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Depolymerization of fractionated wood by hydrothermal liquefaction

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Hydrothermal liquefaction of fractionated wood

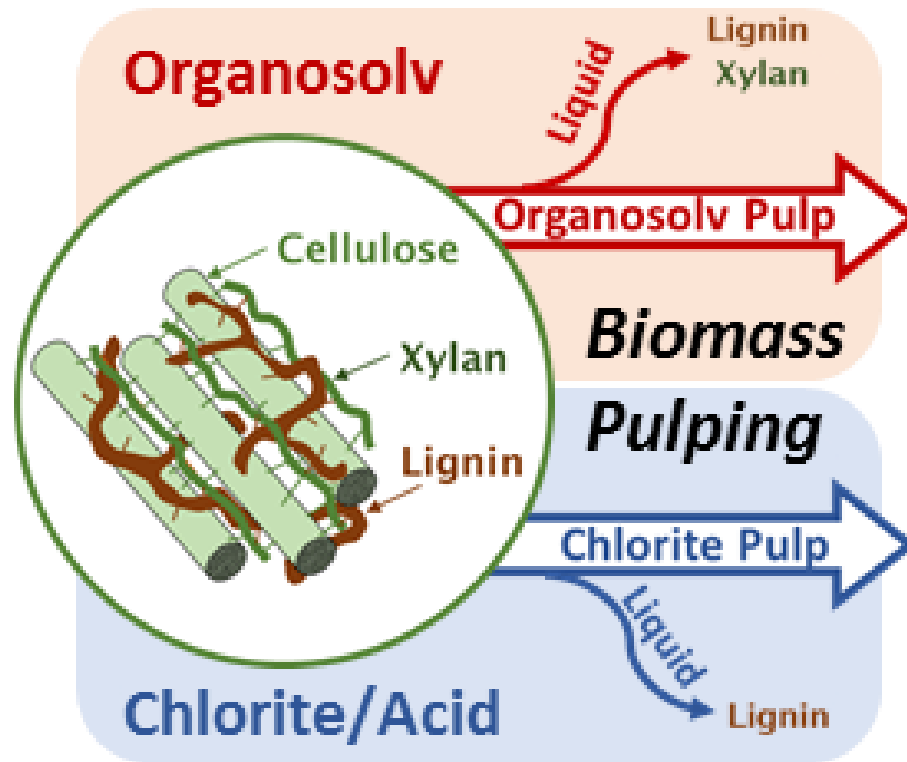
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Yun YU, Hongwei WU, Anthony DUFOUR

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Laboratory of Reactions and Processes Engineering (LRGP)

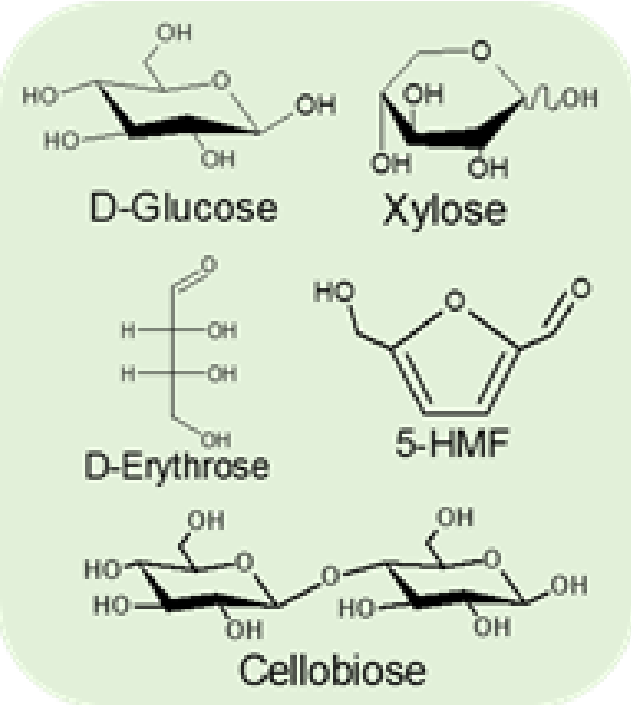
Nancy, France

Hydrothermal liquefaction of cellulosic pulps



Hot-compressed water

Pulp Depolymerization



1. Liquefaction of pure cellulose (operating conditions)
2. Fractionation of beech wood and then hydrothermal liquefaction of the cellulosic pulp
 - Batch reactor
 - Semi-continuous reactor

