

# 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.



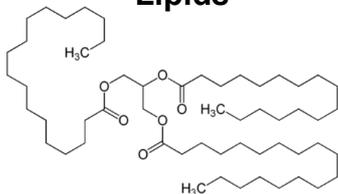
This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 818413

# Hydrothermal liquefaction

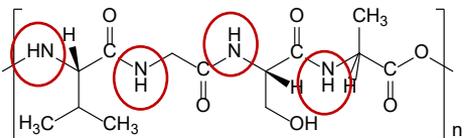
## From Polymers to Monomers

### Biogenic polymers

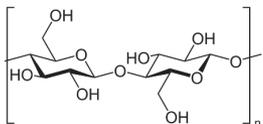
#### Lipids



#### 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**

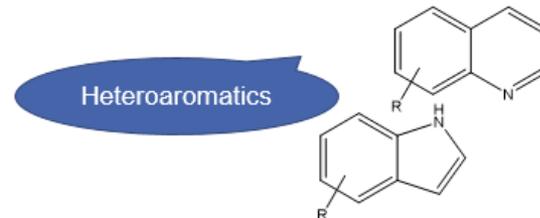
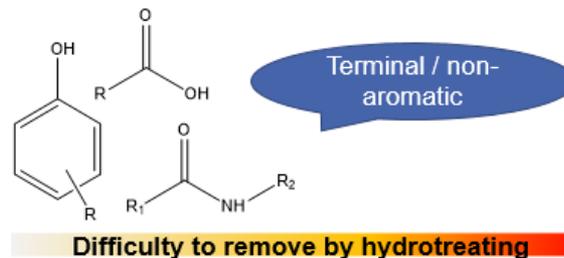
# Hydrothermal liquefaction

## Heteroatoms in compounds



Biocrude product – Elemental composition

	Petroleum <sup>2</sup>	HTL Biocrude <sup>3</sup>
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

**H<sub>2</sub>**

**Blending**



<sup>2</sup>[J. Speight "The Chemistry and Technology of Petroleum" 2006]

<sup>3</sup>[Sewage sludge HTL - Campaign from 2019]

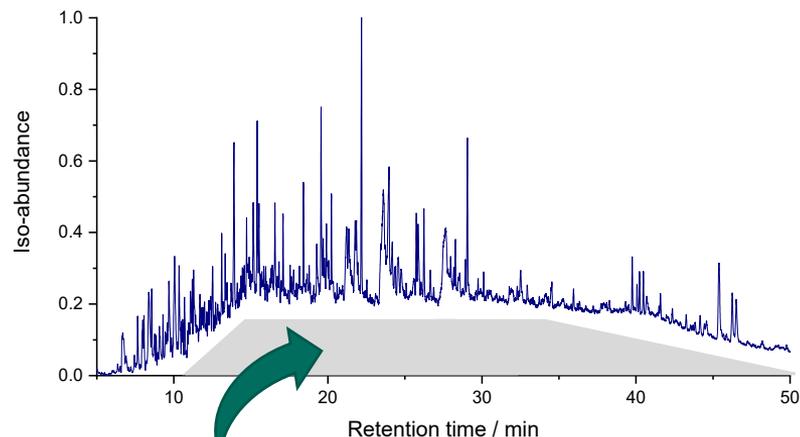
# Biocrude Characterization

## Complex chemical composition

HTL Biocrude<sup>3</sup>

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 ?

Basic ?

Aromatic ?

Acidic ?

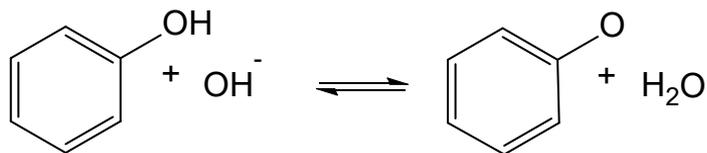
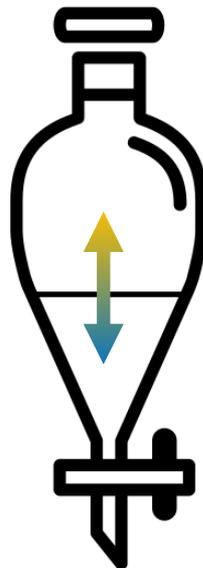
# Simplify by Separation

