

## Ethanol from Nordic wood raw material by simplified alkaline soda cooking pre-treatment

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### HIGHLIGHTS

- ▶ Ethanol prod via alkaline pretreatment enables a coproduction of valuable lignin.
- ▶ Lignin content of 2.5% on pulp was found optimum for aspen and 5% on pulp for pine.
- ▶ Aspen required lower alkali charge and lower temperature compared to pine.
- ▶ Yield gain of fermentable sugars is 1–2% charging AQ(0.1% on wood) in pretreatment.
- ▶ Ethanol yield between 81.6% and 87.8% on theoretical max was obtained.

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### ABSTRACT

Ethanol production from lignocellulosic raw materials will generate multiple streams, since only a certain fraction of the material can be converted into sugars and then fermented to ethanol. This requires a 'poly-generation' approach, where by-products also must have high value (e.g. lignin, sugars from hemicellulose). To reach the large scale required for profitability, it is proposed that the best way is to integrate the new processes with existing industries, preferably those that already operate biomass-to-materials or biomass-to-fuels plants. One of the largest industry branches in this respect is the pulp and paper industry. Production of second generation ethanol (or other products) via sugars from lignocellulosic materials includes a relatively costly pre-treatment of the raw material in order to separate the lignin from the cellulose. This separation of the wood components already takes place in the chemical pulp mill, and the long proven technology in pulp production known as soda cooking (pre-treatment under alkaline conditions) is further evaluated in this study. It can be directly integrated into the recovery of chemicals and energy in the pulp mill. The pre-treatment of the lignocellulosic material studied in this work is alkaline and sulphur-free, and results in a technically pure cellulose to be fed to the hydrolysis stage, which makes it different compared to most of the other processes that aim to produce ethanol from lignocelluloses. The process chain from enzymatic hydrolysis to ethanol is very similar to that being used today for grain ethanol.

The aim of this study was to define the conditions in alkaline pre-treatment stage for the separation of wood to a carbohydrate fraction for hydrolysis and ethanol production, and to a lignin fraction for the production of lignin products. Aspen (*Populus tremula*) and pine (*Pinus sylvestris*) wood from Nordic mills were studied. The reference case was alkaline pre-treatment according to the well-known soda pulping technique. The pulps of alkaline pre-treated aspen could be enzymatically hydrolysed very efficiently and fermented to ethanol with high yields (82–88% ethanol yield from theoretical maximum). It should be possible to use raw material of lower quality and cost than wood from the pulp industry. However, it can then be important to be able to take out non-process elements (NPEs) that otherwise accumulate in the process. This can be done by introducing an acidic prehydrolysis stage prior to the alkaline fractionation. The content of Mg and Mn ions in the wood was possible to reduce by 85–90% and Ba and Ca ions by 75–80%. Potassium was virtually completely removed during the acidic pre-treatment stage.

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## 1. Introduction

The demand for liquid biofuels in Europe is heavily increasing. At the same time the pulp and paper industry undergoes a change that leads to an increased need of finding new product niches. A major barrier for the deployment of wood-based fuel-ethanol is its high production cost. In particular, the pre-treatment is one of the most expensive processing steps in cellulosic biomass to fermentable sugars conversion. Thus, the wood pre-treatment step, preceding the hydrolysis and fermentation steps, has a great potential for improvement. In an alkaline pre-treatment process, wood can be fractionated to relatively pure carbohydrate and lignin fractions by the well-known soda cooking and kraft processes. The solid fibrous carbohydrate fraction can be further subjected to enzymatic hydrolysis, fermentation and distillation for the production of ethanol. "Black liquor" i.e. spent pre-treatment liquor, may be processed to sulphur-free lignin (if soda cooking is used) and energy, see Fig. 1 [1].

Besides the ethanol a nearly sulphur-free lignin with low ash content (1% or lower) and high dryness (65–70%) can be recovered as a potentially valuable by-product that can be used as a fuel or as an intermediate for chemicals production. The kraft cooking process that is the most used method in pulp mills today gives a lignin containing sulphur, but if the separation of the wood components is performed using the soda cooking process there is a possibility to obtain a lignin that is sulphur free. This is an advantage in some future possible applications of the lignin as a high-value added product, e.g. carbon fibres.

