



**AALBORG UNIVERSITY**  
DENMARK

**Aalborg Universitet**

## **Using isopropanol as capping agent in hydrothermal liquefaction of kraft lignin**

Ahlbom, Anders; Hasani, Merima; Theliander, Hans; Nielsen, Rudi P.; Maschietti, Marco

*Publication date:*  
2022

[Link to publication from Aalborg University](#)

*Citation for published version (APA):*

Ahlbom, A., Hasani, M., Theliander, H., Nielsen, R. P., & Maschietti, M. (2022). *Using isopropanol as capping agent in hydrothermal liquefaction of kraft lignin*. Poster presented at 16<sup>th</sup> European Workshop on Lignocellulosic and Pulp, Gothenburg, Sweden.

### **General rights**

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal -

### **Take down policy**

If you believe that this document breaches copyright please contact us at [vbn@aub.aau.dk](mailto:vbn@aub.aau.dk) providing details, and we will remove access to the work immediately and investigate your claim.

# Using isopropanol as a capping agent in hydrothermal liquefaction of kraft lignin

## Introduction

To make use of aromatic units from lignin as chemicals or fuel additives, a first step is to break down the molecular structure of the lignin. Hydrothermal liquefaction (HTL), where the lignin is depolymerised in hot pressurised water, is one way to break the lignin structure. However, the depolymerised material is reactive and suffers from repolymerisation which forms undesired char.

We investigated mitigation of the repolymerisation by adding isopropanol (IPA) as a capping agent in HTL of kraft lignin.<sup>1</sup>

