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Research paper

Steam explosion of sweet sorghum stems: Optimisation of the production of sugars by response surface methodology combined with the severity factor

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ABSTRACT

The purpose of this work is to determine the best conditions allowing the optimal recovery of the first and second generation sugars found in sweet sorghum. This optimization was performed using the severity factor, combined with response surface methodology (RSM) and based on a two-variable central composite design. Dry sorghum was first impregnated with water and then submitted to steam processes. Sugars were recovered in solution, both after the impregnation step and after the steam treatments. A central composite design and response surface methodology was applied to analyze the effects of the reaction time (2–8 min) and the temperature (200–220 °C) on the mass loss and sugar yields. Mass loss and different sugar yields were also correlated to the severity factor and compared to the RSM analysis. Similarly, the response surfaces and the isoresponse curves from the experimental design allowed understanding the mass loss as well as understanding several sugar yields according to the temperature and reaction time. The best operating conditions (i.e. for a severity factor of 3.6 or 2 min at 215 °C) allowed recovery of 37.3 wt% total sugars based on dry sorghum.

1. Introduction

First generation biofuels, especially bioethanol, is based on sugars and starch fermentations using different feedstocks, which in turn depend on their availability in specific areas worldwide. Nowadays, maize and sugarcane are the two major feedstocks used for first generation ethanol production in the Americas (Oliveira et al., 2013; Solomon et al., 2007). First generation ethanol is currently the biofuel with the biggest production volume on a global scale. However, alternative crops such as sugar beet, wheat, grain sorghum, rye or triticale can also be used for first generation ethanol production (Ananda et al., 2011; Naik et al., 2010). The concerns about first generation ethanol production in terms of land use and competition with agriculture, in addition to environmental issues such as global warming, greenhouse-gas emissions and carbon balance have led to the transition from first generation to advanced biofuels. The latter can be produced from various lignocellulosic biomass (Zabed et al., 2016) since it is the most abundant renewable raw material available on earth. In addition, it contains many carbohydrates (occurring as cellulose and hemicelluloses) that could be converted into second generation ethanol.

Sorghum (*Sorghum bicolor* (L.) Moench) is a drought- and heat-tolerant multi-purpose crop, which can be grown on a wide variety of soils. The two most popular types of sorghum are grain sorghum and sweet sorghum (Rooney et al., 2007; Vasilakoglou et al., 2011). The former is the fifth most important cereal crop in the world, after corn, rice, wheat and barley, and its grains are used as dietary staple by more than 500 million people in more than 30 countries of the semi-arid tropics (Henley, 2011) or as feed for animals (Rooney et al., 2007; Steduto et al., 1997). Sweet sorghum produces lower grain yields, but higher sugar content in its juices as well as more important yields of green biomass and this variety is essentially used for forage, silage and sugar syrup production. Sweet sorghum is a promising energy crop in Canada since it has minimal fertilizer and water requirements and a relatively high biomass productivity. In addition, sorghum has a short growing period (3–5 months) and its stalks are rich in readily fermentable sugars (Koradiya et al., 2016; Liu and Shen, 2008; Regassa and Wortmann, 2014). In addition to these free sugars, sorghum stalks contain cellulose and hemicelluloses, both of which are polymers composed of carbohydrate monomers (hence second generation sugars). Sweet sorghum could therefore be used for the simultaneous production of first and second generation ethanol (Barcelos et al., 2016; Wu et al., 2013).

Part of the free sugars found in dry sorghum can easily be extracted by soaking the grinded stalks in water prior for them to be pressed. The free sugars can then be recovered in water that can be concentrated prior to fermentation. However, the sugars contained in cellulose and hemicelluloses cannot as easily be extracted. In these macromolecules, sugars units are linked to each other refraining their direct

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solubilisation in water. In addition, lignin can as well limit the accessibility to these constituents explaining why various pretreatments technologies and delignification processes have been developed over the years in order to split biomass into its main constituents (Kamireddy et al., 2013; Rocha et al., 2012). These preliminary steps are particularly important in a biorefinery concept where all the fractions from biomass (lignin, hemicelluloses, cellulose and extractives) require to be used in order to make biorefineries economically realistic (Beauchet et al., 2013).

Steam treatments consist in heating biomass using saturated steam, followed by an explosive decompression of the pressured system. Such process is known to induce a good fractionation of biomass and solubilisation of hemicelluloses and even lignin (Jacquet et al., 2015; Lavoie and Beauchet, 2012; Oliveira et al., 2013). Steam treatments can be performed with or without catalyst and in such cases, steam treatments presents the advantage of using no additional chemicals (except water), while allowing high yield of hemicelluloses conversion to monomeric sugars (Agudelo et al., 2016). Un-catalyzed steam processes are also known as autohydrolysis due to the catalytic action of acids released from acetyl and formyl groups (as well as other functional groups from the hemicelluloses), that catalyses hemicelluloses hydrolysis. Uncatalyzed steam explosion can be used as a first step when a selective isolation of hemicelluloses is targeted. If hemicelluloses are extracted using an uncatalyzed treatment, a second one (base catalyzed) allows removing lignin as well (Lavoie et al., 2011, 2010)

In this work a steam process was optimized to allow maximum sugar recovery from dry sorghum biomass. Severity factors, ranging from 3.25 to 4.44, were correlated with the total sugar production. The release of inhibitory compounds was also studied since they can have an impact on the downstream fermentation process (Panagiotopoulos et al., 2011). The analysis of the results, depending on the severity factor, as well as following experimental design and response surfaces methodology, allowed defining the optimal conditions to maximize sugar production.

