A PRELIMINARY STUDY ON THE DELIGNIFICATION OF WOOD BLOCKS VIA MICROWAVE ASSISTED ATMOSPHERIC ORGANOSOLV METHOD FOR THE PRODUCTION OF HOLOCELLULOSIC SCAFFOLDS

TUFAN SALAN, M. HAKKI ALMA

KAHRAMANMARAS SUTCU IMAM UNIVERSITY, DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING, FACULTY OF FORESTRY, KAHRAMANMARAS, TURKEY

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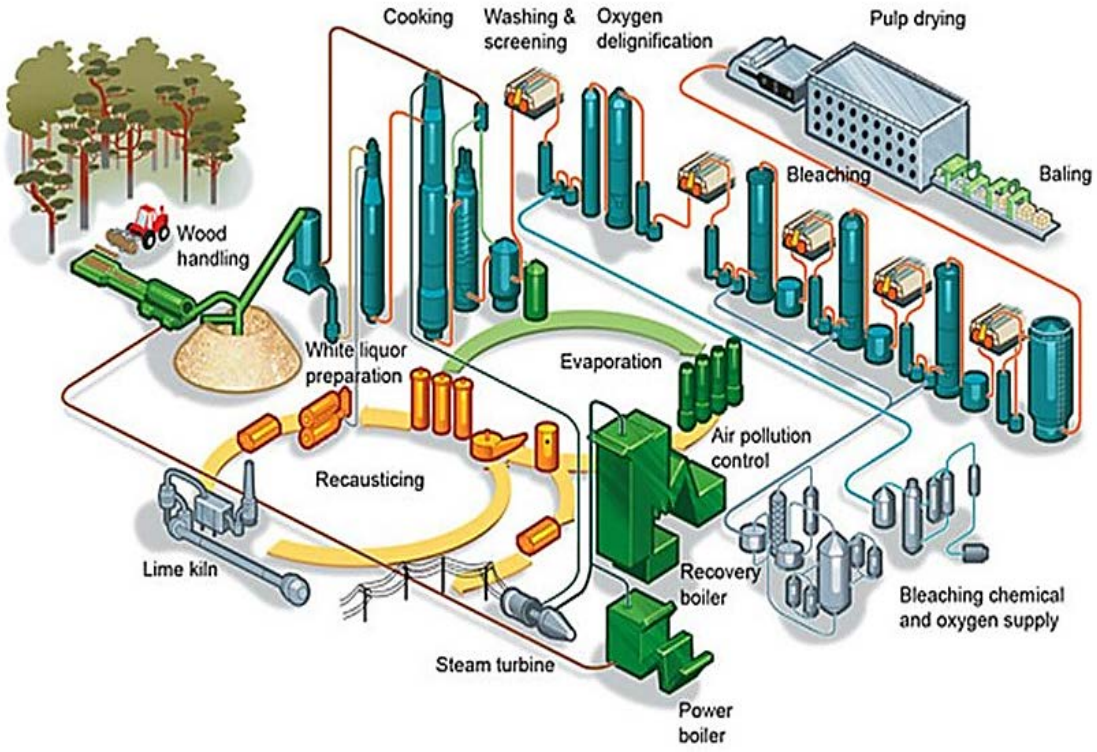
Delignification of Wood

