

CONDITIONS OF A SIMULATED MOVING BED CHROMATOGRAPHY UNIT FOR THE PURIFICATION OF FRUCTO-OLIGOSACCHARIDES

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INTRODUCTION

- Fructo-oligosaccharides (FOS) gained in the last years a great commercial interest due to its beneficial properties in the human health as prebiotics.
- In large scale, FOS are produced by fermentative processes. However, the composition of the final broth includes not only FOS, such as kestose (GF₂), nystose (GF₃) and fructo-furanosilnystose (GF₄), but also di- and mono-saccharides, namely sucrose (S), fructose (F) and glucose (G) that do not contribute to the prebiotic activity and must be removed.
- Simulated moving bed (SMB) chromatography appears to be an efficient downstream process for the recovery and fractionation of sugars in an industrial scale. As adsorbent, sulfonated poly(styrene-co-divinylbenzene) (PS-DVB) resins in potassium form are used. The mechanism of separation using these resins occurs by size exclusion and restricted diffusion effects.

AIMS

- Found a suitable resin to use in a SMB unit.
- Determine the adsorption parameters for *Dowex 50W-X2* and *Dowex Monosphere 99K/320* resins in potassium form.
- Estimate the parameters by numerical simulation using the least-squares method to minimize the distance between the experimental and simulated data.



