



Steam pretreatment of spruce forest residues: Optimal conditions for biogas production and enzymatic hydrolysis



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ABSTRACT

Steam refining of non-debarked spruce forest residues was investigated as pretreatment for enzymatic hydrolysis as well as for biogas production. Pretreatment conditions were varied in the range of 190–220 °C, 5–10 min and 0–3.7% SO₂ according to a statistical design. For both applications highest product yields were predicted at 220 °C and 2.4% SO₂, whereas the reaction time had only a minor influence. The conformity of the model results allows the conclusion that enzymatic hydrolysis is a suitable test method to evaluate the degradability of lignocellulosic biomass in the biogas process. In control experiments under optimal conditions the results of the model were verified. The yield of total monomeric carbohydrates after enzymatic hydrolysis was equivalent to 55% of all theoretically available polysaccharides. The corresponding biogas yield from the pretreated wood amounted to 304 mL/g_{ODM}. Furthermore, furans produced under optimal process conditions showed no inhibitory effect on biogas production.

It can be concluded that steam refining opens the structure of wood, thus improving the enzymatic hydrolysis of the polysaccharides to fermentable monomeric sugars and subsequently enabling a higher and faster production of biogas. Anaerobic fermentation of pretreated wood is a serious alternative to alcoholic fermentation especially when low quality wood grades and residues are used. Anaerobic digestion should be further investigated in order to diversify the biorefinery options for lignocellulosic materials.

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1. Introduction

Today, there is no dispute that the use of renewable energy has to increase in order to reduce the CO₂ net emissions and the dependence on finite fossil fuels. Consequently, in 2008 the EU passed a directive in order to enhance the contribution of renewable energies (such as wind power, hydro power, solar energy, geothermal energy and biomass) to 20% of the total energy needs by 2020. Additionally, a 10% use of green fuels in transport is included within the overall EU objective (European Commission, 2008). The share of renewable energy sources in Europe's primary energy consumption increased from 4.4% in 1990 to 9.5% in 2010 (Eurostat, 2012). Thus, to achieve the 20% share of renewable energy in 2020 significant efforts will still be needed in the future. In Europe, 70% of the energy from renewable resources is gained by the use of

biomass. One considerable advantage of biomass compared to the other renewable energy sources is the fact that dried biomass or biobased liquid fuels can be stored and thus can be utilised as and when they are needed. Currently the production of bioenergy from wood is mainly based on incineration, while biofuels or chemical products from biomass are mainly based on annual plants like corn, wheat or sugar beet. In order to reduce the raw material competition for biomass in energetic, material, and food applications it is desirable to increase the input of residual woody biomass to obtain energy or chemicals.

Conversion processes based on fermentation require the hydrolysis of cellulose and hemicelluloses into their corresponding monomeric carbohydrates in order to make them accessible for microorganisms. However, in case of lignocellulosic biomass the matrix of lignin and hemicelluloses around the cellulose fibrils, the low pore volume of the cell walls, and the high crystallinity of the cellulose fibrils limits the rate and extent of the enzymatic hydrolysis (Chang & Holtzapfle, 2000). Therefore, different physical, chemical or physicochemical pretreatment methods such as mechanical comminution (chipping, grinding, milling), steam

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explosion, organosolv pulping, alkaline hydrolysis as well as diluted and concentrated acid hydrolysis have been investigated to enhance the enzymatic hydrolysis of lignocellulosic materials (Galbe & Zacchi, 2007; Hendriks & Zeeman, 2009; Kumar, Barrett, Delwiche, & Stroeve, 2009; Taherzadeh & Karimi, 2008). Besides dilute acid hydrolysis, steaming (steam explosion or steam refining) with and without addition of acid catalyst is the most investigated pretreatment method to enhance the accessibility of lignocellulosic materials for subsequent biological degradation processes (Galbe & Zacchi, 2002; McMillan, 1994). In case of wood the steam pretreatment is viewed as a cost effective process with a simple set up and low need for chemicals (Clark & Mackie, 1987; Jorgensen, Kristensen, & Felby, 2007; Sun & Cheng, 2002). In the steam explosion method biomass is treated under high pressure with saturated steam. Then the pressure is suddenly reduced, which makes the material undergo an explosive decompression. However, some investigations have shown that the explosion step is not necessary in order to increase the digestibility of wood (Brownell & Saddler, 1987; Brownell, Yu, & Saddler, 1986). Refining of the steamed material in a laboratory defibrator is a possible alternative to the explosion step. Schütt, Westereng, Horn, Puls, & Saake (2012) compared steam refining with steam explosion using the same raw material, namely not debarked poplar, and the same steam conditions. Regardless of the method used for the pretreatment, the results from enzymatic hydrolysis showed no significant difference concerning the carbohydrate yields and the overall product balance.

