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# Deep eutectic solvent pre-treatment of residual biomass streams – effects on anaerobic degradability –

Jana Schultz,

Asli Isci, Marvin Scherzinger, Simel Bagder Elmaci, Dicle Delal Aslanhan,  
Miyase Deniz Cam, Ozge Sakiyan, Martin Kaltschmitt

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ORCID: <https://orcid.org/0009-0003-5039-0772>



- Management of wastes and residues from
  - Agricultural production
  - Processing of agricultural goods
- Provision of renewable energy
- Provision of organic fertilizer
  
- Treatment of lignocellulosic biomass for
  - Production of value-added products
  - Improvement of anaerobic degradability

- Mix of hydrogen bond donor and hydrogen bond dacceptor
- Melting point of mixture < melting point of individual substances
- Good solvation properties
  
- Advantages over conventional solvents
  - Biodegradability, biocompartibility
  - Low toxicity
  - High thermal stability
  - Low volatility
  - Non-flammability

Cork dust

Olive pruning

Common reed



Comminution

DES pre-treatment

- 1:10 solid : solvent ratio
- 1:2 Choline chloride : formic acid
- 90 °C / 110 °C / 130 °C
- 20 min / 40 min / 60 min

# Deep eutectic solvent pre-treatment of residual biomass streams

Materials and methods

Cork dust

Olive pruning

Common reed



Comminution

DES pre-treatment

Solid/liquid separation → Valorization of liquid fraction

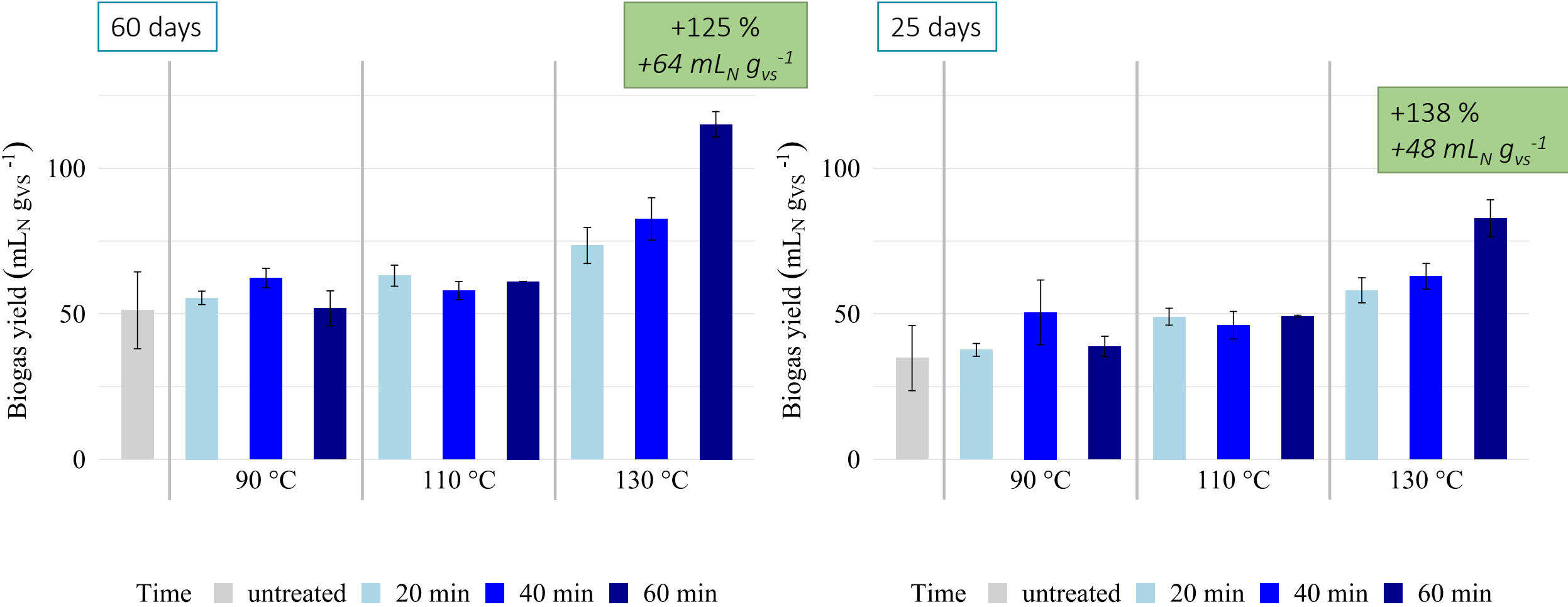
Anaerobic digestion of solid residuals

- $37 \pm 1$  °C
- 60 days
- Gas compositional analysis



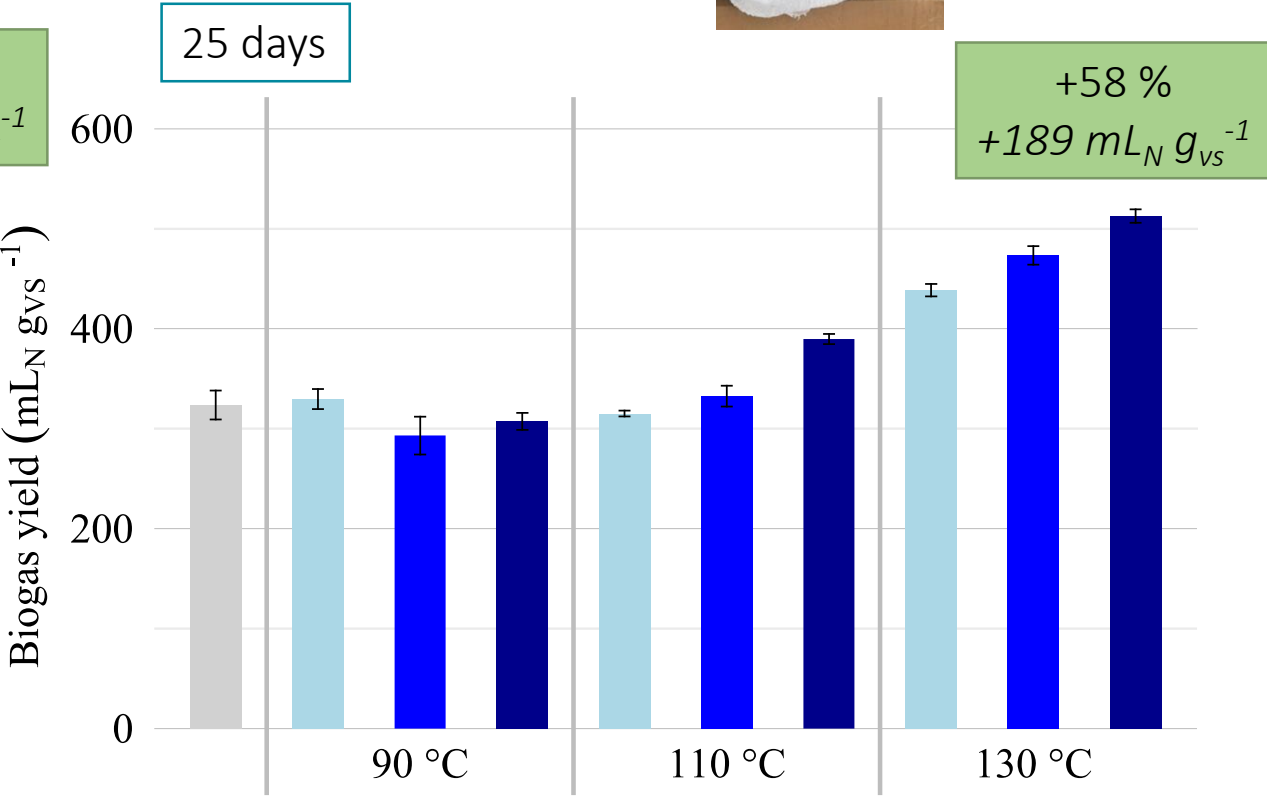
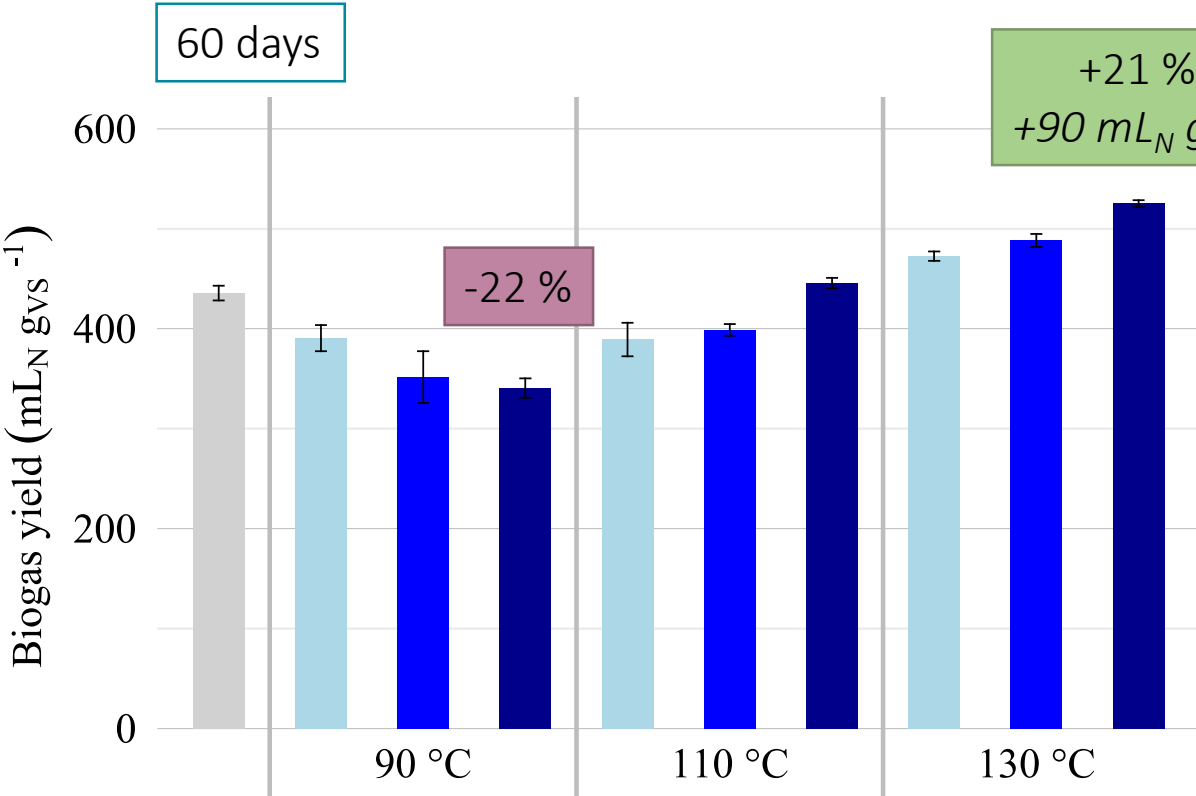
# Anaerobic degradability of DES pre-treated biomass

