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The effect of power-ultrasound on the pretreatment of acidified aqueous solutions of banana flower-stalk: Structural, chemical and statistical analysis

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ABSTRACT

Various pretreatment techniques can change the physical and chemical structure of lignocellulosic biomass and improve the hydrolysis rates. High-intensity ultrasound could be a promising technique in the biomass pretreatment process. The objective of this work was to study the effect of biomass concentration, pH, ultrasonic power level and sonication time on the production yield in total sugars (S_T) and reducing sugars (S_R) during the pretreatment of banana flower-stalk biomass. A qualitative evaluation was carried out by scanning electron microscopy, showing a disruptive effect on the biomass structure at high ultrasonic power levels and low biomass concentrations. An experimental design with three-levels for the four-variables was used in order to set the conditions for the pretreatments. Stepwise regression (SRG) and an artificial neural network (ANN) were applied in order to establish mathematical models that could represent and be used to study the dependence of the factors on both the S_T and S_R yields. The statistical results indicated that the ANN approach provided a more accurate estimation than SRG.

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1. Introduction

Technologies for the conversion of biomass resources into fuels and chemicals such as ethanol have sparked growing interest as a means of reducing dependency on fossil fuels, and of developing environmentally friendly and renewable energy sources. Such biomass resources include energy crops, agricultural crops, forestry residues, aquatic crops and residues from processing industries (Arvanitoyannis, 2008; Lee et al., 2013).

Banana production could be considered as an important biomass feedstock source. This fruit occupies first place amongst the tropical fruits in terms of production and exportation, with levels of around 107 Mt and 16 Mt, respectively. The principal producing countries are India, Brazil, Ecuador, Costa Rica, Philippines and Colombia, representing more than 80% of world production (FAOSTAT, 2013). The banana plant and its associated residual biomass are amylaceous and lignocellulosic materials, including the fruit itself, the peel, flower-stalk, trunk, leaves and pedicel (Vaughan and Geissler, 2009). Velásquez-Arredondo et al. (2010) and Brent-Hammond et al. (1996) researched the application of

(Mosier et al., 2005). Nowadays, the combination of pretreatment techniques has become common practice due to the technological challenges that have arisen from research into second-generation ethanol production (Macrelli et al., 2012).

banana wastes in the second-generation ethanol industry, and the capacity of their lignocellulosic biomass to produce fermentable

Biomass conversion in the second-generation ethanol industry

comprises the pretreatment of the biomass, conversion of the cellu-

lose and hemicellulose into fermentable sugars and fermentation of

the sugars into biofuels (Hendriks and Zeeman, 2009; Kumar et al.,

2009). Sugars can be obtained by using physical (Zheng et al., 2012;

Kumar et al., 2011), physicochemical (Olsen et al., 2012), chemical

(Velásquez-Arredondo et al., 2010; Tadesse and Luque, 2011) and

biological (Tian et al., 2012) pretreatment methods, or a combina-

tion of these (Passoth et al., 2013; Obama et al., 2012). Pretreatment

processes for biomass hydrolysis generally refer to breaking of the

naturally resistant carbohydrate lignin shield that limits accessibil-

ity of the enzymes or chemicals to the cellulose and hemicellulose

sugar, showing promising results for the industry.

Thus the application of power ultrasound is an alternative to the above-mentioned methods, potentiating the production of fermentable sugar in the biomass pretreatment. For instance, high-intensity ultrasound has been reported as a pretreatment in







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the conversion of the following Indian wastes: Areca nut husk, bon bogori and moj (Sasmal et al., 2012), pulp mill waste (Park et al., 2012) and dry maize milling (Khanel et al., 2007) into glucose, and has been related to the consequent increase in biofuel yield.

The pretreatment of biomass with ultrasound uses the principle of cavitation, that is, the spontaneous formation, growth and collapse of millions of microscopic vapor bubbles in the liquid. The collapse or implosion of these localized cavities produces high temperatures and pressures of microsecond duration, producing the hot-spot effect in the cold liquid. These high cooling rates prevent the organization and crystallization of the precipitated products, resulting in mostly amorphous particles (Sawant et al., 2008; Pinjari and Pandit, 2010).

