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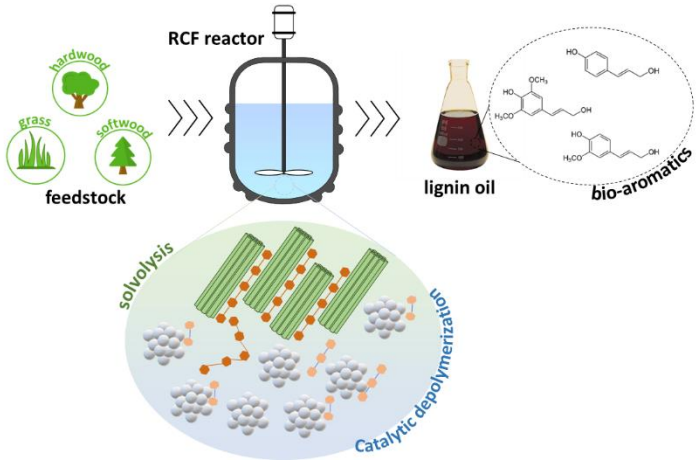
Experimental investigation of the reductive catalytic fractionation of biomass

Aim

The objective of this thesis is to investigate the reductive catalytic depolymerization of different lignin sources in batch reactors. Competitive reaction pathways during depolymerization, i.e. solvolysis and catalytic hydrogenolysis, as well as the role of the catalyst will be assessed through a screening of in-house synthetized catalysts and comprehensive characterization of the product pool.

Justification

Due to depleting oil reserves and the increasing effort to reduce CO_2 emissions, the chemical industry has started shifting towards more sustainable resources and processes. Non-edible biomass such as lignocellulose is one of the most promising alternatives to fossil resources, where the aromatic fraction of lignocellulosic biomass, lignin, represents the most abundant renewable source of aromatics on earth. Reductive Catalytic Fractionation (RCF) is a Lignin-First (*LF*) biorefinery concept that combines biomass fractionation, through solvolytic transformations, with heterogeneously catalysed lignin depolymerization and stabilization [1]. The ultimate goal is the production of added-value building blocks from biomass, e.g. phenolic monomers and dimers, which can be further converted into novel materials or chemicals.



RCF has initially been investigated for raw biomass, aiming for the preservation of native lignin throughout the extraction and ultimately improving the performance of the catalytic depolymerization step. However, potential synergies arise when lignins from engineered biomass (e.g. technical lignin) are considered. To cope with the feedstock heterogeneity, several catalysts configurations are typically proposed, including noble (Pd, Ru) and non-noble (Ni) redox catalysts on various supports. Although it is known that lignin extraction depends entirely on the solvolytic conditions, the depolymerization step comprises two different routes, namely, solvolytic and catalytic hydrogenolysis. Unravelling whether ether bonds are cleaved solvolytically or catalytically is key for a fundamental understanding of the process and catalyst design.

In this thesis, an experimental investigation of the RCF process is proposed. Several operating conditions will be tested, such as lignins from different biomass sources, solvolytic environments, temperature, H_2 source and the catalyst. The effect on the resulting product pool will be assessed via molecular weight (MW) characterization through gel permeation chromatography (GPC), and gas chromatography combined with mass spectroscopy (GCxMS) [2]. Moreover, nuclear magnetic resonance (NMR) analysis will be conducted for further identification of the lignin main inter-linkages and functionalities.

Program

- Literature survey on lignin chemistry, RCF biorefining and analytical methods to characterize lignin oils;
- Lab-scale synthesis and testing of various catalyst configurations;

- Experimental work on lignin catalytic depolymerization to assess on the catalyst role at various operating conditions;
- Comprehensive characterization of the depolymerized lignin oils and kinetic assessment.

References

(1) Renders, T.; Van den Bossche, G.; Vangeel, T.; Van Aelst, K.; Sels, B. Reductive catalytic fractionation: state of the art of the lignin-first biorefinery. *Curr Opin Biotechnol* **2019**, *56*, 193-201. DOI: 10.1016/j.copbio.2018.12.005.

(2) Tolbert, A.; Akinosho, H.; Khunsupat, R.; Naskar, A. K.; Ragauskas, A. J. Characterization and analysis of the molecular weight of lignin for biorefining studies. *Biofuels, Bioproducts and Biorefining* **2014**, *8* (6), 836-856. DOI: 10.1002/bbb.1500.