Optimized use of hemicellulose within a biorefinery for processing high value-added xylooligosaccharides

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Wheat straw was used to produce xylooligosaccharides (XOS) for its prebiotic effect. Steam explosion (SE) pre-treatment has been used to separate the main components of lignocellulosic material: cellulose, hemicellulose and lignin. SE conditions used gave a cellulose recovery as solid fraction (91%) and a xylan recovered in the liquid fraction (LF) (36%). Previously to XOS production by enzymatic hydrolysis of xylan, the LF was subjected to a cleaning step with ion exchange resin to remove soluble inhibitors. The commercial enzyme endo-1,4-β-xylanase was used individually and in combination with β-glucosidase to produce compounds with a low degree of polymerization (DP2-DP6). The process achieved a conversion rate of 91% to XOS with 5 h of hydrolysis supplementation, whatever generated 8.9 g of XOS per 100 g of raw material.

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1. Introduction

In 2014, Europe produced approximately 249 million tons of wheat, and Spain produced approximately 6.5 million tons of lignocellulose biomass of wheat straw (FAOSTAT, 2014). The use of this lignocellulosic biomass has been gaining interest from the point of view of a biorefinery. This concept is similar to that of a traditional refinery, where using biomass and several products as biofuels, chemicals and other biomass-derived products can be obtained (Uhllein and Schebek, 2009).

This research is performed within the biorefinery concept and aims to obtain high value added compounds, such as xylooligosaccharides, from wheat straw (WS) with prebiotic activity. Thus, the utilization of the hemicellulosic fraction (xylan) helps to optimize the production costs of biofuels from lignocellulosic biomass.

Xylans are heteropolysaccharides that are composed of a backbone chain of (1 → 4) linked β-D-xylpyranosyl units and α-L-arabinofuranose, α-D-glucopyranosyl uronic acid and acetyl groups as substituents. Xylan chains can be broken down using different methods, such as chemical processing, enzymatic hydrolysis (EH) or a combination of both to produce XOS.

A prebiotic is defined as a non-digestible food ingredient that benefits the host by selectively stimulating the growth and/or activity of one or a limited number of bacteria in the colon, thus improving the host’s health (Gibson and Roberfroid, 1995). Non-digestible oligosaccharides of low molecular weight carbohydrates are considered to be prebiotic. The criteria to determine this limit are not based on physical or chemical properties; thus, oligosaccharides are considered to be carbohydrates with a low molecular weight (Mussatto and Mancilha, 2007). According to IUPAC, oligosaccharides are the polymerized form of saccharide, with 2–10 monosaccharide residues (IUPAC, 1982).

Some examples of prebiotics are inulin, fructooligosaccharides and galactooligosaccharides (Rastall, 2010). Xylooligosaccharides (XOS) are categorized as emerging prebiotics (Patel and Goyal, 2012). Xylooligomers with prebiotic activity from the hemicellulose fraction of the lignocellulosic material (LM) are considered interesting by the food, cosmetic, pharmaceutical and agriculture industries. In pharmaceutical industries, XOS are used because of its biological properties such as anticarcinogenic, prevention and treatment of gastrointestinal infection, ability to decrease glucose levels and cholesterol, immunomodulatory or antimicrobial activity (Patel and Goyal, 2011; Kumar et al., 2012). In cosmeceuticals are used for skin care because of anti-oxidant properties. In agricultural industries, xylooligomers can be used as ripening agents and yield enhancers (Moure et al., 2006). As well, in Japan, XOS have been approved for Food for Specified Health Uses (FOSHU) as ingredient for food to modify gastrointestinal conditions (Mäkeläinen et al., 2009).

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Currently, both in vitro and in vivo studies are underway regarding the prebiotic effect of xylooligosaccharides. For example, a colon simulator was used to study potential prebiotic compounds; the numbers of Bifidobacterium genus were significantly increased in all XOS used (Makelainen et al., 2010). The results of in vivo fermentation assays in the intestine of rats detected slower growth of Bifidobacterium with XOS higher substituted (Van Craeyveld et al., 2008). The addition to diets a mixture of inulin and xylooligosaccharides, in human, resulted in lowering the lipopolysaccharides level in blood plasma (Lecerf et al., 2012).

In this work, the use of agricultural residues (wheat straw) as natural source for obtaining oligosaccharides as alternative to tedious classical synthetic routes (Smoot and Demchenko, 2009) that involves intermediate reactions producing unstable compounds at low yields (Meyers and Werbinsky, 2011) is proposed.

Over the last decade, obtaining XOS from LM has been one of the main targets in the search for high value added compounds. Diverse biomasses have been used to produce XOS, such as eucalyptus (Moura et al., 2008), poplar wood (Chen et al., 2016), rice straw (Moniz et al., 2014), corn cobs (Samanta et al., 2012), wheat straw (Akpinar et al., 2009; Faryar et al., 2015), industrial waste from the melting industry (Gullón et al., 2011), wheat bran (Immerzeel et al., 2014), and sugarcane bagasse (Mandelli et al., 2014).

Lignocellulosic materials are formed mainly by cellulose, hemicellulose, lignin and other components at a low percentage. The composition of wheat straw is between 32 and 40% cellulose, 23–26% hemicellulose (xylan 19–23%), and 18–26% lignin (Linde et al., 2008; Akpinar et al. 2009; Alvira et al., 2013; Kristensen et al., 2008). The composition of structural carbohydrates and lignin from wheat straw may vary according to the climate conditions, fertilized used during culture, mineral contents of the soil or maturity that grain has reached at the time of harvest (Talebnia et al., 2010).