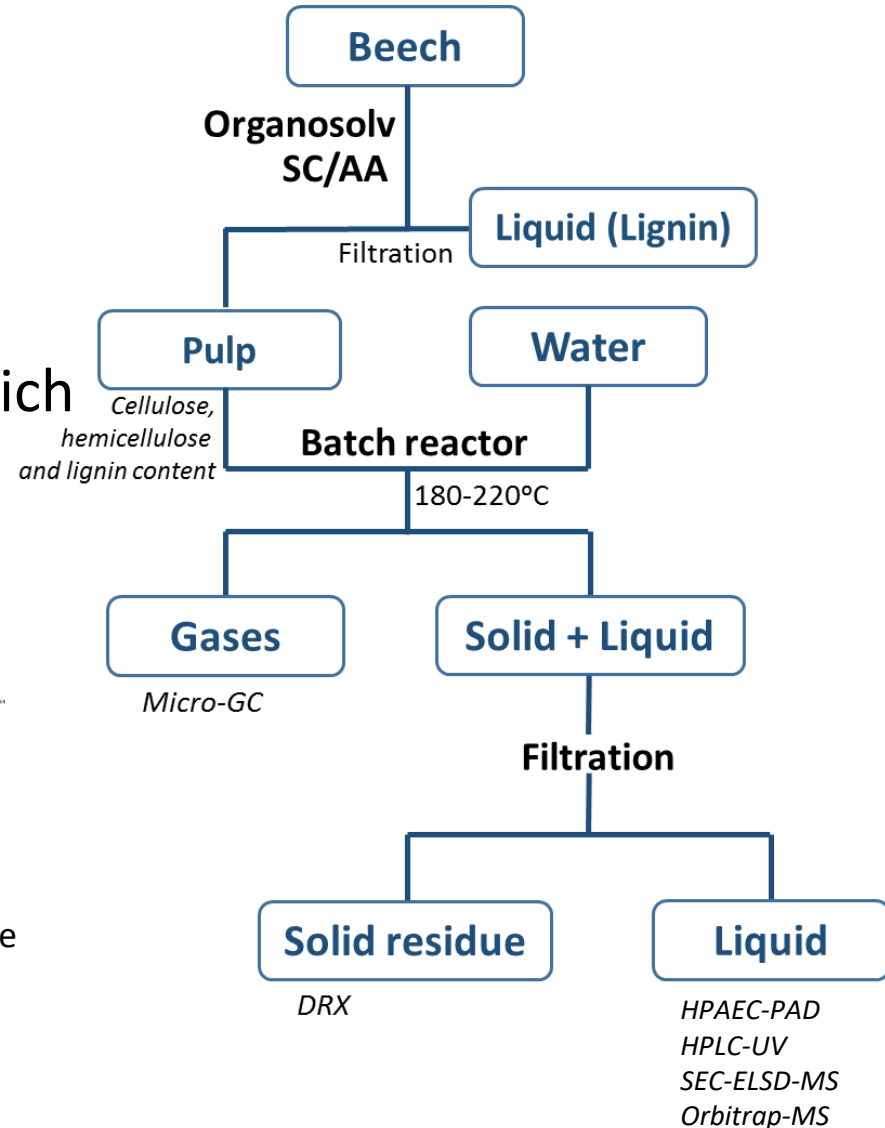
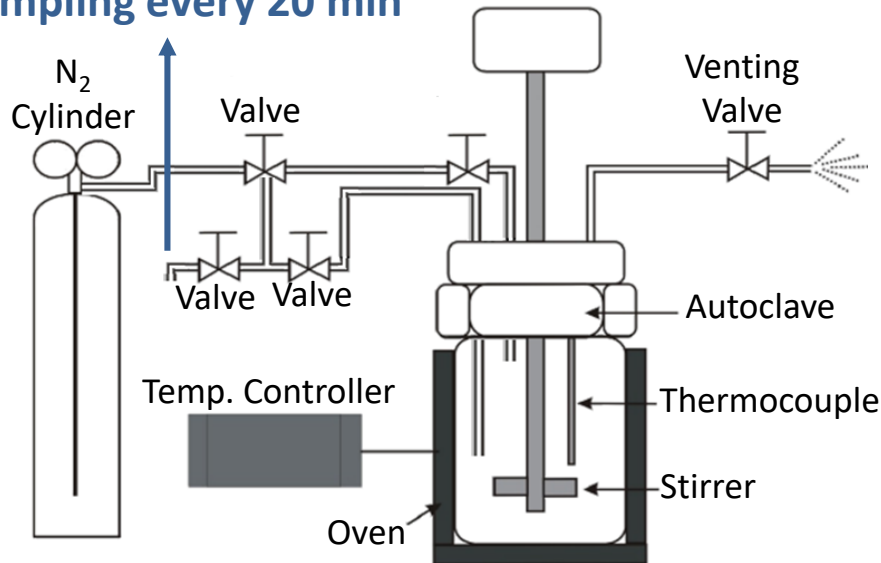
Hydrothermal Liquefaction (Batch)

Fractionating:

- Organosolv (EtOH/H₂O)
180°C, 1h, H₂SO₄
- S. Chlorite/A. Acid
70°C, 6h (glass reactor)

Solid= Cellulosic pulp, Liquid= lignin rich

Sampling every 20 min



Analytical methods

Gas products

- Permanent gases
- **μGC-TCD**

Soluble products

Composition Screening

- **SEC-ELSD/MS**

Carbohydrates/Volatiles

- **Total carbohydrates**
- **HPAEC-PAD**
- **LC-LTQ-Orbitrap-MS**

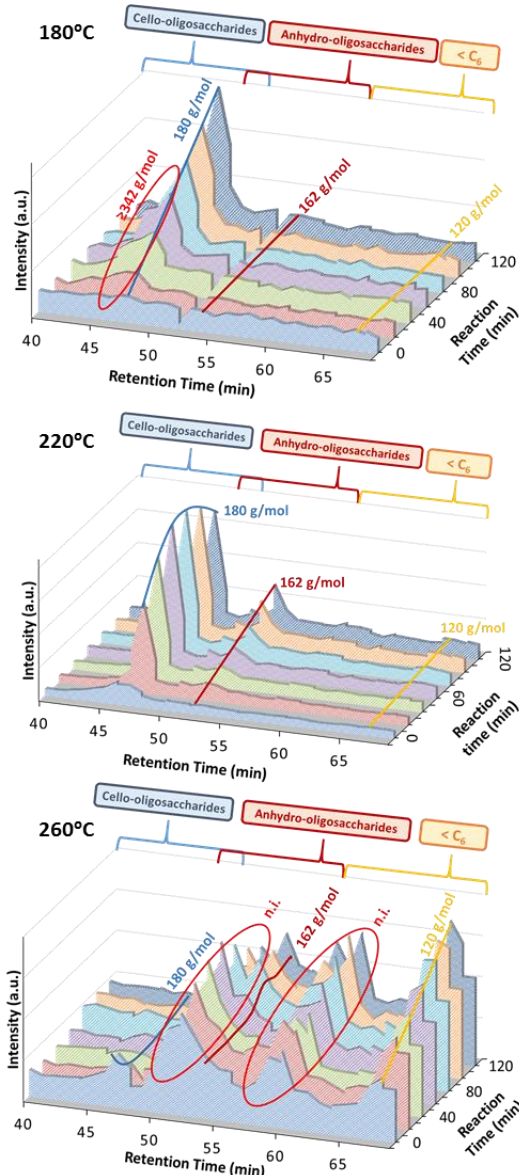
Organic acids

- **HPLC-UV-RID**

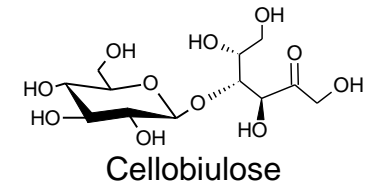
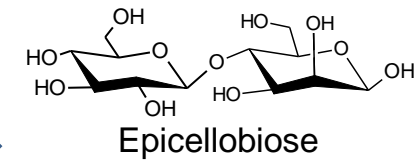
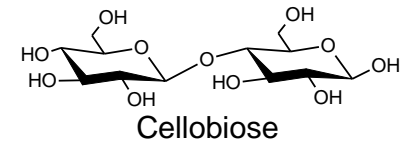
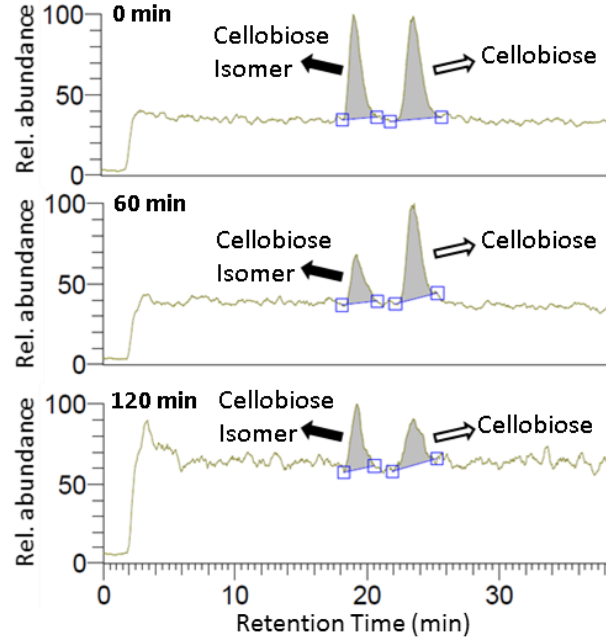
Solid residue

