	3.0	0.42 ± 0.02	4.5 ± 0.35

[a] Relative area was calculated by comparing to standard solutions. Analysis was performed in triplicate. [b] Mean ± standard error.

was 19,138.8 U/mL (approximately 20,000 U/mL) under optimal conditions. An average of 65% xylanase activity remained after treatment at 55 °C for 30 min at the pH range of 3.0 to 7.0.

Sugars from XES and XES-Containing Bread. XES was the hydrolyzate obtained from the xylanase treatment of WBF. A representative chromatogram of XES analysis is presented in **Figure 1** and the average concentration reported in **Table 2**. The concentration of XOS was equivalent to about 51.3% of the total sugars in the XES with concentrations of 1.61, 0.77, 1.65, and 0.80 mg/mL for xylobiose, xylotriose, xylotetraose, and xylopentaose, respectively. Among the XOS, di- and tetra-oligoxylose appear to be present in similar concentrations: xylobiose (1.61 mg/mL, 17.1%) and xylotetraose (1.65 mg/mL, 17.5%). Xylobiose is of particular interest because of the stimulatory effect on the selective growth of human intestinal bifidobacteria, which are important for the maintenance of healthy intestinal microflora (*37*). Monosaccharides (xylose, glucose, and arabinose) constituted the remaining 48.7% with xylose being the highest concentration (39.2%). Thus, XOS and xylose were the major components of the XES constituting 90.5% of the hydrolyzate. Aachary and Prapulla (*31*) found xylobiose as the major component of the XOS mixture produced by enzymatic conversion of corncob xylan using crude endoxylanase from *A. oryzae* MTCC 5154.

Control bread did not contain XOS (data not shown). Bread containing XES was prepared substituting XES for water using the same Farinograph optimum water absorption as that in the control bread. XES contained 9.42 mg of total sugars per mL of hydrolyzate, of which 4.83 mg/mL of hydrolyzate was XOS. The dough formula of bread containing XES had a total concentration of 179.2 mg XOS/100 g dough. A representative chromatograph of the sugar analysis from bread containing XES is reported in **Figure 2**, and the retention time and concentration of each sugar are listed in **Table 3**. In bread, the main sugars were disaccharides (5.77 mg/mL, 59.3%) followed by xylopentaose and arabinose (1.40 mg/mL, corresponding to 14.4% each). The disaccharide peak represents a mixture of xylobiose and maltose since the retention time of xylobiose and maltose were similar, and they coeluted at 11.7 min (**Figure 2**). The separation of these two components was not pursued in this study. The amount of xylose present in bread was 1.8%, much lower than that found in the XES. This suggests that xylose concentration reduced during fermentation and presumably was consumed by yeast. In contrast, the disaccharide (mainly xylobiose and maltose) content in bread was 42.2% higher compared to that in XES, which were mainly xylobiose. Loveday and Winger (*38*) reported that in a dough containing yeast, sucrose concentration was reduced to 12.9% of the original concentration during the mixing step and declined during the following fermentation step. The authors suggest that the rapid disappearance of sucrose might be due to β-fructosidases already present in yeast cells. Langemeier and Rogers (*39*) suggested that baker's yeast prefers monosaccharides and that maltose increased significantly at the end of fermentation in a straight-dough system.

Fermentation Properties (*Hm*, *Hm′*, and CO_2 Production) of Dough. Dough fermentation properties included *Hm*, which is the height of maximum dough development during fermentation, and *Hm′*, the height of maximum gas formation. The effect of XES on *Hm* and *Hm′* is presented in **Figure 3**. The addition of XES significantly improved *Hm* but did not affect *Hm′*. The dough containing XES had a higher CO_2 production compared to

