Oligosaccharide and furan obtaining from apple pomace by means of a biphasic reaction system

Itsasne Beitia[1,2], Amaia Quintela[2], Itziar Egüés[1,2], Iván Rivilla[2], Jalel Labidi[1,2]

[1]Chemical and Environmental Engineering Department, University of Basque Country UPV/EHU, Plaza Europa 1, 20018, Donostia-San Sebastián, Spain

[2] Ma+D-Waste Recovery Engineering, Edificio Joxe Mari Korta—Avda Tolosa 72, 20018 Donostia-San Sebastián, Spain

Oligosaccharides are short chains of sugars linked by glycosidic bonds, which play crucial roles in cellular communication, molecular recognition, adhesion functions, and serving as prebiotics in the digestive system. Due to its prebiotic and antimicrobial activities, they have gained interest in pharmaceutical, cosmetic or food industries[1].

The **AIM** of this work is the extraction of oligosaccharides by means of a biphasic system and production and separation of furans (furfural and hydroxymetylfurfural (HMF)). Oligosaccharides are detected in the aqueous phase, while furans should go to the organic phase.

Materials and methods

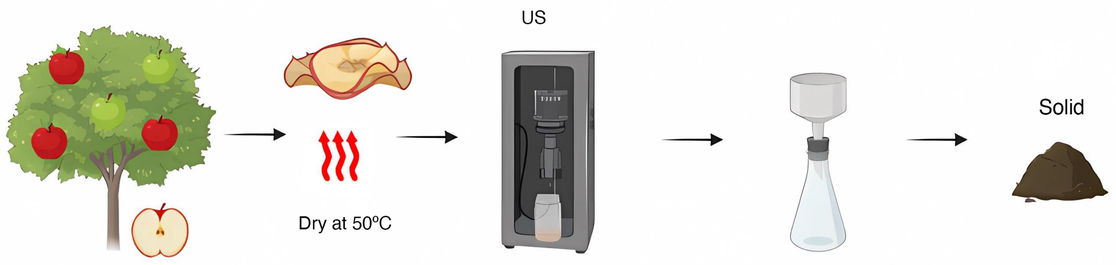
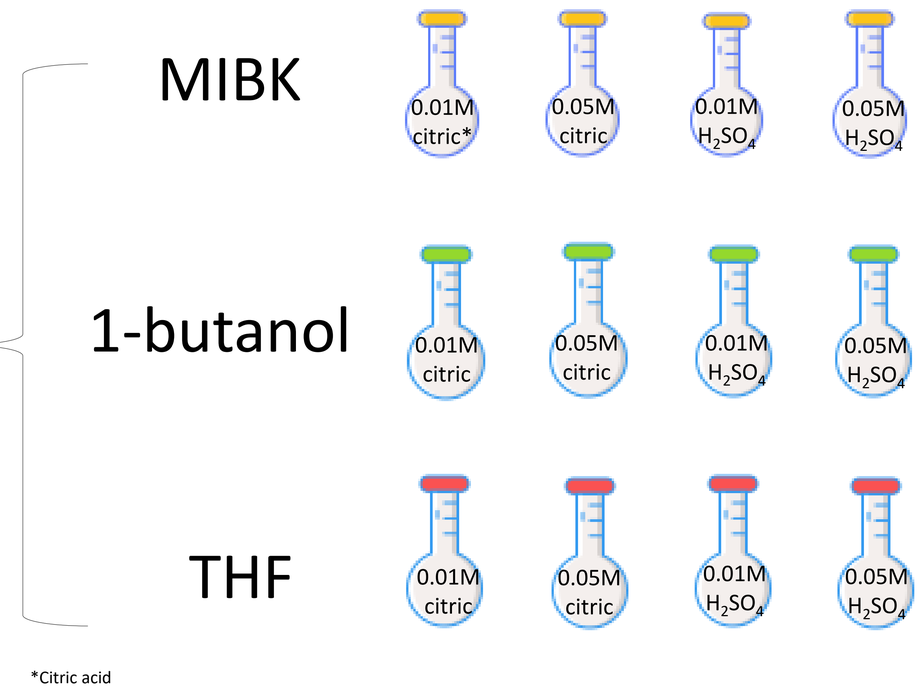


Figure 1. *Schematic representation of apple pomace obtaining and preparation for the biphasic system.*

Biphasic system: Organic + acid aqueous solvent +



*Citric acid

- Microwave assisted extraction (MAE) experiments conditions: 180°C, 15 mins, 500W, solid + biphasic system.
- Purification: liquid-liquid extraction. The aqueous phase obtained containing oligosaccharides was analyzed by high performance liquid chromatography (HPLC). The organic phase containing furans was analyzed by gas chromatography-mass spectrometry (GC-MS).
- In order to detect oligosaccharide extracted quantity, an acid posthydrolysis at 121°C and 30 minutes was carried out for the aqueous phase, in the autoclave. This results were also analyzed by HPLC and they were subtracted to the ones obtained by the previous hydrolysis.
- HPLC was carried out using two different columns. Hydrogen (H) column to make sure all furans go to the organic phase and plumb (Pb) column to analyze oligosaccharides.