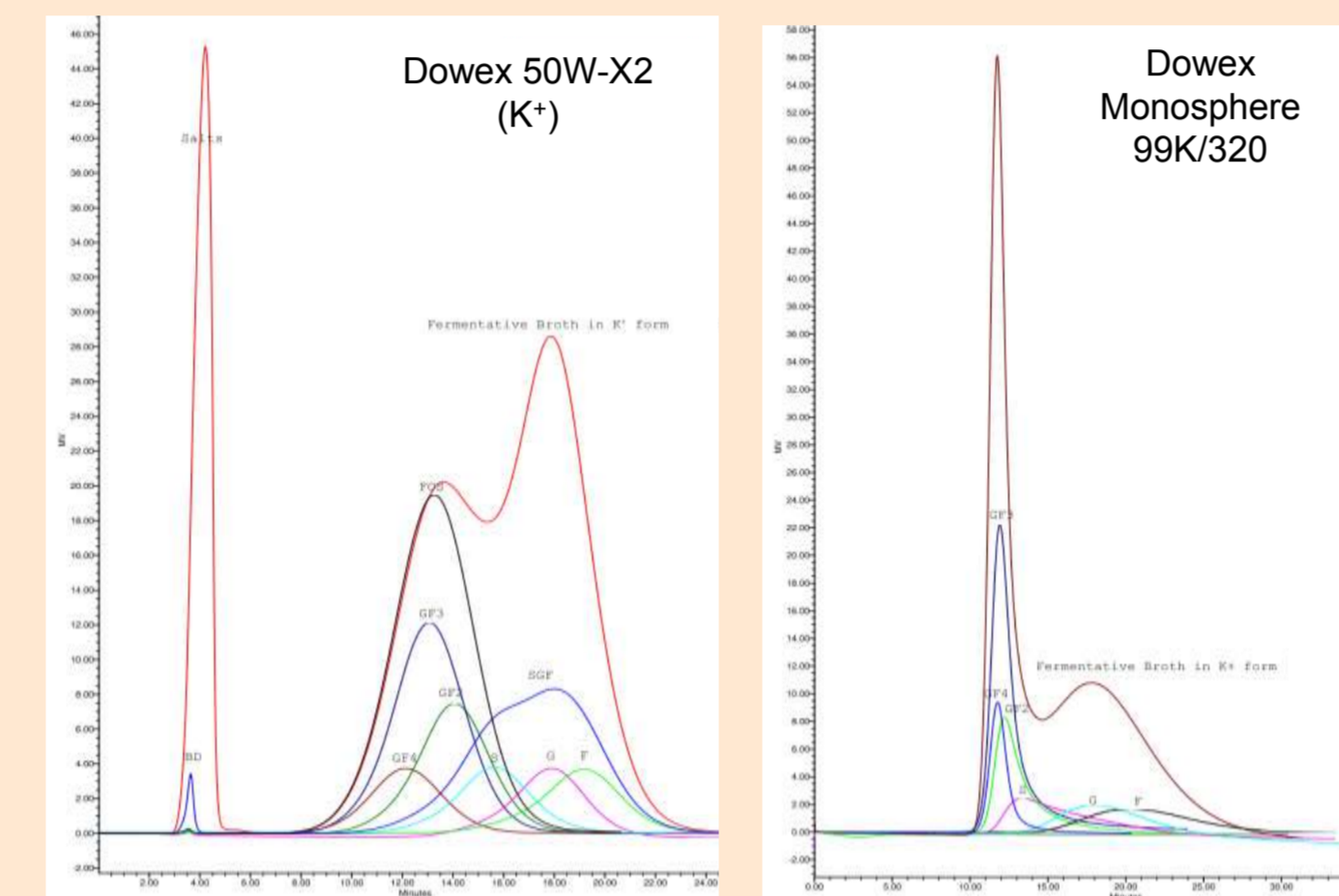
Chromatographic columns in the SMB unit

CONCLUSIONS

- Dowex Monosphere 99K/320* was found to be more suitable to work in the SMB unit as compared to *Dowex 50W-X2* due to a greater resistance at high pressure.
- Very sharp concentration profiles travel through the column and non-oscillatory schemes are necessary to avoid spurious oscillations and unrealistic negative concentration values. This scheme is quite efficient and leads to the following time evolution at the column outlet. The results obtained by numerical simulation compared with the experimental data showed a relatively good fit using the model parameters determined in the batch experiments.

EXPERIMENTAL RESULTS

Elution profile of sugars



Adsorption Isotherms

Linear adsorption

$$q_i = K \cdot C_i$$

Retention Time Method

Legend:

c_i : concentration of the component i in the liquid phase
 q_i : concentration of the component i in the solid phase
 K : Distribution coefficient

Resins characterization

	Dowex 50W-X2 (K ⁺)	Dowex Monosphere 99K/320
Particle Size (μm)	37-74	320
Porosity	0,154	0,387
F	0,812	0,468
G	0,745	0,342
GF	0,628	0,091
GF2	0,547	0,029
GF3	0,495	0,014
GF4	0,446	0,006
SGF	0,742	0,331
FOS	0,519	0,001

Preparative Test on the SMB at 20 mL/min

Dowex 50W-X2

- Possible to separate sugars from salts.
- High compressibility of the particles.
- Back pressure > 60bar (up to the maximum allowed by the SMB).

Dowex Monosphere 99K/320

- Broader peaks for G, F and S due to a slower kinetics caused by large particles.
- Sharper peaks to FOS.
- Constant porosity.
- Back pressure < 60 bar.

NUMERICAL SIMULATION

Mathematical model

$$\frac{\partial}{\partial t} \left(\epsilon C_i \right) + \frac{\partial}{\partial z} \left(V C_i \right) = \frac{\partial}{\partial z} \left(D_{eff} \frac{\partial C_i}{\partial z} \right) + k_{Fi} (C_i - q_i)$$

Legend:

t : time
 ϵ : porosity
 z : axial coordinate
 V : fluid velocity
 k_{Fi} : mass transfer coefficient

