Food Chemistry 235 (2017) 175–180

Contents lists available at ScienceDirect

Food Chemistry

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

Short communication

Environmental-friendly technologies for the production of antioxidant xylooligosaccharides from wheat chaff

Mirjana G. Antov *, Tatjana R. Đorđević

University of Novi Sad, Faculty of Technology, Blvd. Cara Lazara 1, 21 000 Novi Sad, Serbia

ARTICLE INFO

Article history:
Received 11 January 2017
Received in revised form 26 April 2017
Accepted 11 May 2017
Available online 12 May 2017

Keywords:
Xylooligosaccharides
Wheat chaff
Antioxidants
Enzymatic hydrolysis
Ultrasound

ABSTRACT

Ultrasound irradiation and enzymatic hydrolysis were applied for the production of antioxidant xylooligosaccharides from wheat chaff. To facilitate enzyme action, 3% (w/w) suspension of raw material was pretreated by ultrasound at 25 Hz, 540 W for 10 min. Ultrasound treatment released xylooligosaccharides with predominant xylotriose into liquid fraction which expressed 1.03 ± 0.01 (μmol AAE/g) ABTS radicals-scavenging activity. Endo-xylanase action on pretreated wheat chaff released 21.76 ± 1.42 and 32.3 ± 0.75 mg reducing sugars equivalents/g after 24 h when applied at dosages 0.15 and 0.3 U/g. respectively. With increase in reaction time portion of xylotriose and xylotetraose in hydrolysates was increase relatively to xylopentose as well as quantities of xylose and glucose. Extremely significant inverse correlation between monosaccharides/oligosaccharides ratio and ABTS radical-scavenging activity was determined. Results indicated potential of environmental friendly ultrasound and enzymatic technologies for the production of xylooligosaccharides from wheat chaff for potential food application.

1. Introduction

Increased research and commercial interest in functional food came as a result of raised awareness of relation between health and nutrition issues particularly considering the effects of some food ingredients on the prevention of diseases. Among them, xylooligosaccharides (XOS) gained considerable attention being nutraceutical that can be produced from lignocellulosic biomass. According to criteria given by Gibson, Probert, Van Loo, Rastall, and Roberfroid (2004) and Cummings and Stephen (2007)) xylooligosaccharides are classified as prebiotics and they possess great prebiotic potential (Aachary & Prapulla, 2011) even stronger than that of dietary fibre because of faster fermentation kinetics (Gullon et al., 2011).

Their prebiotic nature and beneficial effect of human health are expressed through stimulation of growth of health-promoting bacteria in intestinal tract and decrease of number of pathogenic and putrefactive bacteria, then through immunological, anti-inflammatory, anticancerous, antiallergic and antioxidant action, improvement of bowel function and bioavailability of calcium, reduction of blood cholesterol levels, etc. (Aachary & Prapulla, 2011; Azevedo-Carvalho, de Oliva Neto, da Silva, & Pastore, 2013). In addition, xylooligosaccharides have moderate degree of sweetness, stability over wide pH and temperature ranges, inhibitory effects on starch retrogradation and can be used to improve some nutritional and sensory properties of food (Voragen, 1998). These are the reasons for xylooligosaccharides to be regarded as suitable for the incorporation in food to provide or enhance its functional properties and to enable good gut health which can act as guard from pathogenic microorganisms (Samanta, Jayapal, Jayaram, et al., 2015).

Generally, xylooligosaccharides can be produced from xylan using chemical or enzymatic methods both of them having their own advantages and shortcomings. Although cheaper than enzymatic, chemical hydrolysis of xylan may lead to production of monosaccharides and toxic compounds. On the other side, enzymatic hydrolysis is more specific and easier to control, it is conducted under mild conditions not requiring special equipment and can be considered as environmental friendly (Capla, Pandit, & Shah, 2012).

Xylan as substrate for XOS production originates from plant material and when its source is lignocellulosic agro-waste it is beneficial from environmental and economic reasons (Samanta, Jayapal, Jayaram, et al., 2015). However, direct enzymatic hydrolysis of xylan in such materials is difficult because of lignin which presents barrier to enzyme attack so pretreatment is necessary to break down the complex network of polymerization in biomass and facilitate the exposure of more cellulose and hemicelluloses

* Corresponding author.
E-mail addresses: mantov@uns.ac.rs (M.G. Antov), tatjanadjordjevic@uns.ac.rs (T.R. Đorđević).

http://dx.doi.org/10.1016/j.foodchem.2017.05.058
0308-8146/

for enzymatic hydrolysis (Azevedo-Carvalho et al., 2013; Ravindran & Jaiswal, 2016). Among various options for pretreatment of lignocellulosic materials for hemicellulose bioconversion ultrasound technology recently occupied much attention (Kaweeai et al., 2016). Because it reduces chemical loading and reaction time ultrasonic irradiation of lignocellulosic materials are considered as green technology (Busemaker & Yhang, 2013) whose application leads to improved process yield by reducing the structural rigidity, mass-transfer resistance and enzyme consumption.

The aim of this study was to investigate the possibility of producing xylooligosaccharides from wheat chaff by the action of xylanase. Wheat chaff is a dry, scaly protective casing surrounding ripe wheat seed which represent under-utilized lignocellulosic material although it is abundant in regions with intensive wheat production. It is usually used as ruminant feed ingredient or ploughed in soil as waste material. Nevertheless, because of high carbohydrate content (Bledzki, Mamun, & Volk, 2010; Griffin, Nicholson, Mott, Tolan, & Anand, 2011) it can potentially be used as raw material for the production of valuable compounds. Reported studies have been envisaged wheat chaff as material for heavy metal adsorption (Sud, Mahajan, & Kaur, 2008) and as a source of polymers for biocomposites (Mamun & Bledzki, 2014). Recent study revealed potential use of waste liquid streams from wheat chaff pretreatments as a source of xylooligosaccharides with high antioxidant capacity (Đorđević & Antov, 2016).

