

Biphasic Hydrothermal Fractionation of Alternative Feedstocks for Man-Made Cellulose Fibers and Platform Chemicals: Process and Scale-Up Perspectives

Andrés Acosta, Ph.D
9th-10th June 2026



Man-made cellulosic fibers (MMCFs)



Natural Cellulosic Fibers^[1]



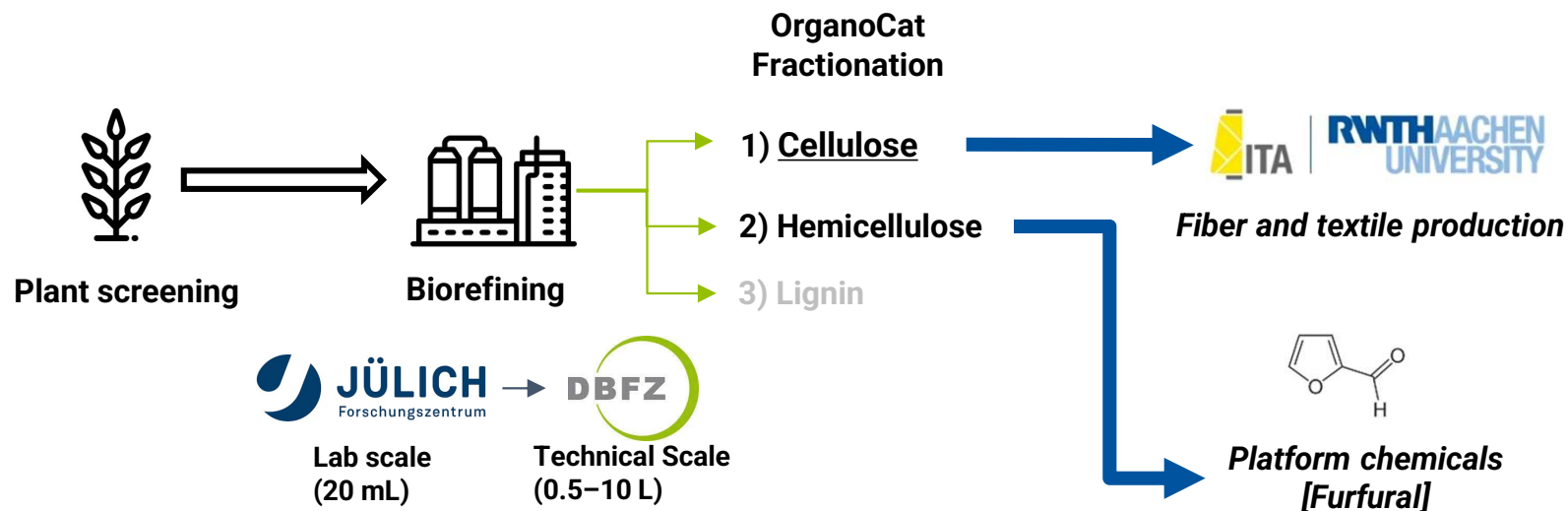
Regenerated Cellulosic Fibers^[1]

- Global textile fibers:
113 → 134 Mt/year by 2030 ^[2]
- Additional demand: +20 Mt/year
- Cotton limited growth: 26–28 Mt/year ^[2]
- Cotton cannot close the gap alone
- Wood-based dissolving pulp under pressure (climate stress, bioeconomy) ^[3]
- Need for alternative cellulose sources (residues) → Fast-growing biomass → MMCF potential.