Wood Decay by White Rot Fungi

Traditional Kraft, Soda, and Sulfite Processes



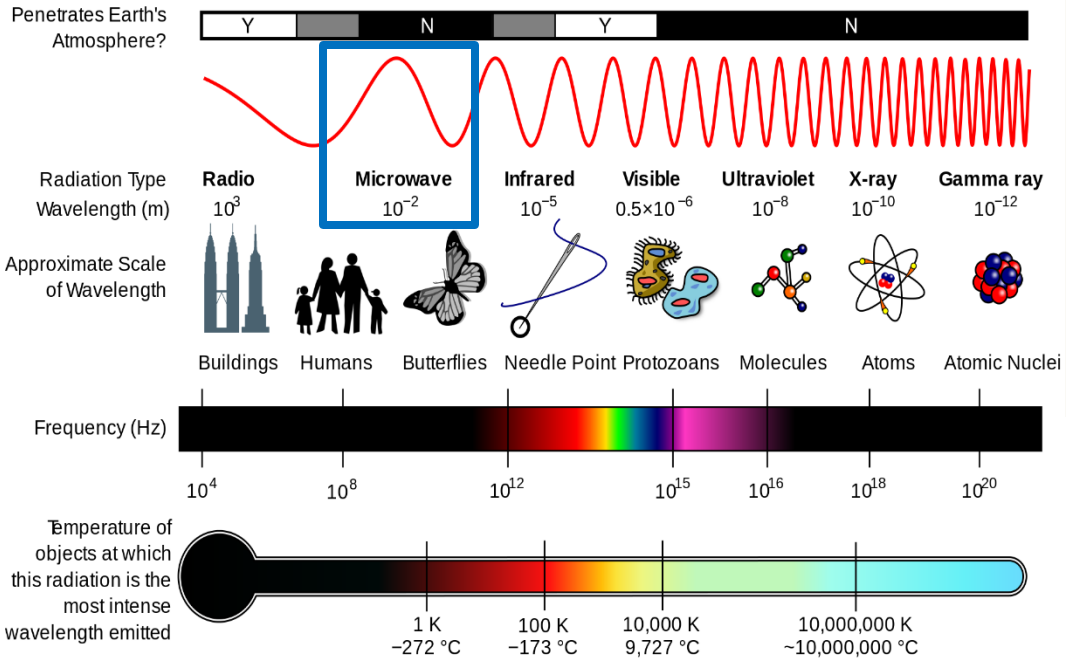
Pulp and Paper Industry

- **The traditional kraft, soda, and sulfite processes have long been used for delignification of wood in the pulp and paper industry.**
- **Important drawbacks: air and water pollution due to organic sulphur and chlorine bleaching compounds…**

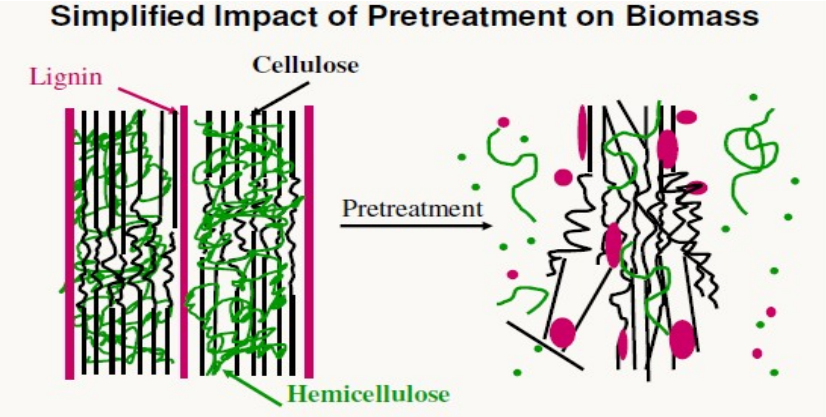
Delignification of Wood

- **Due to environmental concerns, organosolv delignification has attracted much interest as an environmental-friendly method.**
- **However, the organosolv process requires high temperatures, long reaction times, and high pressures.**
- **Due to these challenges, some scientists have conducted microwave energy to enhance the lignin extraction yield via the efficient internal heating with rapid reactions.**