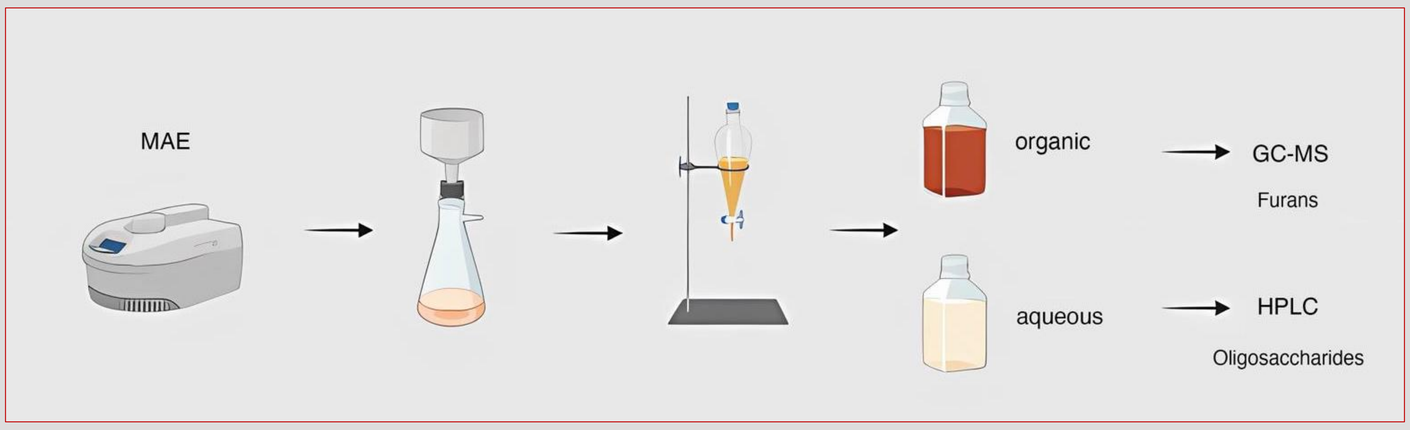


Figure 2. *Schematic representation of MAE followed by separation for furan and oligosaccharide obtaining.*

Results and discussion

Table 1. *Results obtained by HPLC with H column for furans.*

Sample (g/L)	[Acetic] (g/L)	[HMF] (g/L)	[Furfural] (g/L)
MIBK_0.01M citric	0.86	0.63	0.09
MIBK_0.05M citric	0.96	0.64	0.14
MIBK_0.01M H_2SO_4	0.91	0.57	0.11
MIBK_0.05M H_2SO_4	1.28	1.16	0.26
But_0.01M citric	-	-	-
But_0.05M citric	-	-	-
But_0.01M H_2SO_4	-	-	-
But_0.05M H_2SO_4	-	0.49	0.46
THF_0.01M citric	-	-	-
THF_0.05M citric	-	-	-
THF_0.01M H_2SO_4	-	-	-
THF_0.05M H_2SO_4	-	-	-

- : Non detected

Table 2. *Results obtained by HPLC with Pb column for oligosaccharides.*

Sample (g/L)	[GO] (g/L)	[XO] (g/L)	[ArO] (g/L)
MIBK_0.01M citric	1.83	0.16	0.46
MIBK_0.05M citric	1.00	0	0
MIBK_0.01M H_2SO_4	2.03	0	0.40
MIBK_0.05M H_2SO_4	5.96	0	0
But_0.01M citric	14.77	8.90	10.89
But_0.05M citric	2.49	0	4.72
But_0.01M H_2SO_4	2.94	0	2.67
But_0.05M H_2SO_4	8.66	1.36	3.05
THF_0.01M citric	12.95	0	0
THF_0.05M citric	15.04	13.13	19.43
THF_0.01M H_2SO_4	15.68	15.40	18.39
THF_0.05M H_2SO_4	0	0	0

HPLC

- Results obtained with H column have shown furans in the aqueous phase of MIBK, so this solvent has been discarded.
- In the case of Pb column, **Table 2** shows the oligosaccharide (glucose, xylose and arabinose) obtained, where some results were negative. Notably, 1-butanol has shown the best results, including the fact that for THF the 2 phases were not separated in the extraction and NaCl had to be added.

GC-MS

- The only acid solvent able to produce furans was 0.5 M H_2SO_4.

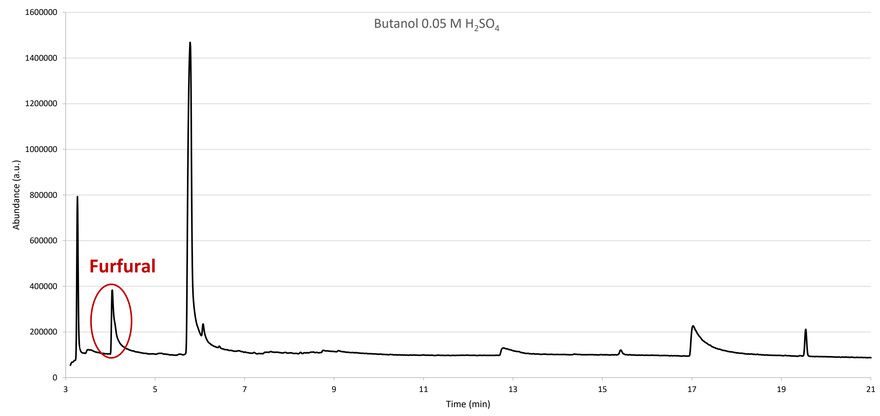


Figure 3. *Results obtained with GC-MS for butanol 0.05 M H_2SO_4.*

Conclusions and future prospect

- 1-butanol 0.05 M H_2SO_4 has shown to be the most effective biphasic system for obtaining oligosaccharides and for the production and separation of furans.
- For future work, the use of microwave for oligosaccharide extraction should be optimized through experimental design.

Acknowledgement

The authors would like to thank Diputación foral de Gipuzkoa TRANTSIZIO EKOLOGIKOA: EZAGUTZA (2023) for supporting financially this work.

References

1. Dávila, I., Gullón, B., Alonso, J. L., Labidi, J., & Gullón, P. (2019). Vine shoots as new source for the manufacture of prebiotic oligosaccharides. *Carbohydrate Polymers*, *207*(November 2018), 34–43. https://doi.org/10.1016/j.carbpol.2018.11.065