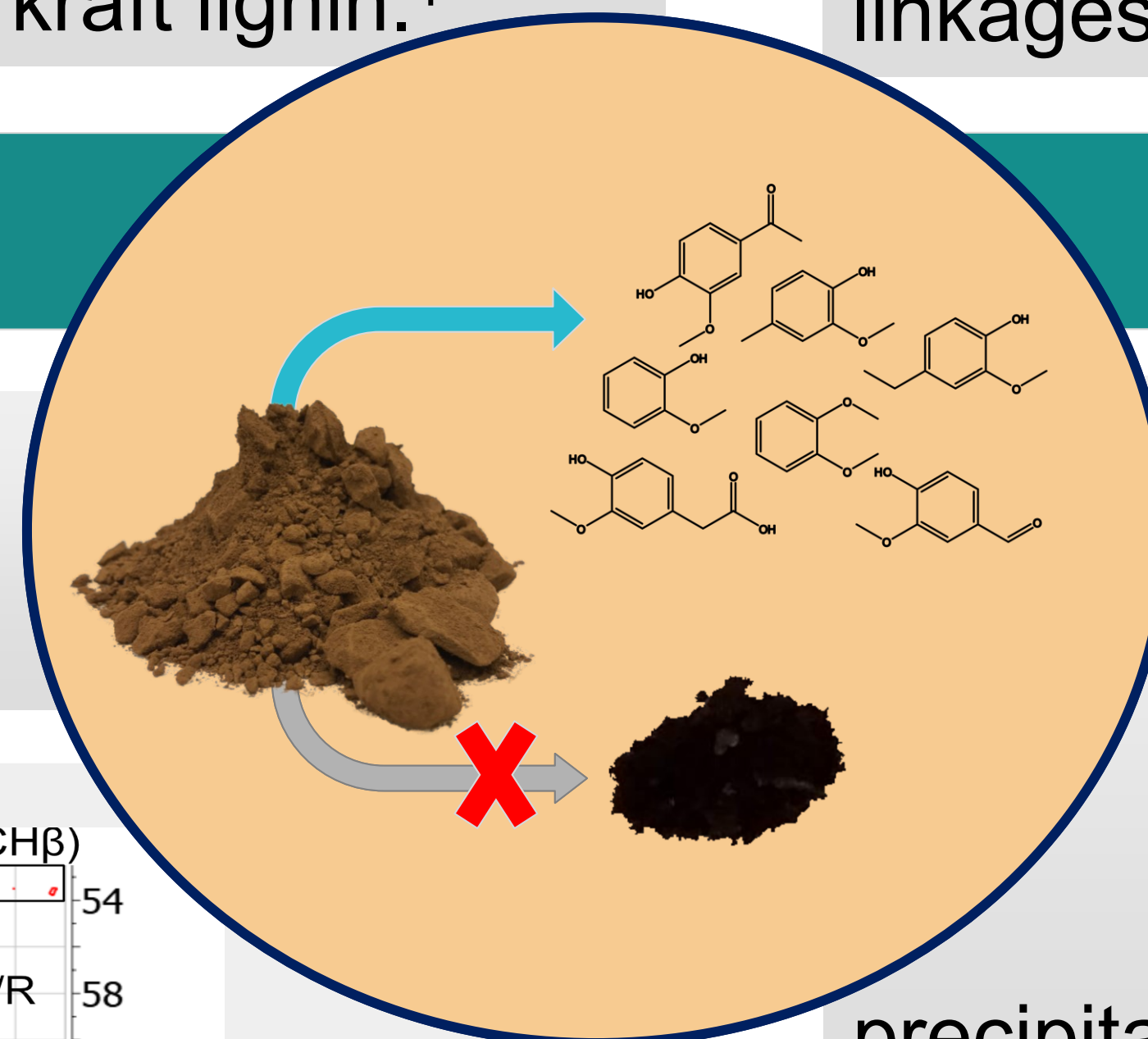
## Conclusions

Hydrothermal liquefaction of kraft lignin, with IPA as a capping agent, did not produce any apparent liquid organic phase. However, the IPA capped unwanted repolymerisation reactions seen by a reduction of the yield and weight average molecular weight of the char.

Also, the reaction conditions, 290-335 °C at 250 bar for 12 min, were severe enough to break the major inter-unit ether linkages in the lignin.

## Structural changes

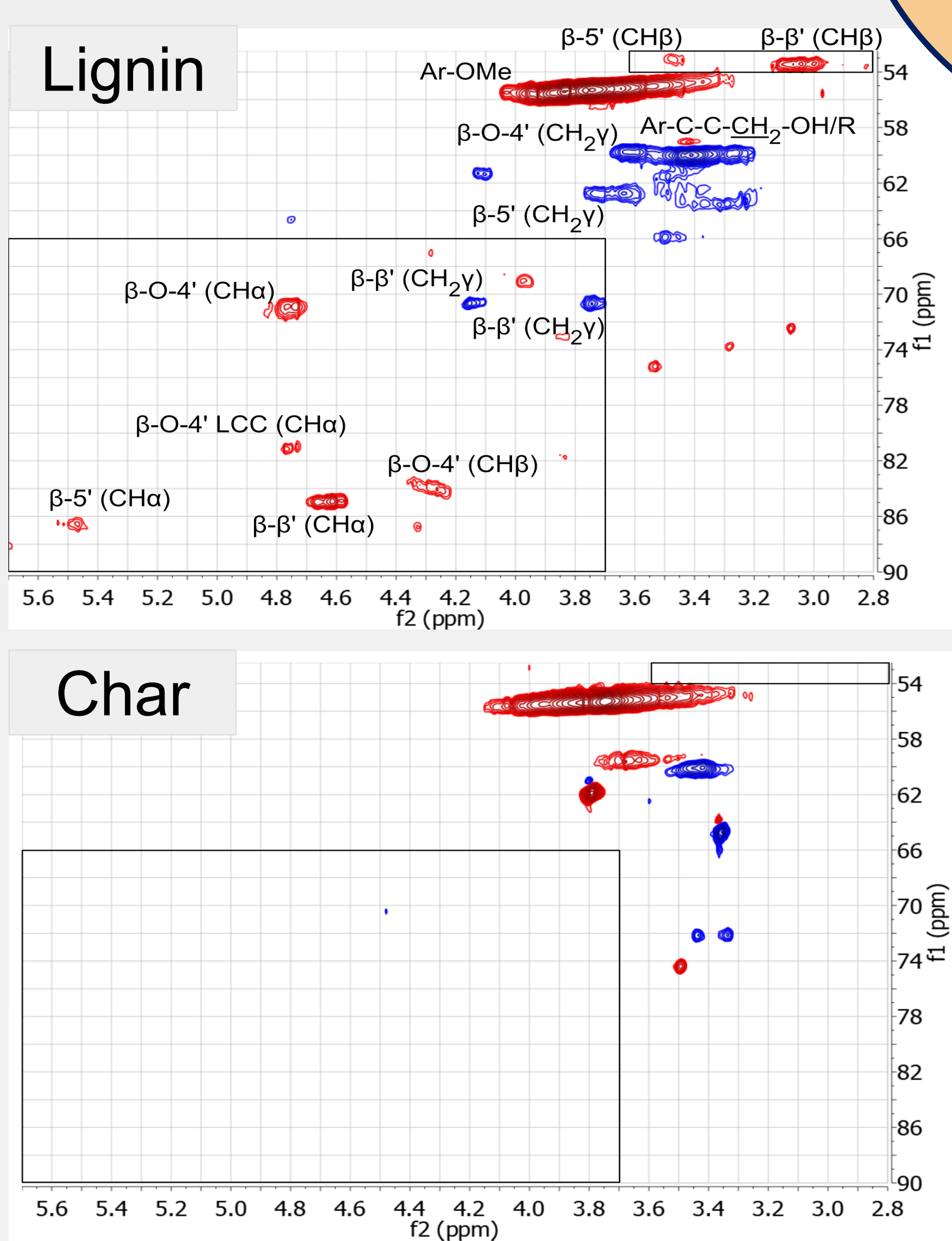
HSQC NMR measurements showed the typical inter-unit ether linkages in the lignin to be broken during the process.