Electromagnetic Spectrum

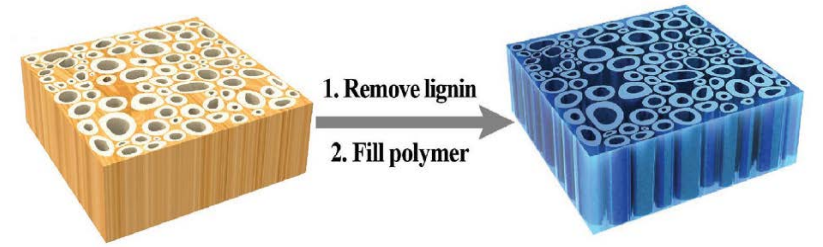


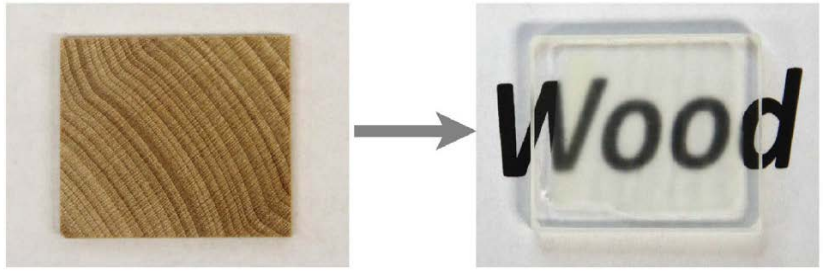
Pre-treatment for the bio-fuel production

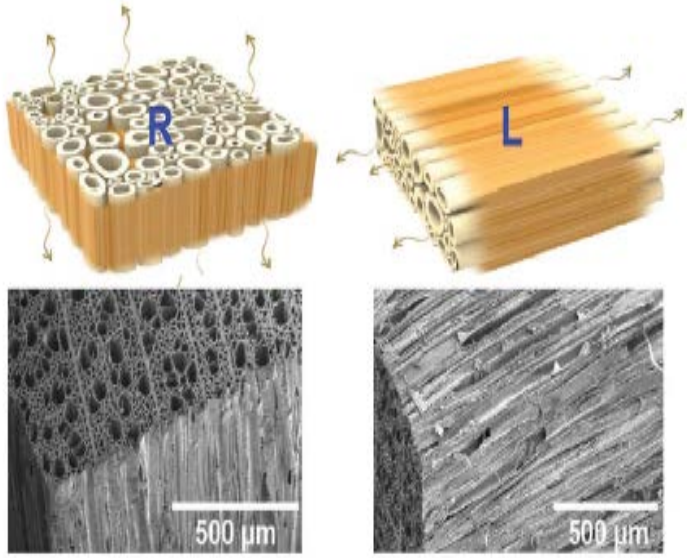


Delignification of Wood

- Most recently, two innovative studies (Li et al. (2016) Biomacromolecules 17(4), 1358-1364 ; Zhu et al. (2016) Adv. Mater., 28(26): 5181–5187) take the delignification processes a step further in this year.
- For the first time, transparent wood composites were produced by removing the lignin and filling the holocellulose structure with a polymer or epoxy resin to obtain high optical transparency.
- Furthermore, structural hierarchy, oriented cell structure, and the nanoscale cellulose fibre network of wood tissue were well preserved.







Source: Zhu et al. (2016) Adv. Mater., 28(26): 5181–5187.

Materials

- **The objective of this study was to evaluate the effects of the microwave enhanced organosolv delignification of wood blocks on some properties of holocellulosic template/scaffold without dissolving or degrading cellulose for potential wood/polymer composite applications.**

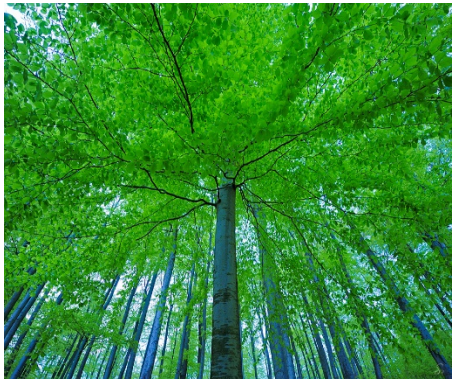
Poplar (Pop)

