



Research paper

Efficient sugar production from sugarcane bagasse by microwave assisted acid and alkali pretreatment



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ABSTRACT

Sugarcane bagasse represents one of the best potential feedstocks for the production of second generation bioethanol. The most efficient method to produce fermentable sugars is by enzymatic hydrolysis, assisted by thermochemical pretreatments. Previous research was focused on conventional heating pretreatment and the pretreated biomass residue characteristics. In this work, microwave energy is applied to facilitate sodium hydroxide (NaOH) and sulphuric acid (H₂SO₄) pretreatments on sugarcane bagasse and the efficiency of sugar production was evaluated on the soluble sugars released during pretreatment. The results show that microwave assisted pretreatment was more efficient than conventional heating pretreatment and it gave rise to 4 times higher reducing sugar release by using 5.7 times less pretreatment time. It is highlighted that enrichment of xylose and glucose can be tuned by changing pretreatment media (NaOH/H₂SO₄) and holding time. SEM study shows significant delignification effect of NaOH pretreatment, suggesting a possible improved enzymatic hydrolysis process. However, severe acid conditions should be avoided (long holding time or high acid concentration) under microwave heating conditions. It led to biomass carbonization, reducing sugar production and forming 'humins'. Overall, in comparison with conventional pretreatment, microwave assisted pretreatment removed significant amount of hemicellulose and lignin and led to high amount of sugar production during pretreatment process, suggesting microwave heating pretreatment is an effective and efficient pretreatment method.

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1. Introduction

In recent years, there has been increasing interest in the use of agricultural waste, as feedstock for second generation biofuels production. Sugarcane is the major bioenergy crop in Brazil, where it has been successfully used for the production of bioethanol [1]. The development of new technologies for lignocellulosic ethanol production from sugarcane bagasse is of special interest, since it would increase the efficiency of ethanol production without expanding the agricultural areas, avoiding the current conflict

produced by change in land use to meet growing energy demands [1].

Lignocellulosic biomass is a recalcitrant material, composed of cellulose, hemicellulose and lignin, organised in a network of polymers that evolved to develop recalcitrance against enzyme hydrolysis produced by microorganism in nature [2]. To release the sugars locked in biomass in industrial processes, various pretreatments have been proposed and trialled at laboratory and pilot scale. Among them are steam explosion [3], ammonia fibre explosion [4], hot water [5], supercritical CO₂ [6], biological [4] and acid or alkaline pretreatments [7,8] and others. An effective pretreatment must meet the following requirements: 1. Improve sugar production or the ability to subsequently form sugars by hydrolysis; 2. Avoid degradation or carbohydrate loss; 3. Avoid by-product formation, such as inhibitory chemicals to the subsequent

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hydrolysis and fermentation processes; 4. Be cost effective [4]. Pretreatments may alter the structure of cellulosic biomass to make it more accessible for enzymes, as well as decrease the degree of polymerization and cellulose crystallinity. Additionally, it can selectively remove hemicellulose and lignin from the lignocellulosic matrix [3,9]. Compared to the other pretreatment methodologies listed above. Acid and alkaline pretreatments are considered effective, which explains their extensive use in most cases during biomass pretreatment [2,9–17]. For instance, Marasabessy et al. reported that, with 30 min 0.9% (w/v) H₂SO₄ pretreatment at 178 °C before enzymatic hydrolysis, 100% of all pentoses present in *Atropa curcas* fruit hull were released (71% yield and 29% degradation to furfural) after 24 h enzymatic hydrolysis. Meanwhile, 83% of the hexoses (78% yield and 5% degradation to 5-hydroxymethylfurfural) is achieved [18]. Zhu et al. studied microwave assisted dilute NaOH (1%) pretreatment of wheat straw, and the weight loss of hemicellulose and lignin after pretreatment is 76–84.4% and 81–86% respectively [17].

