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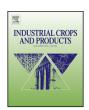
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Bench scale steam explosion pretreatment of acid impregnated elephant grass biomass and its impacts on biomass composition, structure and hydrolysis

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#### ABSTRACT

In the present study, an acid mediated steam explosion process was evaluated for pretreatment of elephant grass biomass in a bench scale reactor. Different combinations of  $H_2SO_4$  concentration, reaction time and temperature (leading to different values of combined severity factor – CSF) were used for biomass pretreatment, and the impact of the resultant pretreatment conditions on biomass composition, structure and enzymatic hydrolysis was assessed. The optimal pretreatment conditions consisted in performing the steam explosion at 161 °C during 11.5 min, and using the biomass impregnated with 0.5%  $H_2SO_4$  (CSF = 1.42). Under these conditions, a pretreated solid containing 52.13% (w/w) cellulose and 31.00% (w/w) lignin was obtained. The modifications in the biomass structure resulting from the different pretreatment conditions were evaluated by scanning electron microscopy, thermogravimetric analysis and nuclear magnetic resonance spectroscopy analysis. Enzymatic hydrolysis of the pretreated material resulted in a cellulose saccharification yield of 55%. These results allow a better understanding about the pretreatment and enzymatic saccharification of elephant grass biomass and also reveal a great potential of this raw material for use in a biorefinery.

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#### 1. Introduction

There is a worldwide interest in changing the current unsustainable economy based on fossil fuels to a more sustainable economy based on renewable biomass in order to ensure a sustainable low-carbon economy for the future with potential environmental, economic, and social benefits (Mussatto, 2016a). In this sense, lignocellulosic biomass including forest and agricultural residues, municipal wastes and food industry leftovers have attracted attention as alternative renewable feedstocks (Kataria et al., 2013).

Pretreatment of lignocellulosic biomass is one of the key steps in the overall process for biomass conversion since it promotes a deconstruction of the plant cell wall structure causing lignin

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removal and resulting in a cellulosic polymer more exposed to the action of enzymes in the subsequent stage of enzymatic saccharification (Mussatto and Dragone, 2016). Various technologies including thermochemical (using acid or alkali), organosolv, liquid hot water, steam explosion, ammonia fiber explosion and biological processes are available for biomass pretreatment (Mussatto, 2016b). Among these options, steam explosion is one of the most promising technologies for use in a biorefinery since it is already used on commercial scale and it has been demonstrated to be effective for the removal of lignin and hemicellulose from different feedstocks, improving the cellulose digestibility. In a steam explosion process, high pressure saturated steam (0.69-4.83 MPa) and temperature (160-260 °C) are used to treat wet biomass during a short period of time (from seconds to few minutes) and then the pressure is suddenly dropped to atmospheric pressure. This sudden drop in pressure disrupts the cell wall structure releasing hemicellulose and lignin and promoting a major exposure of the cellulose fibers, which favors the enzymatic digestibility and glucose yield during hydrolysis (Duque et al., 2016). How-

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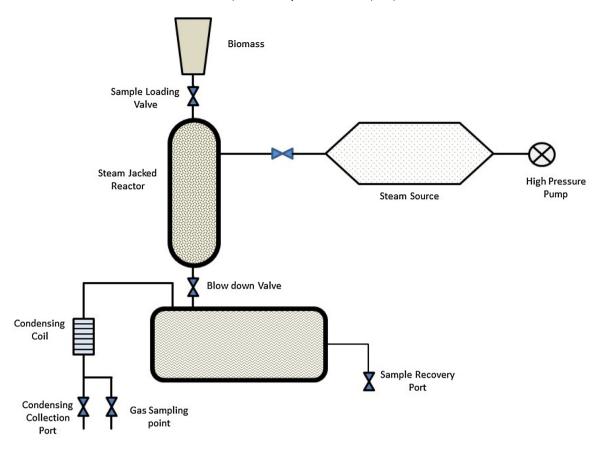


Fig. 1. Schematic representation of the steam explosion equipment used for pretreatment of elephant grass biomass.

ever, there is not a pretreatment condition suitable for all types of biomass and studies are needed to optimize the process conditions to each lignocellulosic feedstock. Biomass can also be soaked with different chemicals (acid or base) before steam explosion pretreatment in order to improve the delignification (Duque et al., 2016).

Several feedstocks have been investigated for steam explosion pretreatment including wheat straw (Zhang et al., 2008), Salix (Diop et al., 2015), corn stalk (Sun et al., 2015), orange peel (Wang et al., 2015), among others. Elephant grass (Miscanthus giganteus) has been more recently introduced as an energy crop and it can be an interesting alternative to other lignocellulosic materials as it does not need much maintenance, can be cultivated in diverse environmental conditions and presents high productivity (30 Tons/hectare) (Somerville et al., 2010). Elephant grass is a perennial grass of subtropical origin, which can grow up to three meter high and is commonly found in The Netherlands and in Western Europe. High carbohydrate content, low lignin content and low cultivation costs make this grass a good candidate for use in a biorefinery.

In the present study, elephant grass was pretreated in a bench scale steam explosion reactor and different conditions of  $\rm H_2SO_4$  concentration, reaction time and temperature were evaluated in order to select the best ones for use on the pretreatment of this feed-stock. The effect of pretreatment on cellulose and lignin contents of the resulting samples was assessed. Biomass structural changes due to the pretreatment were evaluated by scanning electron microscopy, thermogravimetric analysis, and nuclear magnetic resonance spectroscopy. Finally, the enzymatic saccharification of the pretreated sample was assessed.

#### 2. Material and methods

#### 2.1. Feedstock

Elephant grass (*Miscanthus giganteus*) was supplied by NNRGY Crops (The Netherlands). The biomass had an average particle size of 2–3 cm.

#### 2.2. Steam explosion pretreatment

A bench scale reactor installed at the Bioprocess Pilot Facility (BPF, Delft) was used for the pretreatment reactions (Fig. 1). The complete experimental equipment was composed of a 40-L reactor (25-L working volume) able to operate at high temperature and pressure using saturated steam (30 bar), a steam generator, a cyclone of about 15001, a control panel to control the runs, and a container to collect the pretreated biomass.

Before the steam explosion pretreatment, 3 kg of biomass were soaked with 30 kg of  $\rm H_2SO_4$  solution (at 0.5, 2.5 or 4.5 wt%) at room temperature, overnight. Then, the biomass was separated by filtration and its moisture content was determined (28–32% w/w). The soaked biomass was added to the pretreatment reactor and the desired conditions of temperature and time were set to pretreat the biomass with high pressure steam. Once the set time lapsed, a sudden reduction of the pressure was promoted and the pretreated biomass was transferred to the cyclone. Thereafter the pretreated biomass was washed with tap water until the pH of the washing liquid was approx. 5–6, and dried overnight in a ventilation dryer at 70 °C. The dried biomass was stored at room temperature in air tight plastic buckets for further analysis.

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