

Hydrothermal Liquefaction of Sewage Sludge

Fractional Extraction and Characterization of Nitrogen-Compounds from the Biocrude matrix



[SWR aktuell - 2022]

HTL



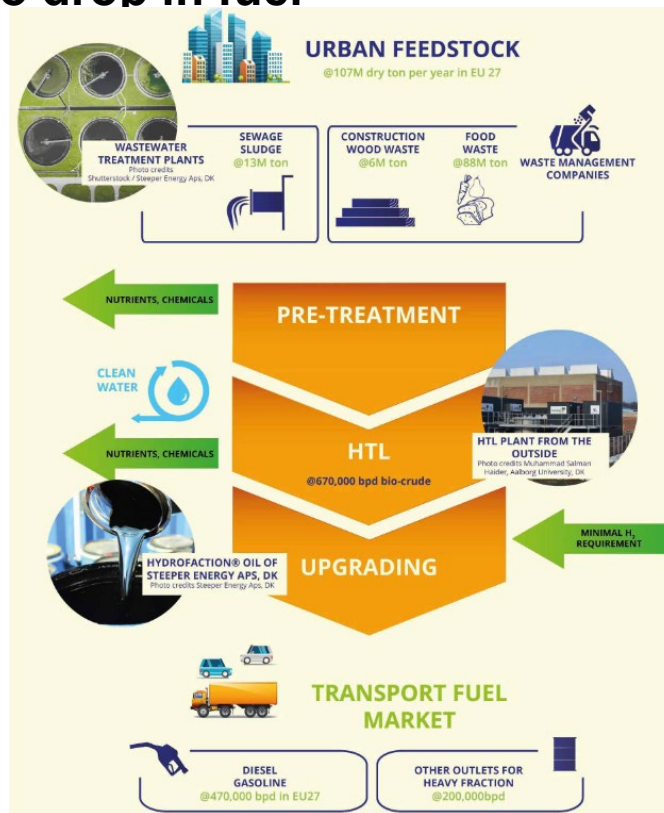
[Steeper Energy – 2018]

The NextGenRoadFuels (NGRF) Project

Converting urban Waste into drop in fuel

NGRF is a Horizon 2020 project to develop a competitive European technology platform for a sustainable liquid fuel production.

NGRF aim to prove the HTL pathway as an efficient route to produce high-volume, cost-competitive, drop-in synthetic gasoline and diesel fuels, as well as other hydrocarbon compounds from urban waste residues.



etaflorence  renewableenergies

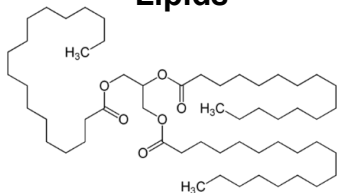


Hydrothermal liquefaction

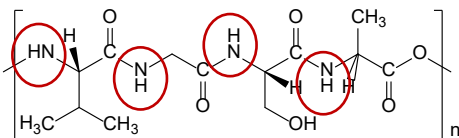
From Polymers to Monomers

Biogenic polymers

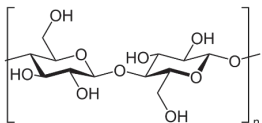
Lipids



Proteins



Polysaccharides



Use properties of
pressurized **water**
at **elevated**
temperatures.



