Fructo-oligosaccharides separation and purification by Simulated Moving Bed Chromatography

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INTRODUCTION

Fructooligosaccharides (FOS) are prebiotic oligosaccharides with increased interest for food and pharmaceutical applications. FOS can be produced by fermentative processes from sucrose and, at the end of the process, are mixed with fructose (F), glucose (G), sucrose (GF) and salts, representing 40-60 % (w/w) of the total sugars [1].

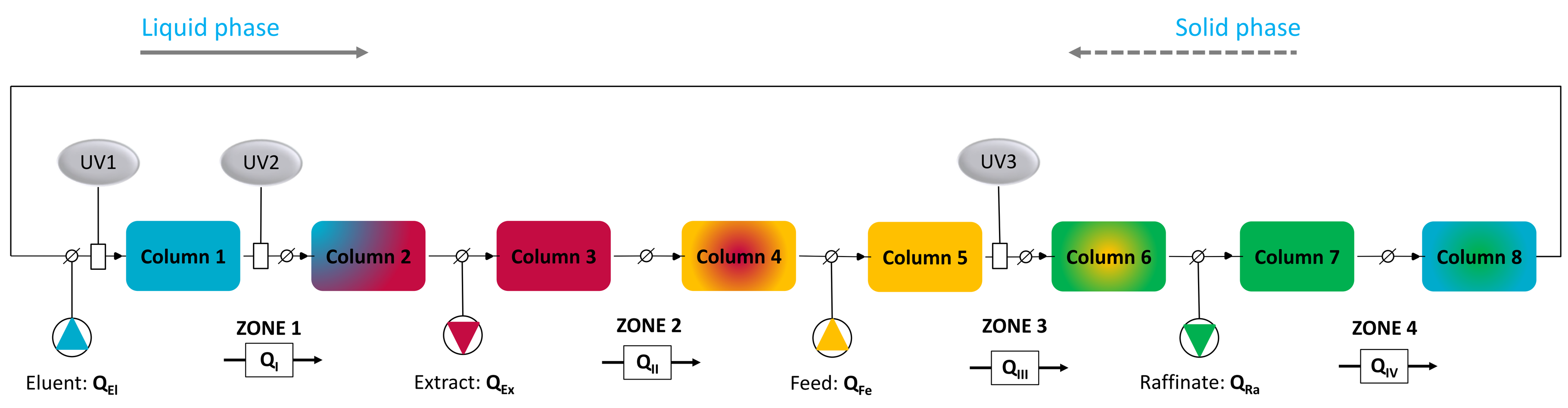
The purification of FOS from the other sugars represents an important increase on the economic value of the final product, which can be further used in diabetic and dietetic food [2]. Different strategies have been developed for this purpose, including microbial treatment [3], ultra and nano-filtration, activated charcoal systems [4], or ion-exchange chromatography [5]. Ion exchange resins are generally used in batch or continuous chromatographic processes, as Simulated Moving Bed (SMB) chromatography, to purify sugars. Compared to classical batch preparative processes, SMB provides a better utilization of the adsorbent and reduces eluent consumption, and requires no organic eluent to achieve high productivities.

A screening of different commercial resins was previously performed where the Diaion 535Ca resin showed the best performance in the separation of the oligosaccharides [5]. Eight columns were packed with the resin and used in the SMB plant.

The present work aimed at:

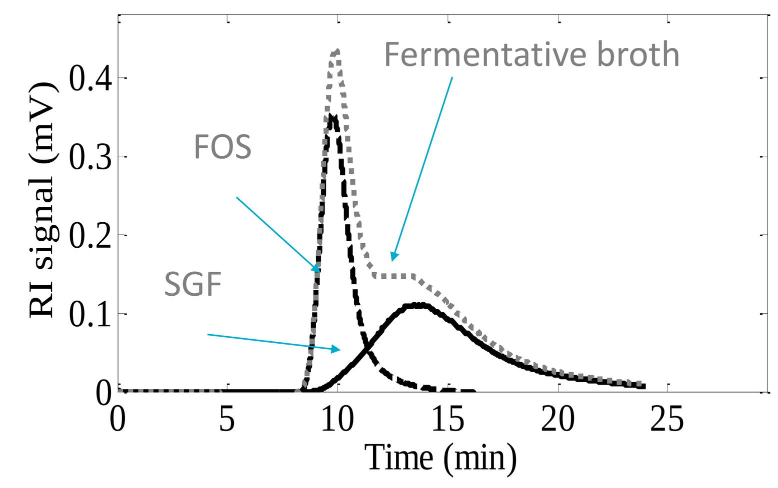
- Evaluate the separation of a binary sugar mixture of fructose/sucrose (~50/50 %) and FOS from a fermentative broth;
- Determine the operating points to improve the SMB separation of mixtures;
- Determine purity, yield and productivity of the sugars collected in the raffinate (GF and FOS) and extract (F and SGF – Sucrose/Glucose/Fructose) streams at the steady state.