As an initial step to increase the efficiency of enzyme action wheat chaff pretreatment by ultrasound will be applied. Time course of generation of xylooligosaccharides by endo-xylanase action will be monitored at various enzyme concentrations. Correlation between monosaccharides/oligosaccharides ratio and antioxidant activity of prepared enzyme hydrolysates will be determined.

2. Experimental

2.1. Raw material

Wheat chaff used as raw material in this study was kindly supplied by A.D. "Mlin" (Žabalj, Serbia). Raw material was grounded by kitchen grinder (MKM6000, Bosch) to pass 0.8 mm sieve and stored at −20 °C. Composition of wheat chaff was determined according to NREL Laboratory Analytical Procedure (NREL, 2017). Moisture content of wheat chaff was determined to be 6.6%.

2.2. Ultrasound (US) pretreatment of wheat chaff

In order to prepare material for the forthcoming step of enzymatic hydrolysis wheat chaff was subjected to pretreatment by ultrasonic waves under following conditions – frequency 25 kHz, power 540 W, duty cycle 0.67, duration 10 min, by immersing titanium cylindrical horn of ultrasonic homogenizer (JY96-IIN, Ningbo Scientz Biotechnology) in 3% (w/w) suspension prepared in bi-distilled water. Simultaneously, control was carried out under the same conditions but with no ultrasound. Separation of solid material after experiments with and with no US treatment from liquid fractions was accomplished by filtration through laboratory filter paper (Macherey-Nagel MN 651/120) and obtained filtrate and control filtrate were further analyzed. Ultrasound pretreated wheat chaff was then repeatedly rinsed with bi-distilled water until no reducing sugar was detected in waste flow by DNS method (Miller, 1959).

2.3. Enzymatic hydrolysis

The hydrolysis experiments were performed in 5% (w/w) suspension of pretreated wheat chaff in 0.1 M acetate buffer (pH 5) at 50 °C with mild shaking using endo-xylanase (NS 22083, Novozymes' Cellulosic Ethanol Enzyme Kit). According to manufacturer's data endo-xylanase from NS 22083 is purified and posses high specificity toward soluble pentosans. endo-xylanase activity was determined to be 36 ± 0.6 U/mL according to Bailey, Biely, and Poutanen (1992) with birchwood xylan as substrate. One unit of xylanase activity was defined as the amount of enzyme that liberates 1 mmol of reducing sugars determined by DNS method (Miller, 1959) with xylose as standard per minute under the assay conditions.

Enzymatic hydrolysis was carried out at two enzyme doses – 0.15 U/g or 0.30 U/g calculated on pretreated substrate dry matter, and enzyme hydrolysates were prepared individually for each sampling interval. Simultaneously, control was carried out under the same conditions but with no enzyme. For enzyme inactivation reaction mixtures were heat treated in a boiling water bath for 10 min, then centrifuged at 4500×*g* for 10 min (Sorvall, RC-5B) and obtained supernatant was used for further analysis.

2.4. Sugar analysis

Concentration of reducing sugars in samples - filtrate obtained by US treatment and supernatants after enzymatic hydrolysis, was determined by DNS method (Miller, 1959) with xylose as standard. In addition, sugars in filtrate after pretreatment and in supernatants after enzymatic hydrolysis were analyzed by HPLC system (Waters 1515, US) with Waters 2414 RI detector using Zorbax column in isocratic mode at flow rate of mobile phase 1 mL/min (acetonitrile: water = 60: 40 (v/v)). Analytical standards for glucose and xylose (Supelco, Sigma), xylotriose, xylotetraose and xylopentose (Megazyme, Ireland) were used for sugars analysis. On the base of the results of quantitative analysis, masses of xylose, glucose and xylooligosaccharides were determined (and expressed as mg per g of pretreated material). Using these values monosacchar ides/oligosaccharides (M/O) ratios were calculated for supernatants prepared by enzymatic hydrolysis.

2.5. ABTS radical-scavenging activity assay

The ABTS radical-scavenging activities of samples, filtrate prepared by US pretreatment of wheat chaff and supernatants produced by hydrolysis of pretreated material by endo-xylanase, were determined according to the method of Re et al. (1999) with slight modifications. A stock ABTS radical solution was prepared by mixing ABTS aqueous solution (final concentration 7 mmol/L) with potassium persulphate (final concentration 2.45 mmol/L). This mixture was incubated for 16 h at room temperature in the dark. After incubation, the bi-distilled water was mixed with the stock solution of ABTS until it displayed an absorbance of 0.70 ± 0.02 at 734 nm. Samples were dissolved in phosphate saline buffer (PBS), pH 7.0, to make final concentration 0.5 mg reducing sugar equivalents/mL. A 50 μL sample was mixed with 950 μL of the diluted ABTS radical solution. The solution was mixed on the vortex for 30 s and incubated in a dark environment for 6 min at room temperature. Then, the absorbance was measured at 734 nm. Control solution was prepared using 50 μL of PBS instead of the sample while PBS was used as blank. All determined antioxidant activities were mean values ± standard deviation of at least three measurements and are expressed in μmol of L-ascorbic acid equivalents (AAE) per g of dry matter.

2.6. Fourier transform infrared (FT-IR) spectroscopy

The FTIR absorption spectra from 4000 to 400 cm^{-1} were collected in transmission using a Thermo-Nicolet Nexus 670 FTIR spectrometer (Waltham, MA, USA) at resolution 4 cm^{-1} while