- Structure
- **DRX**

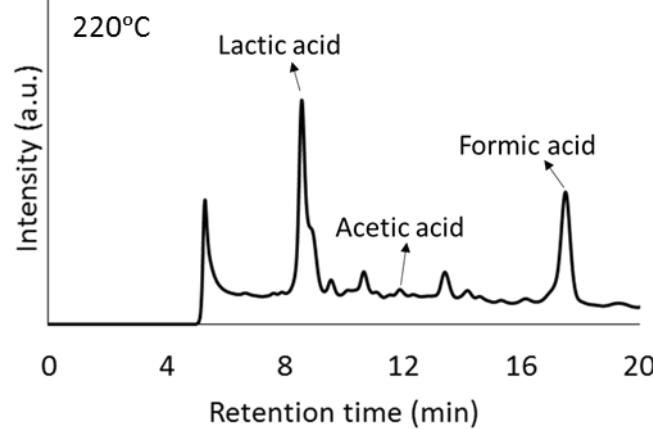
SEC/SEC-MS



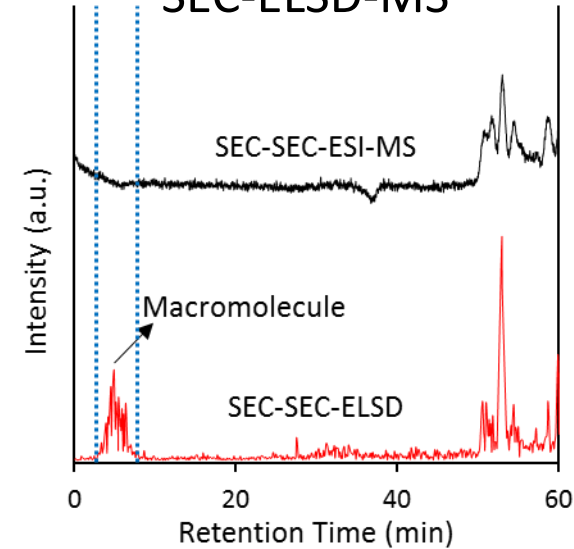
LC-LTQ-Orbitrap MS



HPLC-UV

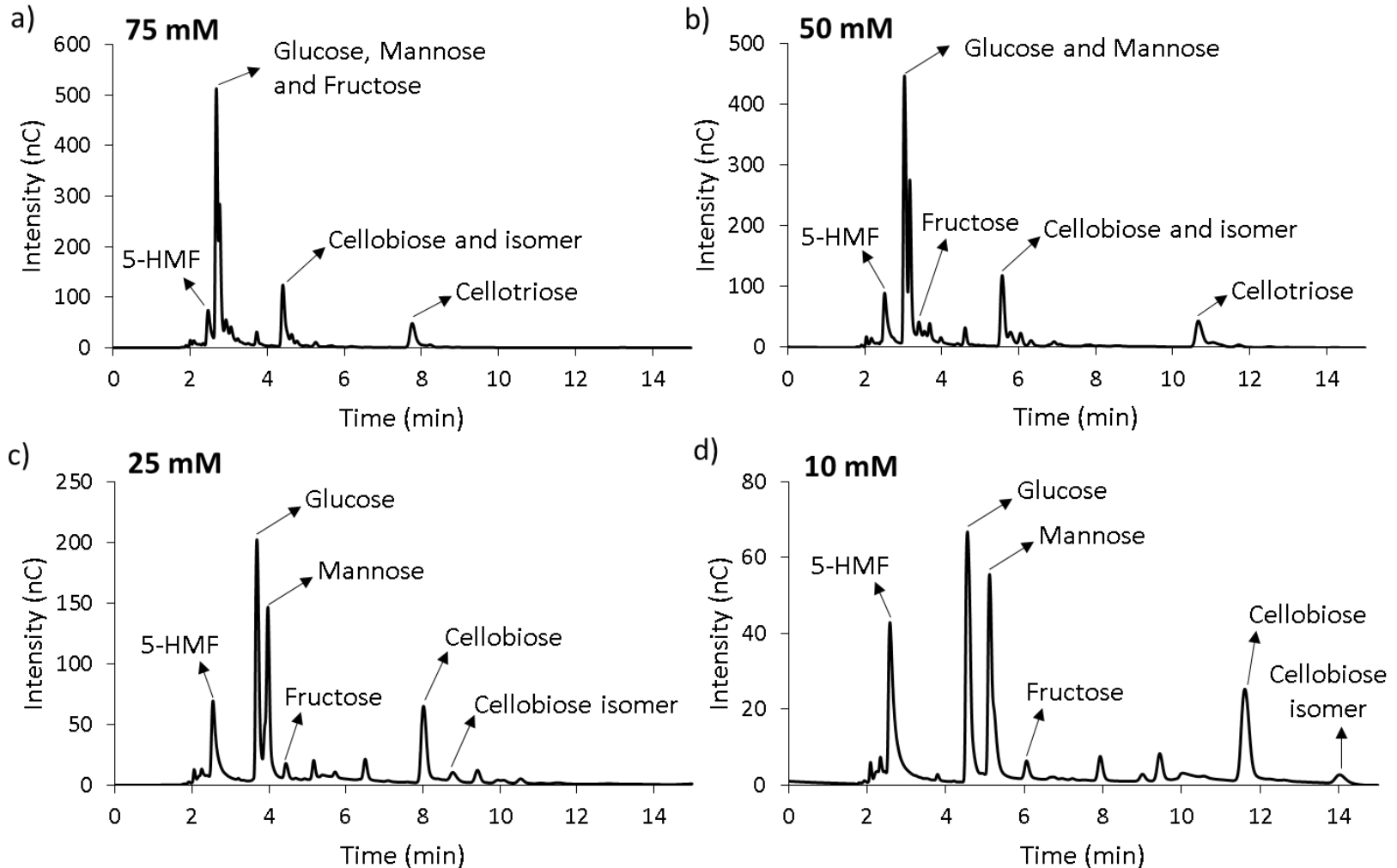


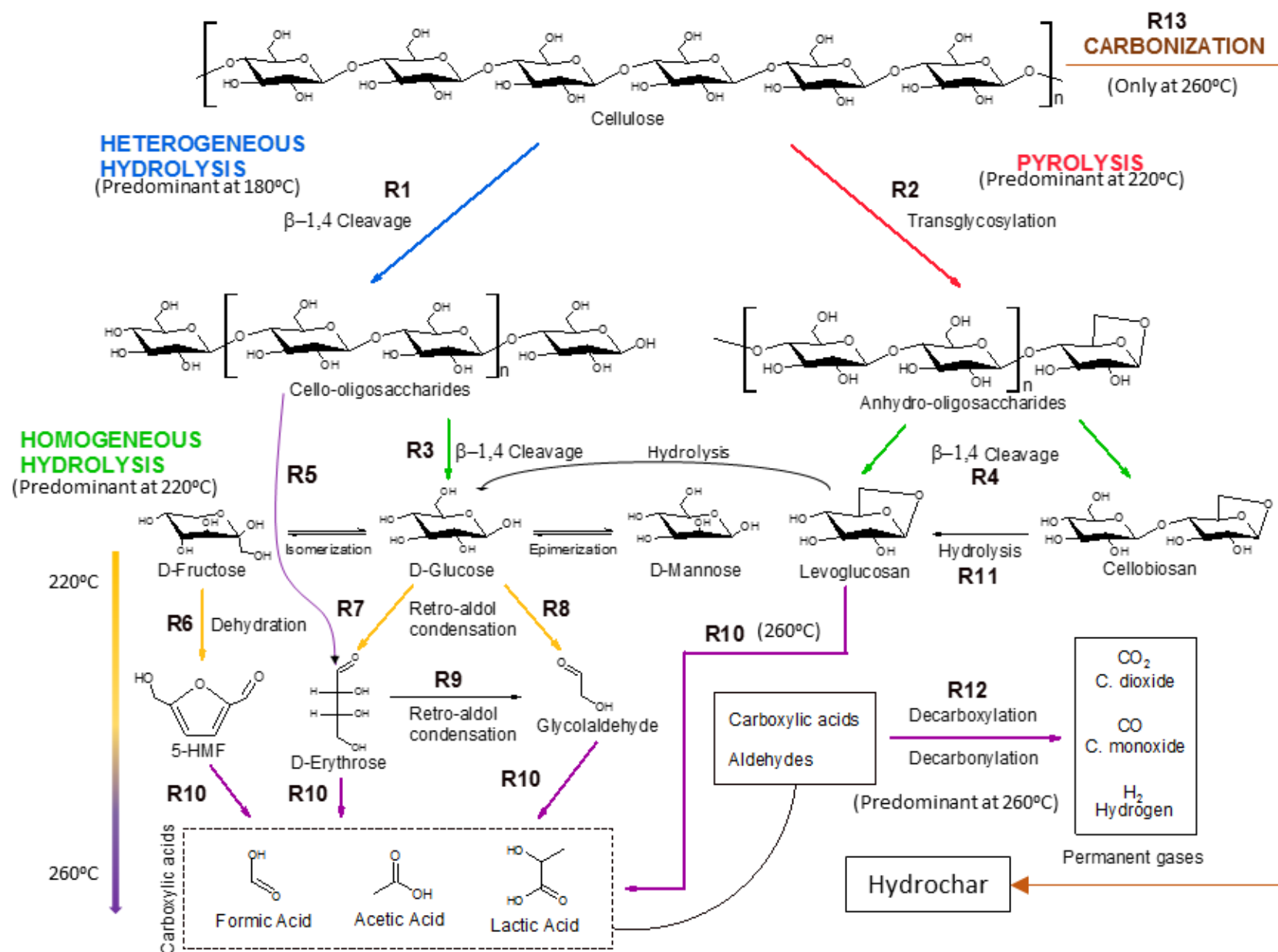
SEC-ELSD-MS