1. Depolymerisation
2. Recombination
3. Condensation



Biocrude product

>10 000 of
different
compounds

Wide range of N-
compounds

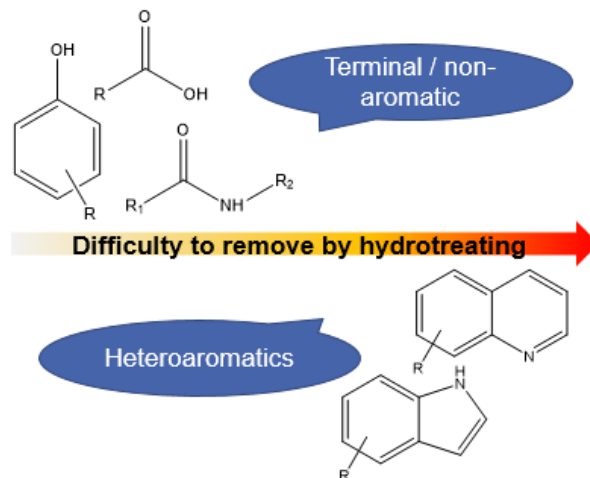
Hydrothermal liquefaction

Heteroatoms in compounds



Biocrude product – Elemental composition

| | Petroleum ² | HTL Biocrude ³ |
|-----------------|------------------------|---------------------------|
| Carbon (wt.%) | 83 – 87 | 76.0 |
| Hydrogen (wt.%) | 10 – 14 | 11.0 |
| Nitrogen (wt.%) | 0.1 – 2 | 3.8 |
| Sulfur (wt.%) | 0.05–6 | 1.0 |
| Oxygen (wt.%) | 0.05 – 1.5 | 8.2 |



Refining:

- Distillation
- HDS

Upgrading:

- HDO
- HDN

H₂

Blending



²[J. Speight "The Chemistry and Technology of Petroleum" 2006]

³[Sewage sludge HTL - Campaign from 2019]

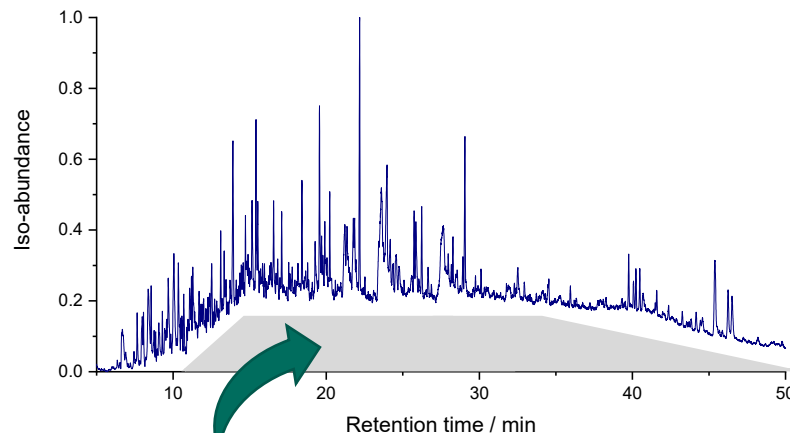
Biocrude Characterization

Complex chemical composition

HTL Biocrude³

| | |
|-----------------|------|
| Carbon (wt.%) | 76.0 |
| Hydrogen (wt.%) | 11.0 |
| Nitrogen (wt.%) | 3.8 |
| Sulfur (wt.%) | 1.0 |
| Oxygen (wt.%) | 8.2 |

Analysis of
chemical
composition



? Poor
Resolution



N?

multi-heteroatom
compounds

Aliphatic ?

Aromatic ?

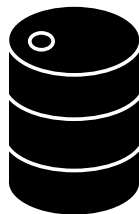
Basic ?

Acidic ?

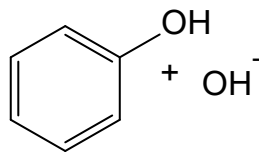
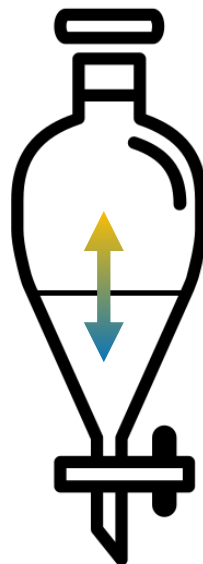
Simplify by Separation