## Solubility of polar heteroatom compounds

Non-polar  
(organic)  
solvent

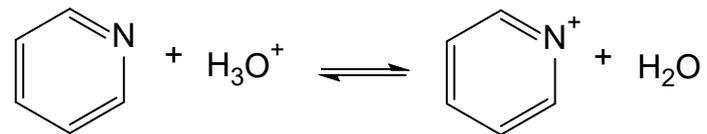


polar  
(aqueous)  
solvent



Phenol

Phenolate

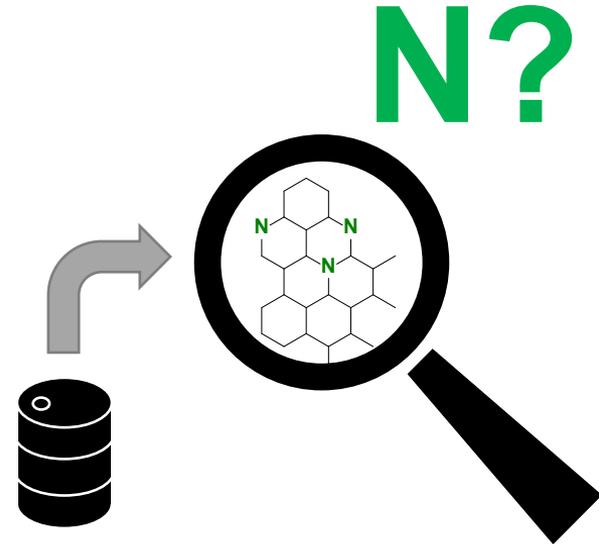


Pyridin

Pyridinium

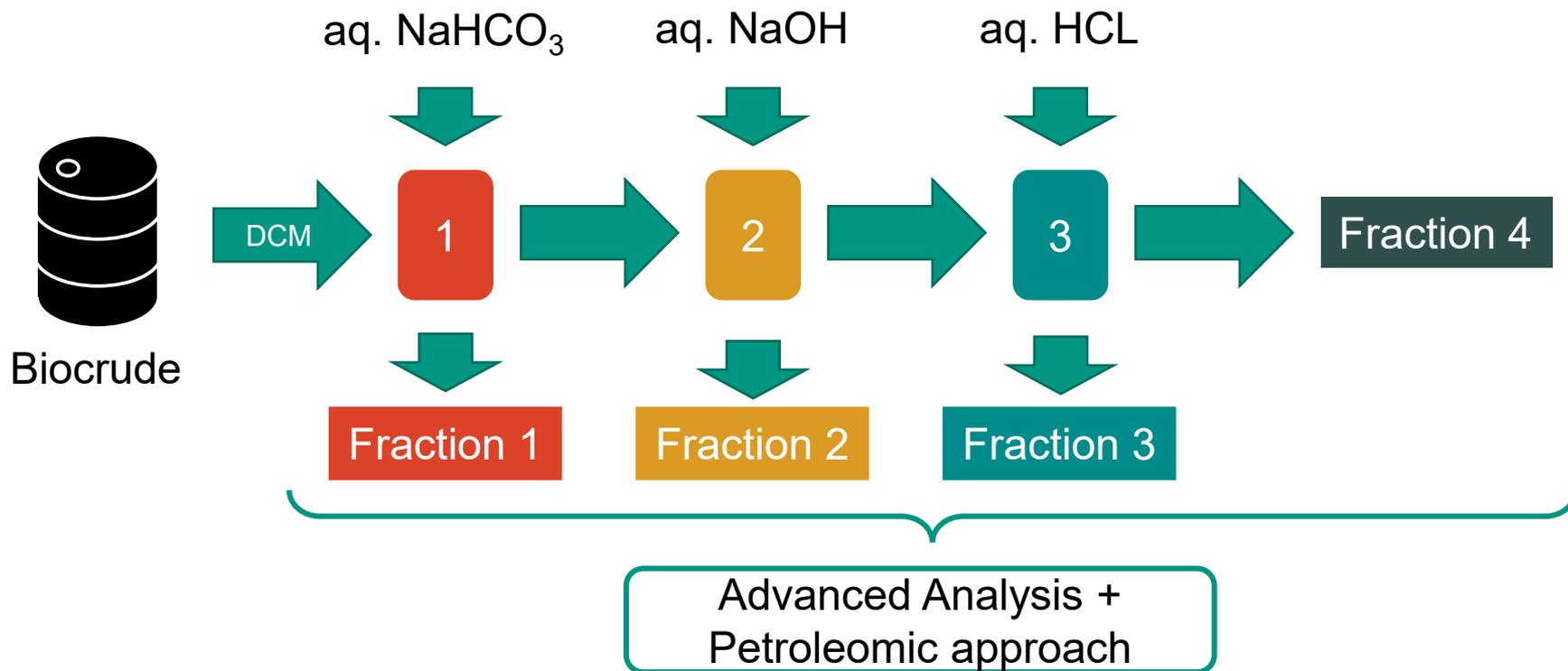
