



Suitability assessment of a continuous process combining thermo-mechano-chemical and bio-catalytic action in a single pilot-scale twin-screw extruder for six different biomass sources



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HIGHLIGHTS

- Adaptation of lignocellulosic biomass deconstruction process on pilot scale extruder.
- Combination of alkali pretreatment and biocatalytic action in extruder.
- Validated of the process on six different lignocellulosic biomasses.

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ABSTRACT

A process has been validated for the deconstruction of lignocellulose on a pilot scale installation using six types of biomass selected for their sustainability, accessibility, worldwide availability, and differences of chemical composition and physical structure. The process combines thermo-mechano-chemical and bio-catalytic action in a single twin-screw extruder. Three treatment phases were sequentially performed: an alkaline pretreatment, a neutralization step coupled with an extraction–separation phase and a bioextrusion treatment. Alkaline pretreatment destructured the wall polymers after just a few minutes and allowed the initial extraction of 18–54% of the hemicelluloses and 9–41% of the lignin. The bioextrusion step induced the start of enzymatic hydrolysis and increased the proportion of soluble organic matter. Extension of saccharification for 24 h at high consistency (20%) and without the addition of new enzyme resulted in the production of 39–84% of the potential glucose.

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1. Introduction

The production of both new feedstock and clean energy from biomass is faced with the problem of whether to promote food or energy. In the context of sustainable development and environmental protection, it is essential to turn to nonedible feedstocks such as agricultural residue, forest residue, industrial waste, dedicated energy crops and municipal solid waste (Ragauskas et al., 2006). Cellulose, hemicelluloses and lignin in the plant cell wall are tightly associated. Classical production processes to generate monomers of interest, including enzymatic hydrolysis and fermentation, work poorly on the lignocellulosic biomass. The conversion of renewable biomass to fuels and chemicals requires the

deconstruction of lignocellulose assembly (Himmel et al., 2007). Increasing the efficiency of lignocellulosic conversion to sugars presents a major challenge. There are many research groups working on new processes for obtaining fuels from biomass and several reviews on developing pretreatment strategies have been published (Mood et al., 2013; Singh et al., 2014; Ravindran and Jaiswal, 2015).

The types of biomass to be used depends on the desired area of application. In the field of energy, these are sustainable, accessible and available materials which are rich in cellulose. The most studied biomass for the production of energy has been sugarcane bagasse (SCB), of which the estimated annual dry mass production was ~279 million metric tons (MMT) in 2011 (bagasse and leaves) (Chandel et al., 2012). Cardona et al. published a review in 2010 on various pretreatment processes to produce bioethanol using this material and proposed future strategies (Cardona et al., 2010). SCB has since been used as a model to develop the important steps

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of the process to produce bioethanol. Rocha et al. (2012) studied the efficiency of a pretreatment process combining steam explosion and alkaline delignification reactions. Zhu et al. (2012) pretreated SCB with $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2$ and ionic liquid resulting in efficient hydrolysis and bioethanol production. Maryana et al. (2014) evaluated the impact of alkaline pretreatment on the chemical composition and structure of SCB. Sambusiti et al. (2015) studied the effect of different milling methods on the physicochemical composition, enzymatic hydrolysis, bioethanol production and energy efficiency of the process.

Oil palm empty fruit bunches (OPEFB) is another biomass feedstock that has been extensively studied for potential second generation bioethanol production, in part because of its availability (23.4 million tons produced in Indonesia in 2011: Millati et al., 2014). It has been subjected to most conventional pretreatments: AFEX (Lau et al., 2010), Acid hydrolysis (Millati et al., 2011), alkaline pretreatment (Han et al., 2011), and aqueous ammonia pretreatment (Jung et al., 2011). More recently, Sudiyani et al. (2013) have obtained promising results using an integrated process that included alkaline pretreatment at the pilot scale. Chiesa and Gnansounou (2014) compared dilute acid and dilute alkali pretreatment and showed that dilute alkali pretreatment performed poorly due to the significant lignin content of the OPEFB. Kristiani et al. (2015) studied the effect of combining alkaline treatment with an irradiation pretreatment process using an electron beam machine. This combination affected the structure of OPEFB by decreasing the lignin content and changing the crystallinity index.

Wood residues are another potential energy source. Eucalyptus globulus is one of the most commercially important hardwood species. In 2004, there were about 2.5 million hectares planted worldwide (Catry et al., 2013). Eucalyptus production and processing generates a large amount of wood residues, such as sawmill residues or bark and branches currently left in the field. The residues can reach 30% of the total harvested biomass (15–25 ton/ha/year) Lima et al. (2013). Eucalyptus wood and bark are harder and denser than grass or cereal biomass, and are resistant to microbial and enzymatic action as a result of their higher lignin content. Zhu and Pan (2010) has presented a comprehensive discussion of the key technical issues of woody biomass pretreatment including: dilute acid, acid-catalyzed steam explosion, organosolv, sulfite, and alkaline pretreatment. Work on eucalyptus is ongoing to improve the enzymatic accessibility of the biomass such as: hydrothermal, dilute acid, and alkaline pretreatment (de Carvalho et al., 2015), hydrothermal microwaves using acidic ionic liquid as catalyst (Xu et al., 2015), and steam explosion processing (Romaní et al., 2013).