Results - Cork dust



# Anaerobic degradability of DES pre-treated biomass

Results - Olive tree pruning

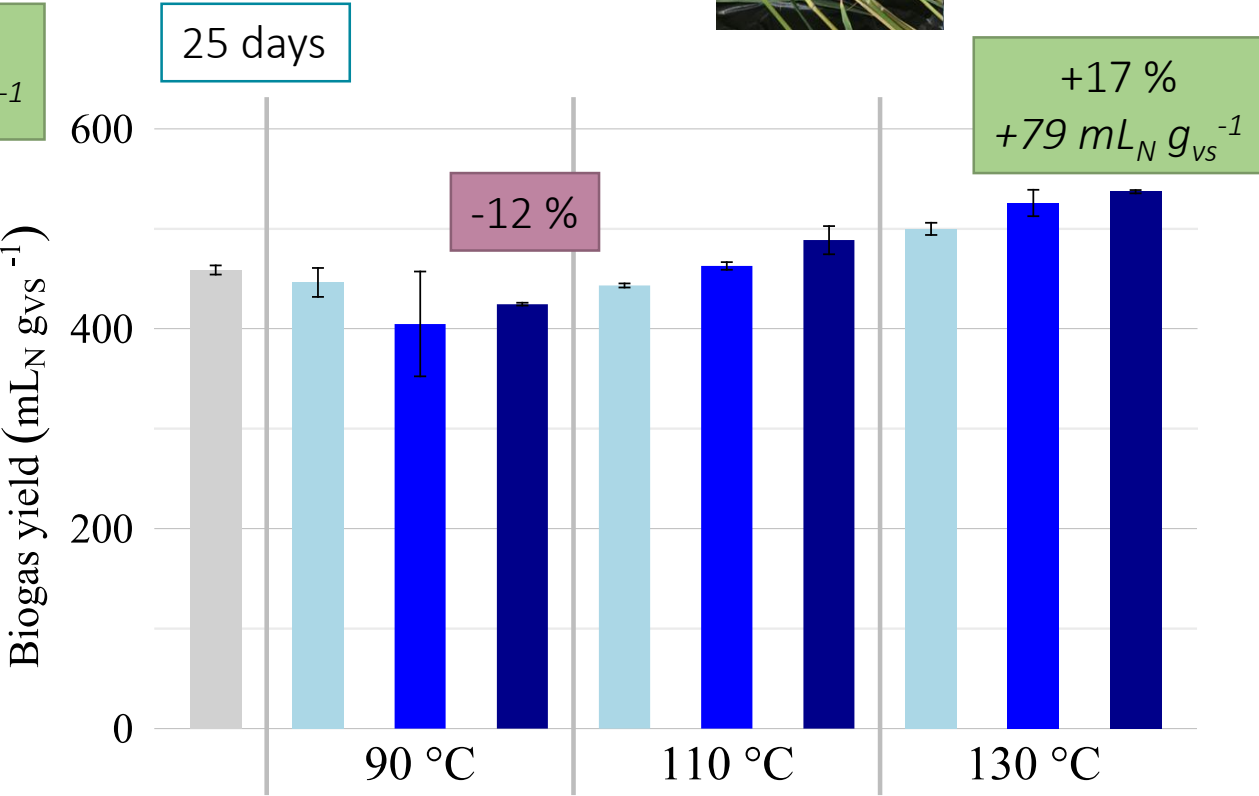
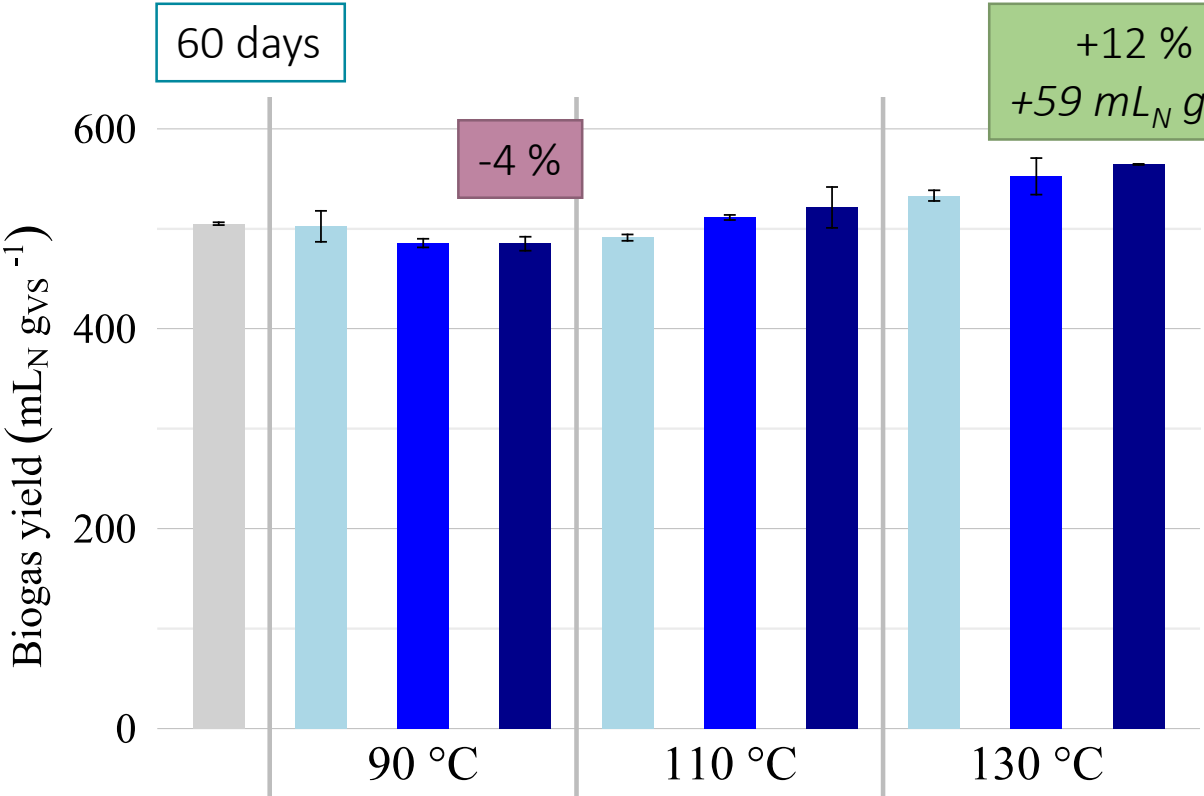


Time   ■ untreated   ■ 20 min   ■ 40 min   ■ 60 min

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# Anaerobic degradability of DES pre-treated biomass