Solubility of polar heteroatom compounds

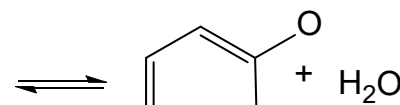
Non-polar
(organic)
solvent



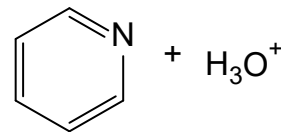
polar
(aqueous)
solvent



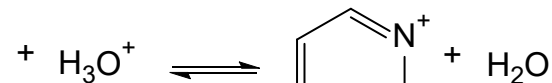
Phenol



Phenolate



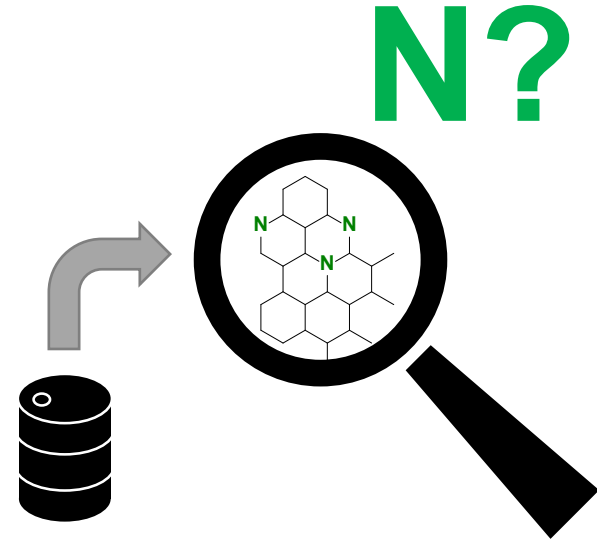
Pyridin



Pyridinium

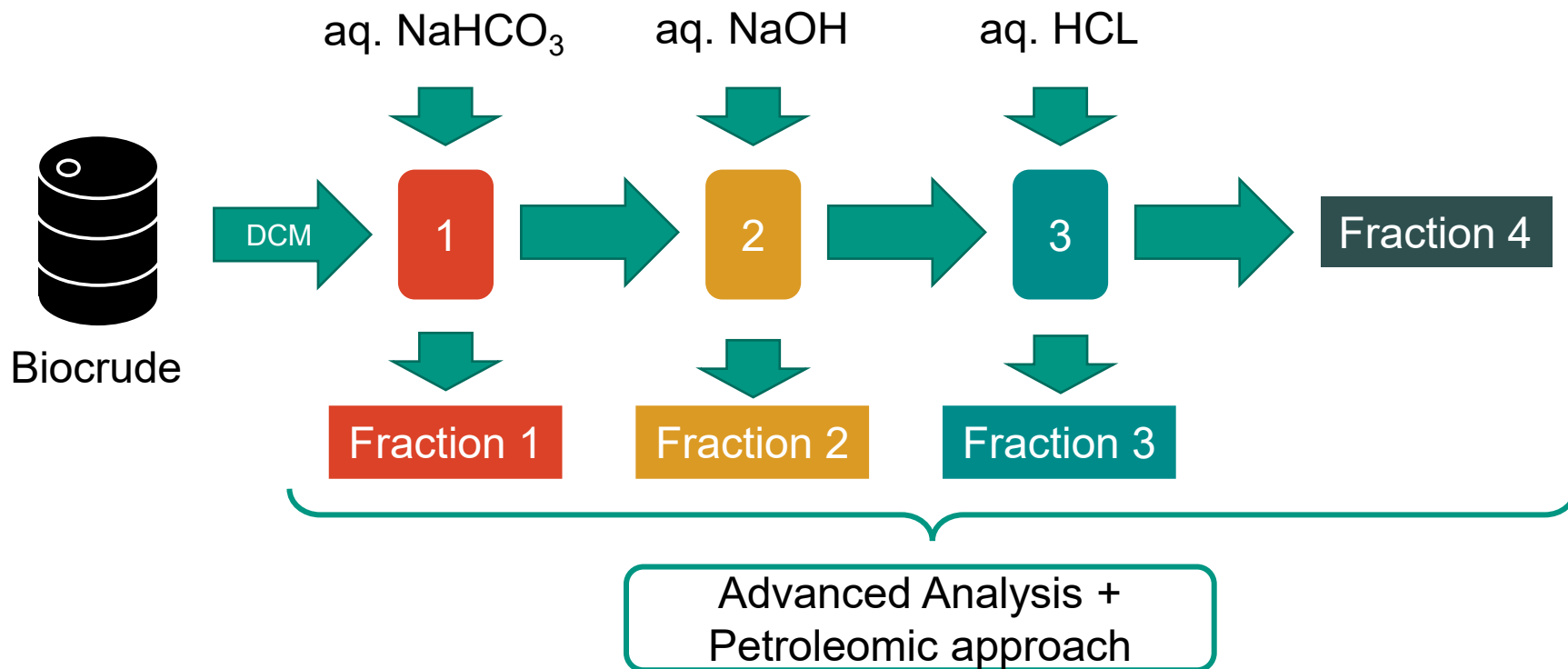
Scope of this presentation

- Present a scheme for the separation of biocrude by polarity.
- Identify in which fraction carbon (C) and nitrogen (N) is recovered.
- Identify species of N-compounds in these fractions.
- Evaluate the aromaticity distribution of N-compounds over the fractions.



