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Mild-temperature dilute acid pretreatment for integration of first and second generation ethanol processes

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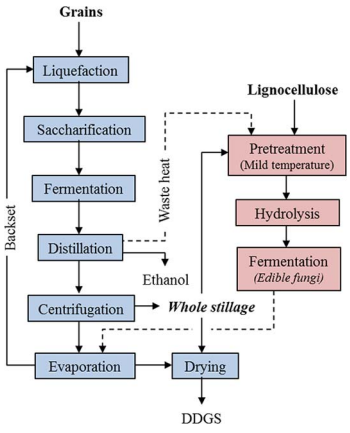
GRAPHICAL ABSTRACT



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ABSTRACT

The use of hot-water (100 °C) from the 1st generation ethanol plants for mild-temperature lignocellulose pretreatment can possibly cut down the operational (energy) cost of 2nd generation ethanol process, in an integrated model. Dilute-sulfuric and -phosphoric acid pretreatment at 100 °C was carried out for wheat bran and whole-stillage fibers. Pretreatment time and acid type influenced the release of sugars from wheat bran, while acid-concentration was found significant for whole-stillage fibers. Pretreatment led up-to 300% improvement in the glucose yield compared to only-enzymatically treated substrates. The pretreated substrates were 191–344% and 115–300% richer in lignin and glucan, respectively. Fermentation using *Neurospora intermedia*, showed 81% and 91% ethanol yields from wheat bran and stillage-fibers, respectively. Sawdust proved to be a highly recalcitrant substrate for mild-temperature pretreatment with only 22% glucose yield. Both wheat bran and whole-stillage are potential substrates for pretreatment using waste heat from the 1st generation process for 2nd generation ethanol.

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1. Introduction

The commercialization of 2nd generation ethanol process using lignocellulosic materials as feedstocks has got several decades of research and industrial attention. One of the major constraints of the second generation ethanol process includes the recalcitrant structure of lignocellulosic materials that requires a relatively harsh initial pretreatment step, entailing high quality steam requirements (usually around 160–260 °C) (Xu and Wang, 2017; Zabed et al., 2017) with or without the addition of chemical catalyst (Kumar et al., 2009; Taherzadeh and Karimi, 2008). The potential of any pretreatment technologies for commercial production is based not only on the total fermentable sugar production, but also on energy consumption (Chen et al., 2017; Zhu and Pan, 2010). Hence, pretreatment is considered as one of the most important and expensive process units in the second generation ethanol process, with a high incidence in the operational cost in the form of energy (heat) (dos Santos Rocha et al., 2017; Vargas et al., 2015). An alternative energy and cost efficient approach of pretreatment is to use the waste heat (ca 100 °C) from the existing first generation ethanol plants to facilitate a mild temperature lignocellulose pretreatment. However, pretreatment at ca 100 °C is little studied until date. Few examples of the reported lignocelluloses pretreatment at temperatures ca 100–130 °C includes, the bisulfite pretreatment of corncob residues for furfural production at 100 °C (Xing et al., 2016); dilute acid pretreatment of corn stover at 120 °C (Hong et al., 2016); and sodium carbonate pretreatment of rice straw within 90–130 °C (Dehghani et al., 2015).

The use of secondary or waste heat at 100 °C could hence facilitate the 'integrated' process (Fig. 1) of lignocellulose to ethanol at the 1st generation ethanol plants (Lennartsson et al., 2014). There are also logistic advantages to integrating first and 2nd generation ethanol production. For example, the logistic network for transporting wheat to the plant could also be used to transport wheat bran. As feedstock for the integrated 1st generation ethanol process another potential candidate is whole stillage. Whole stillage is the saccharified and fermented whole grains after separation of ethanol during the distillation process in the 1st generation ethanol plants, and is thus rich in fibers. It is also already heated up, with a temperature usually around 100 °C (depending on the distillation pressure) and can be sent directly for pretreatment (Fig. 1). Furthermore, part of the required facilities such as evaporators, distillation columns and dryers are already in place for the 1st generation process and could be shared (Fig. 1). The feasibility of a biorefinery that includes both first and second generation process has been described for sugarcane (Dias et al., 2012, 2013; Macrelli et al., 2014, 2012) and corn (Čuček et al. (2011)) based ethanol processes. The ethanol production in an integrated approach, using grains (wheat meal) and wheat straw as the raw materials (Erdei et al., 2010, 2013), has also been reported. However, the high-energy requirement for pretreatment still remains as a bottle-neck in all the integration or biorefinery processes described so far in the literature.

