

# Evaluation of a green pressurized reaction media (subW-CO<sub>2</sub>) and pressurized microwave-assisted reaction for furfural production from corn stover and its derivatives sugars

A.E. Illera<sup>1\*</sup>, H. Candela<sup>1</sup>, P. Barea<sup>1</sup>, A. Bermejo-López<sup>2</sup>, Ó. Benito-Román<sup>1</sup>, R. Melgosa<sup>1</sup>, S. Beltrán<sup>1</sup>, M.T. Sanz<sup>1</sup>

<sup>1</sup>Department of Biotechnology and Food Science, University of Burgos, Pl. Misael Bañuelos s/n, 09001 Burgos, Spain

<sup>2</sup>Department of Chemical Engineering, University of the Basque Country UPV/EHU, Barrio Sarriena, s/n, 48940 Leioa, Bizkaia, Spain

\*Corresponding author: aeillera@ubu.es

CO<sub>2</sub> EIFS 2024  
3<sup>er</sup> Encuentro Ibérico de Fluidos Supercríticos



## CORN STOVER AND ITS DERIVATIVES SUGARS



Corn stover is a **lignocellulosic biomass** with a valuable carbohydrates composition.

In corn production, residues constitute **50 %** of the total weight: **Corn rachis and corn stover**



Corn stover (leaves and stems)

### CORN STOVER (CS) COMPOSITION

Component		g/100g dry-CS
Hemicellulose	<b>Xylans</b>	25 ± 3
	<b>Arabinans</b>	4.1 ± 0.5
	Acetyl	4.2 ± 0.2
Cellulose	Glucans	41.4 ± 4
	Total lignin	18.7 ± 3
Protein		3.2 ± 0.2
Ash		2.7 ± 0.4

## FURFURAL FROM HEMICELLULOSE

- ✓ Furfural is considered one of the **top value-added chemicals** derived from biomass.
- ✓ It is the **dehydration product of pentoses** (xylose and arabinose in corn stover).
- ✓ C<sub>5</sub> polysaccharides are first **hydrolyzed from the hemicellulose** to then produce furfural.

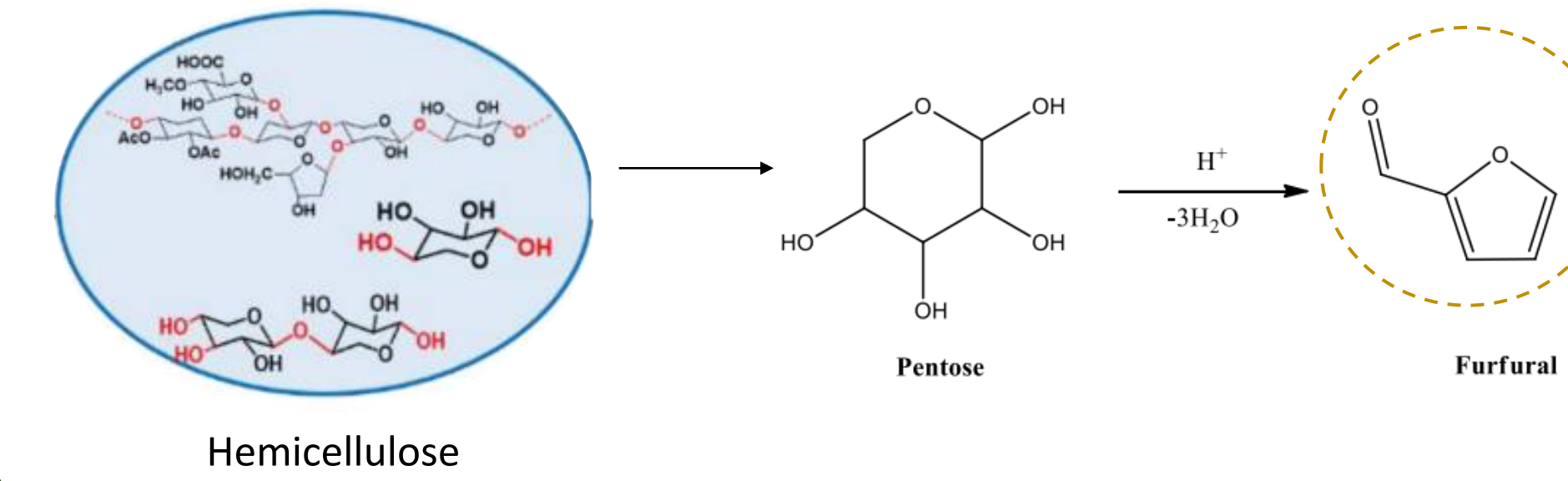


Figure 1. Furfural production from hemicellulose.