Results - Common reed

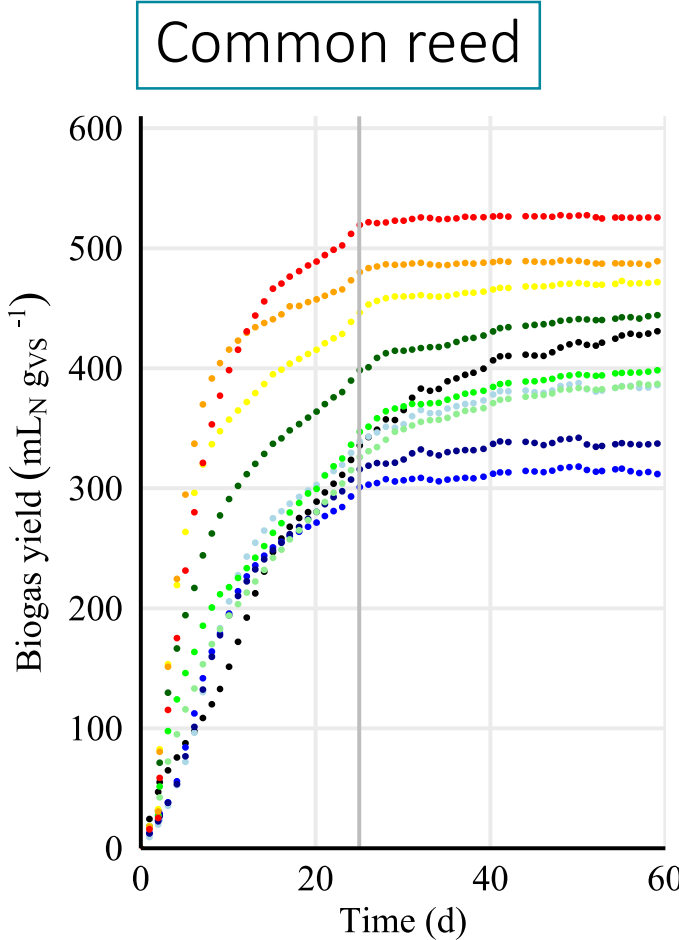
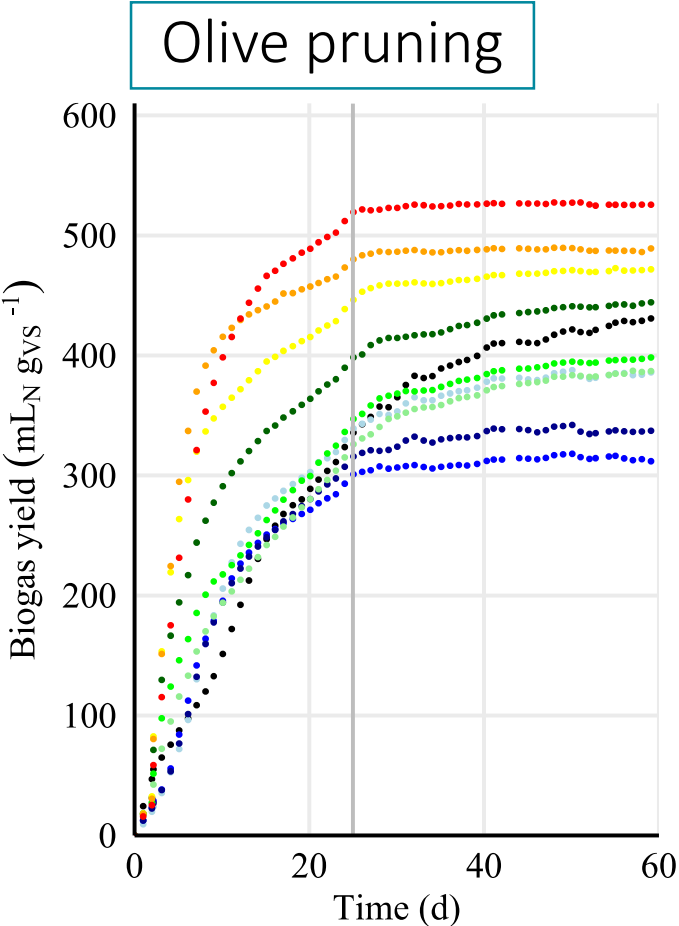
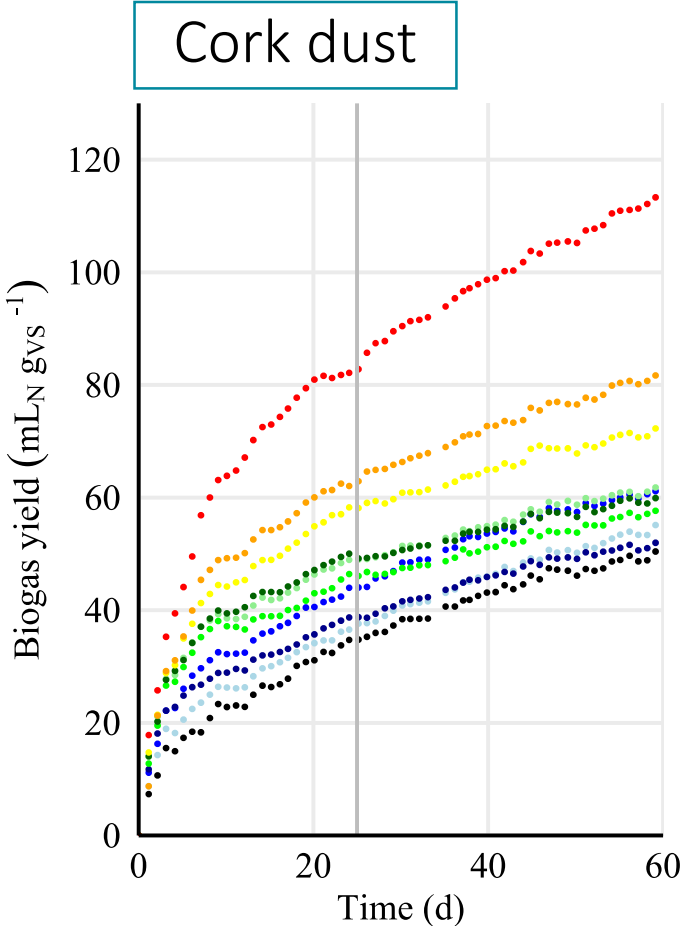


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# Anaerobic degradability of DES pre-treated biomass