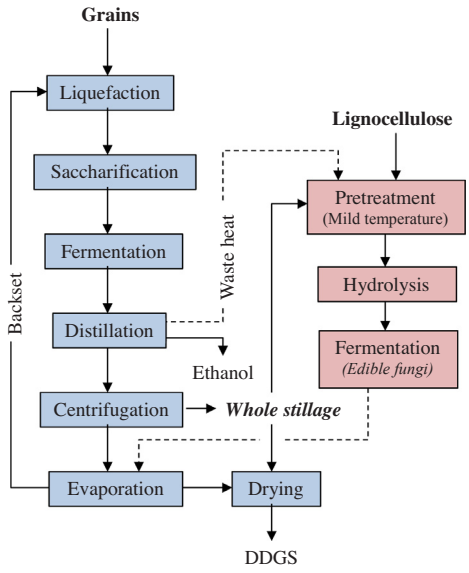

Fig. 1. Proposed integration between a first (left) and a second (right) generation ethanol process utilizing mild temperature (100 °C) dilute acid pretreatment for the lignocellulosic feedstock. Integration between the two processes are marked with dotted lines.

The present study, hence evaluates the use of a mild temperature (100 °C) dilute acid pretreatment, for the integration of lignocellulose to the 1st generation wheat based ethanol facilities. The effect of pretreatment time, acid type and concentration on lignocellulose biomass such as whole stillage fibers, wheat bran and a more recalcitrant material in the form of sawdust, was studied using an experimental design. Furthermore, a pentose utilizing edible filamentous fungal strain, *Neurospora intermedia* was used for fermentation after the pretreatment.

2. Materials and methods

2.1. Lignocellulosic materials

The three different lignocellulosic materials, namely wheat bran (Granngården, Sweden), lignocellulosic fibers of whole stillage (Lantmännen Agroetanol, Sweden) and softwood sawdust (from a local sawmill outside Borås, Sweden) were used in the present study. Lignocellulosic fibers from whole stillage were separated by sieving (1 mm^2 pore size area) and washed thoroughly with distilled water, prior to use. All substrates were dried to constant weight at 45 °C. Due to its size heterogeneity, sawdust was sieved after drying to obtain a homogenous mixture of 2 mm particle size. Pure commercial Avicel was also used to determine the effect of mild temperature dilute acid pretreatment on pure cellulose. Characteristics of the untreated lignocellulosic substrates are shown in Table 1.

2.2. Pretreatment experimental design and statistical analysis

The 2×3^2 full factorial experimental design was developed for the pretreatment using MINITAB® 17 (Minitab Inc., Sate College, PA, USA). Factors examined were type of acid (phosphoric and sulfuric acid), acid concentration (1, 1.5, and 2% (w/w)) and retention time (1, 4.5, and 8 h), where the response variable was yield of released sugars after enzymatic hydrolysis. The selection of factors and the range of variables were based on preliminary experiments conducted (data not shown). Lignocellulosic materials and pure Avicel cellulose at 15% (w/w) solid loading were added to 250 ml screw-cap bottles with a total working volume of 100 g (substrate suspensions in deionized H_2O). Pretreatment of the substrates were carried out at 100 ± 2 °C in a circulating (stirring speed of 125 rpm) water bath (Grant OLS-Aqua pro, UK). After the pretreatment step, the bottles was immediately quenched

Table 1
Composition of untreated substrates used in the study.

Components (%)	Wheat bran	Whole-stillage fiber	Sawdust
Moisture	9.07 ± 0.08	–	51.76 ± 0.62
Ash	5.71 ± 0.07	7.19 ± 0.06	0.27 ± 0.04
Glucan	20.38 ± 1.66	13.12 ± 0.21	31.66 ± 0.71
Xylan	15.61 ± 1.95	18.90 ± 0.81	5.06 ± 0.91
Arabinan	8.78 ± 1.46	12.69 ± 1.42	–
Mannan	–	–	10.28 ± 0.20
Galactan	–	–	1.46 ± 0.22
Crude protein	14.68 ± 0.10	30.16 ± 0.20	–
Starch	17.00 ± 0.16	–	–
Lignin	10.46 ± 0.10	18.17 ± 0.33	28.30 ± 0.04