# Scope of this presentation

- 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.



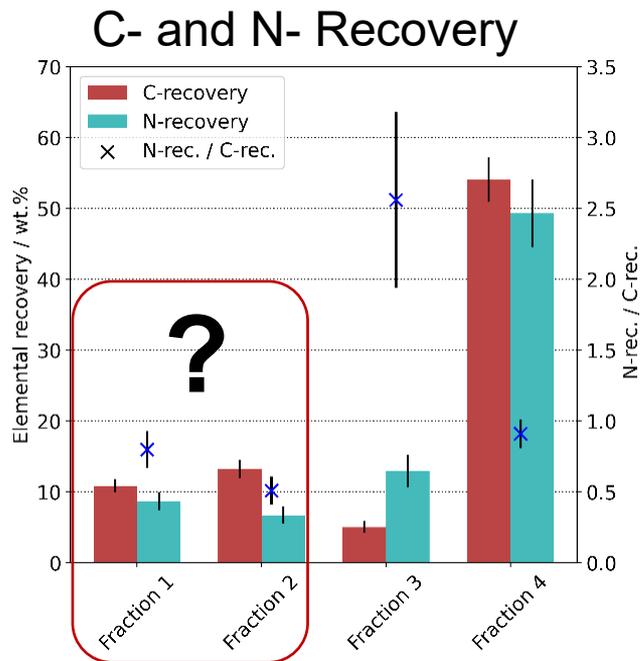
# Applying the Separation

## Sequential extraction of polar compounds



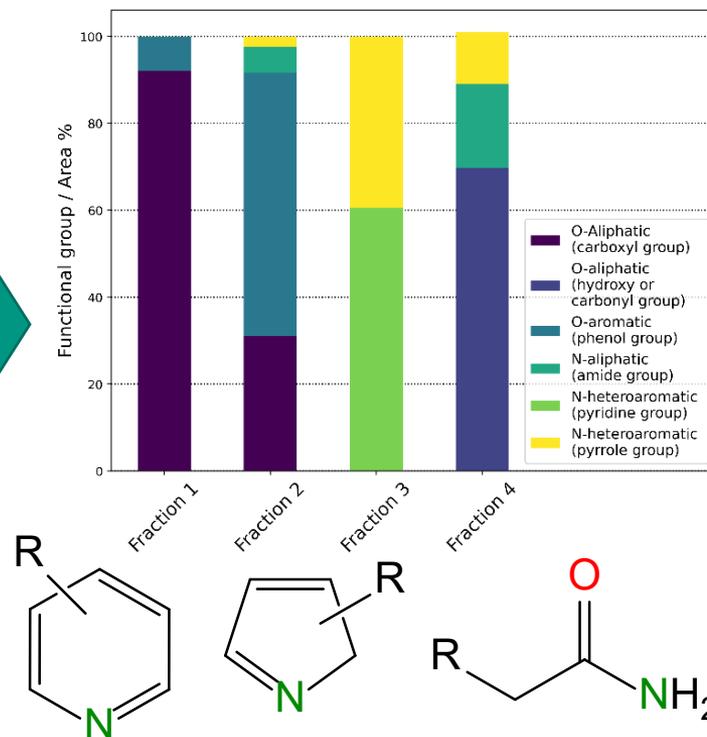
# Solvent extraction and polar-aqueous solutions