Longitudinal (L)

Radial (R)

Organic Solvents

- **Water**
- **Ethanol**
- **Glycerin**



Beech (Bch)

Longitudinal (L)

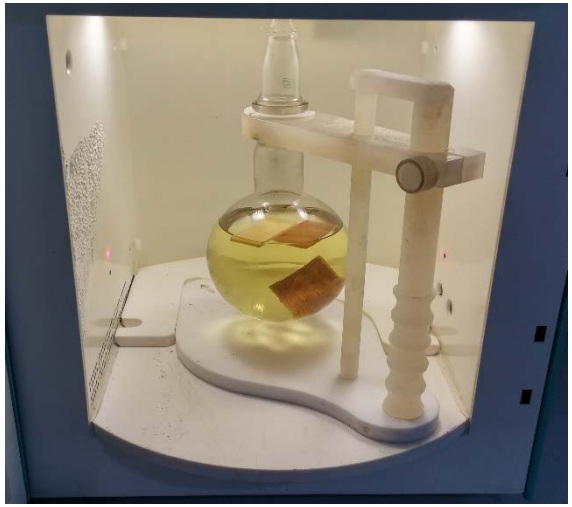
Chemicals Used

$NaOH/Na_2S$

Methods

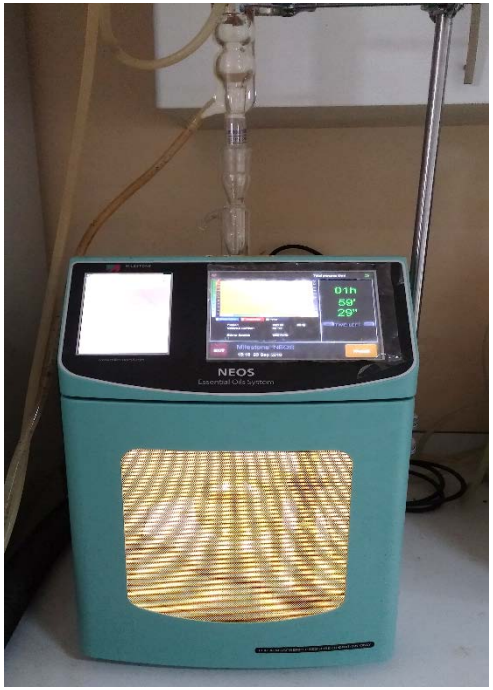
The samples were immersed in:

- Aqueous solution of ethanol (75 %, v/v) with a sulfidity of 20 % and 50 %
- Aqueous solution glycerin (85 %, v/v) with a sulfidity of 20 %
- Water solution of $NaOH/Na_2S$ with a sulfidity of 40 %



The samples were extracted in:

- A microwave oven (NEOS) at the power level of 350 W for 2 h under reflux and continuous stirring.



- At the end of the reaction, samples were separated from extraction solution and washed with the aqueous ethanol.
- The wood blocks were then placed in a bleaching solution of H_2O_2 and treated in the microwave oven at the power level of 150 W for 1.5 h.
- The weight loss (WL) percentage of the wood blocks was calculated as a degree of delignification for different experiments.

Results

- The visual appearance of the wood blocks after the delignification indicated that the lignin, colored polymer of the wood, was removed and wood blocks turned into a yellowish color.
- After that with the effect of bleaching process the color of the wood blocks becomes lighter and whiter due to the colorless cellulose template.

