Chemical Hydrolysis of *Fagus sylvatica* Wood: Dilute Acid vs. Alkaline Treatment

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Background and Aim

Lignocellulose present in plants such as wood, grasses and agricultural residues, is the most abundant source of molecules required for production of fuels and high value - added products. Lignocellulosic biomass is composed of three polymers: cellulose, hemicellulose and lignin. Cellulose is a non-branched polymer consisting of glucoses (hexoses). Hemicellulose is a complex carbohydrate containing xylose (pentose) as the main sugar. Lignin is a biopolymer with aromatic alcohols as basic monomeric units. Due to complex polymeric structure, lignocellulosic materials are resistant to chemical treatment. A number of methods are harnessed to efficiently hydrolyse lignocellulose, amongst which dilute acid and alkaline treatment, are the most common. The objective of this study was to evaluate the effect of dilute acid and alkaline treatment on hydrolysis rate of polymeric components in *Fagus sylvatica* wood.

Materials and Methods

❖**Hydrolysis process:** Wood material was soaked in 3 % H_2SO_4 or 7 % NaOH solution at a liquid/dry matter of 20/1 in double-necked boiling flasks situated on heating plates. After soaking, the temperature and time of hydrolysis was set to 100 °C and 1h. During hydrolysis, stirring was continuously applied and set at 150 rpm. After 1 h, hydrolysis was stopped by removing flasks from heating plates and allowed to cool at room temperature. The pulps were filtered under vacuum and hydrolysates were collected for further analysis. Solid remnants were washed with distilled water and collected for sugar analysis.

❖**Sugar content analysis:** Sugars content in solids was determined after 1 h of prehydrolysis of solid samples in 72 % H_2SO_4 at 30 °C followed by 3h hydrolysis in 4 % H_2SO_4 at 100 °C. Sugars were determined as alditol acetates by Gas Chromatography, equipped with FID detector.

❖**Lignin analysis:** Solid samples were hydrolysed with 72 % H_2SO_4 for 1 h at 30 °C followed by 1h hydrolysis in 4 % H_2SO_4 at 121 °C in autoclave. Subsequently, solids were transferred onto filtering crucibles, washed with distilled water and dried at 105 °C to constant weight for Klason lignin determination. The filtrate was used to measure acid soluble lignin by means of UV spectrophotometer at 205 nm.

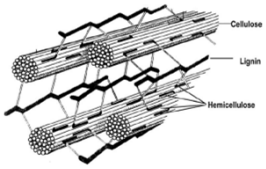
❖**Furan analysis**: Concentrations of 2 – furfural and hydroxymethylfurfural in dilute acid hydrolysate were determined by means of High Performance Liquid Chromatography, equipped with DAD detector set at 284 nm.

❖**Extractives analysis:** Raw material was extracted with hot water at 100 °C and hot ethanol in a Soxhlet apparatus.

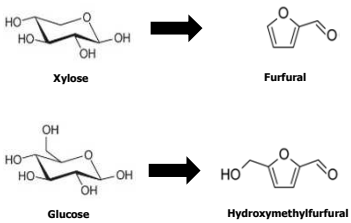
❖**Ash analysis:** Ash content was determined after combustion of samples at 525 °C for 4 h.

Results

Fagus sylvatica

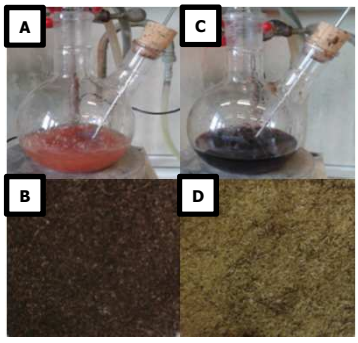


Lignocellulose structure



Component	Content* [%]
Water extractives	4.0 ± 0.1
Ethanol extractives[1]	1.1 ± 0.1
Glucose	48 ± 2.0
Xylose	18 ± 1.0
Klason lignin	20 ± 1.0
Acid soluble lignin	3.7 ± 0.3
Ash	0.38

*Expressed on dry material basis
[1]After water extraction



A – Dilute acid hydrolysis
B – Material after dilute acid hydrolysis
C – Alkaline hydrolysis
D – Material after alkaline hydrolysis

Removal %	Dilute acid	Alkaline
Xylose	71	59
Glucose	4	0
Klason lignin	0	11

Dilute acid treatment	
Compound	Release* [%]
Hydroxymethylfurfural	0.10
Furfural	0.03

*Expressed as % of dry material

Conclusions

- 1 h hydrolysis at 100 °C with the use of 3 % H_2SO_4 resulted in 71 % removal of xylose and 4 % removal of glucose with Klason lignin remained intact.
- The presence of sugar degradation products: 2 - furfural and hydroxymethylfurfural was detected in dilute acid hydrolysate.
- 1 h hydrolysis at 100 °C with the use of 7 % NaOH caused 59 % xylose removal and 11 % removal of Klason lignin with no effect on glucose.
- Dilute acid hydrolysis proved to be more efficient in removing xylose but alkaline hydrolysis additionally showed to remove Klason lignin.