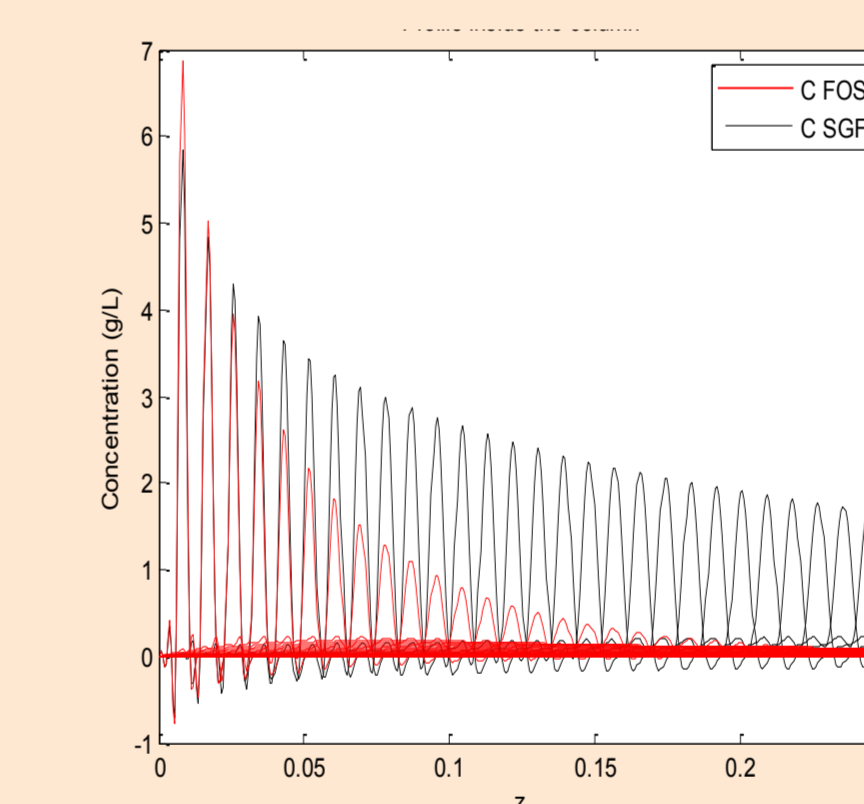
Mass transfer kinetics

LDF model

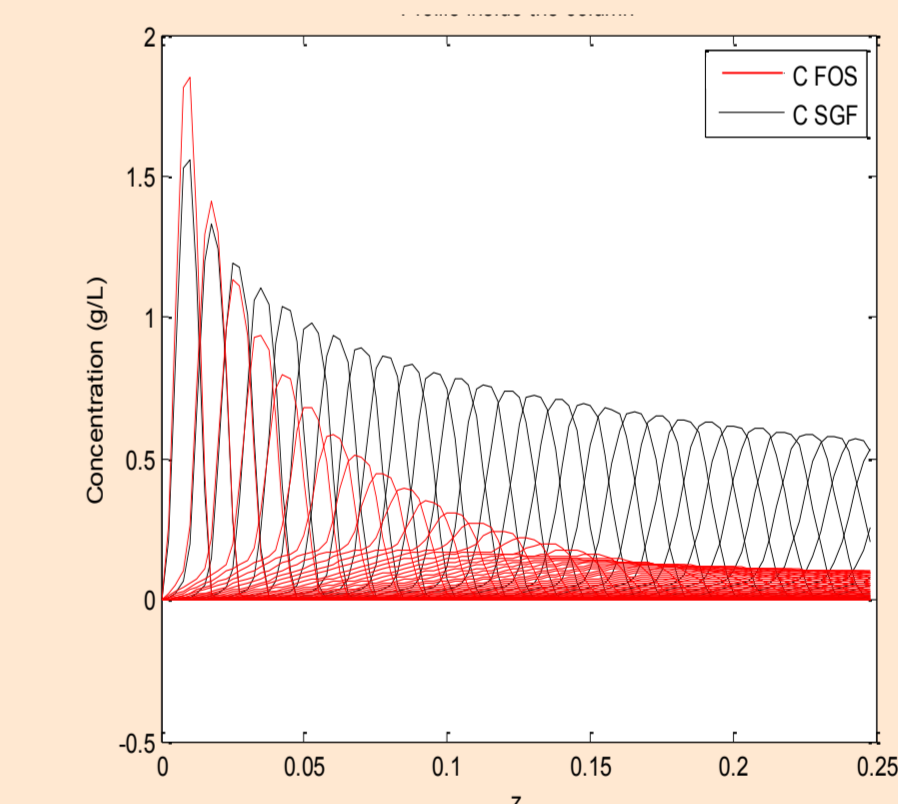
PDEs models are solved in Matlab with the toolbox MatMol (www.matmol.org)