There is a growing interest in using microwaves as a tool to bring about various chemical processes, both in synthetic chemistry and in the treatment of lignocellulosic biomass [19,20]. Microwaves are electromagnetic waves, whose frequencies lie between 300 MHz and 300 GHz by general definition. In the case of conventional heating, heat is transferred by means of convection, conduction, and radiation from the surfaces to the inner areas of the materials. Thus, while in conventional heating, the energy transfer is the result of thermal gradients, microwave energy is transferred directly to the material being heated by electromagnetic radiation [21]. Under the influence of the incident radiation, a substance possessing dipole moments and sufficient mobility will align itself to the electromagnetic field by rotation. Further, the rotation will give rise to frictions between neighbouring molecules and increase the temperature. Therefore, microwave heating is more direct, rapid and uniform than conventional heating [21]. Significant research has been carried out in the field of microwave assisted pretreatment, with different feedstock including sugarcane bagasse [2], switchgrass [22], wheat straw [17], and so on. Hu and Wen found that the total sugar amount (glucose and xylose) from pretreatment and hydrolysis of switchgrass was 58.5% of the maximum sugars in the material [22]. Nikolic et al. studied microwave pretreatment for corn starch and the results showed that the glucose concentration in pretreatment liquor was increased by 8.48% compared to untreated control sample and the percentage of theoretical ethanol yield was 92.27% after 44 h of the simultaneous saccharification and fermentation (SSF) [23]. Lu et al. reported that the glucose yield of rape straw from enzymatic hydrolysis was enhanced by 56.2% (11.5% for raw rape straw) after microwave pretreatment [24]. Chen et al. studied the effect of microwave assisted sulphuric acid pretreatment for sugarcane bagasse, and revealed that at 190 °C the fragmentation of particles become very pronounced and almost all hemicellulose was removed and the crystalline structure of cellulose disappeared [25]. These studies show that microwave combined with thermochemical processes could be a promising pretreatment method.

As it is well known, conventional heating systems have the disadvantage of being time consuming and also lead to significant sugar degradation [26–30]. Microwave heating, on the contrary, is faster, and is gaining growing attention, being applied for pyrolytic and hydrolytic activation [19,20,31,32]. Most of the current microwave assistance studies were concentrated on pyrolytic activation of lignocellulosic material, but hydrolytic activation has been less studied. Besides, previous research concentrated on sugar production from enzymatic hydrolysis of biomass after pretreatment. In our study, rapid microwave heating is used to enhance the pretreatment process and improve the sugar production. Only a

few results have been reported on sugar production during the pretreatment process under microwave conditions. In the present work, sugarcane bagasse is processed under a range of microwave assisted acid (H₂SO₄) and alkaline (NaOH) conditions. The sugar production and the chemical changes produced during pretreatments were evaluated in order to explain the unique microwave effects on biomass composition and structure, while informing the optimisation of sugar production. The current study is very useful for the comprehension of the microwave effect on the sugar production enhancement from lignocellulosic material during pretreatment and the unique microwave effects on biomass composition and structure.

2. Material and method

2.1. Raw material

Sugarcane bagasse was provided by the Cosan Mill (Ibaté, SP, Brazil). The raw material previously washed and roughly ground was dried in a convection oven at 60 °C for 24 h and further ground by knife milling into small pieces (625 μm × 188 μm avg.). The biomass compositions of raw sugarcane bagasse are cellulose (25 ± 2.7%), hemicellulose (48 ± 2.3%), lignin (31 ± 1.2%) and ash (0.83 ± 0.03%).

2.2. Microwave pretreatment and conventional thermochemical pretreatment method

Microwave acid digestion vessel (Vessel 4781, Parr Instruments) was used as a reactor and a domestic microwave oven (SAMSUNG Model CM1629A; Dimensions: W 464 × D 557 × H 368 mm; power output: 1600 W) was used as a microwave source. A total weight of 0.2 g of sugarcane bagasse was immersed in 8 ml 0.2 M or 0.4 M H₂SO₄ or NaOH. The pretreatment was carried out at 320 W for various holding time (3–10 min), resulting in more heat being transferred to the sample as time increased. The internal temperature of the pressure vessel was estimated by using an indirect method and found it to be 170 °C ± 5 °C when the microwave heating time was 3–5 min [33]. Pretreatment temperature was not measured. The biomass solid residue resulting from the pretreatments was separated from the liquor by centrifugation, and then rinsed with ethanol (3 × 10 ml), and dried in oven at 50 °C for 24 h. Both the liquor and the solid residues were analysed.

Conventional thermo-chemical pretreatment is conducted in the acid digestion vessel (Vessel 4745, Parr Instruments) under 120 °C for 40 min. Same biomass loading, sample separation, washing and drying procedures are performed in order carry out following analysis.

2.3. Chemical analysis of the liquors from pretreatment media

The liquor resulting from the acid and the alkaline pretreatments were neutralized to pH 7 by 1 M HCl and 1 M NaOH solutions, respectively. Then the analyses of monosaccharides were carried out by using a Dionex High Performance Anion Exchange Chromatography (ICS-3000PC, Thermal scientific, USA) equipped with electrochemical detector [34].

2.4. Lignin quantification

Lignin was quantified as follows: 3.5 mg of biomass (untreated or pretreated) was dissolved in acetyl bromide solution (25% v/v acetyl bromide/glacial acetic acid), then 1 ml of NaOH 2 M and 175 μl hydroxylamine hydrochloride of 0.5 M were added in a 5 ml volumetric flask. The solution was taken to 5 ml with acetic acid

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