AltCell2 Project



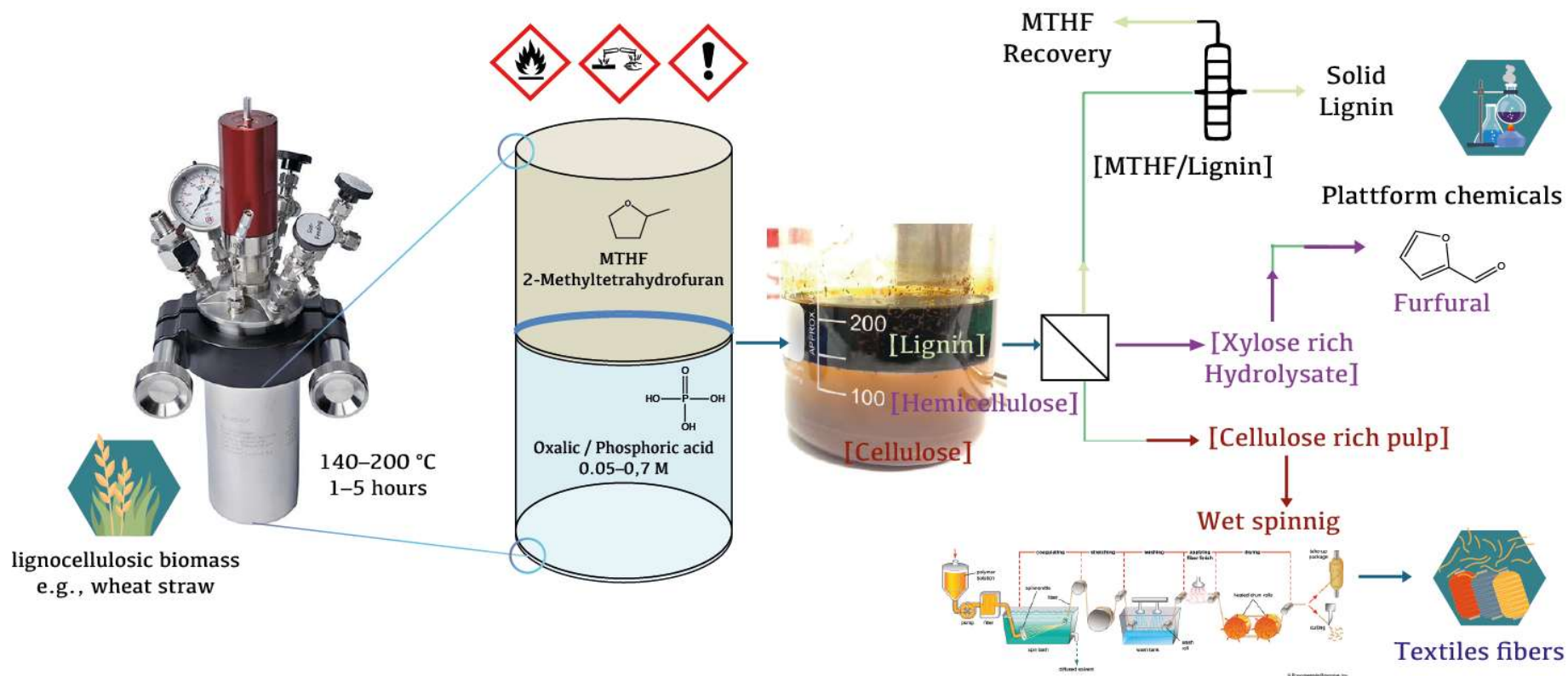
Bundesministerium
für Forschung, Technologie
und Raumfahrt



Product examples



Biphasic Hydrothermal Fractionation



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0.5 L Upscaling



- System modified for biomass fractionation.
- Maximum filling volume estimation
- Phase separation behavior

- Solution: MTHF (1:1 ; 2:1)
- Acid solution (0.05–0.74 M)
- Temp (140, 160, 200, 220 °C)
- Particle size (0.5–1 mm)
- Reaction time (1–3 hr)

- ✓ 2-MTHF safety window
- ✓ Pressure profile generation
- ✓ Solvent expansion / density change
- ✓ Aspen Plus® modelling input

Modules for fractionation system (10 L)



- ✓ Reaction, Stirring & Collection module (RSCM)

1. Aspen-assisted volume estimation

Density change · thermal expansion
20–200 °C · 2-MTHF/H₃PO₄ phases.



- ✓ Hydrothermal membrane system (HTMV)

2. Fixed-volume reactor balance

Hot liquid volume · biomass moisture · dry
biomass displacement · headspace.