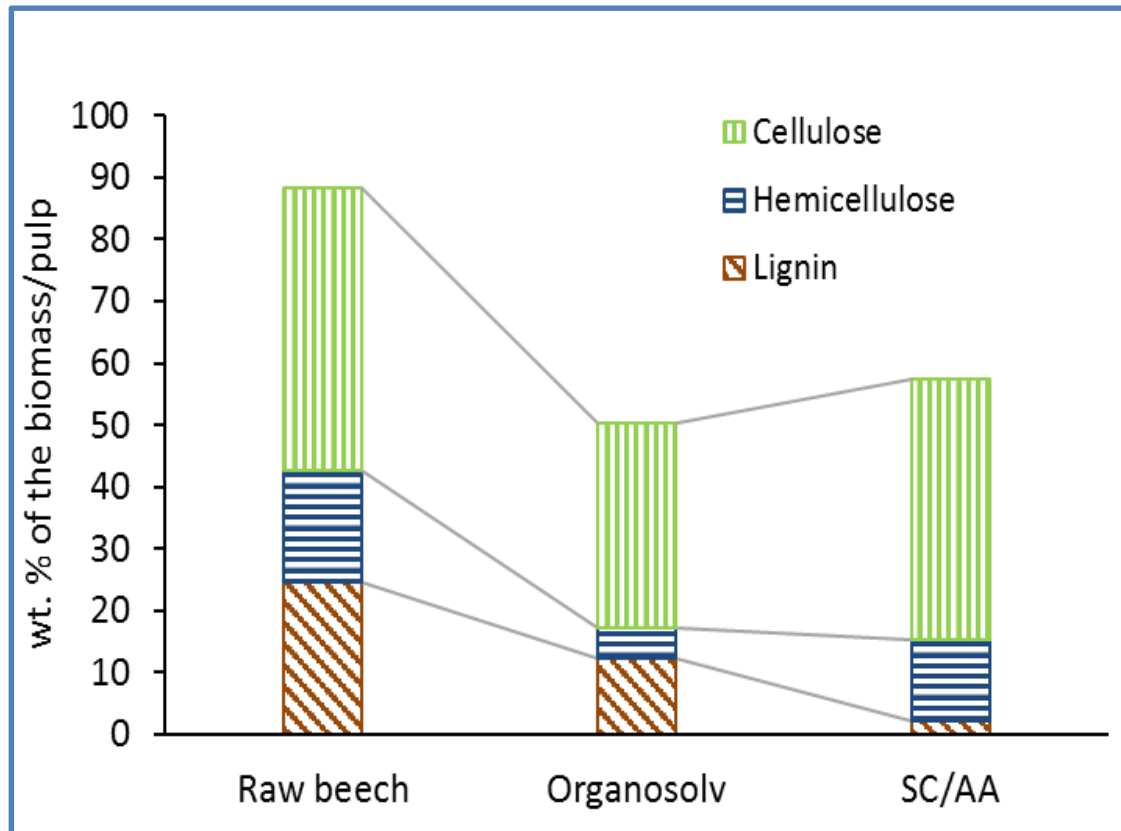
Buendia-Kandia, F., Mauviel, G., Guedon, E., Rondags, E., Petitjean, D., & Dufour, A. (2017). Decomposition of cellulose in hot-compressed water: detailed analysis of the products and effect of operating conditions. *Energy & fuels*.