SMB PILOT PLANT

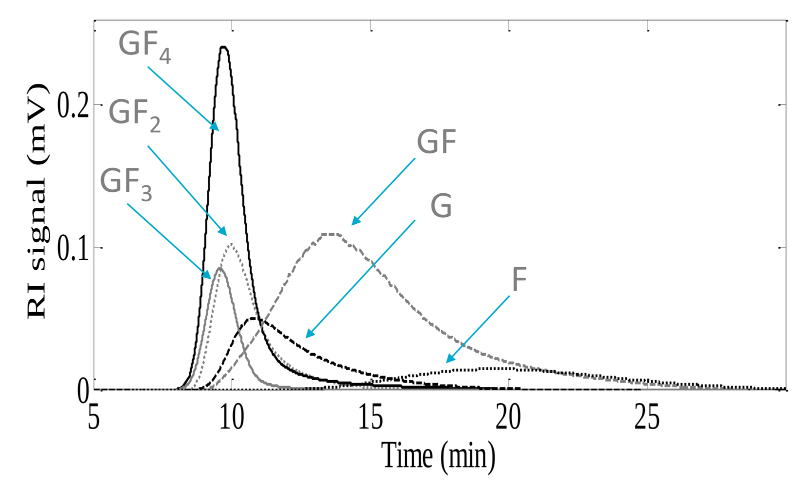


ELUTION PROFILES OF SUGARS

Chromatographic signal of the fermentative broth

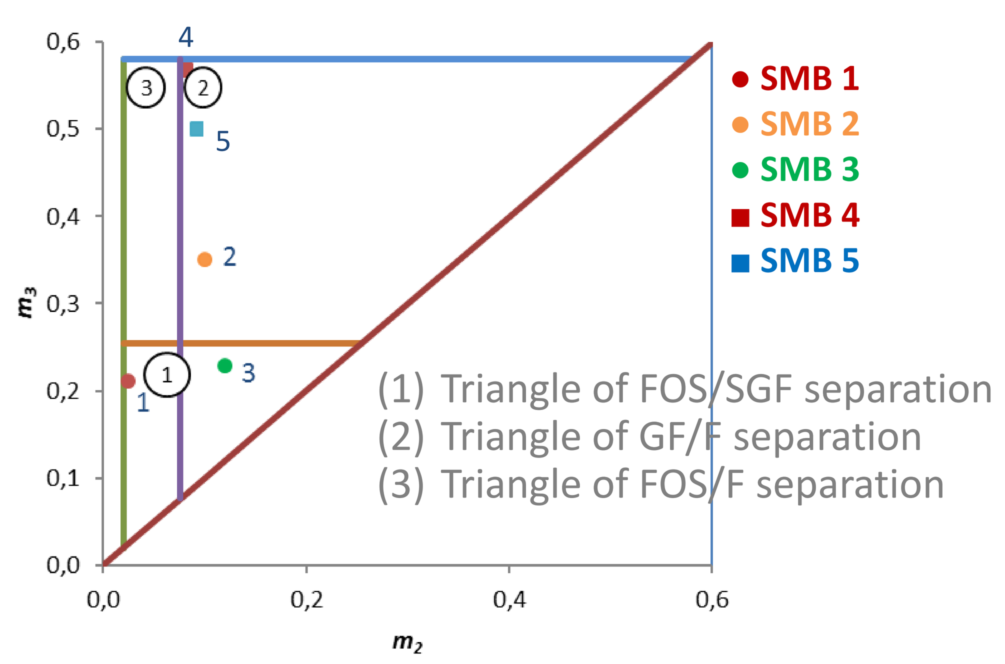


Elution profile of pure sugars injected in a Diaion UBK535Ca



RESULTS AND DISCUSSION

Separation regions and operating points



- ✓ Adsorption equilibrium isotherms were determined using Retention Time Method (RTM) for SGF, FOS, F and GF;
- ✓ Henri constants and security factors were used to predict sets of values (m_1, m_2, m_3, m_4) that provide the complete regeneration of solid and liquid phases;
- ✓ Flow-rate conditions were chosen to select the operating points from the complete separation zone (m_2, m_3);