3. Pressure validation



4. Effective gas/vapor volume

Pressure-calibrated headspace · VLLE
behavior

5. Validated scale-up loading

Empirical correction · safe filling envelope.

Results



Table 1: Fractionation performance indicators

Process step	Mass balance equation
Pulp yield	$Y_{zs} = \frac{m_{zs}}{m_{ws}} \times 100 \%$
Cellulose recovery	$R_{cell} = \frac{m_{cell,out}}{m_{cell,in}} \times 100 \%$
Hemicellulose removal from pulp	$RM_{hemcell} = \left(1 - \frac{m_{hemcell,out}}{m_{hemcell,in}}\right) \times 100 \%$
Lignin removal from pulp	$RM_{lign} = \left(1 - \frac{m_{lign,out}}{m_{lign,in}}\right) \times 100 \%$
Lignin yield in organic phase	$Y_{lign} = \frac{m_{lign,recov}}{m_{lign,in}} \times 100 \%$
Sugar yield relative to the dry matter content of wheat straw	$Y_s = \left(\frac{m_H + m_{PW}}{m_{ws}}\right) \times 100 \%$
Xylose recovery	$\eta_{xyl} = \left(\frac{m_{xyl,recovered,total}}{m_{xyl,theoretical}}\right) \times 100 \%$

For $x \in \{cell, lign, hemcell\}$ holds: $m_{x,in} = m_{ws} \cdot w_{x,ws}$ and $m_{x,out} = m_{zs} \cdot w_{x,zs}$
H: Hydrolysate; PW: Process water (washing stage)

Table 2: High temperature MTHF fractionation–dry-matter basis (10 L system)

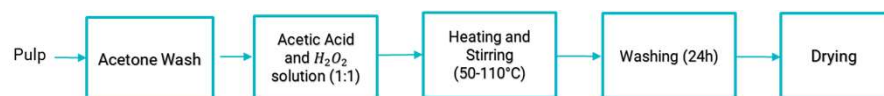
Experiment	P _{max} [bar]	Y _{zs} [%]	R _{cell} [%]	RM _{hem.cell} [%]	RM _{lign} [%]	Y _{lign} [%]
1:1 140 0.74	16 ± 1	52 ± 0	84 ± 1	78 ± 1	62 ± 3	27 ± 1
2:1 140 0.74	16	52	76	86	66	17
1:1 200 0.05	27	48	91	84	81	56
2:1 200 0.05	29 ± 1	46 ± 1	69 ± 1	93 ± 2	80 ± 1	48 ± 1

- ✓ **Pulp yield** remains in a narrow range 48–52 %
- ✓ Interesting cellulose recovery: higher severity [Ratio 1:1 | 200 °C | 0.05 M] with up to 91 %.
- ✓ **Higher temperature improves fractionation:** hemicellulose removal remains high, while lignin removal increases strongly from ~62–66% at 140 °C to ~80–81% at 200 °C
- ✓ **Phase ratio matters:** increasing from 1:1 to 2:1 reduces cellulose recovery, so more solvent/aqueous phase is not automatically better