The process will also give the possibility to separate the hemicelluloses of the wood as a third product besides the sugars and lignin. The hemicelluloses can be used as fibre additive, gas barriers, hydrogels and thermoplastics.

Harmsen et al. [2] prepared a literature review on physical and chemical pre-treatment processes for lignocellulosic biomass as part of the BioSynergy project. This review concluded that organosolv and alkaline pre-treatment, are the most suitable pre-treatment processes if one wants to produce lignin in sufficient quality for the production of chemicals. In most other processes the lignin generally remains with the cellulose fraction, giving an impure lignin product containing un-hydrolysed sugar polymers as well as other organics. However, the organosolv process has a high cost of organic solvents, necessitating high solvent recovery, which causes increased energy consumption. Mosier et al. [3] reviewed process parameters and their fundamental modes of action for different pretreatment methods such as steam explosion, liquid

hot water pre-treatment, acid pre-treatment and alkaline pre-treatment. He concludes that pre-treatment processing conditions must be tailored to the specific chemical and structural composition of the various, and variable sources of lignocellulosic biomass.

The objectives of this study were to define the conditions in the alkaline pre-treatment stage for the separation of wood to a carbohydrate fraction for the ethanol production and to a lignin fraction. Also combinations with ethanol or anthraquinone addition and with acidic pre-treatment were studied, and a comparison between hardwood and softwood raw material was made.

## 2. Materials and methods

Wood of aspen (*Populus tremula*) and pine (*Pinus sylvestris*) from Swedish mills were used. Aspen and pine logs were laboratory chipped, screened, dried and hand sorted at Innventia. The chips were analyzed with respect to lignin content, carbohydrate composition and extractives, see Table 1.

### 2.1. Alkaline pre-treatment

Alkaline pre-treatment was carried out according to the well-known sulphur-free soda cooking process with sodium hydroxide (NaOH) as active chemical. 100 or 300 g dry-weight chips were charged per batch in 0.8 and 2.5 L steel autoclaves, respectively. The autoclaves were evacuated for 30 min and the alkaline pre-treatment liquor was sucked into the autoclaves so that a liquor-to-wood ratio of 4:1 was reached. An N<sub>2</sub>-pressure of 0.5 MPa was applied for 30 min. The pressure was levelled out before placing the autoclaves in a glycol bath at 120 °C for 35 min. The temperature was increased by 5°/min to pre-treatment temperature, 170 °C for aspen wood and 180 °C for pine wood. The conditions used in the alkaline pre-treatment stage are reported in, e.g. Table 2.

#### 2.1.1. Alkaline pre-treatment with ethanol addition

The addition of alcohols to the soda cooking enhances the rate and selectivity of delignification [4,5]. In kraft cooking a positive effect of the alcohol addition on the delignification has been found [6]. The effect of alcohol on the delignification was explained mainly as a change in the solubility parameters of the pulping medium and not as an alternation in the chemical reaction of lignin.

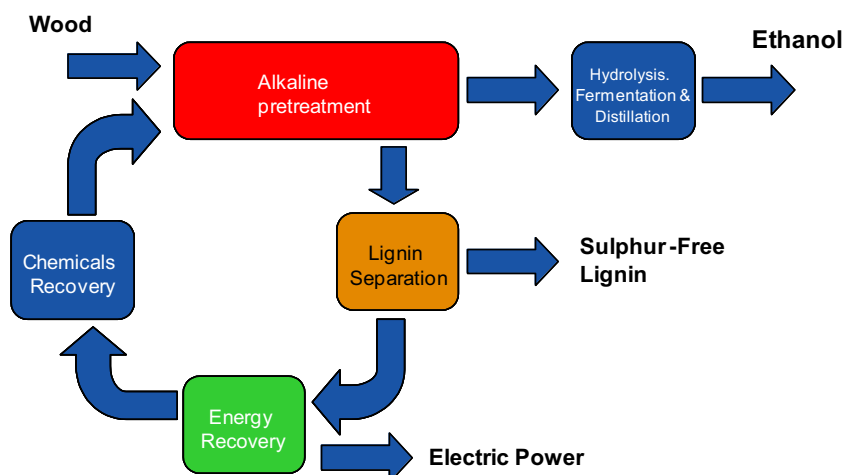


Fig. 1. Conceptual design of the alkaline pre-treatment with ethanol production.

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