Analysis of carbohydrates by HPAEC-PAD





Beech wood fractionation



Composition of the solid fraction showing cellulose, hemicellulose and lignin content

Delignification yields

Organosolv = 50.4 wt.%

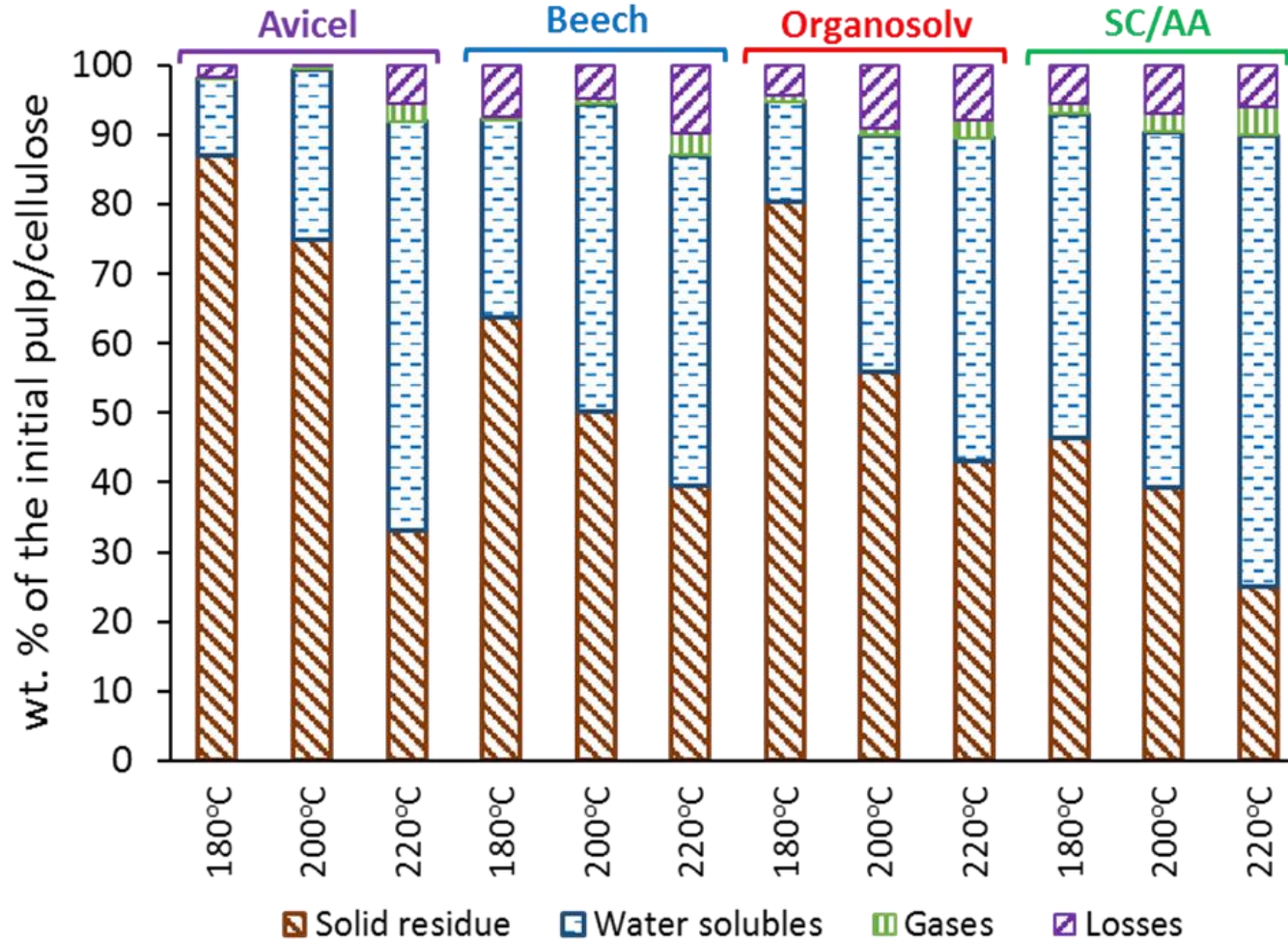
Chlorite/acid = 91.3 wt.%

Cellulose recovery

Organosolv = 62.3 wt.%

Chlorite/acid = 67.2 wt.%

Hydrothermal liquefaction of pulps

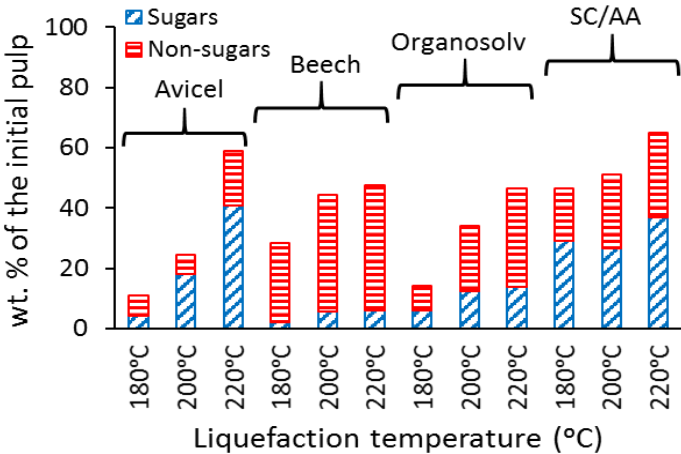


Mass balance after hydrothermal liquefaction

Buendia-Kandia, F., Brosse, N., Petitjean, D., Mauviel, G., Rondags, E., Guedon, E., & Dufour, A. (2019). Hydrothermal conversion of wood, organosolv, and chlorite pulps. *Biomass Conversion and Biorefinery*.