In the pretreatment of organic matter in solution with different ultrasonic power levels, Jiang et al. (2011) demonstrated that the effect on the biomass of exposure to low-intensity ultrasound for long periods is similar to that of exposure to high-intensity ultrasound for short periods. Other studies have shown that the combination of ultrasound and chemical methods increases the efficiency of the biomass pretreatment (Esfahani and Azin, 2012; Yunus et al., 2010).

Fig. 1 shows an example of the use of high-intensity ultrasound as a biomass pretreatment linked to a second-generation ethanol industry. According to the scheme, high-intensity ultrasound could be used as a single process or as a combined process together with chemical pretreatments, with a subsequent biological pretreatment (Kumar et al., 2009). An important benefit of high-intensity ultrasound is the recycling of the biomass, such that ultrasound may act in the breakage of the raw material and, at the same time, in the biological inhibition of the centrifuged wine, thus making the process more environmentally friendly.

Although the application of ultrasound has produced some important results for the bioethanol industry, few studies have been carried out to evaluate the effect of this method on the material, due to the difficulty in calculating the acoustic properties, and also because these properties may differ significantly from one material to another (Hodnett and Zeqiri, 1997).

Therefore this study aimed to determine the effects of the ultrasonic power level, biomass concentration, pH and sonication time on the production yield in both total and reducing sugars during the biomass pretreatment of banana flower-stalk. Scanning electron microscopy was used to evaluate the structural effects on the material, and the stepwise regression and artificial neural network approaches were used to analyze the correlation between the experimental results and the variables considered.

2. Materials and methods

2.1. Raw material and sample preparation

Banana flower stalks (*Musa acuminata* AAA cv. Dwarf Cavendish) with a moisture content of $92.8 \pm 0.5\%$ w/w (d.b.) were acquired from local farmers and from the industry Banatech (Guapiaçu, São Paulo State, Brazil), and kept in a refrigerator at $15 \,^{\circ}$ C for 24 h prior to use. After cleaning, the flower stalks were cut longitudinally in the direction of the fibers and dehydrated in a forced convection dryer at 40 $^{\circ}$ C and 2 m/s for 24 h. The dried flower stalks, with a moisture content of $9.2 \pm 0.3\%$ w/w (d.b.), were then ground in a knife mill (model MA380, Marconi, Piracicaba, Brazil) to a powdered biomass with a particle size ranging from 50 to 200 μ m.

This powder was used to prepare acidified aqueous solutions of flower-stalk biomass (AFSB) at concentrations of 0.04, 0.06, 0.08, 0.10 and 0.12 g/mL. Acidification of the AFSB was carried out before mixing, using 500 mL of deionized water with the addition of 0.001% H₂SO₄, and stabilizing for 3 days to obtain pH values of 3.0, 5.0, and 7.0, corresponding to concentrations of 1.49 ± 0.10 , 0.31 ± 0.02 and 0.00 ± 0.00 mol/L of H₂SO₄, respectively.

2.2. Experimental design

Four factors: biomass concentration (g/mL), ultrasound power (W), pH and time (min) were experimentally combined to establish their effects on the following responses: production yield in total sugar (S_T , mg/g) and production yield in reducing sugar (S_T , mg/g). An experimental design was used combining different levels of the factors (Table 1), obtaining 72 experiments divided into two sections. In the first section, the combination of three levels from the four factors was established, superscript "*a*" totalizing 36 experiments, with 12 experiments being repetitions of the central point. In the second section, the combination of intermediate levels of the factors: biomass concentration (g/mL) and ultrasound power (W) was established, with the three levels of the factors: pH and time, superscript "*b*" totalizing 36 experiments.

2.3. Biomass pretreatment

A high volume ultrasonic liquid processing system (model VCX 1500HV, Sonics & Materials Inc., USA) was used for the pretreatments. The equipment has a piezoelectric lead zirconiumtitanium crystal converter connected to a titanium alloy probe (model Ti-6Al-4V, Sonics & Materials Inc., USA). It was operated in the pulse mode (8 s with ultrasound and 2 s without ultrasound) to avoid overload during the pretreatments, provided by a high-volume



Fig. 1. Scheme for the conceptual bioethanol industry options for the pretreatment of lignocellulosic biomass with high-intensity ultrasound.

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