## SubW-CO<sub>2</sub> SYSTEM

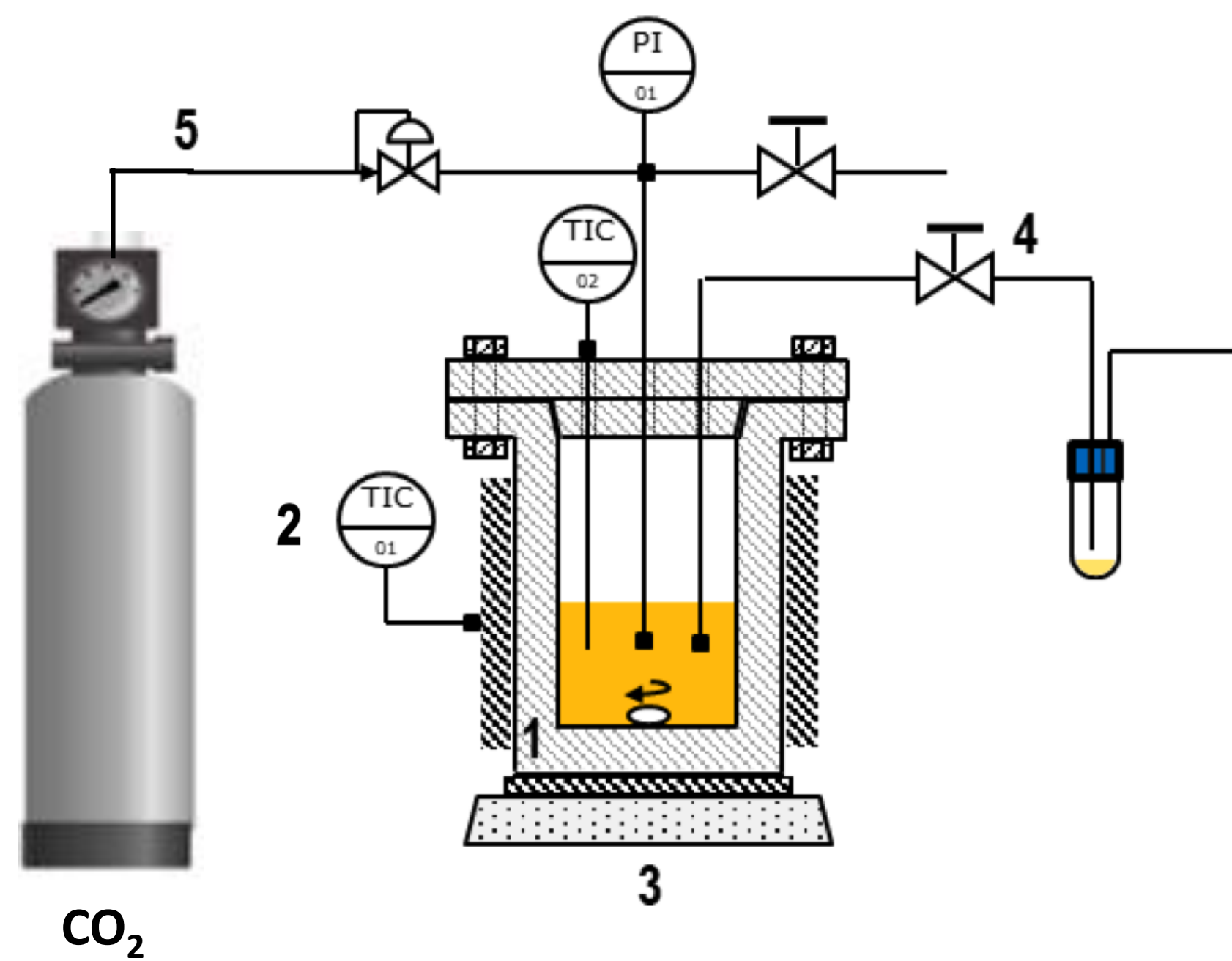


Figure 2. Diagram of the laboratory-scale subcritical water equipment. 1: pressure vessel; 2: heating jacket; 3: magnetic stirring; 4: sample valve; 5: pressurized gas system.