In the present study steam refining was tested as pretreatment for the conversion of spruce forest residues into biogas. The steam refining conditions time, temperature and SO_2 -charge were optimized by an experimental design based on the response surface methodology (Box, Hunter, & Hunter, 1978). Since debarking of forestry residues is technically difficult and uneconomic the spruce wood was used without debarking in this investigation. The effectiveness of steam refining was assessed by biogas experiments as well as by conducting separate enzymatic hydrolysis tests in order to determine the accessible carbohydrate yields. In doing so, it should be evaluated if the separate enzymatic hydrolysis can be used as a fast test method in order to evaluate the degradability of pretreated lignocelluloses in the time consuming biogas process. Furthermore, furfural and 5-hydroxymethylfurfural (5-HMF) contents of the extracts after steaming were determined and their inhibitory influence on the biogas process was investigated.

2. Materials and methods

2.1. Raw material

Spruce forest residues originate from Reinbek Forest near Hamburg (Germany). The trunks and branches with diameters between 2 and 15 cm were chipped with their adherent bark in a shredder typical for landscaping applications. The shredded material used for steam refining experiments showed an inhomogeneous particle size from 10 to 60 mm in length and from 5 to 15 mm in thickness and contained about 8% bark. The wet chips were stored in a cooling chamber and were defrosted one day before the steam experiments.

2.2. Analytical methods for raw material characterisation

The extractives of the wood and bark fractions were analyzed by accelerated solvent extraction (ASE 200, Fa. Dionex). Therefore, about 1 g of milled sample materials were extracted successively by petrol ether, acetone:water (9:1) and water at 100 bar and 100 °C for 40 min. The total extractive content was determined by adding

the single extraction yields. Ash contents were determined by combustion at 525 °C (Tappi Standard - T211om-93, 1993).

Carbohydrate and lignin contents were analyzed by a two-stage acid hydrolysis. 200 mg of dry material were hydrolyzed with 2 mL of 72% H_2SO_4 at 30 °C for exactly 1 h to break up the crystalline structure of the cellulose. The samples were then diluted with water to a concentration of 4% H_2SO_4 and put in an autoclave for 40 min at 120 °C for the hydrolysis to monomeric sugars. After cooling, the samples were filtered on a No. 4 sintered glass crucible. The residue was weighed on an analytical balance (AG 245, Mettler Toledo) and represents the acid-insoluble lignin content. The carbohydrate content in the hydrolysates was analyzed by borate-anion-exchange-chromatography with after-column derivatization and detection of the separated monosaccharides at 560 nm as described previously (Sinner & Puls, 1978; Sinner, Simatupang, & Dietrichs, 1975; Willfor et al., 2009).

2.3. Steam refining pretreatment

Steam refining was performed in a 10 L reactor. Material input per batch was 300 g_{DM} of wet spruce chips and the reactor was completely filled with saturated steam.

Treatments were performed at 190–220 °C for 5–10 min with a SO_2 -charge of 0–3.7% based on dry wood. In total, 25 steam refining experiments were conducted for the optimization of the three process parameters. The addition of SO_2 was carried out prior to the steam pretreatments. The wood was put into a plastic bag and the required SO_2 amount was fed into the bag. Finally, the SO_2 uptake was controlled on a balance.

The reactor contained inside a blade system, which was rotated at 1455 rpm for 30 s at the end of each steam treatment, to defibrate the softened material. By flashing with additional water the reactor was emptied and a washing of the pretreated material occurred. The extract formed during steam treatment and the wash water was separated from the fibers in a spin dryer. This extract contains the water soluble compounds (monomeric or oligomeric sugars, furans and organic acids) after steaming. Yields of the steam pretreated solid fibers and of the liquid extract were determined after drying.

2.4. Application tests of steam pretreated samples

The effectiveness of the steam refining pretreatment was evaluated by the total monomeric carbohydrate yield after hydrolysis of the fiber and extract fraction. Additionally, the produced biogas volume after anaerobic fermentation of the combined fiber and extract fractions was measured.

2.4.1. Total monomeric carbohydrate yield after hydrolysis

The above mentioned total monomeric carbohydrate yields after hydrolysis of the pretreated samples were calculated by the sum of carbohydrates yields after enzymatic hydrolysis of the fiber fraction and after acid hydrolysis of the extract fraction. Preliminary experiments with extracts showed that the monomeric carbohydrate yields of enzymatic hydrolysis are very close to those of acid hydrolysis. Furthermore, HPLC–MS measurements revealed that the hemicellulose degradation products exist as monomers or oligomers in the extract after steam refining. Therefore, it can be assumed that all carbohydrates in the extract are easy to degrade by enzymatic hydrolysis and it is permissible to determine the monomeric yields of the extract directly by acid hydrolysis.

Hydrolysis of the extracts was carried out after freeze-drying. Lyophilized samples (100 mg) were dissolved in 10 mL water, spiked with 1.8 mL of 2 N H_2SO_4 (1.5% H_2SO_4) and autoclaved at 120 °C for 40 min.

Enzymatic hydrolysis of the fibers was conducted with 21 FPU (filter paper units) Celluclast 1.5 L (Cellulase complex, Novozymes

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