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The AlkaPolP process: Fractionation of various lignocelluloses and continuous pulping within an integrated biorefinery concept

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ABSTRACT

The implementation of a lignocellulose-based biorefinery requires an efficient fractionation of its raw material. For this purpose a comprehensive biorefinery concept has been developed based on alkaline polyol pulping (AlkaPolP). It exhibits a high degree of flexibility with respect to the extent of fractionation by adjusting easily controllable process parameters. As shown in this contribution, the AlkaPolP process can be successfully applied for a wide range of lignocellulosic biomass including hardwood, softwood, bark and grasses, all of which yielding high quality product fractions.

With essentially complete delignification the obtained pulp fraction is easily degraded by cellulases. The produced hydrolysates are free of inhibitors and can be used as substrates for a variety of fermentation processes. Without further modification, the AlkaPolP lignin fraction can be readily applied as high value substrate for various processes as confirmed by extensive characterisations in industrial and research laboratories (Dynea, Fraunhofer Institute). The remaining product fraction resulting from the degradation of various polysaccharides is being recovered as carboxylic acids.

Based on extensive experimental results up to mini-plant scale, an integrated biorefinery concept has been developed including a continuous pulping stage and a full scale recycling of process chemicals thus allowing operations in environmentally sensitive rural areas.
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1. Introduction

Due to the finiteness of fossil resources, especially petroleum and natural gas, renewable chemical feedstock sources have to be opened up to guarantee the future raw material supply for the chemical industry. In this regard the utilisation of lignocelluloses appears to be very promising. Because more than 100 billion metric tons of lignocellulosic biomass are globally produced by nature every year it represents a very abundant alternative resource (Lützen et al., 1983). Furthermore, the use of lignocellulose is not burdened by a food-vs-fuel conflict arising when agricultural products are applied for the production of fuels and chemicals.

Lignocellulosic biomass mainly consists of the biopolymers cellulose, hemicellulose and lignin which are partially interconnected and form a tight structure resistant to the influences of solvents and heat (Pedersen and Meyer, 2010). Therefore, the utilisation of carbohydrate as well as aromatic

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lignocellulose constituents within a biorefinery requires a previous efficient fractionation of the raw material. For this purpose a novel process based on alkaline polyol pulping (AlkaPolP) has been developed (Hundt et al., 2013a). As already reported, the AlkaPolP process enables the efficient delignification of hardwood (Hundt et al., 2013b) and softwood (Hundt et al., 2013c) as well as a nearly complete enzymatic hydrolysis of the resulting pulp fraction. The lignin dissolved during the pulping process can be precipitated by neutralisation of the black liquor. As shown for pine wood, the AlkaPolP process can be optimised with the aid of a dimensionless parameter combining the effects of the most important pulping conditions (Hundt et al., 2014).

The results published so far regarding the AlkaPolP process were obtained by experiments in batch processing. However, the performance of pulping in a stirred-tank reactor involves some inherent disadvantages. The time required for heating, cooling, filling and emptying of the reactor impairs the productivity of the process. Differences in the process sequence result in a variable product quality, especially when renewables are used as raw material. Moreover, the liquid-to-solid ratio has to be kept high to guarantee a sufficient mixing of the reagents.

Because the AlkaPolP process can be performed at atmospheric pressure at short times it appears to be predestined for continuous pulping using reactive extrusion. By this means dead times can be avoided and the productivity can be increased at reduced variation in product quality. Furthermore, an extruder enables the processing of reaction mixtures with considerably lower liquid-to-solid ratios resulting in higher product concentrations and thus increased efficiency of downstream processing.