Two green technologies were proposed to produce furfural from xylose and corn stover with a Lewis acid catalyst only using **water as reaction medium**

Xylose load: 11g/L  
Corn stover load: 5 % (w/w)  
CrCl<sub>3</sub> as catalyst (2% w/xylose weight)

SubW-CO <sub>2</sub> SYSTEM		MW SYSTEM	
Volume	200 mL	Volume	30 mL
Pressure	50 bar	Pressure	10 bar
Temperature	180 °C	Temperature	180 °C
Time	120 minutes	Time	5-90 minutes

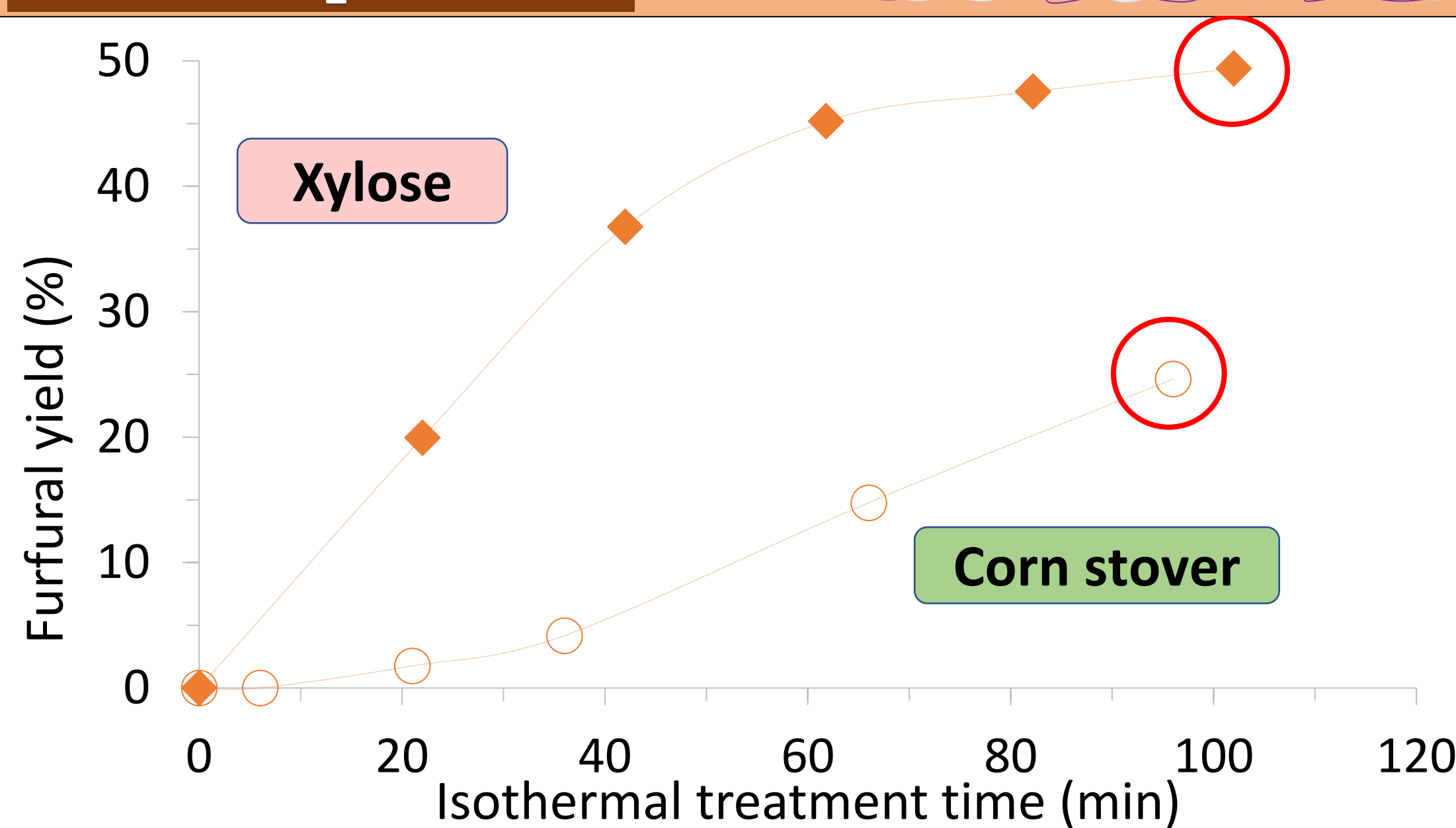
## PRESSURIZED MW SYSTEM



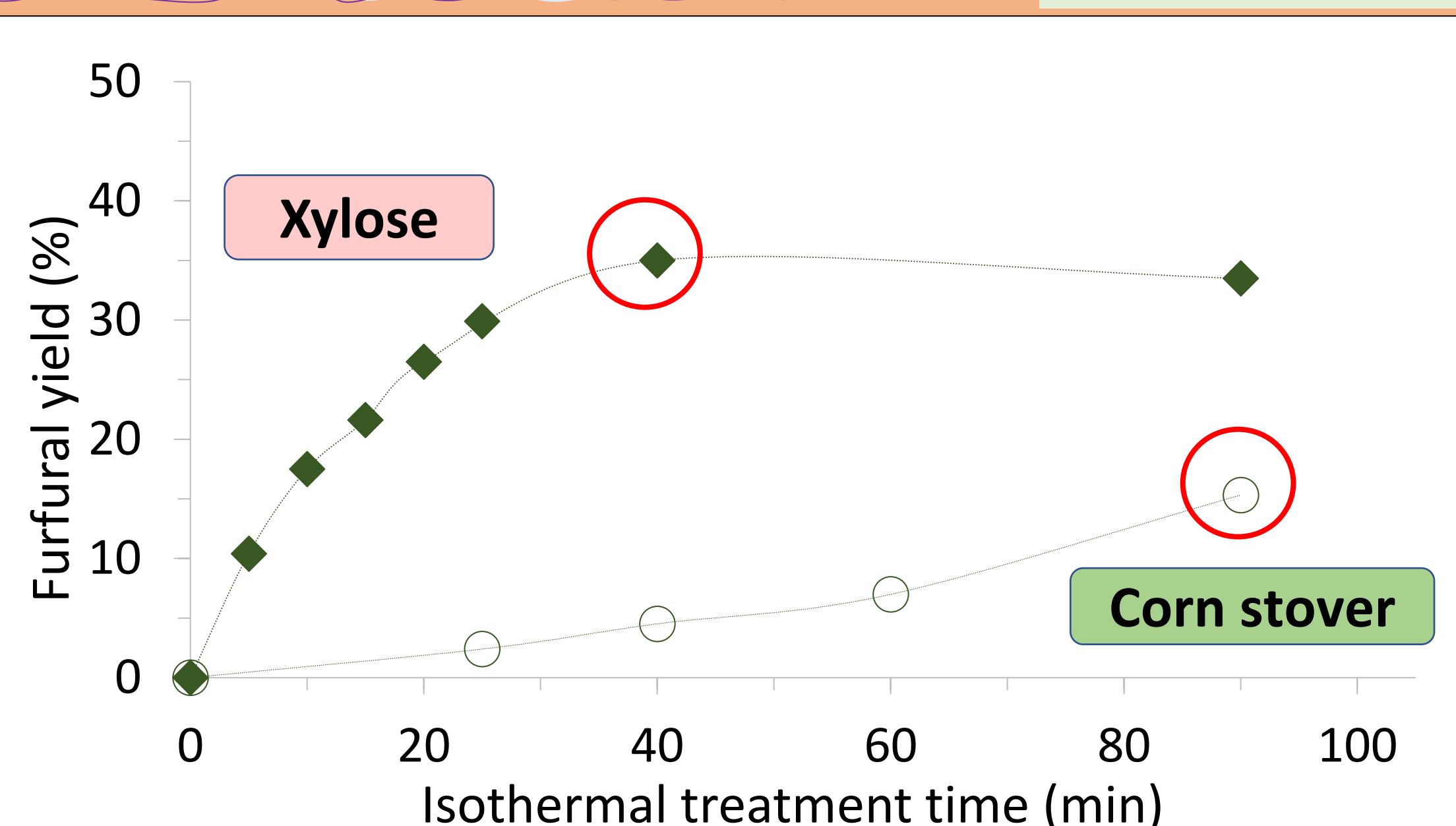
Figure 3. Microwave (MW) equipment.