Applying the Separation

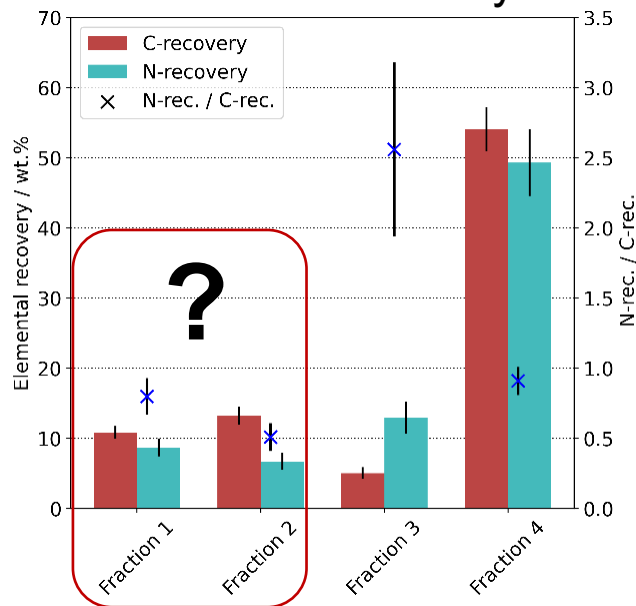
Sequential extraction of polar compounds



Solvent extraction and polar-aqueous solutions

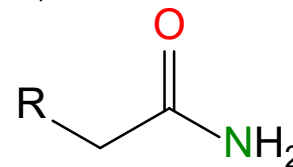
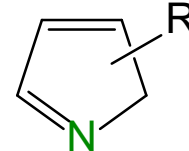
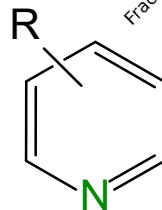
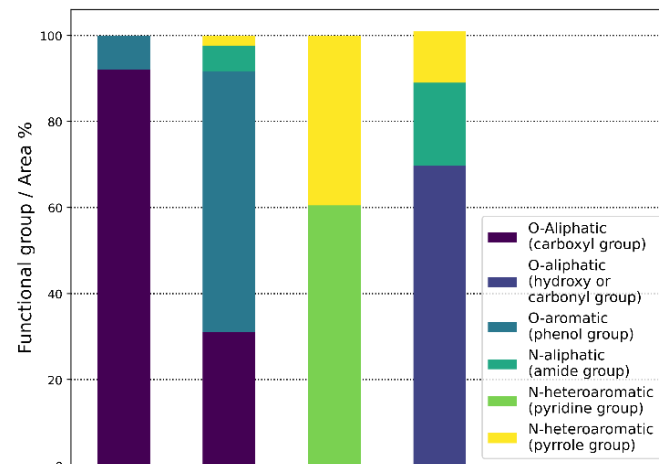
C- and N-recovery into the extracted fractions