Pulp bleaching and biopolymers analysis



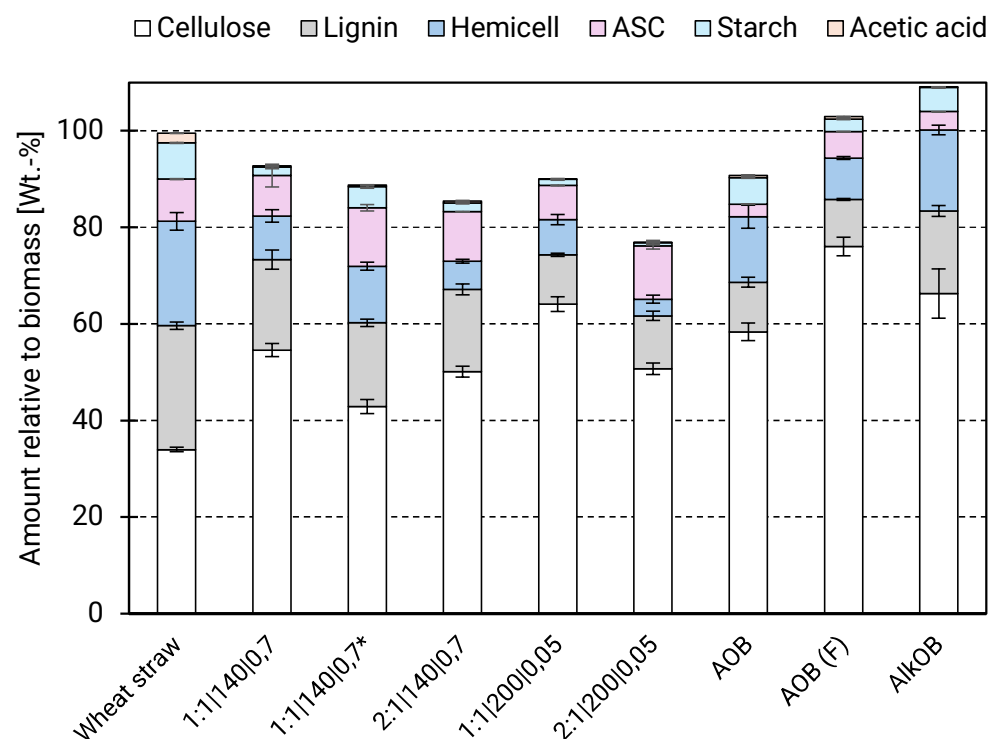
Pulp obtained after fractionation (left) and acidic bleached pulp (right) @ DBFZ



Acidic bleaching (© Andrés C. Acosta, 2026)



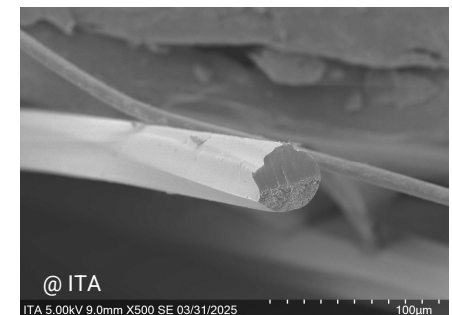
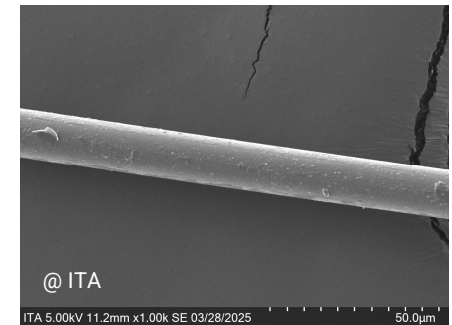
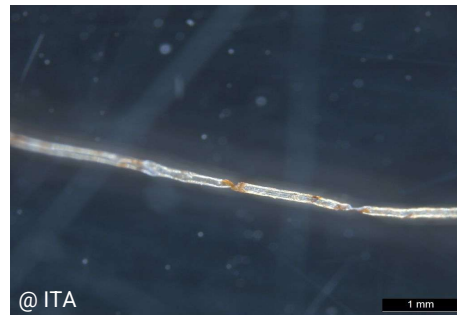
Alkaline bleaching (© ITA, 2026)



Biopolymer composition of pulp and bleached pulp (dry basis)

mean ± standard deviation based on three individual measurements (© Jülich Forschungszentrum; Andrés C. Acosta, 2026).

From Wheat Straw Pulp to Fibers (15 cm)



Gel Permeation Chromatography (GPC)