## C- and N-recovery into the extracted fractions



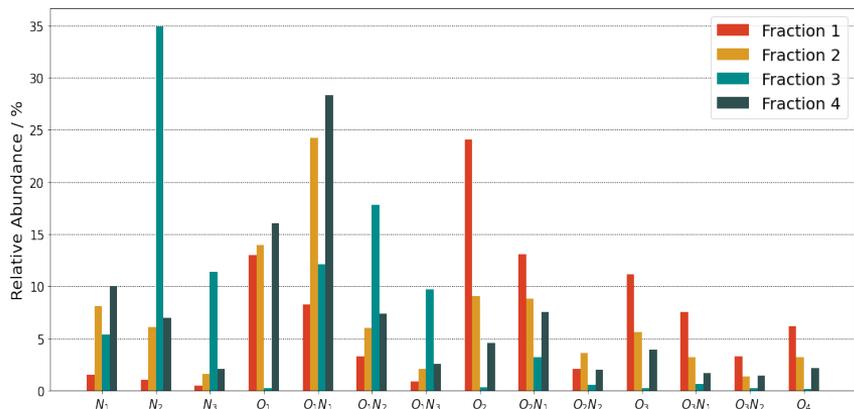
Analysis of chemical composition

### Qualitative GC-MS

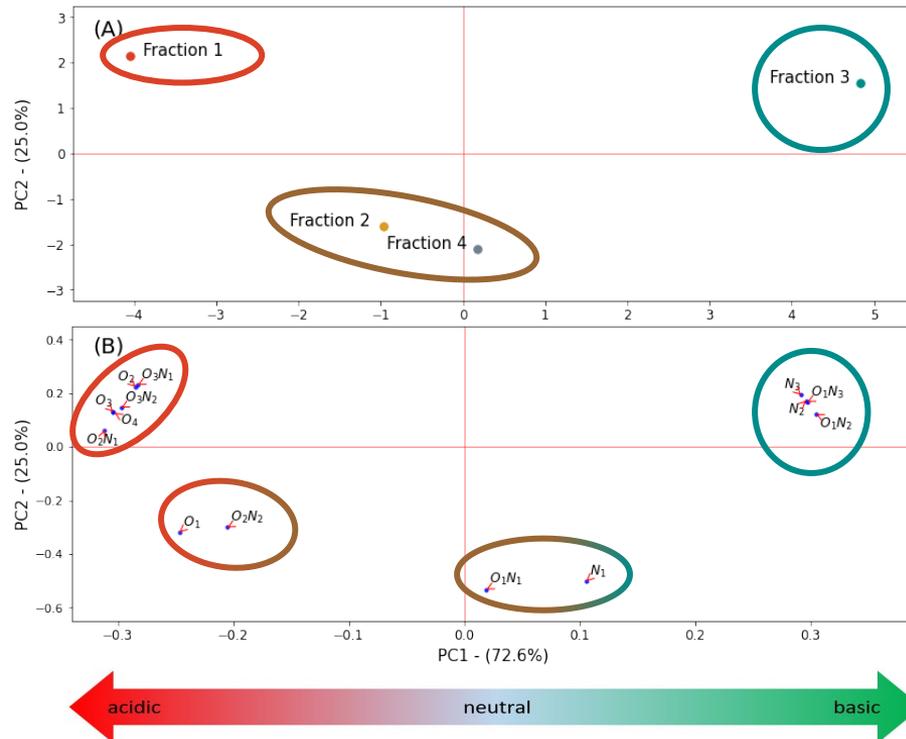