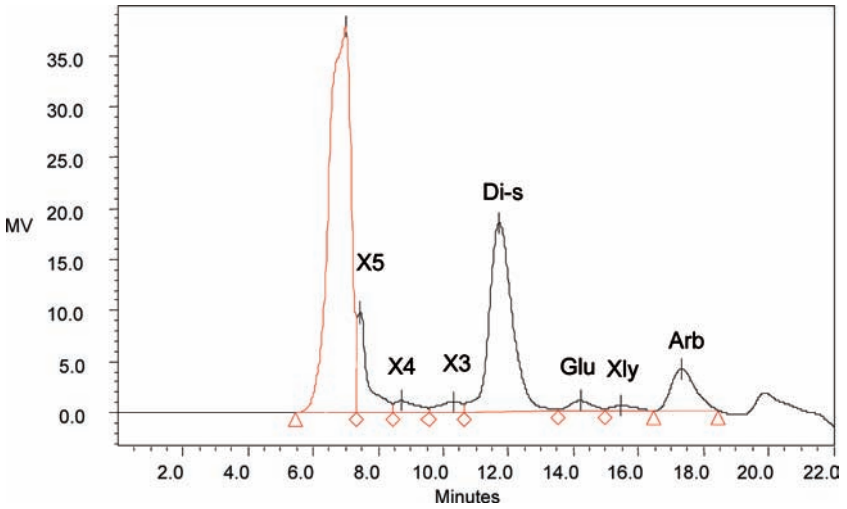

Figure 2. HPLC map of oligosaccharides extracted from bread containing *xylo*-oligosaccharide enzymolysis solution. Conditions are as described in **Figure 1**. Arb, arabinose; Xyl, xylose; Glu, glucose; Di-s, disaccharides; X3, xylotriose; X4, xylotetraose; X5, xylopentaose.

Table 3. Retention Time, Relative Area, and Concentration of Sugars from Bread Containing *xylo*-Oligosaccharide Enzymolysis Solution (XES) of Wheat Bran Fraction by HPLC Analysis

sugars	retention time (min)	relative area[a] (%)	concentration[b] (mg/mL)	concentration[b] percentage (%)
xylopentaose	7.5	6.7	1.40 ± 0.03	14.4 ± 0.92
xylotetraose	8.7	1.6	0.33 ± 0.04	3.4 ± 0.58
xylotriose	10.3	1.5	0.32 ± 0.01	3.3 ± 0.52
disaccharides	11.7	27.7	5.77 ± 0.06	59.3 ± 1.33
glucose	14.2	1.6	0.33 ± 0.01	3.4 ± 0.04
xylose	15.5	0.9	0.18 ± 0.04	1.8 ± 0.52
arabinose	17.3	6.4	1.40 ± 0.01	14.4 ± 1.10

[a] Relative area was calculated by comparing to standard solutions. Analysis was performed in triplicate. [b] Mean ± standard error.

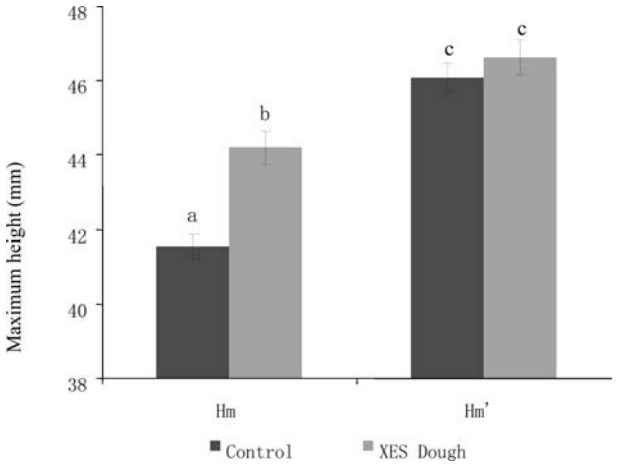

Figure 3. Effect of *xylo*-oligosaccharide enzymolysis solution (XES) from wheat bran fraction on the maximum height of dough (*Hm*) and gaseo us release (*Hm'*) of control and XES dough. Means with a different letter within a group (crumb or crust) are significantly different ($P < 0.05$).

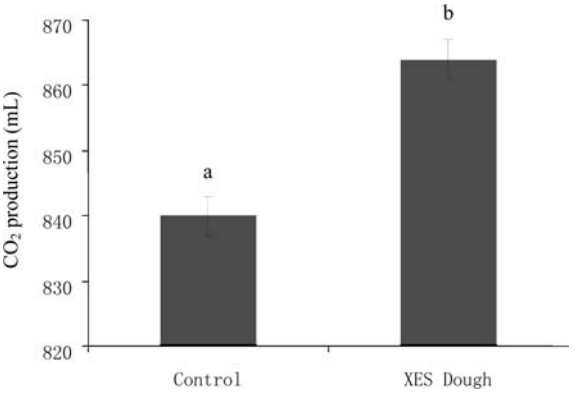

Figure 4. Effect of *xylo*-oligosaccharide enzymolysis solution (XES) from wheat bran fraction on CO_2 production of control and XES dough. Means with a different letter within a group (crumb or crust) are significantly different ($P < 0.05$).

that of the control (**Figure 4**). These results suggested that XES has a pronounced effect on the fermentation properties of dough, such as yeast fermentation and dough development. This could be explained in part by more yeast activity in the presence of XES compared to that in the control.