C- and N- Recovery



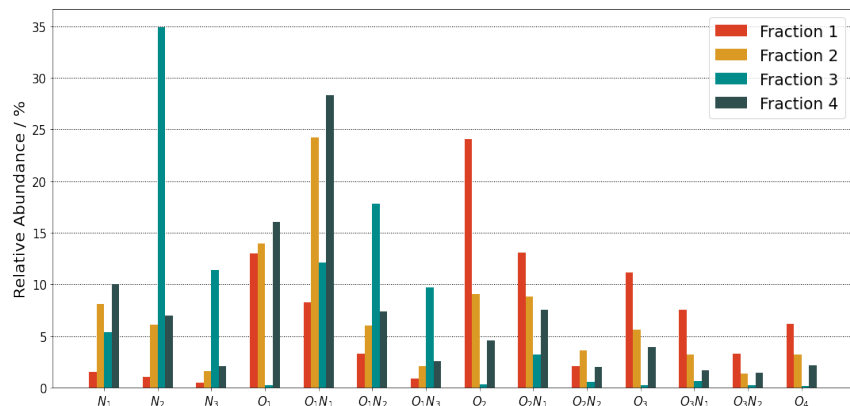
Analysis of
chemical
composition

Qualitative GC-MS

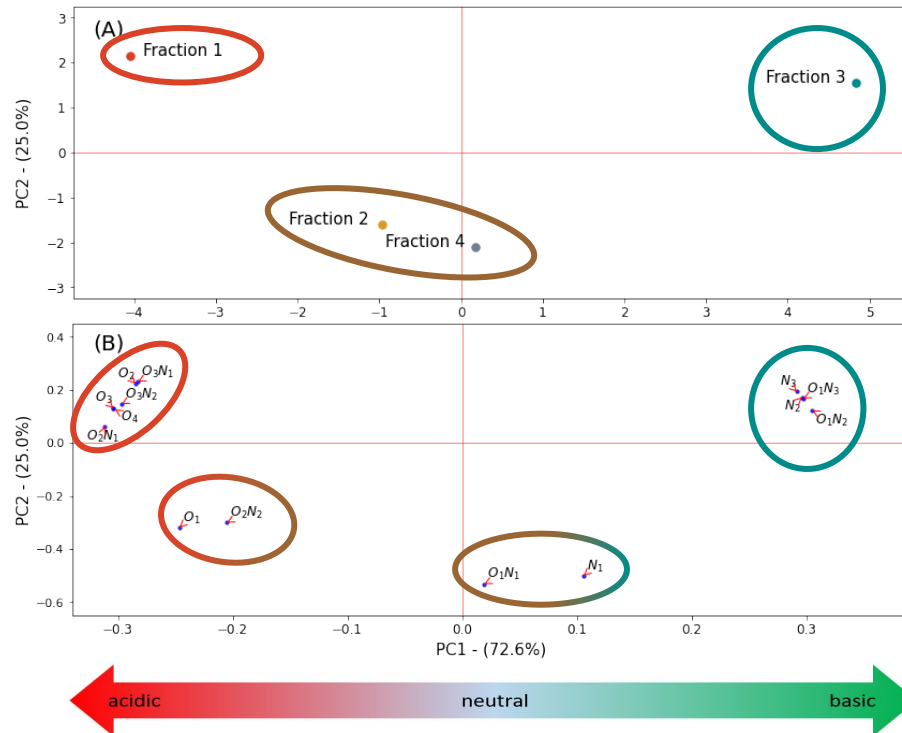