2. Materials and methods

2.1. Raw material

Sweet sorghum (*Sorghum bicolor* (L.) Moench) was grown at the CEROM research center in Saint-Mathieu-de-Beloeil (Québec, Canada, 45.58°N, 73.24°W) and accumulated between 2901 and 3100 CHU (corn heat units) during the growing season (Plouffe et al., 2012). Soil type was a St. Urbain clay loam (very-fine clayey, mixed mesic Typic Humaquapt). Sweet sorghum hybrid CSSH 45 (AERC Inc., Delhi, ON, Canada) was used for a total production of 200 kg (dry mass) for the project. Seedbed preparation consisted of mouldboard ploughing in the fall, harrowing in the spring to stimulate weed germination, and a final harrowing to kill weeds prior to seeding. The seeding rate was 320 m^{-2} pure live seeds. Seeding was performed at a depth of 2.5 cm as soon as soil temperature reached 12 °C (Agriculture Environmental Renewal Canada, 2017), using a Fabro plot seeder (Fabro Enterprises Ltd., Swift Current, SK).

Bentazone [3-isopropyl-1H-2.1.3-benzothiadiazin-4 (3H)-12.2-dioxide] was applied at a rate of 1.08 kg of active ingredient per hectare between the three- and six-leaf stage to suppress dicotyledon weeds. Hand weeding of the sweet sorghum plots was done at the ten-leaf stage. Nitrogen fertilization using nitrate (27–0–0) was performed at a rate of 80 kg N ha^{-1}, with 40 kg N ha^{-1} broadcast at seeding and the remaining N side-dressed at the four-leaf stage. Phosphorus was applied as triple superphosphate (0–46–0) and potassium as potassium chloride (0–0–60) based on soil analyses and local recommendations (Parent and Gagné, 2015).

It is important to note that the use of another sweet sorghum cultivar could give different results because of their different chemical compositions.

Sweet sorghum stems were hand-harvested, using a sickle, the day before a killing frost corresponding to 121 days after seeding. Full size green stems were shipped the same day they were harvested in order to ensure integrity of water-soluble carbohydrates and secondary metabolites content. The green stems were then dried in a ventilated oven at 60 °C for 72 h in order to avoid biodegradation. Then, the samples were stored in a dry and well-ventilated place. Prior to the utilization, raw material was chipped down to a final size of about 2–3 cm and its initial moisture content was tested in triplicate, on an initial mass of approximately 10 g. The initial moisture content of sorghum was 20.27% ± 0.77%.

2.2. Impregnation

Prior to steam processes, dry sorghum requires an impregnation step with water since the tested biomass must contain sufficient moisture to produce the expected effects (volume expansion when liquid water become steam). In addition, and in the case of dry sorghum, the impregnation step allows extracting the free sugars present. These free sugars are then found in the impregnation liquid, which can then be concentrated for fermentation.

As part of the experimental plan aimed at optimizing steam processes on dry sorghum, a batch of biomass was impregnated prior to the following 13 steam explosion tests. A total of 2502.0 g of dry sorghum were impregnated with 25.0 kg of water for 15 h. The solid:liquid ratio was 1:10 and the duration of the impregnation was determined in relation to the results obtained previously (unpublished results). After impregnation at room temperature, the biomass was pressed for 5 min at 689 kPa (100 psi), leading to the recuperation of 23.56 kg of liquid after which an additional 25.0 kg of water was used to wash the impregnated sorghum. Washing was performed for 15 min, then the biomass was pressed again for 5 min at 689 kPa (100 psi), leading to the recuperation of 25.52 kg of washing liquid. Weighing the liquids made it possible to calculate the yields of sugars and inhibitors for this impregnation step. The moisture content of the sorghum after impregnation was tested in triplicate, with an initial mass of about 17 g. The moisture content of sorghum after impregnation was 59.74% ± 0.61%.

The method used for dry sorghum impregnation followed by the steam processes is depicted in Fig. 1, as well as the wash steps. This method has already been used previously with different biomass and was reported by our team (Lavoie et al., 2010; Lavoie and Beauchet, 2012).

2.3. Steam processes

Prior to operation, the double envelope of the reactor was preheated using a steam circulation to bring the reactor to the targeted temperature. The reactor was then filled with steam, which was eventually depressurized without biomass in a specifically designed vessel. Immediately afterwards, the reactor was opened from the top and impregnated sorghum was quickly inserted so that it can maintain its moisture content. 92.6 g of water-extracted sorghum (dry basis) were used for each test. The reactor was then sealed and filled with pressurized steam through an inlet that allows to quickly reach the targeted temperature. Sorghum was cooked at temperatures ranging from 200 to 220 °C, for 2–8 min. When the operation temperature was reached, a stopwatch was triggered and, as soon as the treatment duration has elapsed, the explosion of the biomass was induced by opening the blowdown valve. The biomass was depressurized in 5 kg of water at ambient temperature, which was contained in the explosion tank in order to temper the shock induced by the steam explosion. The reactor was then filled again with steam and as soon as the operating temperature was reached, this vapor was depressurized again in order to empty all the sorghum fibers from the reactor towards the depressurization vessel. Then, the processed mixture containing the exploded