Bread Crumb and Crust Color Characteristics. The crumb and crust color of bread are important characteristics mostly related to customer's acceptability. The brownness indices (*BI*) for crumb and crust of XES bread were 18.3 and 110.2, respectively, while those of the control bread averaged 13.7 and 61.9, respectively (**Figure 5**). A higher BI corresponds to darker bread color. The results suggested that XES had a significant effect on both the crumb and crust color of bread compared to that in the control.

Sensory Analysis. Sensory analysis of control and XES-containing breads were performed by 11 nontrained panelists using a seven-point hedonic scale. The scores for the XES bread ranged from 5.2 to 5.6 (**Table 4**), while that of the control bread were 4.6 to 5.3. No statistical significant differences were found in appearance, flavor, mouth feel, and overall acceptability. XES bread had higher scores for color than the control (5.64 and 4.55, respectively) suggesting that the effect of XES on color is

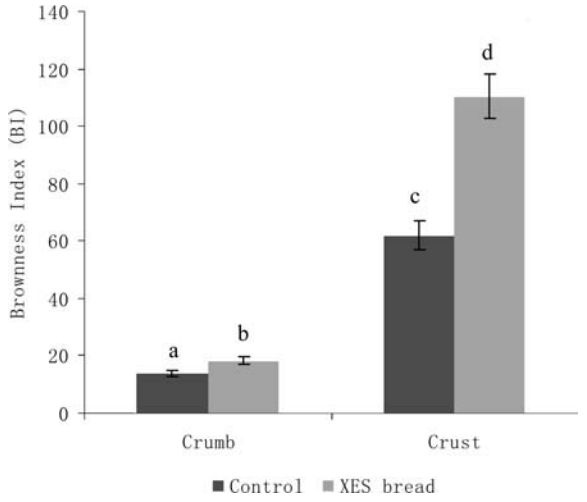

Figure 5. Effect of *xylo*-oligosaccharide enzymolysis solution (XES) from wheat bran fraction on the brownness index (BI) of the bread's crumb and crust. Means with a different letter within a group (crumb or crust) are significantly different ($P < 0.05$).

Table 4. Acceptability Scores of Control and *xylo*-Oligosaccharide Enzymolysis Solution (XES)-Containing Breads[a]

attributes	control bread[b]	XES bread[b]
appearance	5.3 ± 1 a	5.5 ± 1.0 a
color	4.6 ± 0.5 a	5.6 ± 0.7 b
flavor	5.2 ± 0.9 a	5.3 ± 1.0 a
mouth feel	5.1 ± 1.0 a	5.4 ± 0.8 a
overall	5.1 ± 0.8 a	5.218 ± 1.1 a

[a] Seven-point hedonic scale with 1, 4, and 7 representing extremely dislike, neither like nor dislike, and extremely like, respectively. Each value is expressed as mean ± standard error ($n = 11$). Means followed by a different letter within a row are significantly different ($P < 0.05$). [b] Mean ± standard error.

a desirable characteristic, improving its acceptance. This observation agrees with the color differences measured objectively by the brownness index. However, with the brownness index, we are able to estimate a major effect on the crust than in the crumb. The results suggest that substituting water in the bread formula with XES would not affect the acceptability of bread.

In summary, XOS and xylose were the major components of the XES produced by enzymatic hydrolysis of WBF using xylanase from *Trichoderma reesei* Rut-C30. XES-containing bread contains 21.1% of total sugars as XOS, xylotriose, xylotetraose, and xylopentaose, with the latter one accounting for 14.4% of the total sugars. The addition of XES improved the maximum dough height (*Hm*), while the maximum gaseous release height (*Hm*′) remained unchanged. The interest in improving bread products by value added ingredients such as prebiotic XOS will continue to increase. The production of XES and its advantages in bread products justifies more research, i.e., finding the maximum concentration of XOS that is retained after different processes and using different formulas. There is potential for the use of XES in the baking industry.

ABBREVIATIONS USED

XOS, *xylo*-oligosaccharides; XES, *xylo*-oligosaccharide enzymolysis solution; *Hm*, the maximum dough height measured by a Rheofermentometer F3; *Hm*′, the maximum height of gas release measured by a Rheofermentometer F3; WBF, wheat bran fractions; HPLC, high performance liquid chromatography; *BI*, brownness index.

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