Method of lines approach and slope limiters for approximating the spatial operators

Time evolution of the spatial concentration profiles inside a chromatographic column with Dowex Monosphere 99K/320

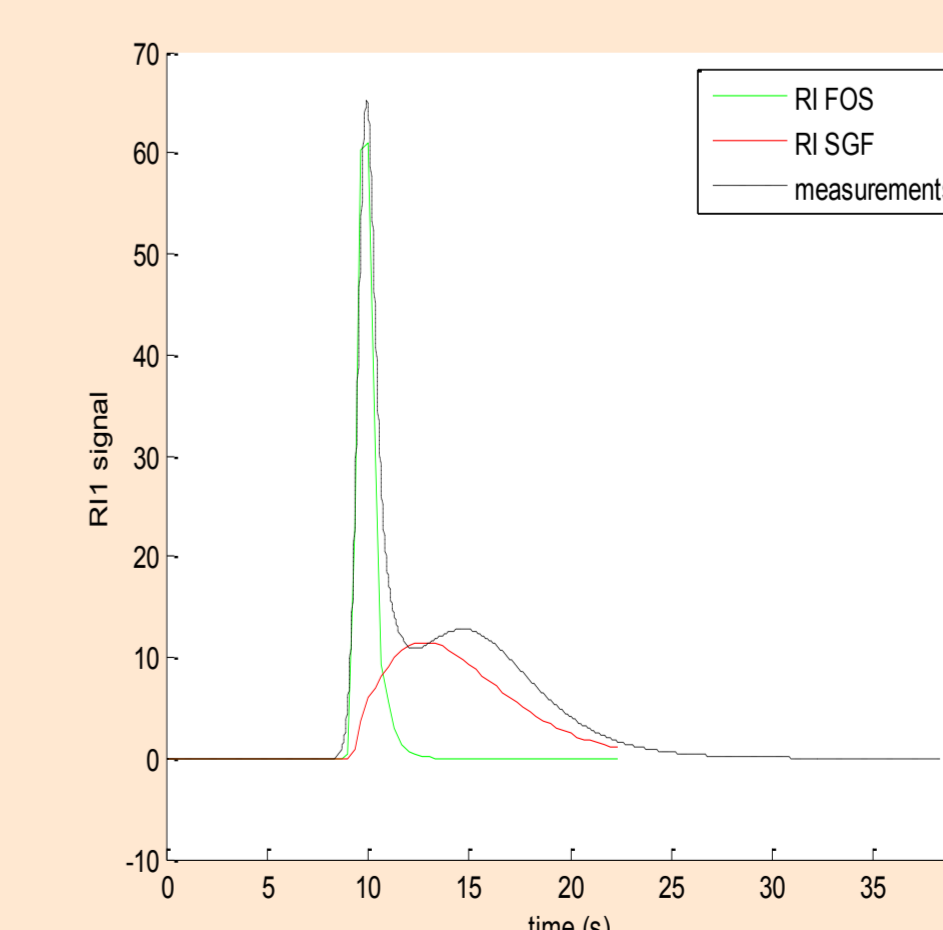


Finite differences with a 4-point biased upwind scheme. $N = 400$ grid points



Koren slope limiter scheme. $N = 100$ grid points

Time evolution of the RI signal at the column outlet with Dowex Monosphere 99K/320



Comparison of real measurements and simulated data for FOS and SGF.