Beech Wood (L)

Before Delignification

After Delignification

Poplar Wood (L)

Before Delignification

After Delignification

Poplar Wood (R)

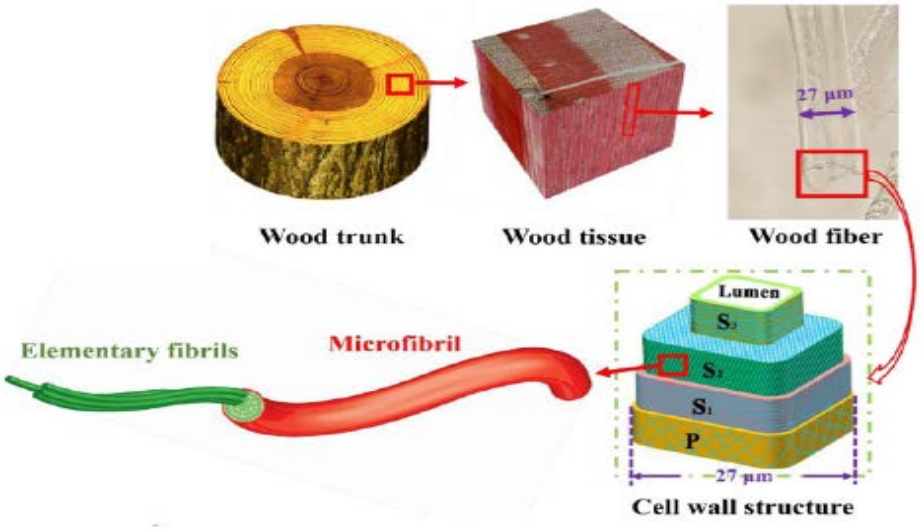
Before Delignification

After Delignification

Appearance of the Solvents After Delignification

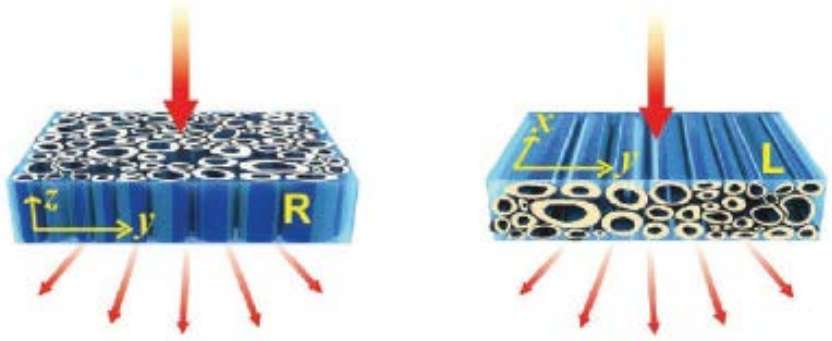
Results

Sample	WL (%)	Sample	WL (%)
Pop-W	8.81	Bch-W	14.87
Pop-E50	11.1	Bch-E50	17.98
Pop-E20	14.35	Bch-E20	21.7
Pop-Gly	12.55	Bch-Gly	15.78
Pop/R-E50	19.73		
Pop/R-Gly	17.72		



Source: Fang et al. (2014) Nano Letters 14(2): 765-773.