Other less studied sources of biomass can serve as a potential energy source. Among these is vineyard pruning (VP). Pruning of grape trees generates high quantities of lignocellulosic biomass (Velazquez-Martí et al., 2011): about 21 million tons of pruning waste are produced each year (Argun and Onaran, 2015). Buratti et al. (2014) pretreated it using steam explosion to produce ethanol and Argun and Onaran (2015) studied its delignification using alkaline peroxide.

Agave bagasse is a residue that accumulates during the production of alcoholic beverages from plants of the agavaceae family. It offers a potential sustainable resource that was estimated to be produced at a rate of around 360 thousand dry tons per year (Caspeta et al., 2014). Nguyen (2014) deposited a patent on a process for producing ethanol using acid-catalyzed steam pretreatment of agave bagasse. Perez-Pimienta et al. (2015) thoroughly characterized agave bagasse following ionic liquid pretreatment.

Sweet corn residue is another potential source of biomass for energy production. The worldwide production of sweet corn was about 2.9 million tons of grain in 2012 (Hansen, 2013). The produc-

tion of sweet corn residue can be estimated to be approximately 6 million tons per year because its weight is twice that of the grain. This biomass is mostly used for forage for ruminant animals and has been little studied for energy production. Its use as a source of sugar for energy production has been tested in previous studies using alkaline pretreatment followed by bio-catalytic hydrolysis in a twin screw extruder (Vandebossche et al., 2014b, 2015).

Increasing the solid concentration of biomass, and thereby decreasing the volume to be treated, could improve the conversion process and lower the costs. However, this increases the viscosity of biomass slurries, making mixing and conveying operations more difficult. Among the processes used to carry out pretreatment with a minimum number of steps, twin-screw extrusion technology offers many advantages and permits working with high solid concentrations. It produces a high shear, rapid heat transfer, and effective and rapid mixing in a continuous operation, with good adjustability of the treatment steps.

Twin-screw extrusion can be used to pretreat different types of biomass for the production of sugars. Several authors have reported this type of application (Vandebossche et al., 2014a; Zheng and Rehmann, 2014). Karunanithy and Muthukumarappan (2013) provide an overview of the combination of extrusion with alkaline pretreatment, including the factors that influence extruder and feedstock parameters and an evaluation of the pretreatment efficiency. A continuous process combining alkaline thermo-mechano-chemical pretreatment, followed by the injection of enzymes into the twin-screw extruder, called “bioextrusion” was developed and tested on different biomass sources such as sweet corn residue (SC), blue agave bagasse, OPEFB as a residue from palm oil manufacture, and barley straw (Vandebossche et al., 2014b). This new process results in excellent mixing of the enzymes with the pretreated biomass at high concentrations, and allows saccharification to begin during bioextrusion (Vandebossche et al., 2015).

This continuous process had been developed and tested at the pilot scale in a single extrusion in this study using different types of biomass.

2. Materials and methods

2.1. Material

2.1.1. Feedstocks

Dehydrated sweet corn (*Zea mays* L. *saccharata*) co-products (SCC) were obtained from industrial corn grain canneries and were provided by SARL Soupro+ (Castelmoron sur Lot, France). They were milled using a hammer mill fitted with a 6 mm grid.

SCB came from Brazil and was provided by EMBRAPA. They were milled using a hammer mill fitted with a 6 mm grid.

Sawdust of *Eucalyptus grandis* (SE) came from Uruguay and were provided by the Instituto Nacional de Investigación Agropecuaria (INIA). They were milled using a hammer mill fitted with a 6 mm grid.

VP came from Chile and were provided by the Instituto Nacional de Investigación Agropecuaria (INIA). They were milled using a hammer mill fitted with a 6 mm grid.

Blue agave (*Agave tequilana*) bagasse (BAB) is the fiber residue from the manufacture of Tequila. It was air dried, and kindly provided by the PATRON Spirits Company in Mexico (Atotonilco, State of Jalisco). It was milled using a hammer mill fitted with a 2 mm grid.

Oil palm (*Elaeis guineensis* Jasq.) empty fruit bunch (OPEFB) is the bunch residue after separation of the fruits for the manufacture of palm oil. It was air dried before being sent from Costa Rica (Palma Tica grupo numar), and was milled using a hammer mill fitted with a 2 mm grid.

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