Solvent extraction and polar-aqueous solutions

Heteroatom classes and their distribution

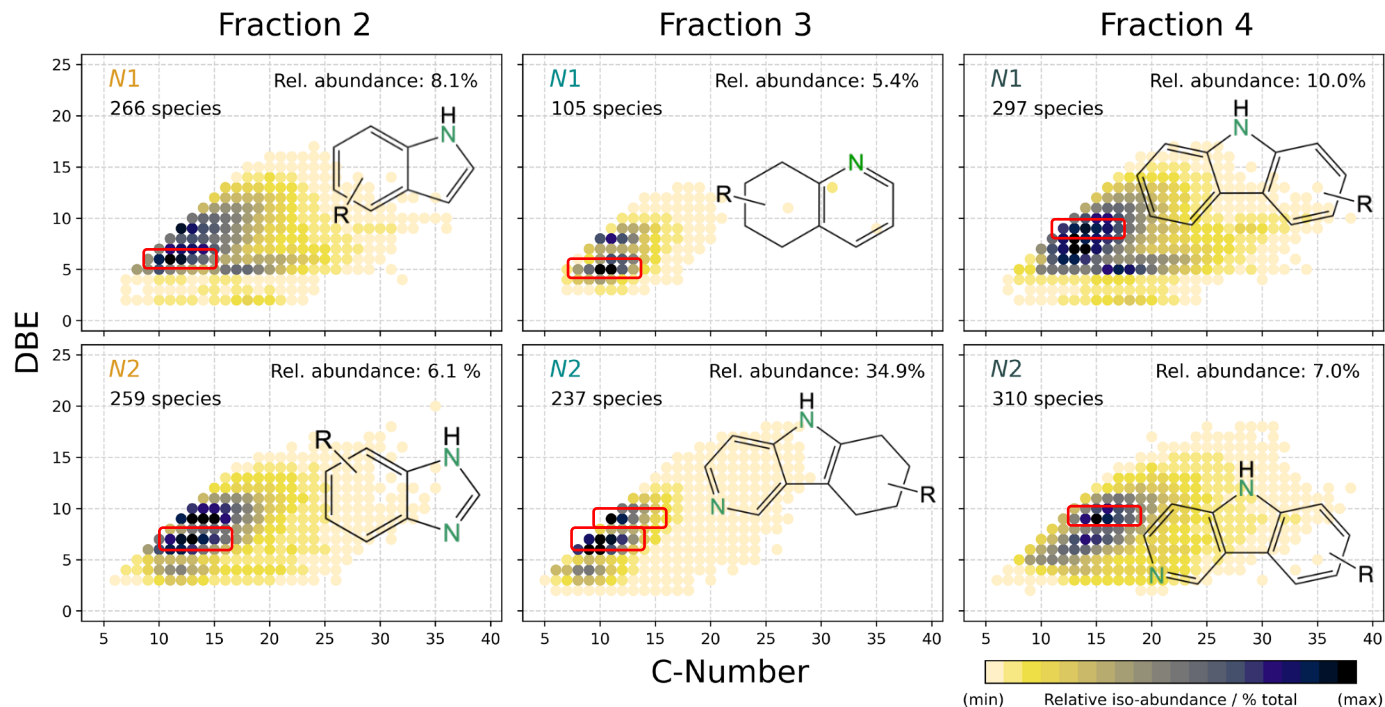


Explorative principal
component analysis (PCA)