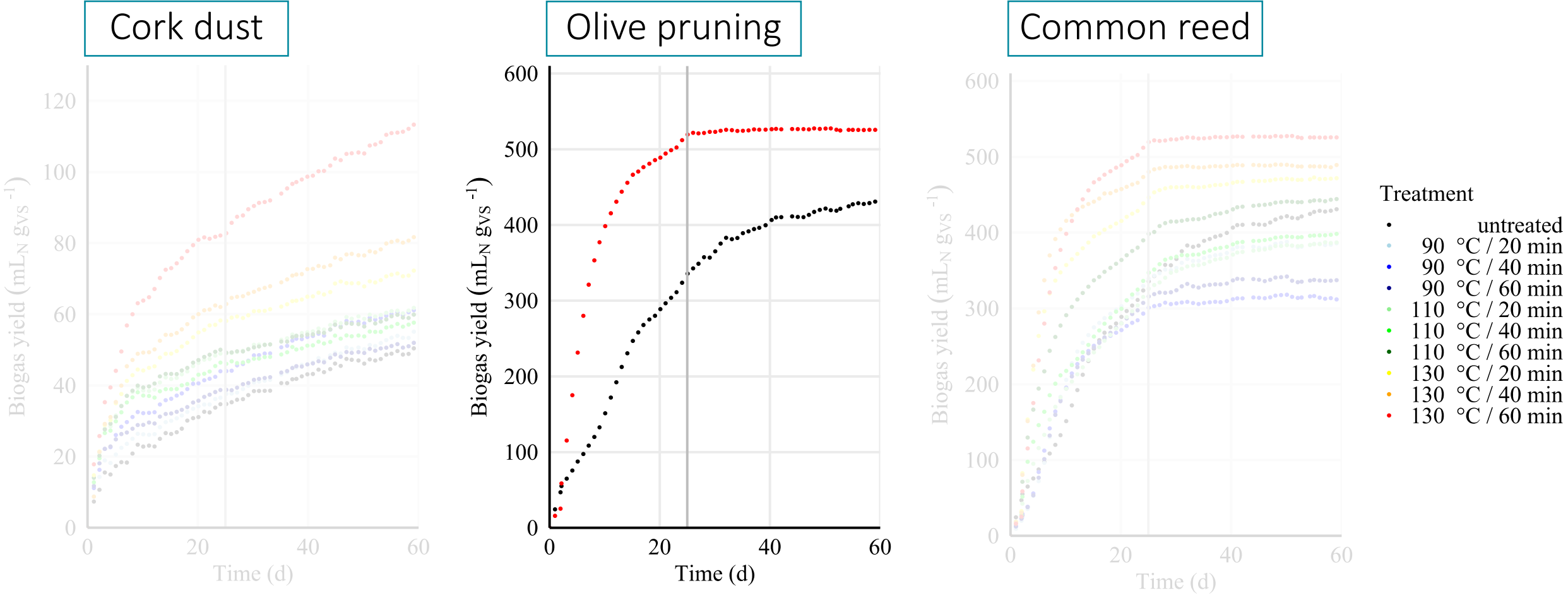
Results - Changes in degradation kinetics



- Treatment
- untreated
  - 90 °C / 20 min
  - 90 °C / 40 min
  - 90 °C / 60 min
  - 110 °C / 20 min
  - 110 °C / 40 min
  - 110 °C / 60 min
  - 130 °C / 20 min
  - 130 °C / 40 min
  - 130 °C / 60 min