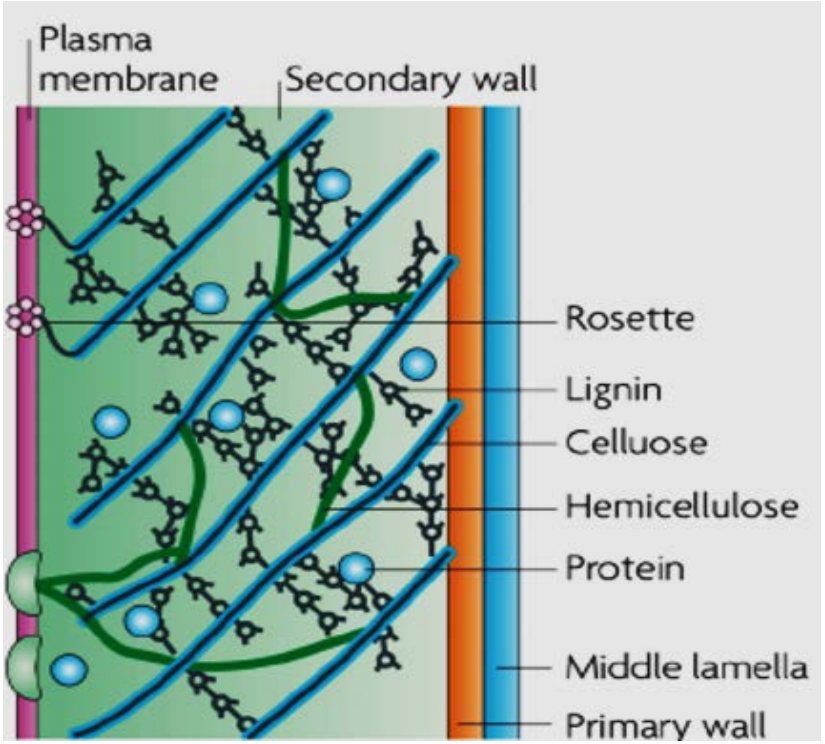
- The highest delignification was obtained via aqueous ethanol solution with the sulfidity of 20 for L-wood samples.
- WL of beech samples was more than poplar.
- When the WL in the R-wood and L-wood of poplar was compared, it was very critical that degree of delignification substantially increased.
- The difference in fiber directions led to different mass transfer behaviour, where lignin could be extracted out much more easily in R-wood due to the cell lumina perpendicular to the plane with a short depth same as the thickness of the wood block.



Source: Zhu et al. (2016) Adv. Mater., 28(26): 5181–5187.

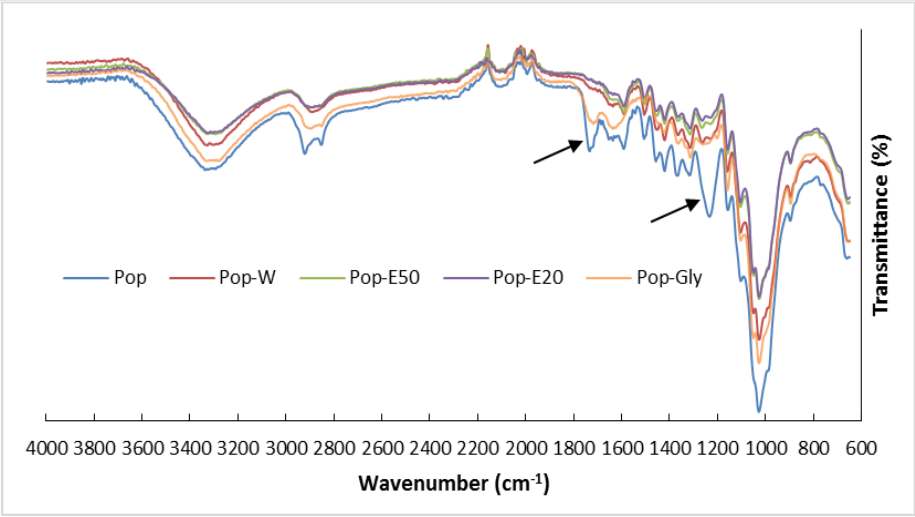
Results

Sample	Shore D	Sample	Shore D
Pop	46.7 ±3.9	Bch	59.7 ±4.5
Pop/R	42.1 ±3.6	Bch-W	54.8 ±3.1
Pop-W	43.7 ±2.7	Bch-E50	50.1 ±3.7
Pop-E50	36.2 ±4.2	Bch-E20	52.1 ±3.6
Pop-E20	37.8 ±2.2	Bch-Gly	49.3 ±5.2
Pop-Gly	38.7 ±3.3	Pop/R-Gly	36.5 ±2.5
Pop/R-E50	34.2 ±3.1		