Pre-treatment is necessary to alter the LM structure and divide it into the main components mentioned above, which can be separately processed into a solid fraction (cellulose) for the production of biofuel as well as high value added products, such as XOS from the soluble fraction (hemicellulose). Pre-treatment with steam (Teng et al., 2010), diluted solutions of mineral acids (Kabel et al., 2007), alkalines (Akpinar et al., 2009) or autohydrolysis (Moniz et al., 2014) (Nabarlatz et al., 2007) have been studied.

Numerous studies have been conducted to obtain XOS with various pre-treatment however; the use of SE pre-treatment without a catalyst has the advantage of being a scalable technology compared to other hydrothermal treatments.

In this study, steam explosion was used and is an effective pre-treatment for a herbaceous biomass (wheat straw) to obtain a cellullosic fraction that can lead to a fraction rich in fermentable sugars (Alvira et al., 2016) as well as a liquid fraction (LF) containing hemicellulose (xylan) to produce xylooligosaccharides (Teng et al., 2010) under enzymatic hydrolysis. LF obtained after pre-treatment not only contains sugars but also degradation compounds such as furans, carboxylic acids and phenolic compounds (Ballesteros et al., 2006). These compounds could affect the enzymes involved in the process and should be removed. So, phenolic compounds may act as inhibitors or deactivators of cellulases/hemicellulases (Ximenes et al., 2010). This author observed that phenols were major deactivator of β-glucosidase (Ximenes et al., 2011). As well, kinetics inactivation of a family GH11 endo-xylanase by phenolic compounds has been verified (Boukari et al., 2011).

Oligomeric phenolic have been proved to have a higher inhibitory effect on EH than the monomeric form (Tejirian and Xu, 2011). To remove most of the degradation compounds generated during pre-treatment, different cleaning step have been employed. An ion exchange resin was used in the cleaning process according to the methodology described (Negro et al., 2014). Compounds such as furfural, 5–HMF, carboxylic acids (acetic and formic acids), phenolic compounds and other soluble inhibitors were removed (Kim et al., 2011).

Enzymatic hydrolysis is a recommended method to obtain XOS because it produces selective and controlled hydrolysis and minimizes the generation of compounds that can be harmful at later stages. Additionally, a soft enzymatic condition (pH and temperature) is required. A xylanolytic enzyme system is used to degrade the xylan of the prehydrolysate. This system includes enzymes with different activities, such as endo-xylanase and β-xylanosidase.

To obtain oligomers with a low degree of polymerization the released monosaccharides have to be minimized a purification process has to be convenient to carry out (Qing et al., 2013). Therefore, in the production of XOS with a low degree of polymerization, the presence of β-xylanosidase needs to be avoided or have a very low activity to avoid the production of monomeric xylose.

The objective of this work is to achieve optimum use of wheat straw residue in the biorefinery concept to obtain high value added compounds (XOS) from the liquid fraction using a scalable technology (SE), unlike other pre-treatments. The proposed process can make biofuel production more profitable.

2. Materials and methods

2.1. Materials

Wheat straw has been used as a raw material to produce a liquid rich in sugars. Straw was provided by Ceder–Ciemat (Soria–Spain). The Standard Laboratory Analytical Procedures for biomass analysis, provided by the National Renewable Energies Laboratory (Colorado, USA) (Sluiter et al., 2010), were used to characterize the raw material. Wheat straw had the following composition (% dry weight): cellulose, 38.2 ± 1.7%; hemicellulose, 25.4 ± 0.01% (xylan, 23.7%); lignin, 20.9 ± 1.7%; ash 5.1 ± 0.07% and 10.5 ± 1.3% extractives.

2.2. Pre-treatment

Wheat straw was previously milled using a hammer mill (Fransson HK-AIR26, 75 kW) fitted with an 8 mm grid. Steam explosion was used as the pre-treatment method to extract hemicellulose from wheat straw. SE is a hydrothermal pre-treatment in which the biomass is subjected to high-pressure saturated steam for a period of time ranging from seconds to several minutes before sudden depressurization. This pre-treatment combines mechanical forces and chemical effects because of the hydrolysis of acetyl groups (autohydrolysis) that are present in hemicellulose. The pre-treatment was carried out in a 10 L reactor at 200 °C for 4 min of residence time and 50 s of heating time; cooling down occurred for 2–3 min until reaching approximately 40–60 °C after decompression. These conditions were selected according to several studies on this material developed at CIEMAT where a wide range of temperatures and residence times were tested (Ballesteros et al., 2006). These operating conditions were selected to recover as much cellulose and hemicellulose as possible. The whole slurry was filtered for solid and liquid recovery. Filtration was performed with a laboratory system vacuum (Aspirator Pump, OAKTON, Mod. WP15–1); a nylon mesh of 200 µm was used. The solid fraction (water insoluble solid) containing cellulose (55.6% dry matter) was used to hydrolyse and ferment to ethanol; the liquid fraction containing soluble hemicellulose (18.0% dry matter) was utilized to produce compounds with a high added value, including prebiotic xylooligosomers. After pre-treatment, most of lignin (28.5%) remains together with cellulose in the solid residue. A minor portion of lignin is solubilised and some phenolic and others degradation compounds could be found in the liquid fraction.
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