



Evaluation of different lignocellulosic raw materials as potential alternative feedstocks in biorefinery processes



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ABSTRACT

In the present work several alternative lignocellulosic materials have been classified according to their type and origin, and chemically characterized in order to propose their proper exploitation as biorefinery feedstock. In addition, different fractionation processes have been applied to some of these raw materials with the purpose of evaluating their efficiency and applicability for different feedstocks. The yield of each applied treatment has been determined according to the delignification ratio and the carbohydrate dissolution degree achieved. The characterisation results indicated that different lignocellulosic feedstocks could be treated together in biorefinery processes because of similar compositions and fractionation behaviour. Furthermore, different pretreatments could be applied for the achievement of specific fractionation results, depending on the desired solid or liquid by-product to obtain.

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1. The pretreatment of the lignocellulosic biomass

The pretreatment of the lignocellulosic feedstock is, probably, the single most crucial step in the biorefinery processes, since it has a large impact on the yield and efficiency of the subsequent treatments (Alvira et al., 2010; Galbe and Zacchi, 2012). It mainly consists on the disruption of the lignocellulosic structure by using chemical, physical and/or mechanical treatments, in order to enhance the separation and removal of its constituting components. This pretreatment stage should have a low capital and operational cost, be effective on a wide range of feedstocks and result in the recovery of most of the lignocellulosic components in a useable form in separate fractions (Agbor et al., 2011).

The current research, concerning lignocellulose pretreatment processes, comprises the study of several and different technologies and reagents (Table 1) with the aim of achieving high yield and cost-efficient pathways for the comprehensive exploitation of lignocellulosic feedstocks.

The most commonly used pretreatment methods are the so-called hydrothermal processes, which consist in the treatment of the biomass with aqueous mixtures of solvents at different reaction times and temperatures. These methods can be roughly classified into neutral, acidic or basic pretreatments (Galbe and Zacchi, 2012). An increase in temperature reduces significantly the pretreatment time. It is also clear that, at neutral pH values, the pretreatment

becomes longer as compared to the requirements at more extreme pH values. Furthermore, pH affects the ratio and selectivity of the dissolved lignocellulosic components. Regarding this fact, the hemicelluloses solubilisation resulted favoured at low and neutral pH (Alvira et al., 2010) whereas better lignin degradation occurred at extreme pH conditions (very high or low pH values).

A severe fractionation stage usually entails several associated issues, such as carbohydrate losses or inhibiting compounds formation, which could lead to worse yields in the following enzymatic/biological production of biofuels and biochemicals. Therefore, the successful and efficiency of the applied pretreatment must be accomplished by the proper selection of treatment conditions for raw materials that present different recalcitrance. An overview of the different fractionation processes applied in the present work, their performance principles, the used operation conditions and the methodology used for the analysis and recovery of product in each case are described below.

1.1. Alkaline treatments

Pretreatments with alkali such as NaOH, KOH, Ca(OH)₂, hydrazine and anhydrous ammonia (Agbor et al., 2011) cause swelling of biomass, increasing its internal surface area and decreasing crystallinity and degree of polymerisation of the cellulose. Alkaline pretreatment disrupts the lignin structure and breaks the linkage between lignin and the carbohydrate fractions, which increases the reactivity of the remaining polysaccharides as delignification occurs (Pedersen and Meyer, 2010).

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Table 1
Advantages and disadvantages of different pretreatment methods.

| Method ^a | Type | Advantages | Disadvantages | Feedstock |
|---------------------|-------------------|--|--|---|
| DAH | Chemical | Good removal of hemicelluloses | Formation of degradation products, corrosion | Agricultural residues |
| OS | Chemical | Causes lignin and hemicelluloses hydrolysis | High cost, used solvents need to be drained and recycled | Agricultural residues and woody biomass |
| MIC | Chemical | Low energy requirement, no corrosion, suitable for lignin and hemicelluloses removal | Time expensive, some saccharide losses | Agricultural residues and woody biomass |
| SE | Physical chemical | Cost-effective, high glucose yields, lignin and hemicelluloses removal | Formation of inhibitors and toxic compounds | Agricultural residues and woody biomass |
| AFEX | Physical chemical | Low formation of inhibitors and high accessible surface area | High cost due solvent, not effective for high lignin containing feedstocks | Agricultural residues |
| MIL | Mechanical | Reduces cellulose crystallinity | High power and energy consumption | Agricultural residues |
| IL | Chemical | Effective dissolution of all lignocellulose components, low degradation products | Expensive technology and recovery required | Agricultural residues and woody biomass |
| COEX | Physical chemical | Increases accessible surface area, cost-effective | Does not affect lignin and hemicelluloses, very high pressure requirements | Agricultural residues |

Adapted from [Alvira et al. \(2010\)](#) and [Pedersen and Meyer \(2010\)](#).