# Anaerobic degradability of DES pre-treated biomass

Results - Changes in degradation kinetics

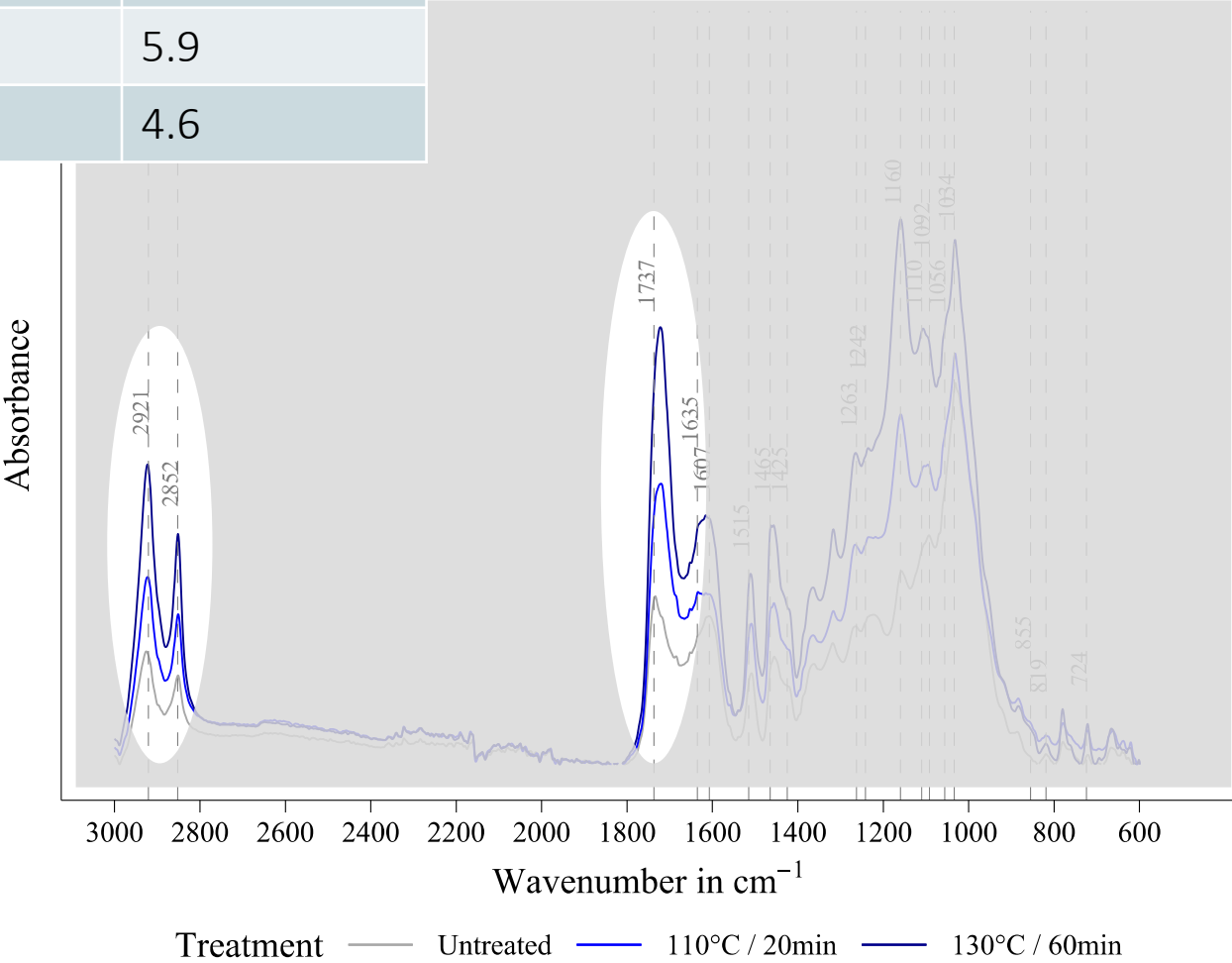


# Anaerobic degradability of DES pre-treated biomass

Results - Cork dust

	Glucose (%)	Lignin (+ suberin %)	Ash (%)
Untreated	9.0	52.0	2.1
110 °C – 20 min	12.6	53.5	5.9
130 °C – 60 min	17.2	56.7	4.6

- Increase in suberin

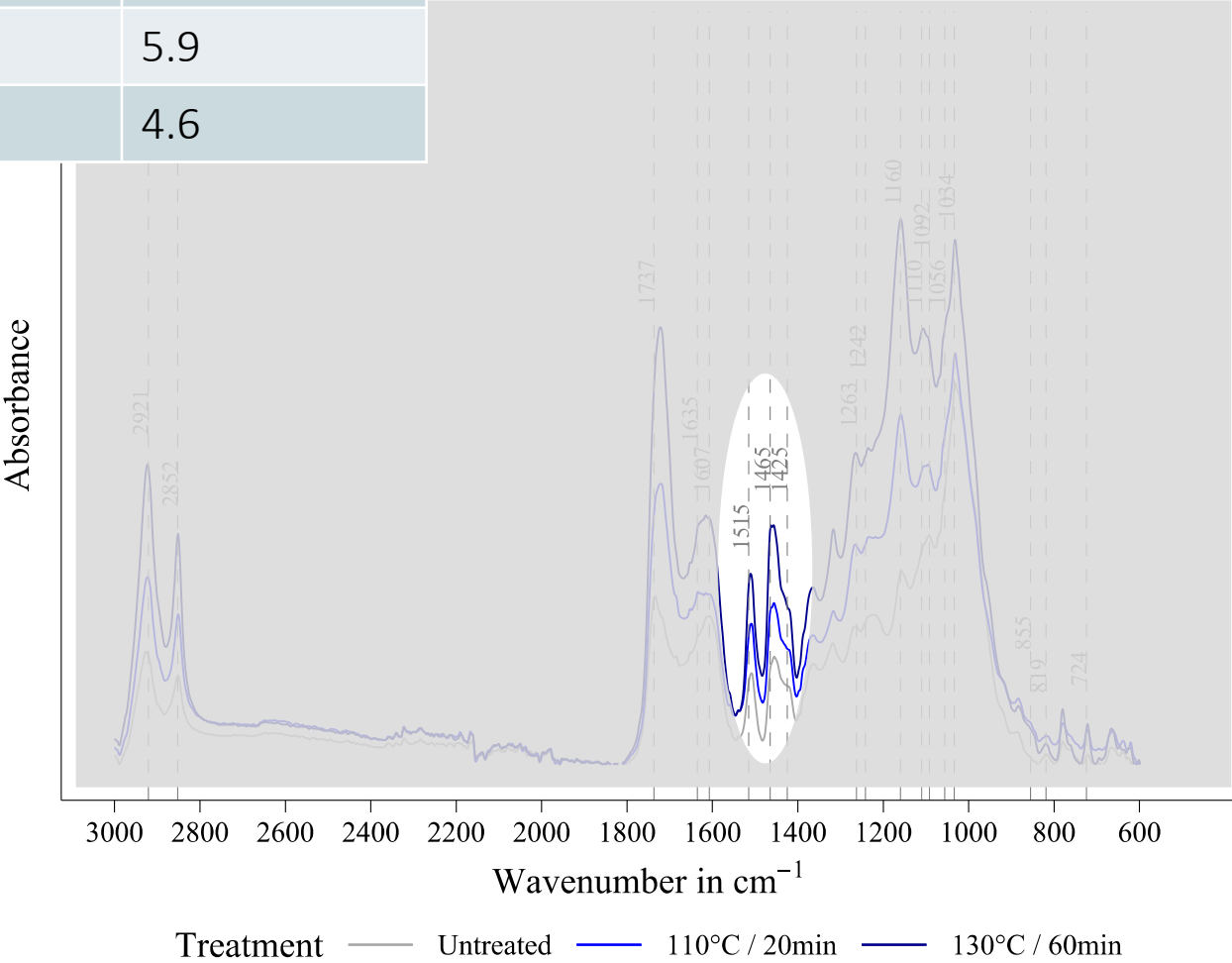


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- Increase in suberin and lignin