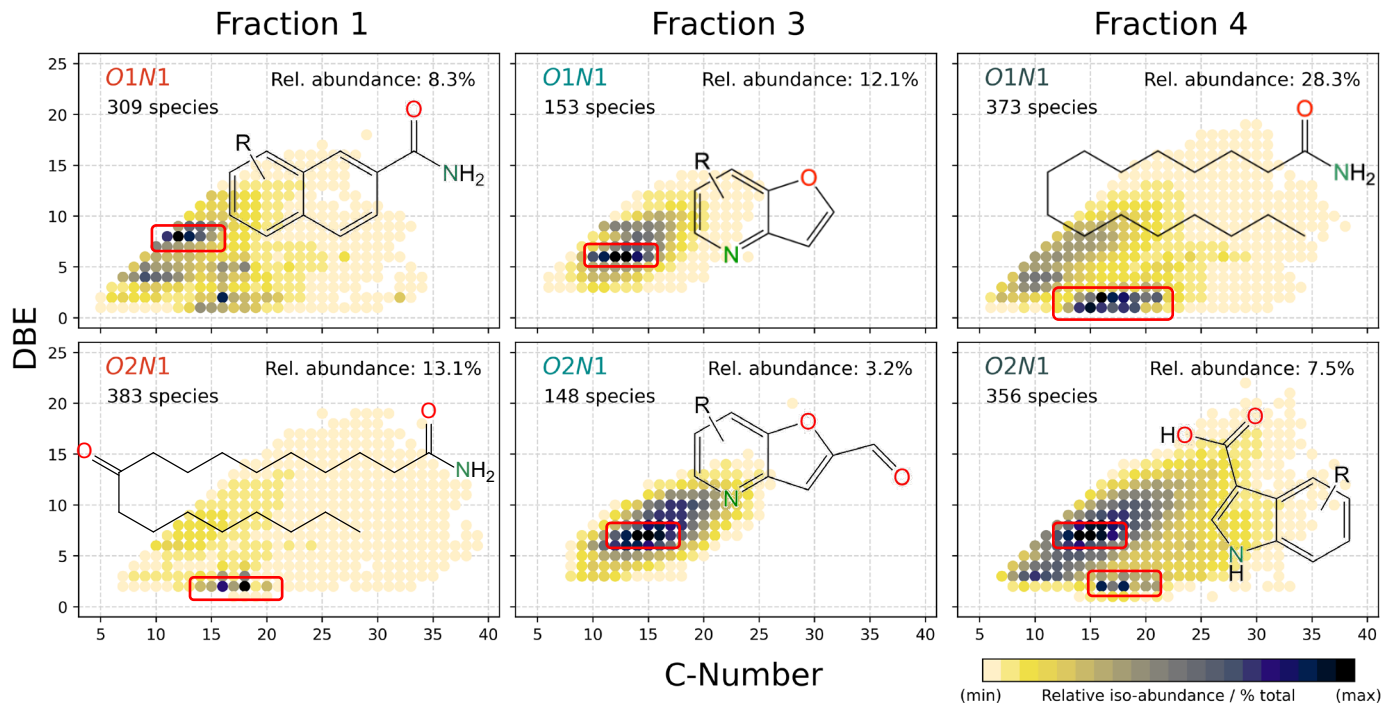
N-compounds in detail

N1 and N2 species



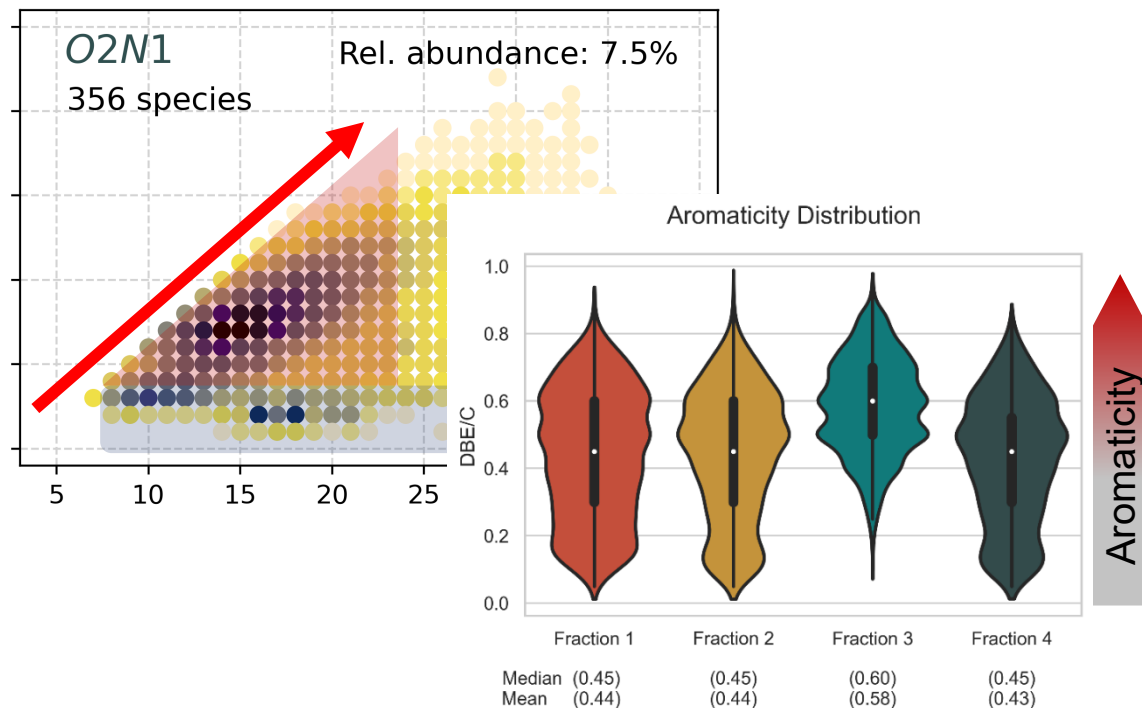
N-compounds in detail

O1N1 and O2N1 species



Aromaticity of N-compounds

Distribution over the fractions



- Definition of aromaticity over the DBE/C value
- High aromaticity found in Fraction 4
- Other Fractions show also aliphatic species

Conclusion and outlook

- N-compounds in biocrude can be separated based on their polarity.
- N-compounds show a strong basic character.
- Species identified in “acidic” Fractions are nearly always combined with oxygen heteroatoms.
- N-compounds with a basic character are nearly always identified as a heteroaromatic.
- Where to apply these results?
→ Downstream HTL: in product separation, LLE or adsorption of biocrude.

Thank You