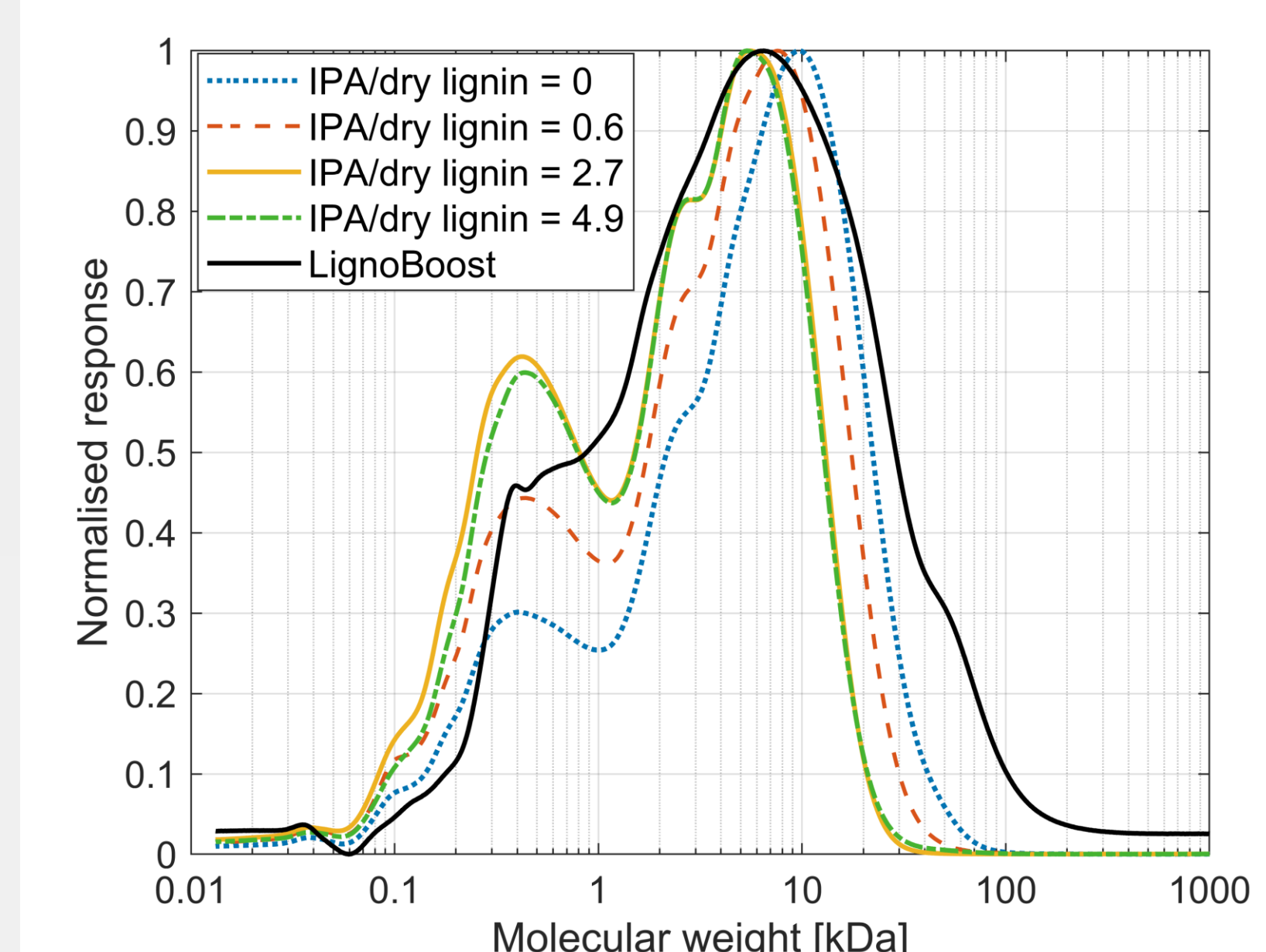
## Molecular weights and yields

The molecular weight distributions showed that the weight average molecular weight of the char was reduced with increased IPA/dry lignin loading.