$$\text{Furfural yield (\%)} = \frac{\text{moles of C in furfural}_{(t)}}{\text{moles of C in pentoses monomers}_{(0)}} \cdot 100$$

## SubW-CO<sub>2</sub> SYSTEM



## MW SYSTEM



## CONCLUSIONS

- Both technologies showed their **viability to produce furfural**.
- **Corn stover produced less furfural** than xylose due to its **more complex matrix**.
- **subW-CO<sub>2</sub>** produced the **highest furfural yield** from xylose (50 %) and corn stover (25 %) when compared to MW.
- The **presence of CO<sub>2</sub>** was a **key parameter** for furfural production, acting as a **Brønsted acid**.
- **subW-CO<sub>2</sub>** produced **less degradation products** than MW.

Subcritical water pressurized by CO<sub>2</sub> showed to be an effective combination for the production of furfural from xylose and corn stover.

- ❖ Xylose yielded **more furfural** than corn stover.
- ❖ **Corn stover** required **more time** than xylose to produce furfural.
- ❖ In **corn stover**, hemicellulose needs to first be **hydrolyzed into pentoses** to then produce furfural.

### Highest furfural yield values

Substrate	MW	subW
Xylose	35 %	50 %
Corn stover	15 %	25 %

MW technology produced lower furfural yield than subW-CO<sub>2</sub>

- ❖ The main difference between the two technologies is the use of **CO<sub>2</sub>** as pressurizing agent in the **subW system**.
- ❖ **Dissolved CO<sub>2</sub>** formed **carbonic acid** in water, which acted as a **Brønsted acid**, favouring furfural production.

## References

- [1] Luo, Y., Zheng, L., Li, X., Liu, X., Fan, J., Clark, J.H., Hu, C. Catalysis Today, 319, 14-24, 2019.
- [2] Li, H.Y., Xu, L., Liu, W.J., Fang, M.Q., Wang, N. Asian-Australasian Journal of Animal Sciences, 27(2), 194-200, 2014.
- [3] Alonso-Riaño, P.; Illera, A. E.; Amândio, M. S. T.; Xavier, A. M. R. B.; Beltrán, S.; Teresa Sanz, M. Sep Purif Technol, 309, 2023.
- [4] Choudhary, V., Mushrif, S.H., Ho, C., Anderko, A., Nikolakis, V., Marinkovic, N.S., Frenkel, A.I., Sandler, S.I., Vlachos, D.G. J. Am. Chem. Soc., 135,10,3997-4006, 2013.

## Acknowledgements

This work was supported by the Agencia Estatal de Investigación (AEI), Ministerio de Ciencia e Innovación (MICINN) and Next Generation UE [grant numbers PID2022-136385OB-I00, PID2020-116716RJ-I00, TED2021-129311B-I00 and PDC2022-133443-I00] and the Junta de Castilla y León (JCyL) and the European Regional Development Fund (ERDF) [grant number BU027P23]. Benito Román post-doctoral contract was funded by AEI through project PID2020-116716RJ-I00. R. Melgosa contract was funded by a Beatriz Galindo Research Fellowship [BG20/00182]. P. Barea predoctoral contract was funded by JCyL and the European Social Fund (ESF) by ORDEN EDU/1868/2022, de 19 de diciembre. H. Candela contract was funded by TED2021-129311B-I00.



NOS IMPULSA  
Junta de Castilla y León