^a DAH, diluted acid hydrolysis; OS, organosolv; MIC, microbial; SE, steam explosion; AFEX, ammonia fiber explosion; MIL, milling; IL, ionic liquids; COEX, CO₂ explosion.

The alkali pretreatment results effective for agricultural residues and herbaceous crops. The reason is the smaller amount of lignin present in these types of materials ([Galbe and Zacchi, 2012](#)). Due to the severity of the alkaline media, high reaction temperatures are usually not required (below 140 °C). This fact, together with the low cost of the commonly used reagents, makes the alkaline treatments easily implementable at industrial scale. However, during these extraction processes, non-desirable reactions occur, such as solubilisation of extracts, dissolution of phenolic compounds, and ashes neutralisations and, therefore, the recovery and subsequent treatment of the resulting liquid and solid fractions could result more laborious and expensive due to purification steps requirement ([Egüés et al., 2012](#)).

1.2. Autohydrolysis

Through the autohydrolysis of lignocellulosic materials, the partial or total solubilisation of hemicelluloses can be achieved. The protons generated by the auto-ionisation of water act as catalysts, disrupting the acetyl groups present in xylans ([Egüés et al., 2012](#)), thus, forming acetic acid that improves the hemicelluloses hydrolysis. The solid fraction resulting from the autohydrolysis, with high cellulose and lignin contents, could be employed as feedstock for bio-ethanol production, in ruminant feed and pulp and paper making, but also for energy generation ([Feria et al., 2011](#)). This auto-catalysed pretreatment is usually carried out between 140 and 210 °C with a residence time that can vary from a few minutes to one hour ([Agbor et al., 2011](#); [Galbe and Zacchi, 2012](#)). These moderate conditions allow to avoid the hemicelluloses degradation and inhibitors formation. The absence of reagents and the low reaction time used makes this fractionation process to be an economic and appropriate method for the industrial pretreatment of herbaceous feedstocks.

1.3. Organosolv processes

Organosolv pretreatment consists in extracting lignin with organic solvents or their aqueous solutions, increasing the cellulosic fraction digestibility. It has been investigated for several woody and non-woody biomasses and resulted effective for hemicelluloses/lignin degradation and cellulose crystallinity reduction ([Geng et al., 2012](#)). Based on the fact that lignin can be solubilised in certain solvents, it can be recovered and used as starting point for chemicals or as fuel ([Galbe and Zacchi, 2012](#)). These

processes can be also classified into neutral, acidic or basic treatments depending on the used organic solvent. Alcohols, organic acids, amines, ketones, phenols and other solvents ([Agbor et al., 2011](#); [González Alriols et al., 2009](#)), could selectively and efficiently extract lignin in a wide range of temperature (100–250 °C). For economic and environmental reasons, most of the used organic solvents need to be recovered, as well as for avoiding potentially inhibitory effects on further enzymatic hydrolysis or fermentation of resulting streams ([Alvira et al., 2010](#); [Galbe and Zacchi, 2012](#)).

2. The use of alternative lignocellulosic biomass

The success of lignocellulose fractionation process obviously depends on the applied conditions (temperature, reaction time, solvents, chemicals, etc) but considerably on the feedstock composition and its treatability or recalcitrance, i.e., how easy is to delignify the raw material and to make more accessible its cellulosic structure for subsequent treatments (digestibility). The recalcitrance is directly related to the crystallinity and polymerisation degree of cellulose, the accessible surface area, the protection of cellulose by lignin and its sheathing by hemicelluloses, the fibre strength, porosity and coarseness of the raw material ([Agbor et al., 2011](#); [Alvira et al., 2010](#)). Chemically, woody biomass presents higher lignin content than agricultural biomass and thus woody biomass results more recalcitrant to microbial and enzymatic digestion than non-woody biomass. The major differences between woody (forestry crops or fruit tree pruning) and non-woody biomass (straw, grasses or stalks) are their physical properties and chemical compositions. Woody structure is physically larger, structurally stronger and denser than agricultural biomass ([Geng et al., 2012](#)).

Wood has been the main industrially used feedstock for pulp and paper production ([Egüés et al., 2012](#)). Nevertheless, forest preservation is becoming compulsory and, therefore, new solutions for reducing CO₂ emissions and deforestation are needed. Renewable lignocellulosic feedstocks, such as crop residues, constitute a suitable option as raw material to produce paper pulp and cellulose–fibre based products ([González Alriols et al., 2009](#)). In this context, the use of new non-edible lignocellulosic materials is a strategic objective for the manufacture of chemicals and fuels. These raw materials constitute an interesting and promising alternative for the replacement of fossil fuels ([González Alriols et al., 2009](#); [Requejo et al., 2012](#)). Lignocellulosic feedstock is composed

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