Operating points selected for SMB experiments

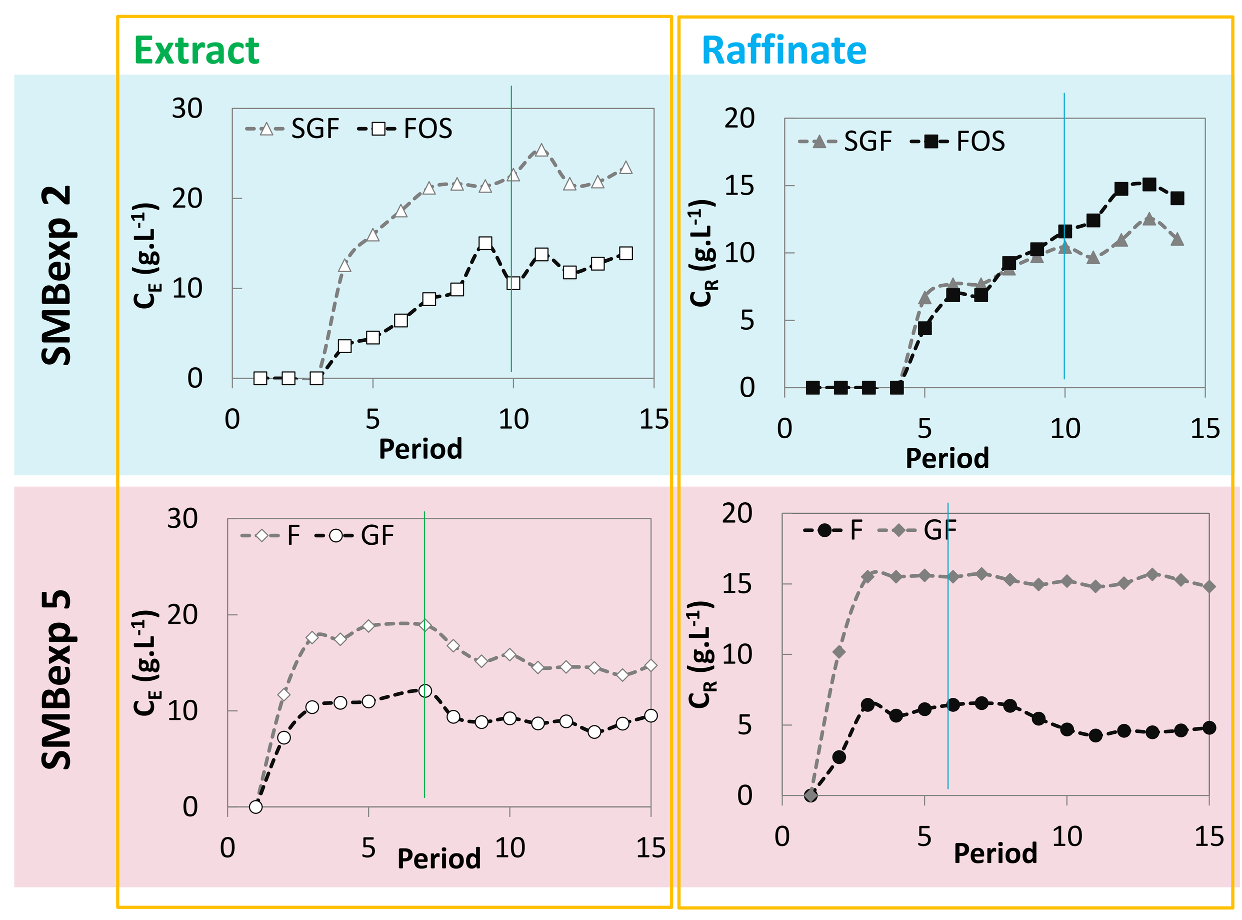
$SMB_{exp.}$	Mixture	Q_{Fe}	Q_{Ra}	Q_{Ex}	Q_{El}	Q_I	Q_{II}	Q_{III}	Q_{IV}
1	Demineralized fermentative broth (SGF and FOS)	3.65	3.70	4.12	4.07	15.83	11.76	15.41	11.71
2		4.96	6.66	7.16	5.45	18.84	13.38	18.34	11.68
3		2.14	4.24	7.16	5.06	18.84	13.78	15.92	11.68
4A	Fructose / Sucrose (50/50 w/w) (F/GF)	9.76	9.92	11.09	10.93	23.70	12.76	22.53	12.61
4B		9.76	9.92	12.76	12.61	25.37	12.76	22.53	12.61
4C		9.76	10.09	12.94	12.61	25.37	12.76	22.53	12.44
5A		8.20	8.75	12.94	12.39	25.37	12.98	21.19	12.44
5B		8.20	8.75	18.80	18.25	31.23	12.98	21.19	12.44