Literature on continuous processing of lignocellulose in an extruder mostly deals with thermo-mechanical pretreatment methods where wet biomass is usually extruded for less than two minutes at temperatures above 100 °C (Yoo et al., 2011; Karunanithy and Muthukumarappan, 2011). For several types of lignocellulose an improved enzymatic hydrolysis was found after thermo-mechanical treatment with (Lee et al., 2009; Karunanithy and Muthukumarappan, 2011; Zhang et al., 2012) and without (Yoo et al., 2011; Karunanithy et al., 2012; Karunanithy and Muthukumarappan, 2010) addition of chemicals. However, low residence times and temperatures do not allow an efficient fractionation of the lignocellulose. As a result of high shear stress the biomass is mainly defibrated and becomes more susceptible to enzymatic degradation because of its increased specific surface (Karunanithy et al., 2012). When using longer extruders and strongly alkaline solutions the continuous fractionation of lignocellulose becomes possible as shown by de Vrije et al. (2002) for *Miscanthus*. However, although a comprehensive literature research has been conducted, no report on continuous pulping of wood chips could be found yet.

The objective of this contribution is to demonstrate that the AlkaPolP process can be successfully applied to all types of lignocellulosic biomass including hardwood, softwood, bark and grasses yielding high quality product fractions. Furthermore, it will be shown that the pulping process can be performed continuously by using the single screw extruder developed in-house. Finally, an integrated biorefinery concept, which was designed on the basis of the continuous AlkaPolP process, is introduced.

2. Materials and methods

2.1. Raw materials

Wood of black locust (*Robinia pseudoacacia*), pine (*Pinus sylvestris*), beech (*Fagus sylvatica*) and birch (*Betula pendula* or *Betula pubescens*), pine bark (*Pinus sylvestris*) and plants of *Miscanthus* (*Miscanthus* × *giganteus*) were used as raw material. The pine wood was obtained in the form of commercial chips produced during operating of a Southern German sawmill (Binderholz GmbH). The other raw materials were harvested in the region of Lusatia, Eastern Germany. If necessary, the lignocelluloses were shredded before screening using a cutting mill equipped with a 10 mm square hole bottom sieve. The fraction between 2 and 4 mm was used for the pulping experiments in order to reduce the impact of the particle size. The preprocessing steps and compositions of all raw materials are given in Table 1.

2.2. Alkaline Polyol Pulping

To describe the influences of time and temperature the severity factor R_0 introduced by Overend and Chornet (1987) was used. The extent of pulping can be expressed by the pulp yield Y_p and the degree of lignin solubilisation *LS*:

$$Y_p = \frac{m_p}{m_{LC}} \cdot 100\%, \quad (1)$$

$$LS = \left(1 - \frac{m_{lig,p}}{m_{lig,LC}}\right) \cdot 100\%, \quad (2)$$

where m_p = pulp mass, m_{LC} = lignocellulose mass, $m_{lig,p}$ = mass of lignin in pulp and $m_{lig,LC}$ = mass of lignin in lignocellulose.

The setup and procedure for batch pulping of lignocellulose in alkaline glycerol has already been described elsewhere (Hundt et al., 2013c). The continuous pulping experiments were performed by using the single screw extruder developed in-house. All parts getting in contact with the reaction mixture were made of stainless steel. The screw (diameter 40 mm, length 3150 mm, trapezoidal thread with 16 mm pitch, 7 mm channel depth and a flight width of 4 mm at the root and 2 mm at the outer diameter) constitutes the core of the extruder. It is driven by a worm geared motor with a power rating of 370 W, a nominal output speed of 14.9 rpm and a torque of 123 Nm. The actual output speed is adjusted steplessly by a variable-frequency drive. The extruder barrel consists of an inner (outside diameter 44.5 mm, wall thickness 2.0 mm) and an outer pipe (outside diameter 48.3 mm, wall thickness 1.6 mm). Slits (3 mm wide and 300 mm long) spaced at 120 degrees intervals along the circumference of the inner pipe increase the mean coefficient of friction between the reaction mixture and the barrel thus enhancing the conveying performance of the extruder.

The pulping temperature is adjusted using three 900 mm long heating jackets also providing thermal insulation of the extruder. Each jacket has a power rating of 1100 W and is connected to a separate controller. Temperature set points and actual values are recorded by a micro process control system (μPCS) that was developed in-house for this purpose. The μPCS also monitors the mass flow of alkaline glycerol into the extruder by recording the weight of the stirred tank where the pulping liquor is preheated to 170 °C. The hot pulping liquor is conveyed via a heated line (177 °C) into the extruder using a gear pump. About 200 mm before the liquor inlet the

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