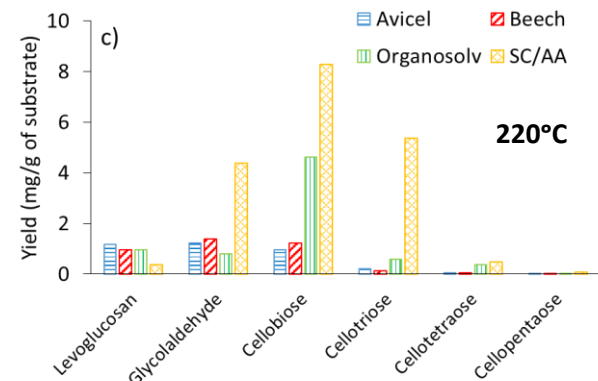
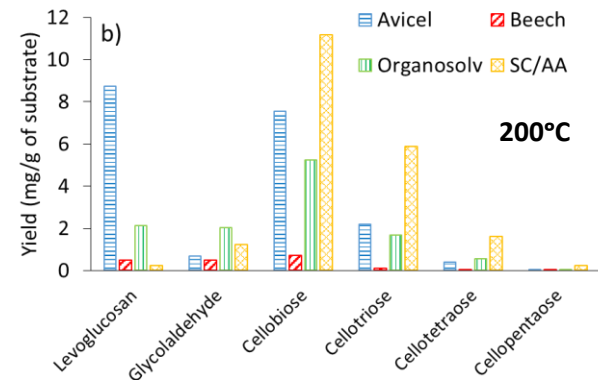
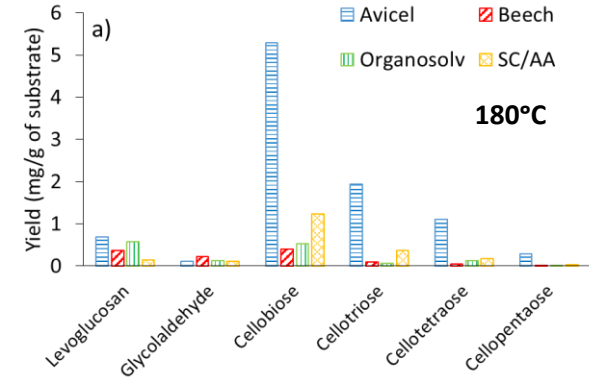
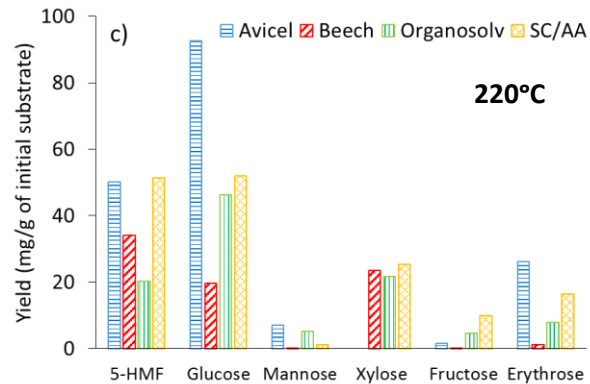
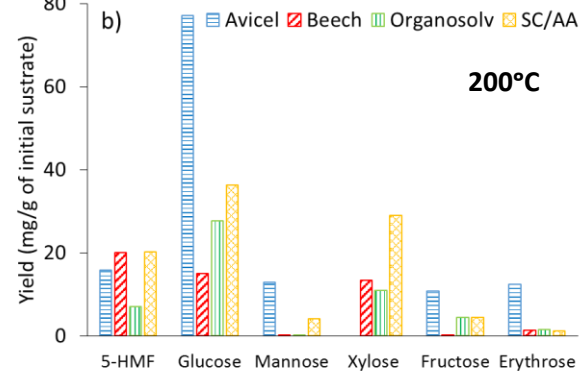
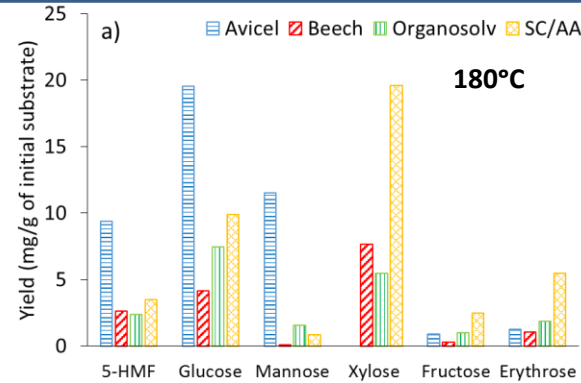
Sugar yield

Total carbohydrates and sugar quantification by HPAEC-PAD after 2 h of reaction



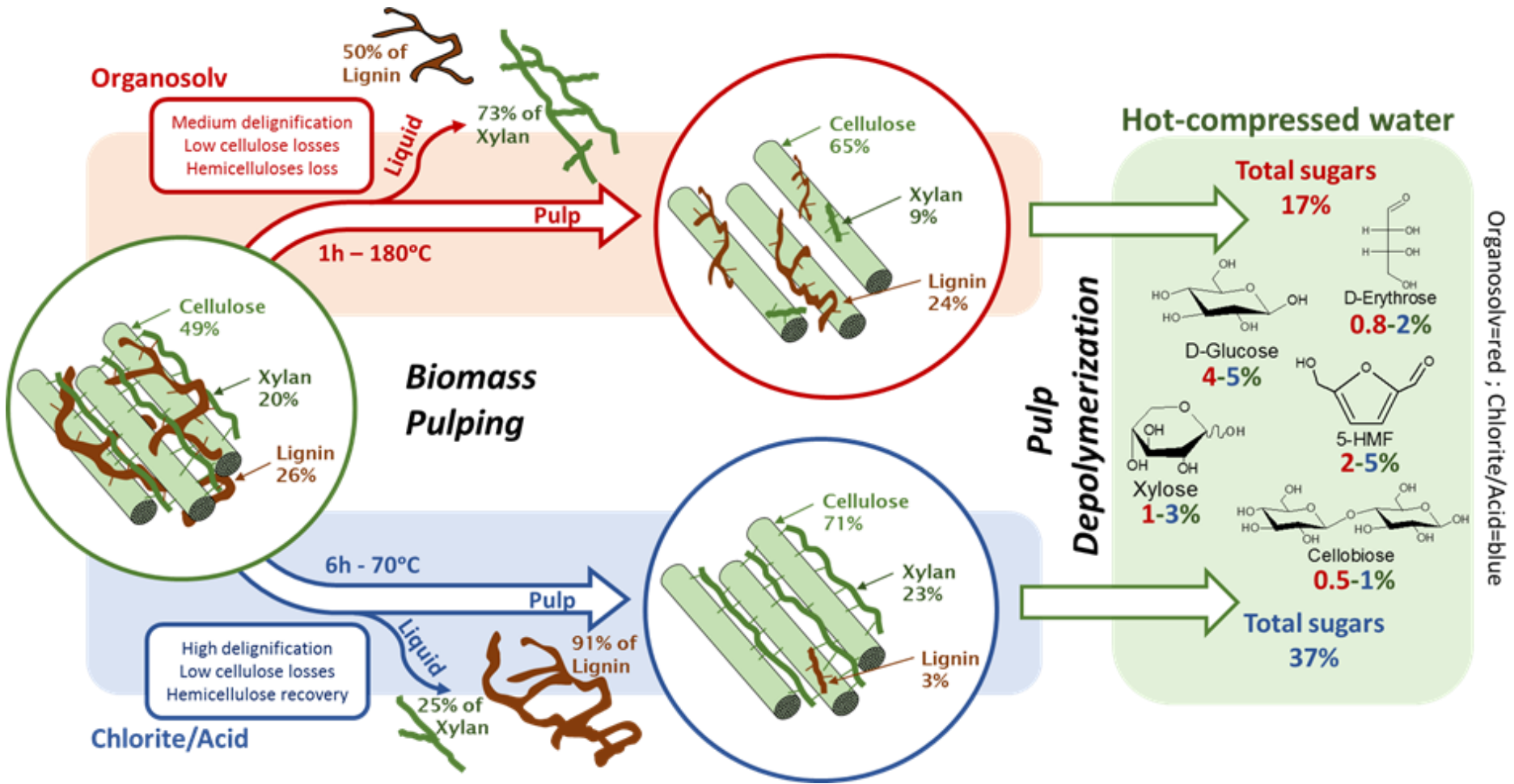
Best case at 220°C:

- Avicel=40.7 %
- Beech=6.1 %
- Organosolv=17.3 %
- SC/AA=36.8 %



Buendia-Kandia, F., Brosse, N., Petitjean, D., Mauviel, G., Rondags, E., Guedon, E., & Dufour, A. (2019). Hydrothermal conversion of wood, organosolv, and chlorite pulps. *Biomass Conversion and Biorefinery*.

From wood to sugars

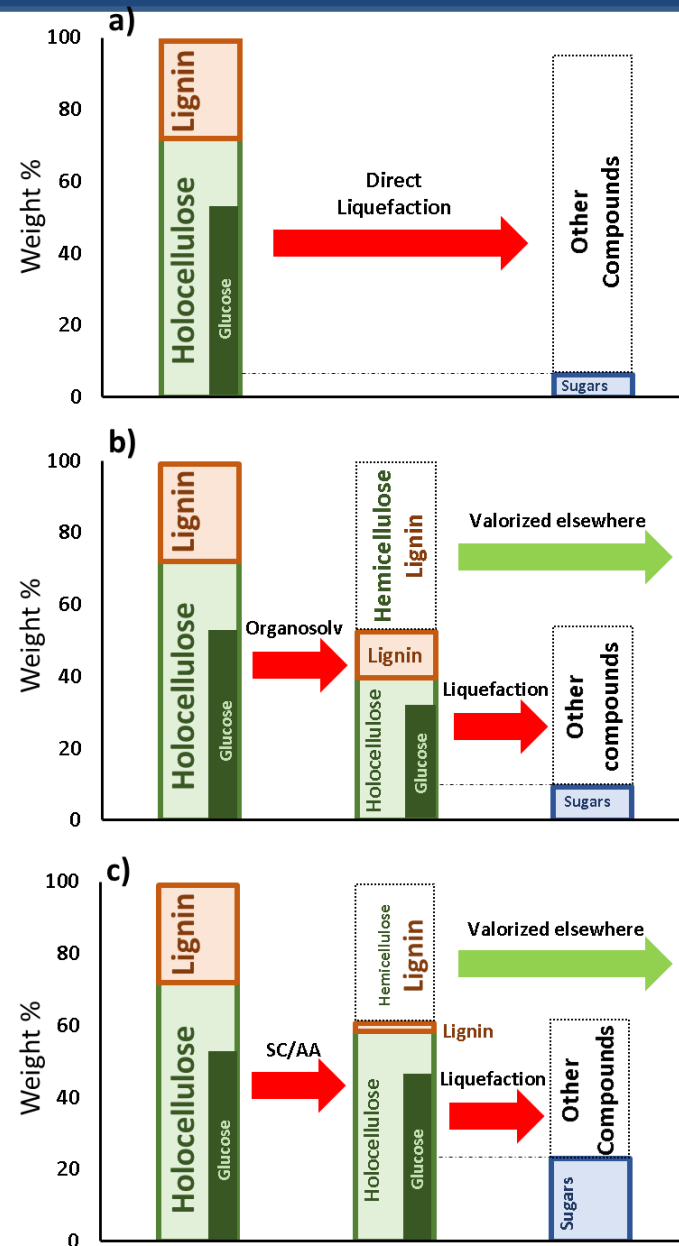


Buendia-Kandia, F., Brosse, N., Petitjean, D., Mauviel, G., Rondags, E., Guedon, E., & Dufour, A. (2019). Hydrothermal conversion of wood, organosolv, and chlorite pulps. *Biomass Conversion and Biorefinery*.