# Solvent extraction and polar-aqueous solutions

## Heteroatom classes and their distribution

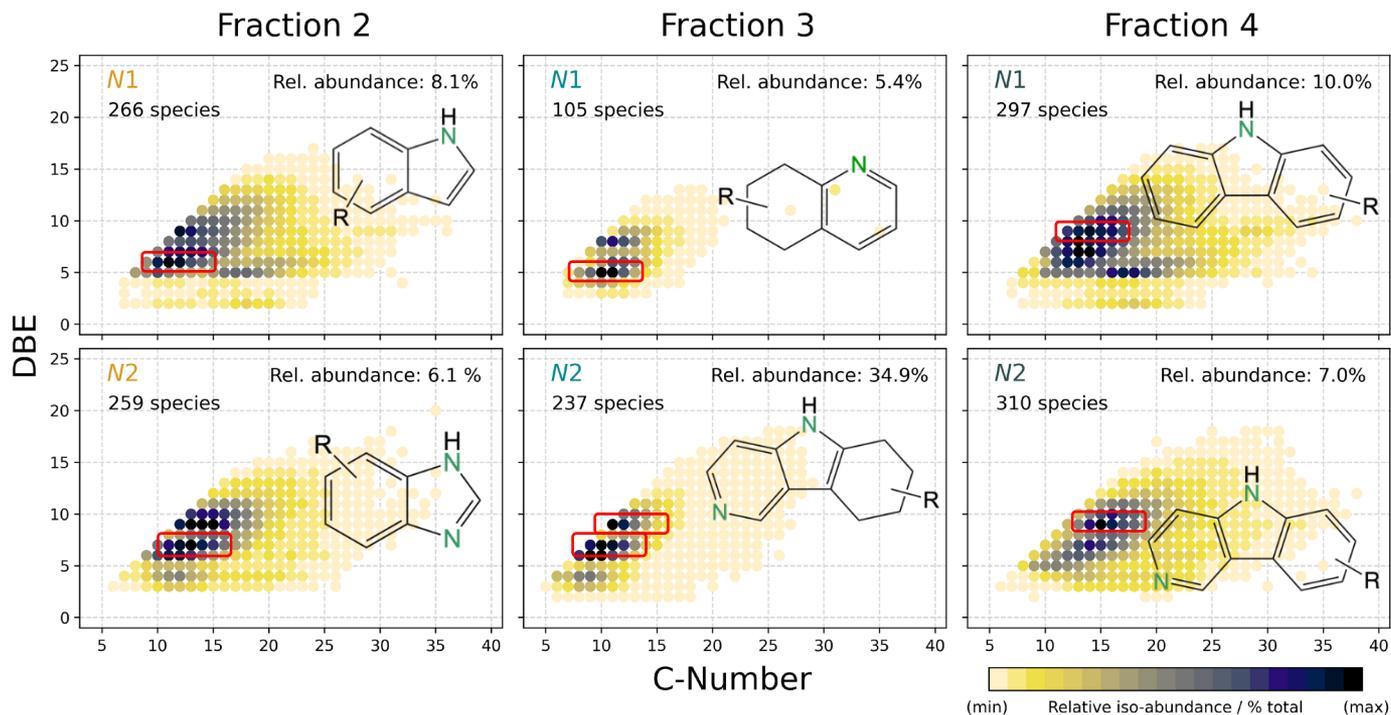


Explorative principal component analysis (PCA)