Performance of SMB separations

$SMB_{exp.}$	Purity (%)		Yield (%)		Productivity ($g.L^{-1}.h^{-1}$)	
	R_{FOS}	E_{SGF}	R_{FOS}	E_{SGF}	R_{FOS}	E_{SGF}
1	42.4	49.9	17.4	48.4	15.1	39.0
2	67.5	67.0	46.3	51.4	53.2	55.0
3	55.7	62.8	30.4	63.1	14.8	29.9
	R_{GF}	E_F	R_{GF}	E_F	R_{GF}	E_F
4A	66.0	64	60.4	79.3	43.1	59.1
4B	69.4	66.1	60.4	85.9	43.1	64.0
4C	69.1	65.1	60.4	86.9	43.1	64.7
5A	70.6	61.0	60.7	90.8	34.7	59.1
5B	76.8	61.3	59.0	97.1	33.7	63.2

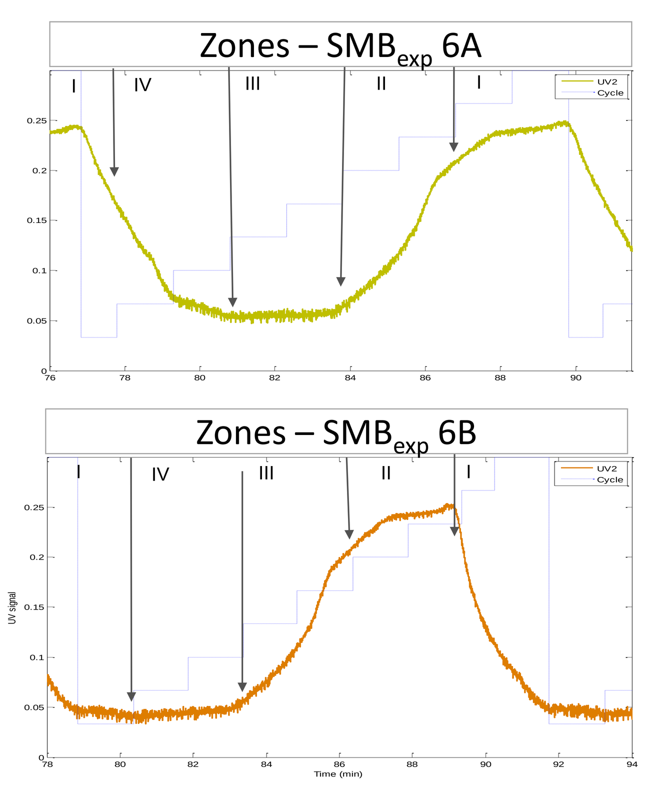
- ✓ FOS and SGF were purified from 50 to 67 % (SMB2);
- ✓ Sugars were diluted during the SMB separation, mainly in SMB3;
- ✓ Operating points selected increased the purity of sucrose in the raffinate stream from 71 to 77 % (SMB5A and SMB5B);
- ✓ Fructose yield and productivity in the extract stream increase from 91 to 97 % and from 59 to 63 $g.L^{-1}h^{-1}$ (SMB5A and SMB5B).

Sugars profiles in extract and raffinate streams



- ✓ The steady state for FOS separation from the fermentation broth was reached after 10 cycles;
- ✓ The steady state for the separation of F and GF from the binary solution was reached after around 6 cycles.

Chromatographic signal of UV_2



- ✓ Increasing the flow-rate in Zone 1, the concentration profile curve moved forward;
- ✓ Columns regeneration occurs between zones 1 and 4.

CONCLUSIONS

- ➢ The use of the Diaion 535Ca resin in SMB studies allowed to increase the purity of FOS and SGF recovered in the raffinate and extract streams respectively;
- ➢ The purity, yield and productivity of fructose was increased by the adjustment of the operating parameters;
- ➢ The accurate selection of the operating points is crucial to improve the separation through SMB;
- ➢ Future works will be focused on the separation of FOS from a fermentative broth containing high-levels of oligosaccharides.

REFERENCES

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