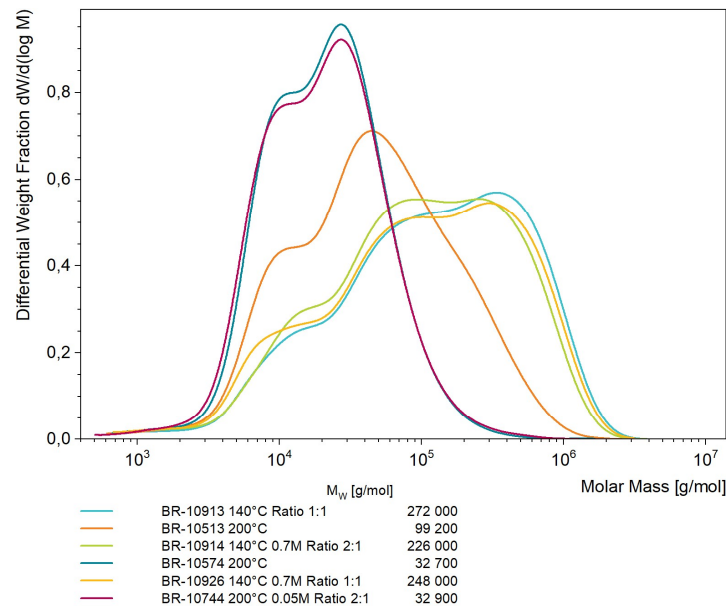


Table 3: GPC analysis of pulps

Experiment	M_n [kg/mol]	M_w [kg/mol]	D^*	DP^{**}
1:1 140 0.74	32	272	8,6	722
2:1 140 0.74	16	52	7,7	628
1:1 200 0.05	27	48	5,2	276
2:1 200 0.05	29	46	2,5	91

* **Dispersity** is defined as the ratio of M_w to M_n .

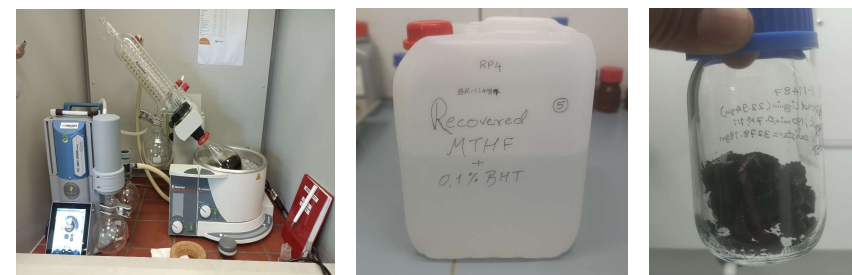
** The **degree of polymerization** was calculated based on the average molar mass of the **anhydroglucose unit** of cellulose

- ✓ Target for processing: $DP > 200$
- High D : broad chain-length distribution
- Low D : more homogeneous cellulose pulp

Xylose rich hydrolysates



View into the modified HTMV (left) and phase separation of the organic phase (right) (© Andrés C. Acosta, 2026).



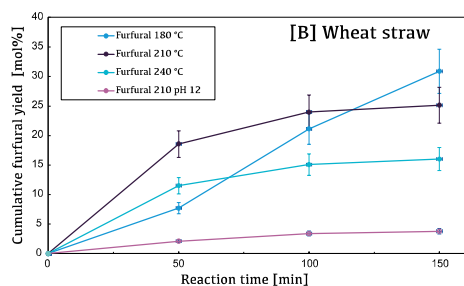
MTHF Recovery (© Andrés C. Acosta & A. Sunil 2026)

Table 3: High temperature MTHF fractionation–dry-matter basis (10 L system)

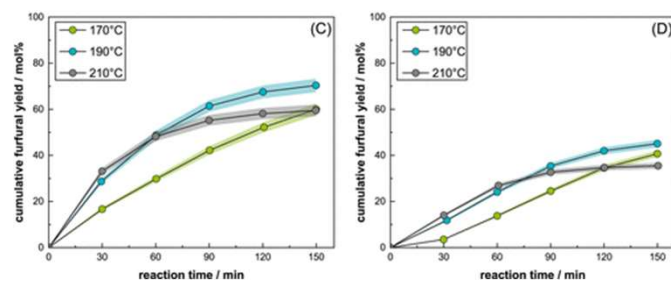
Experiment	Glucose	Xylose	Arabinose	Mannose	HMF	Furfural
	(mg/L)					
1:1 140 0.74	634 ± 66	4315 ± 120	524 ± 37	n.d	n.d	254 ± 75
2:1 140 0.74	658	4290	422	n.d	n.d	459
1:1 200 0.05		n.d	n.d	3030	222	492
2:1 200 0.05	227 ± 42	n.d	69 ± 1	3875 ± 191	80 ± 1	871 ± 21

Sugar monomers measured with HPLC-RID and Furanic aldehydes with HPLC-DAD
Lignin is recovered using the rotary evaporator
Acosta, A. C. (2025). [\[Dissemination video\]](#). Fast Forward Science 2025^[4].

