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Equilibrium Studies of the Adsorption of Fructo-oligosaccharides from a Pure Mixture and a Fermentative Broth on a Dowex Monosphere Calcium Resin

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Introduction

- Fructo-oligosaccharides (FOS) gained in the last years a large commercial interest due to its beneficial properties in the human health as prebiotics.
- Fermentative processes appear to be a good alternative for large scale production of FOS, that include kestose (GF2), nystose (GF3) and fructo-furanosylnystose (GF4). However, the result of such fermentations is a complex mixture containing salts and low molecular weight sugars such as glucose (G), fructose (F) and sucrose (GF), that do not contribute to the beneficial effects and must be removed.
- Simulated moving bed chromatography (SMB) appears to be an efficient downstream process for the fractionation of sugars in an industrial scale. The major challenge when designing the separation process is the choice of an efficient ion-exchange resin. Therefore, the knowledge of the adsorption isotherms of the different compounds present in the mixture is an important parameter to consider when selecting the resin. Moreover, the influence on the adsorption of salts and other sugars present in the mixture must be studied.

Aims

- Modelling of the adsorption isotherms for FOS (from fermentative broth and pure mixtures) onto a Dowex Monosphere calcium resin.
- Determination of the model isotherm parameters using linear and non-linear correlations by minimization of several error functions.

Experimental Methodology

Resin Characteristics

Dowex Monosphere 99Ca/320	
Ionic form	Ca^{2+}
Structure	Gel-type
Matrix	Styrene-DVB
Functional group	Sulfonate
Total capacity (eq/L)	>1.5 (H^+form)
Water content (%)	57-61 (H^+ form)
Volume median diameter (µm)	300-330

Isotherm Models

Curvature	Isotherm Model
Linear	Linear
Upward	Anti-Langmuir
Downward	Langmuir
	Freundlich
	Redlich & Peterson
	Toth

Isotherm Parameters Determination

Linear Correlation
Linearization of the Model Equations

Non-Linear Correlation
Minimization of several error functions

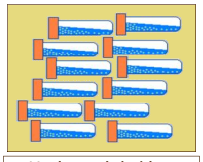
Error Function
Sum square of the errors
Hybrid fractional error function
Marquardt's percent standard deviation
Average relative error
Sum of absolute errors

Smaller Sum of the Errors ⬇ Best fitting of the experimantal data

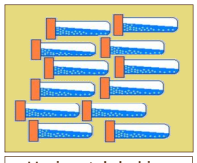
Static - Adsorption /Desorption Method

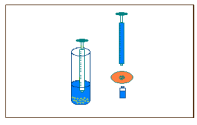
Resin
Dowex® Monosphere® 99 Ca/320

FOS solutions
Fermentative broth (38% FOS + 62% F, G, GF + Calcium ions)
Pure mixture (90% FOS + 10% F, G, GF)
C_i: = 15 – 180 g/L
V_{liquid} = 3.5 mL
$V_{adsorbent}$ = 1.5 mL

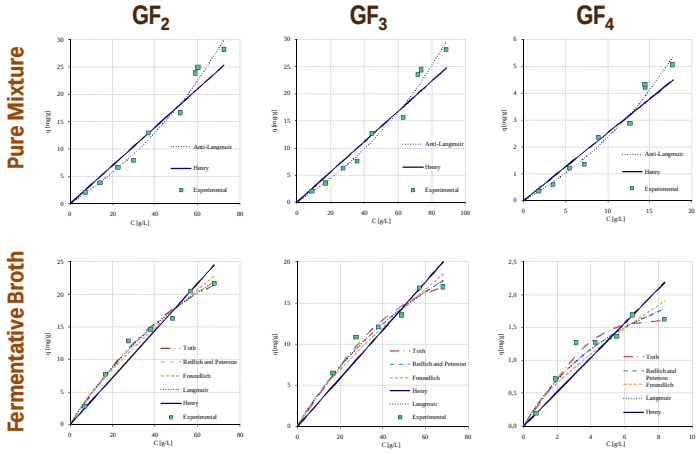
Adsorption

Horizontal shaking
$T_{adsorption}$ = 60 ºC
Contact time = 8h

Washing
Decantation of the liquid phase
Washing the resin with pure water
Addition of 5mL of pure water

Desorption

Horizontal shaking
$T_{adsorption}$ = 25 ºC
Contact time = *overnight*

Quantification

Filtration of supernatant (0.2 µm)
High Performance Liquid Chromatography (HPLC)

Results



Linear Distribution coefficients ($L.kg^{-1}$)

	GF_2	GF_3	GF_4
Fermentative broth	0,36	0,29	0,26
Pure mixture	0,35	0,28	0,25

Conclusions

- FOS present in pure mixture or broth have different adsorption behaviors.
- High ionic strengths and high concentrations of the other sugars seems to influence the adsorption.
- *Toth*, *Langmuir* and *Redlich & Peterson* isotherms were the models that best represented the adsorption of FOS in the broth, while FOS in pure mixture were better represented by the *Anti-Langmuir* isotherm.
- For both mixtures studied, the sugars were adsorbed according to their molecular size and kept a constant selective behavior.
- The non-linear methods were found to be more adequate to estimated the isotherms parameters, being the HYBRID function the one that gives better the results.

References

- GR Gibson (2008) J Clin Gastroenterol **42**:S75-S79
- M Gramblicka, M Polakovic (2007) J Chem Eng Data **52**:345-350
- J Nowak, I Poplewska, D Antos, A Seidel-Morgenstern (2009) J Chrom A **1216**:8697-8704
- M Saska, MD Wu, SJ Clarke, K Iqbal (1992) Sep Sci Technol **27**:1711-1732
- M Hadi, MR Samarghandi, G Mckay (2010) Chem Eng J **160**:408-416

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