Raw Beech Batch Liquefaction

Organosolv Pulp Batch Liquefaction

SC/AA Pulp Batch Liquefaction



Liquefaction of pulps



Semi-continuous Reactor

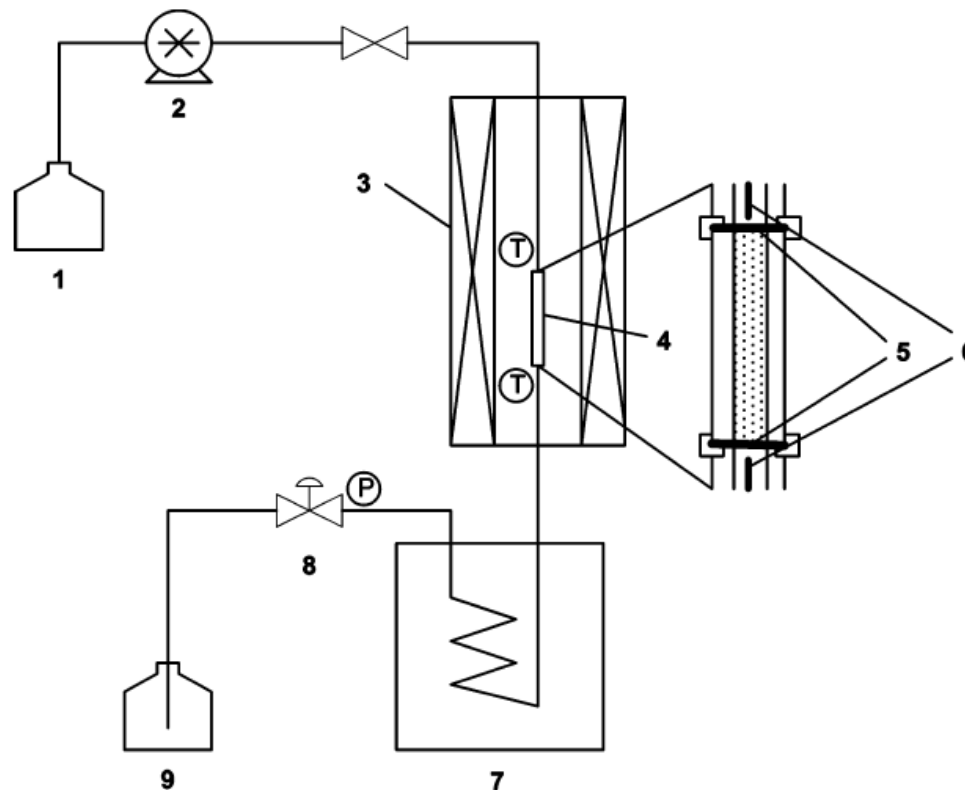
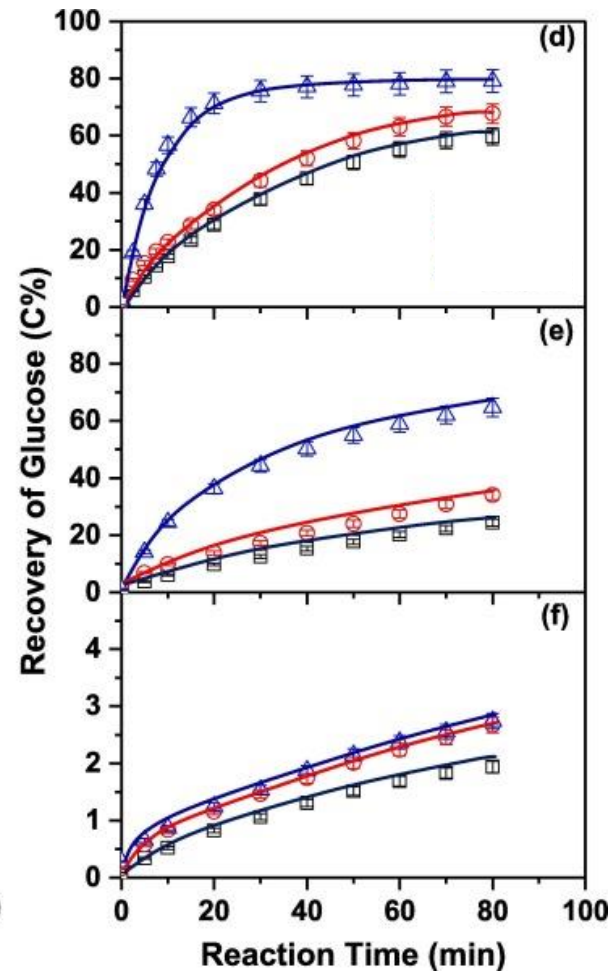
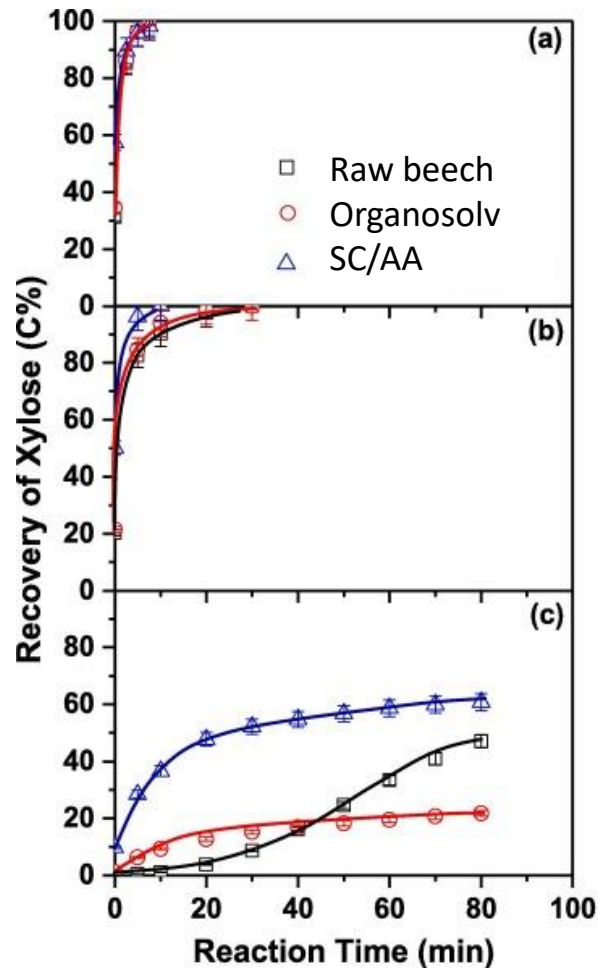


Figure 1. Schematic diagram of the semicontinuous reactor system used in this study: (1) Water reservoir; (2) HPLC pump; (3) infrared image furnace; (4) reactor; (5) sintered stainless steel filter; (6) thermocouple; (7) cooling unit; (8) back-pressure regulator; (9) liquid product collector.

Yu, Y., & Wu, H. (2009). Characteristics and precipitation of glucose oligomers in the fresh liquid products obtained from the hydrolysis of cellulose in hot-compressed water. *Ind. & Eng. Chem. Res.*

Semi-continuous liquefaction

Yields of sugar recovery (relative to the initial sugar content)



250°C

230°C

150°C

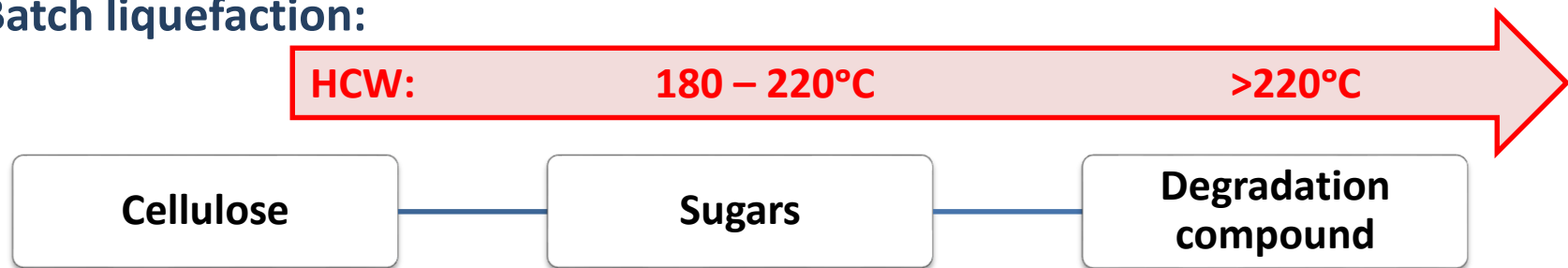
Song, B., Buendia-Kandia, F., Yu, Y., Dufour, A., & Wu, H. (2019). Importance of lignin removal in enhancing biomass hydrolysis in hot-compressed water. *Bioresource technology*

Conclusion

Fractionation:

Delignification yields of 50.4 and 91.3 % were achieved by Organosolv and SC/AA treatments.

Batch liquefaction:



Organosolv and SC/AA liquefaction pulps increased the total sugar yield relative to the raw beech wood, but yields are still low.

Semi-continuous liquefaction:

Around 95 wt.% of xylose and 80 wt.% of glucose can be recovered with temperatures around 250°C.

Thank you