Hydrothermal reactive distillation (HRD)

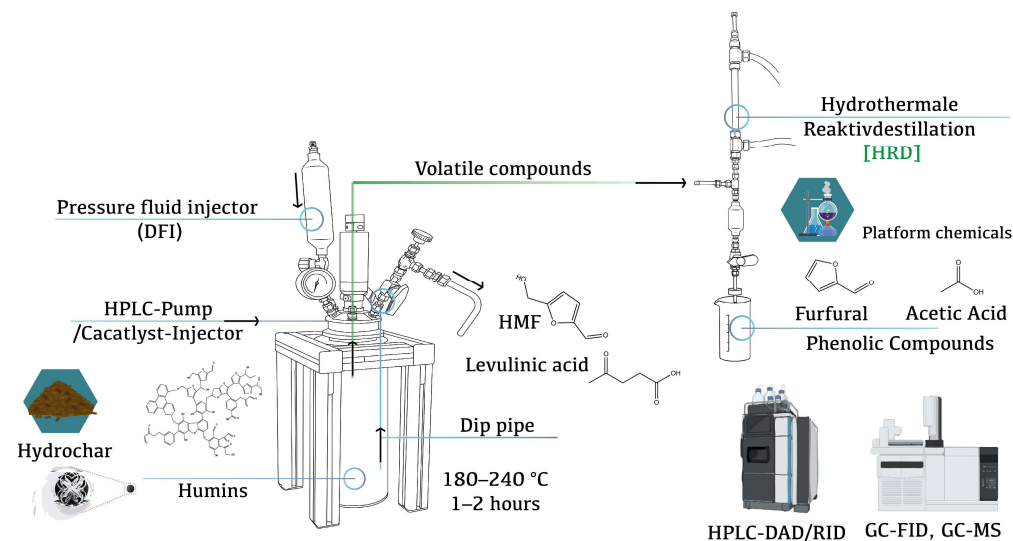


Cumulative furfural yield using Wheat Straw (© Köchermann & Acosta; 2026)



Cumulative furfural yield sepl husk hydrolysate (left) and Organosolv hemicellulose (right) (© Köchermann & Klemm; 2023)^[5]

✓ Hydrothermal reactive distillation for furfural synthesis.

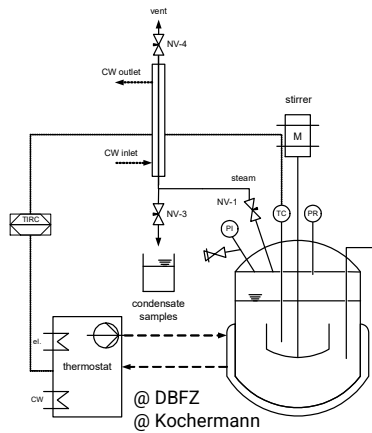


(© Andrés C. Acosta, 2026)

(© Köchermann & Klemm; 2023)^[5]

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Hydrothermal reactive distillation (HRD)



Reflux condenser design and installation in fume hood.



Upscaling HRD system in Hydrothermal membrane system (HTMV) for integration to 10 L reactor



(© Köchermann & Klemm; 2023)^[5]

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Thank you !

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References

- [1] <https://mikem.com/news/what-is-cellulose-fiber/>
- [2] <https://doi.org/10.1016/j.jil.2025.100178>.
- [3] <https://datalab.dbfz.de/resdb?lang=en>
- [4] Acosta, A. C. (2025). [\[Dissemination video\]](#). Fast Forward Science 2025
- [5] <https://doi.org/10.1021/acs.iecr.3c00259>

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