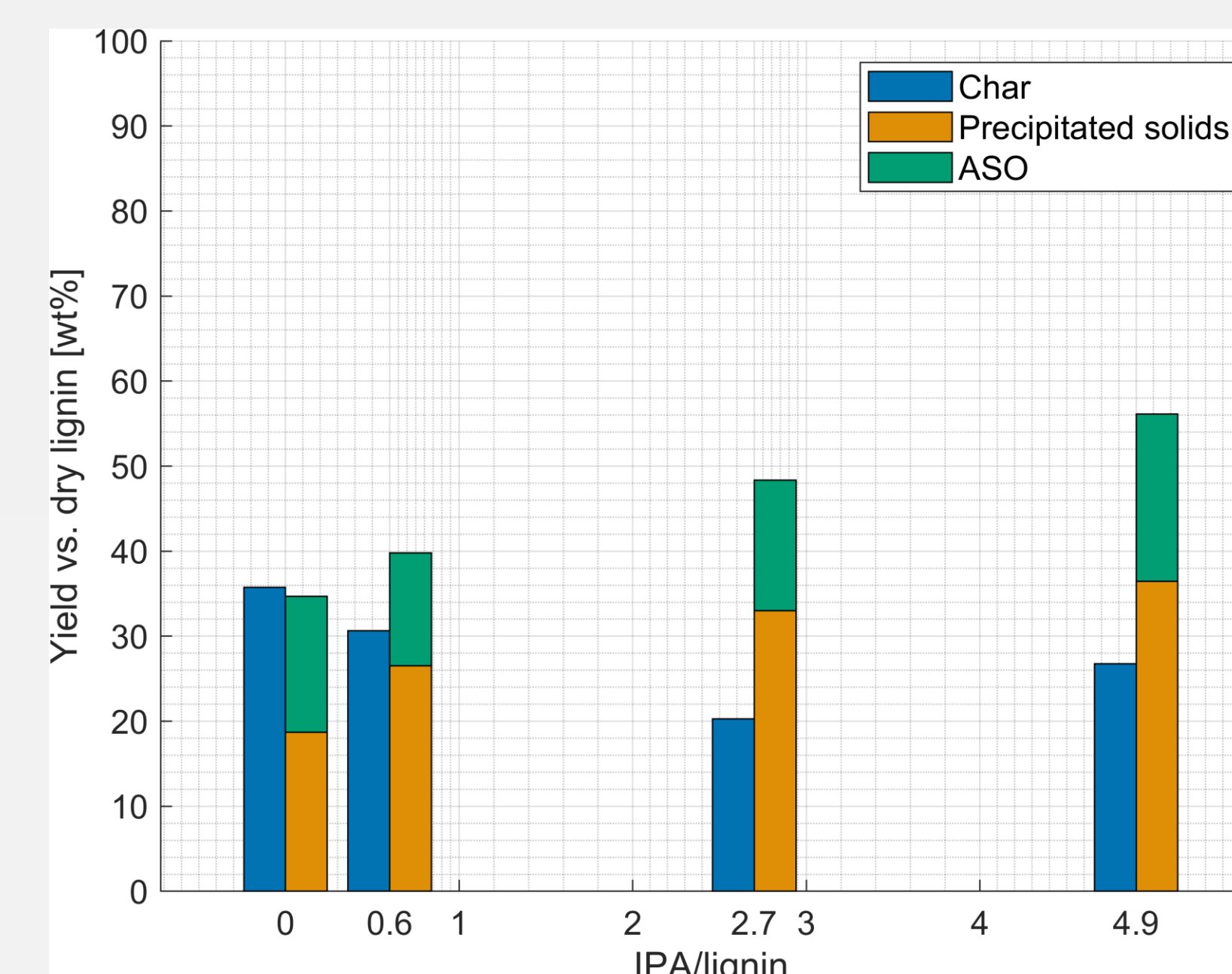
Increasing the IPA/dry lignin loading in the reaction system caused an increase in the yield of precipitated solids while the char yield was reduced. This points to IPA working as a capping agent.



Inter-unit aliphatic region of HSQC spectra of LignoBoost lignin and char with IPA/dry lignin 2.7. Annotations for lignin peaks according to Mattsson et al.<sup>2</sup>



Molecular weights of the LignoBoost lignin and the char fractions.



Yields of the product fractions with ASO being acid soluble organics.

## Methods

Softwood kraft lignin, isolated with the LignoBoost process, was depolymerised using hydrothermal liquefaction at the following conditions

- 290-335°C
- 250 bar
- 12 min residence time
- 0-24 wt% isopropanol (IPA/dry lignin 0-4.9)
- 5 wt% lignin
- 1.6wt% Na<sub>2</sub>CO<sub>3</sub>
- 99 ml batch reactor

The reaction mixture was quickly heated in a custom-made batch reactor by pumping a mixture containing lignin and IPA into a pre-heated water and Na<sub>2</sub>CO<sub>3</sub>-mixture. The reaction product was ejected after the specified residence time and quickly quenched in an ice-bath. Fractionation of the product produced char, precipitated solids and acid soluble organics (ASO).

1. Ahlbom, A., Maschietti, M., Nielsen, R., Lyckeskog, H., Hasani, M. and Theliander, H., **Energies** 14, 932 (2021).
2. Mattsson, C. et al., **Biomass and Bioenergy** 95, 364–377 (2016).