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Haute Ecole Spécialisée de Suisse occidentale
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Master of Science HES-SO in Life Sciences

An Industrial Approach for the Synthesis of functionalized-Xylose via Direct Trans-acetalization

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CHEMICAL DEVELOPMENT & PRODUCTION

HEIA-FR

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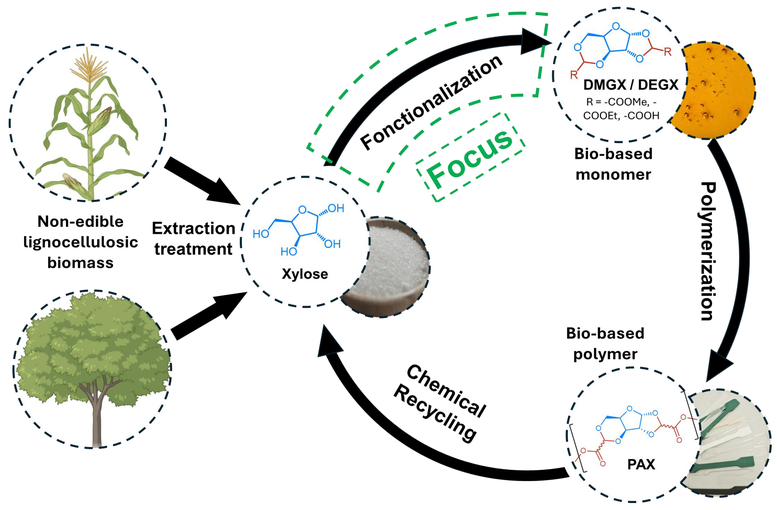
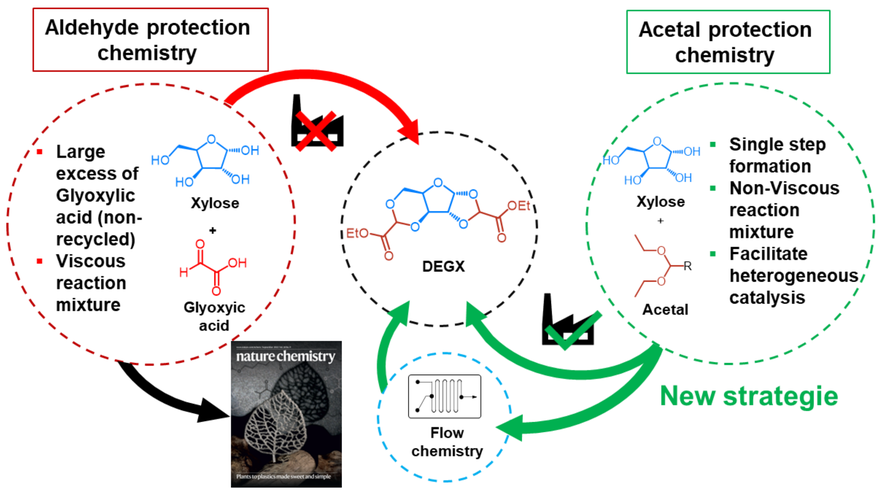
Introduction & Context



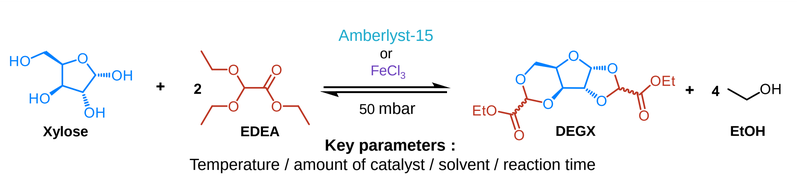
Goal of the projet

- **Developing novel bio-based monomers is crucial** for creating sustainable alternatives to fossil-based chemicals.
- This project explores a **novel synthetic pathway** for producing a xylose-derived monomer through a **trans-acetalization mechanism** using ethyl diethoxyacetate (EDEA) as both a solvent and a reactant.