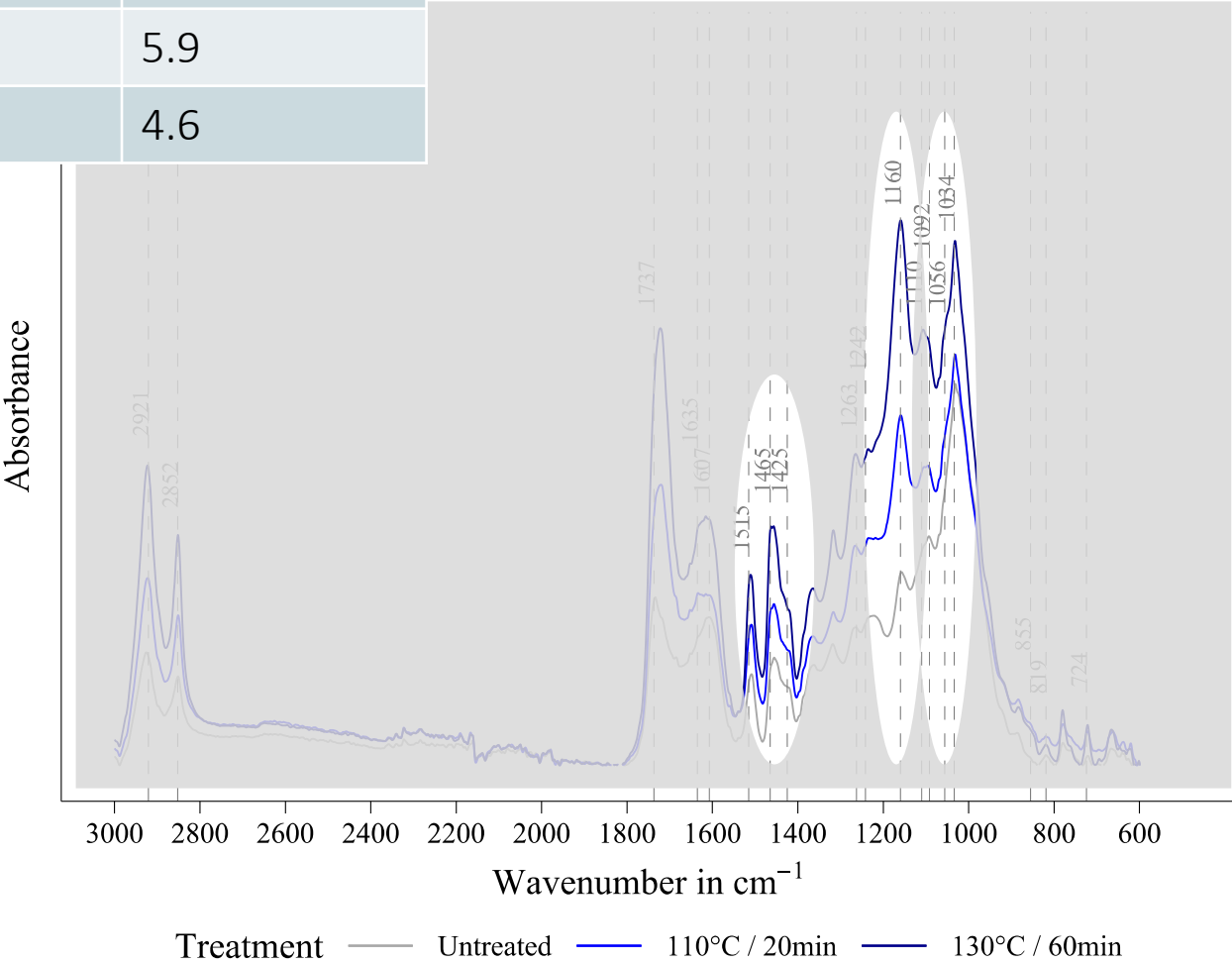


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- Increase in suberin and lignin
- Increase in glucose

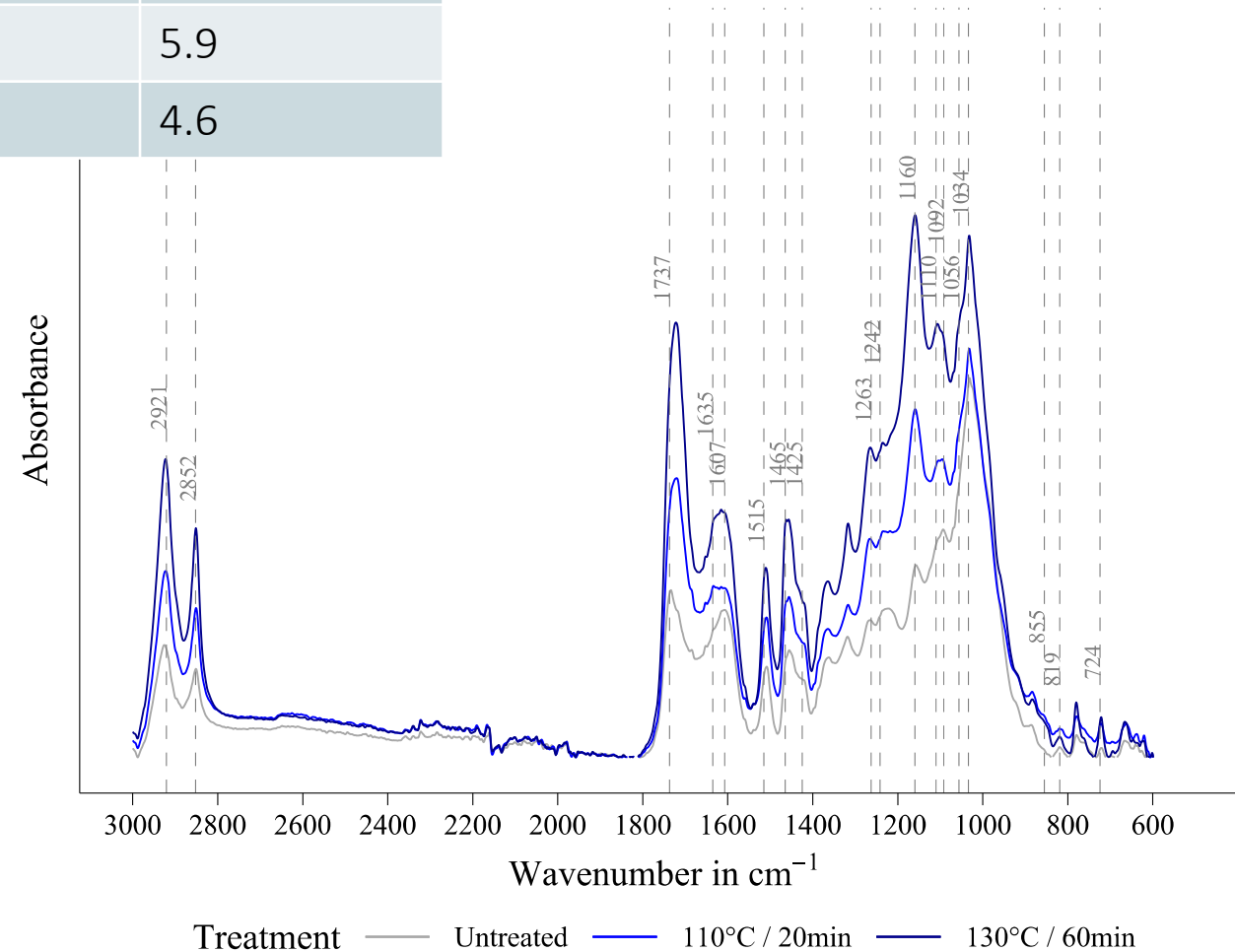


# Anaerobic degradability of DES pre-treated biomass

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- Increase in suberin and lignin
- Increase in glucose
- Relative increase of cellulose to lignin ratio