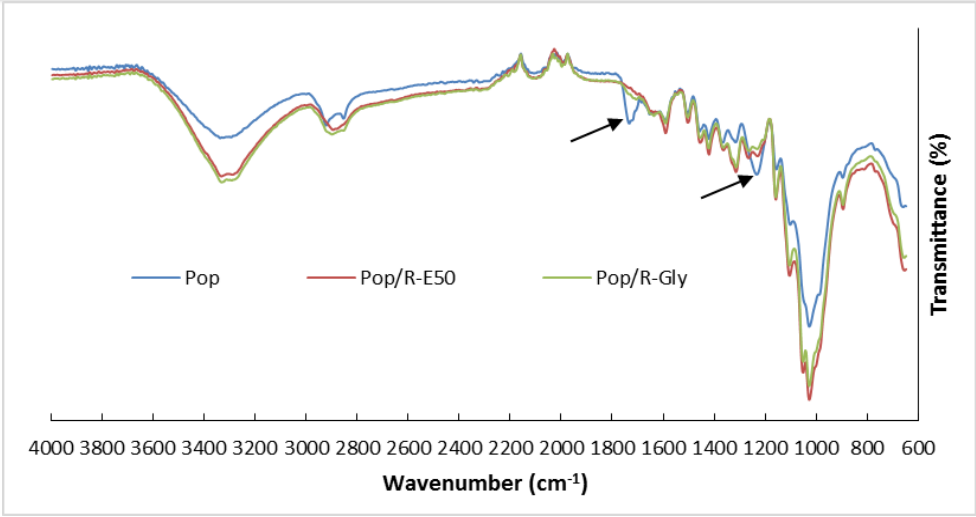


- The lignin polymer fills the spaces in the cell wall between cellulose, hemicellulose and pectin, and supplies the mechanical strength and rigidity of plant walls.
- The importance of this phenomenon was proven with the results of hardness tests.
- The Shore D surface hardness values of the samples diminished correlatively with the delignification degree of samples.

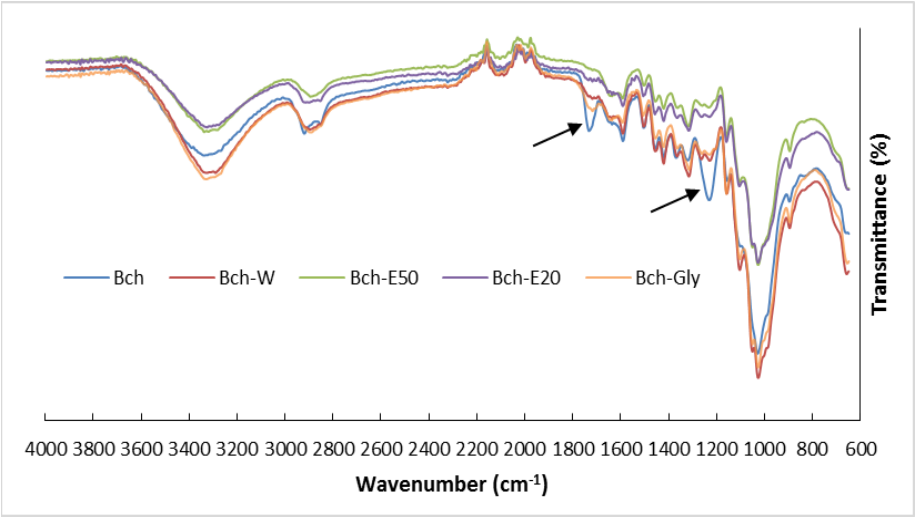
Results



Poplar Wood (L)



Poplar Wood (R)



Beech Wood (R)

- **Finally, FTIR spectrum of the samples showed that some alterations occurred in the chemical structure of wood.**
- **Especially, it was noticed that the peaks of C-O stretching and syringly ring of lignin at the wavenumber of 1230 cm^{-1} and C=O stretching of ketones, carbonyl and ester groups at the wavenumber of 1730 cm^{-1} disappeared after delignification.**

Thank You