RESULTS & DISCUSSION

1.Optimization of the DEGX reaction



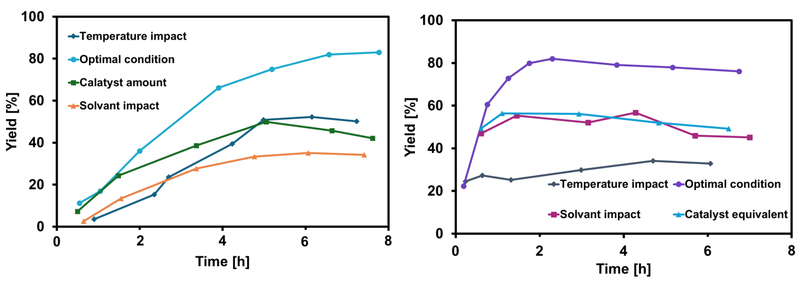
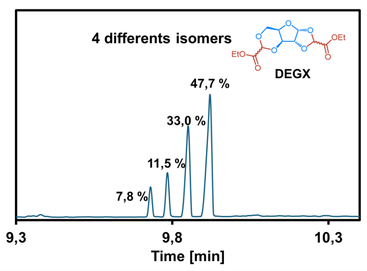


Table 1 : Opitmized condition for DEGX reaction

Catalyst	Temperature [°C]	Equivalent of catalyst [-]	Solvent	Reaction time [h]	Yield [%]
Amberlyst-15	95	0,24	no	6	~80
$FeCl_3$	95	0,4	no	1,5	~80



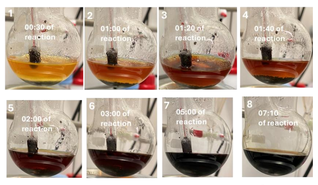
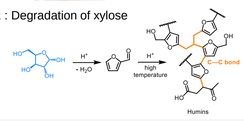


Figure 1 : Degradation of xylose

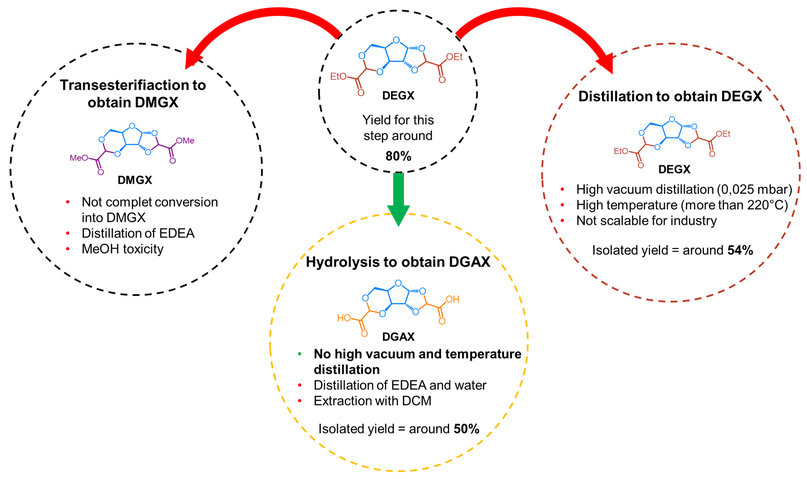


TAKE HOME MESSAGE

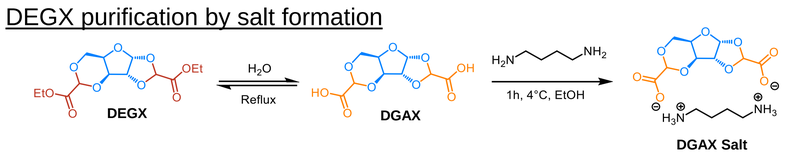
- The reaction **yields up to 80%.**
- Amberlyst-15 can be **recycled**
- Reaction time is **4h faster with FeCl3**
- Xylose degraded into humins