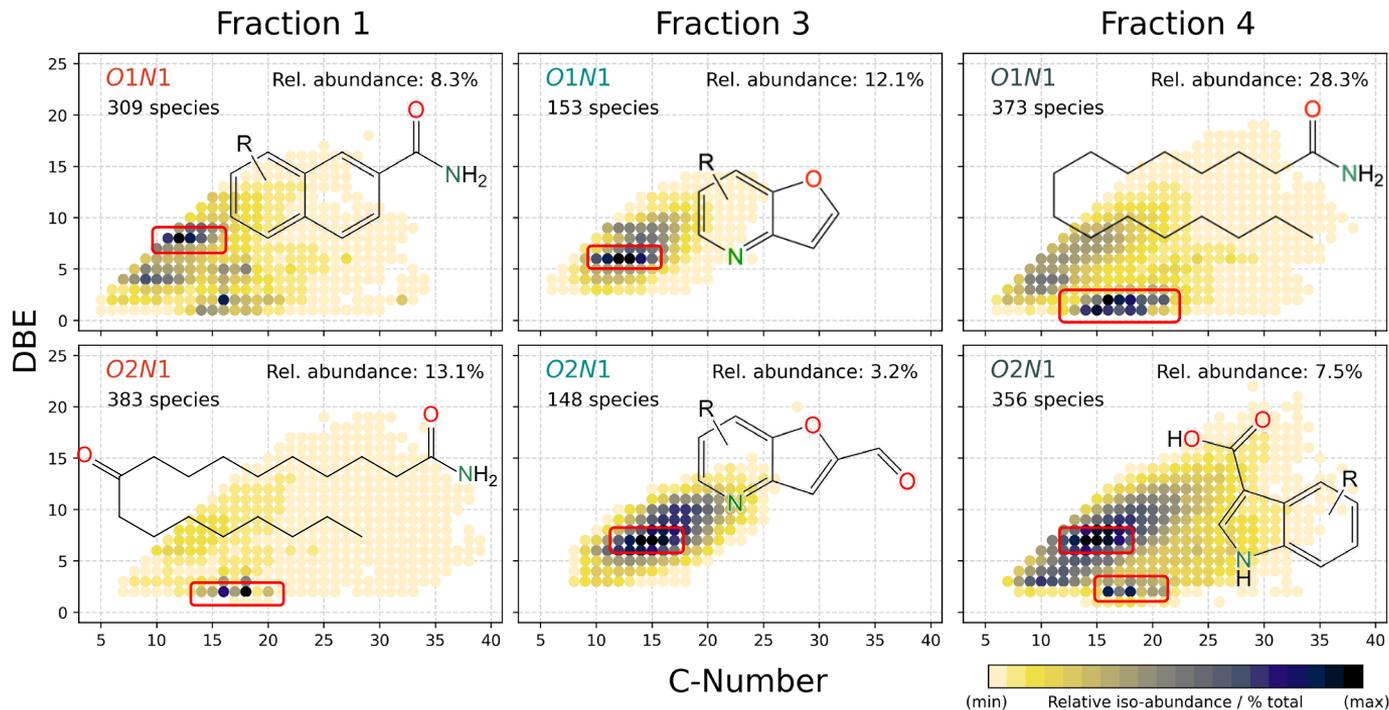
# N-compounds in detail

## N1 and N2 species



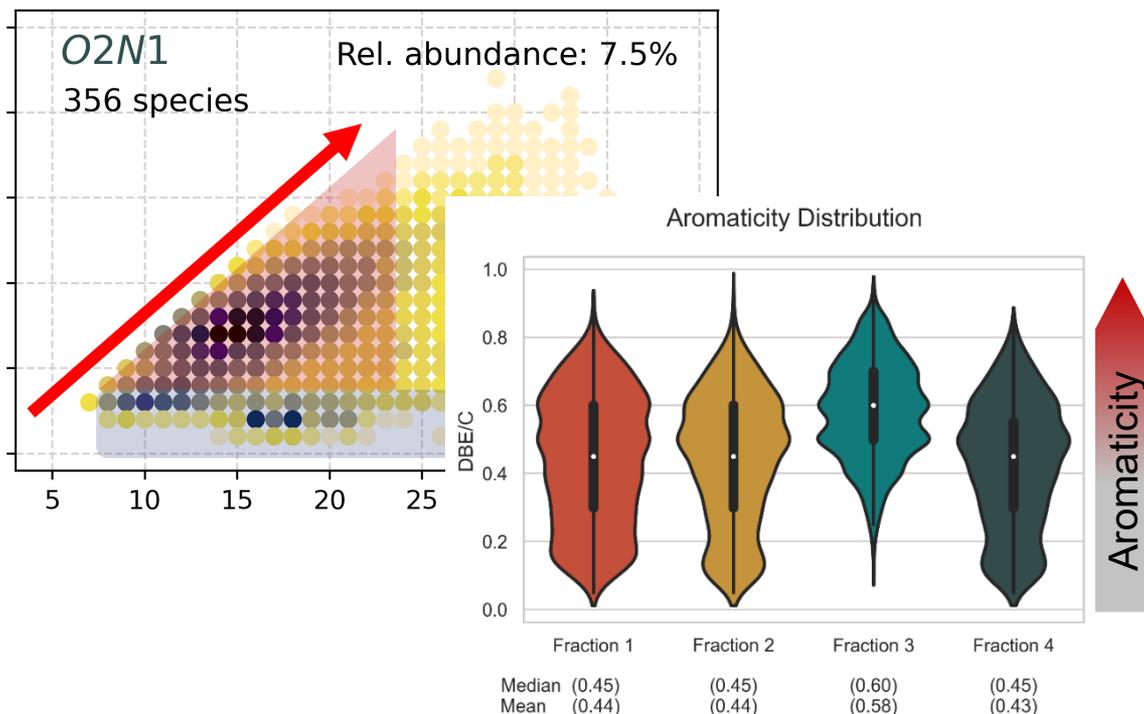
# N-compounds in detail

## 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