- Huge differences among investigated biomass streams
- Two major effects on biogas formation
  - Increase of biogas yield
  - Change of degradation kinetics
- Increase associated with increased cellulose:lignin ratio

Thank you for your attention!

Questions?



Jana Schultz M.Sc.

Technische Universität Hamburg (TUHH)

Institut für Umweltechnik und Energiewirtschaft (IUE)

Eißendorfer Str. 40, D-21073 Hamburg

+49 40 42878 4747 | [jana.schultz@tuhh.de](mailto:jana.schultz@tuhh.de) | [www.tuhh.de/iue](http://www.tuhh.de/iue)

Thank you for your attention!

Questions?

# Anaerobic degradability of DES pre-treated biomass

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Jana Schultz M.Sc.

Technische Universität Hamburg (TUHH)

Institut für Umweltechnik und Energiewirtschaft (IUE)

Eißendorfer Str. 40, D-21073 Hamburg

+49 40 42878 4747 | [jana.schultz@tuhh.de](mailto:jana.schultz@tuhh.de) | [www.tuhh.de/iue](http://www.tuhh.de/iue)

- Abbott, Andrew P.; Capper, Glen; Davies, David L.; Rasheed, Raymond K.; Tambyrajah, Vasuki (2003): Novel solvent properties of choline chloride/urea mixtures. In *Chemical communications* (Cambridge, England) (1), pp. 70–71. DOI: 10.1039/B210714G.
- Garcia, Helga; Ferreira, Rui; Petkovic, Marija; Ferguson, Jamie L.; Leitão, Maria C.; Gunaratne, H. Q. Nimal et al. (2010): Dissolution of cork biopolymers in biocompatible ionic liquids. In *Green Chem.* 12 (3), p. 367. DOI: 10.1039/B922553F.
- Gorke, Johnathan T.; Srienc, Friedrich; Kazlauskas, Romas J. (2008): Hydrolase-catalyzed biotransformations in deep eutectic solvents. In *Chemical communications* (Cambridge, England) (10), pp. 1235–1237. DOI: 10.1039/b716317g.
- Lopes, M. H.; Barros, A. S.; Pascoal Neto, C.; Rutledge, D.; Delgadillo, I.; Gil, A. M. (2001): Variability of cork from Portuguese *Quercus suber* studied by solid-state <sup>13</sup>C-NMR and FTIR spectroscopies. In *Biopolymers* 62 (5), pp. 268–277. DOI: 10.1002/bip.1022.
- Lopes, M. H.; Neto, C. Pascoal; Barros, A. S.; Rutledge, D.; Delgadillo, I.; Gil, A. M. (2000): Quantitation of aliphatic suberin in *Quercus suber* L. cork by FTIR spectroscopy and solid-state <sup>13</sup>C-NMR spectroscopy. In *Biopolymers* 57 (6), pp. 344–351. DOI: 10.1002/1097-0282(2000)57:6<344::AID-BIP40>3.0.CO;2-%23.
- Lynam, Joan G.; Kumar, Narendra; Wong, Mark J. (2017): Deep eutectic solvents' ability to solubilize lignin, cellulose, and hemicellulose; thermal stability; and density. In *Bioresource technology* 238, pp. 684–689. DOI: 10.1016/j.biortech.2017.04.079.
- Pandey, Ashish; Bhawna; Dhingra, Divya; Pandey, Siddharth (2017): Hydrogen Bond Donor/Acceptor Cosolvent-Modified Choline Chloride-Based Deep Eutectic Solvents. In *The journal of physical chemistry. B* 121 (16), pp. 4202–4212. DOI: 10.1021/acs.jpcc.7b01724.
- Pascoal Neto, C.; Rocha, J.; Gil, A.; Cordeiro, N.; Esculcas, A. P.; Rocha, S. et al. (1995): <sup>13</sup>C solid-state nuclear magnetic resonance and Fourier transform infrared studies of the thermal decomposition of cork. In *Solid state nuclear magnetic resonance* 4 (3), pp. 143–151. DOI: 10.1016/0926-2040(94)00039-F.
- Qin, Hao; Hu, Xutao; Wang, Jingwen; Cheng, Hongye; Chen, Lifang; Qi, Zhiwen (2020): Overview of acidic deep eutectic solvents on synthesis, properties and applications. In *Green Energy & Environment* 5 (1), pp. 8–21. DOI: 10.1016/j.gee.2019.03.002.
- Rocha, S. M.; Goodfellow, B. J.; Delgadillo, I.; Neto, C. P.; Gil, A. M. (2001): Enzymatic isolation and structural characterisation of polymeric suberin of cork from *Quercus suber* L. In *International journal of biological macromolecules* 28 (2), pp. 107–119. DOI: 10.1016/S0141-8130(00)00163-X.
- Silva-González, José Alberto; Chandel, Anuj Kumar; Da Silva, Silvio Silvério; Balagurusamy, Nagamani (2020): Biogas in Circular Bio-Economy: Sustainable Practice for Rural Farm Waste Management and Techno-economic Analyses. In : *Biogas Production*: Springer, Cham, pp. 389–414. Available online at [https://link.springer.com/chapter/10.1007/978-3-030-58827-4\\_17](https://link.springer.com/chapter/10.1007/978-3-030-58827-4_17).