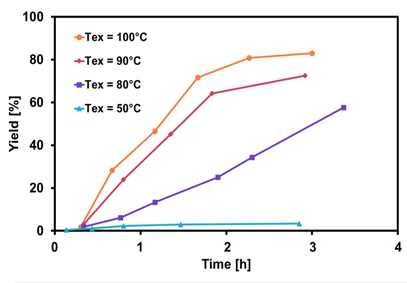
2. Purification of DEGX

Different pathways to purify DEGX



DEGX purification by salt formation





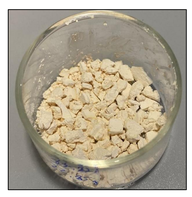


Figure 2 : DGAX Salt from curde DGAX

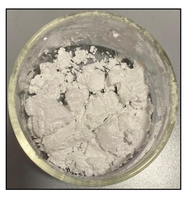


Figure 3 : DGAX Salt from pur DGAX

TAKE HOME MESSAGE

- The saponification **yields up to 83% after 2h**
- DGAX isolated yield is **92% from the crude**
- **DGAX degrades to xylose** above 100°C
- **Hydrolysis without catalyst**

3.DEGX scale-up

Thermal Safety assessment:

Catalyst Addition	ΔT_{ad} [°C]
Amberlyst-15	7
$FeCl_3$	34

TAKE HOME MESSAGE

- **Scale-up to 40g** of xylose
- The reaction **yields up to 70%.**
- **EDEA is recycled** with a yield of 80%

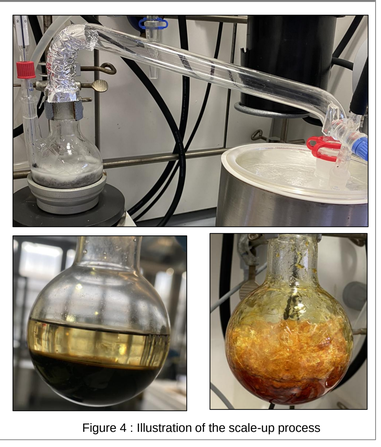


Figure 4 : Illustration of the scale-up process

4. Flow chemistry of DEGX reaction

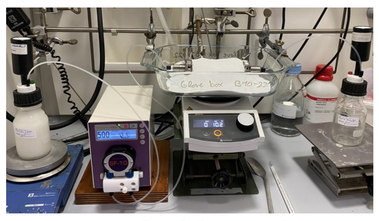


Figure 5 : Flow chemistry Set-up

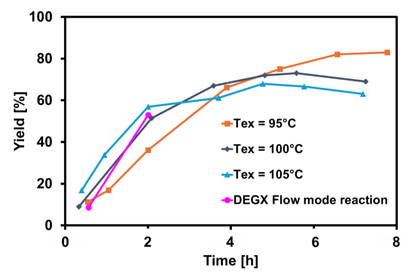


Figure 2 : Impact of the residence time on yield for DEGX reaction

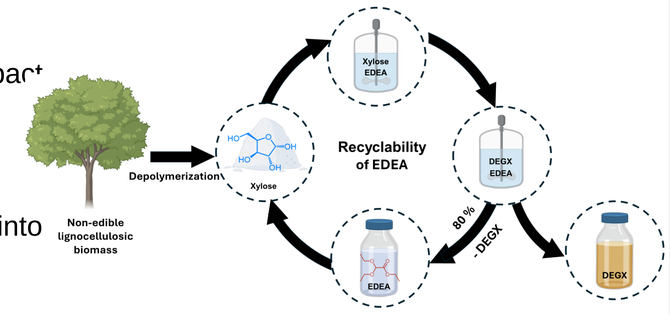
Entry	Flow [mL/min]	Solution type (EDEA + xylose)	External temperature [°C]	Equiv of EDEA [-]	Yield [%]
1	0,5	Suspension	110	6	0
2	0,5	Solution	110	6	45
3	0,25	Solution	110	6	53
4	0,15	Solution	110	6	41

TAKE HOME MESSAGE

- Performance similar to batch reaction
- The residence time impact the yield of the reaction

CONCLUSION

- **Amberlyst-15** and **$FeCl_3$** achieve the same yield, ca **80%**, for the DEGX reaction.
  - The **advantage of $FeCl_3$** in terms of **reaction speed** is counterbalanced by its **thermal instability** and **higher environmental** impact compared to Amberlyst-15.
- **DEGX purification** remains a **major challenge** across all processes, with **hydrolysis** appearing to be the **most industrially viable option**.
- **Flow chemistry** poses challenges for this reaction, but exploratory tests show **promising trends** similar to batch mode. Further investigation into extending the **residence time** could **enhance yield** and process efficiency.



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