Modelling of hierachical pore structures in freeze-dried pectin cryogels

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Summary

- Hierachical pore networks of directionally freeze-dried pectin cryogels show enhanced acoustic absorption characteristics when salt is added before freeze drying [1].
- Added salt and freeze drying lead to laminar pore structures interconnected by micropores that increase in number and size with increasing salt concentration.
- In this work, acoustic characterisation, SEM image feature extraction and CFD modelling is used to understand the effects of the pore geometry on sound absorption.

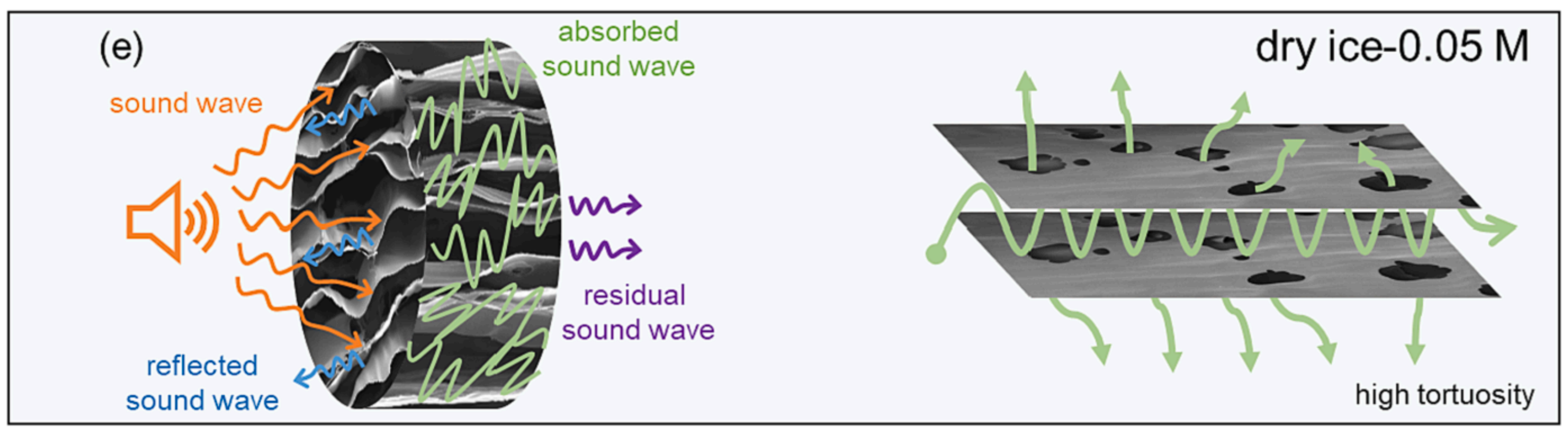

Figure 1 Freeze-dried pectin cryogel pore morphology proposed by Zou et al. [1].

Introduction

- Zou et al. found sound absorption to be best at salt concentrations of 0.05-0.1M (Fig. 2)
- The reason why sound absorption decreases for higher salt concentrations is not yet fully understood.
- Special interest draws flow resistivity and tortuosity, describing how the soundwaves propagate inside of the porous material.
- Comparing estimated JCAL parameters obtained from three-microphone impedance tube, extracted pore geometry from SEM images, and CFD simulation results could give inside on which parameters are affected by the pore geometry.

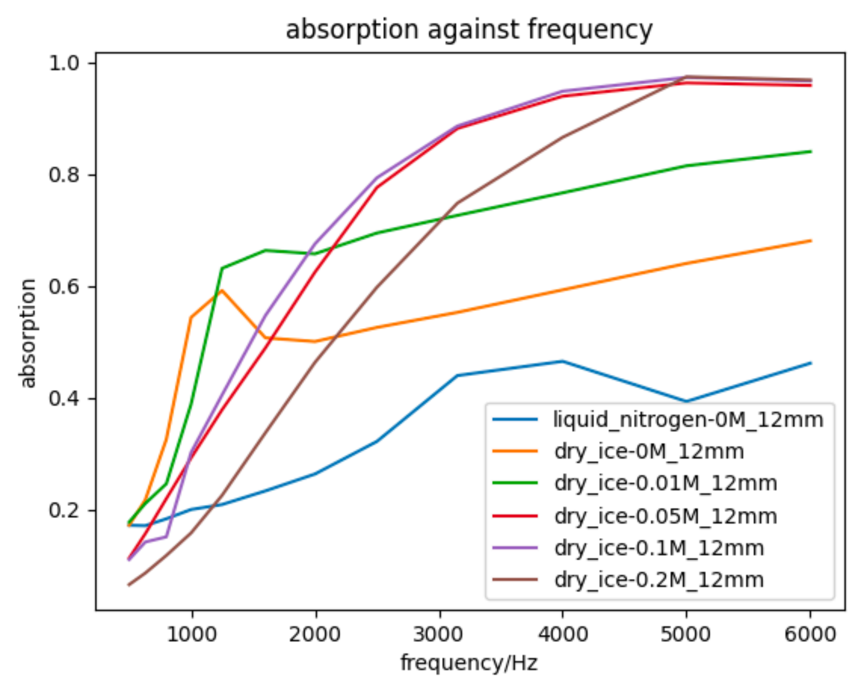

Figure 2 Sound absorption coefficient for cryogels of various salt content [1].

Methodology

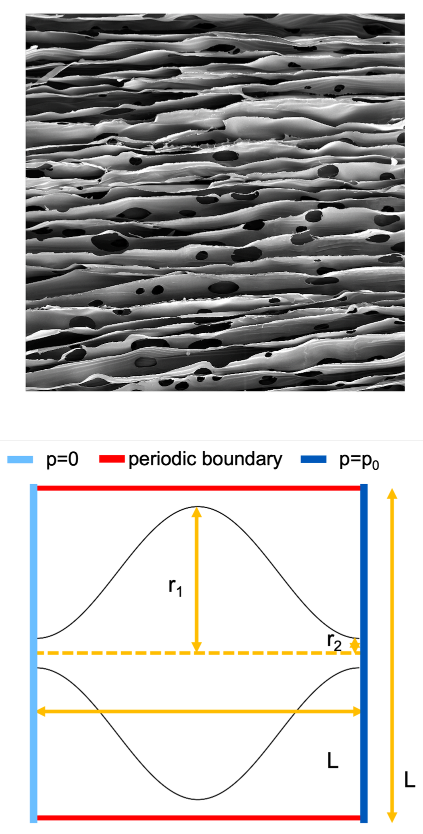

Figure 3 a) SEM image of laminar pores of cryogel b) sinusoidal pore model

- Pectin cryogel is characterised with RoKCell via three microphone impedance tube measurements.
- Flow resistivity and porosity are measured independently.
- Pore cross section areas are extracted from SEM images via image processing and compared to JCAL parameters from RoKCell.
- A 2D CFD model with sinusoidal pore shape unit cell is used to investigate the effect of micropores on laminar pore walls on flow resistivity and tortuosity.
- Sinusoidal pore shape is inspired by SEM images (Fig. 3).
- Viscid and inviscid fluid simulations determine flow resistivity and tortuosity for different neck-belly ratios, increasing micropore numbers and size, aiming to give insight on how pore shape affects JCAL parameters.

Acoustic Characterisation

NaCl concentration $[M]$	Measured flow resistivity σ $[Nsm^{-4}]$	Measured porosity ϕ	Tortuosity α_∞	Viscous char. length $[\mu m]$	Thermal char. length $[\mu m]$	Thermal permeability k'$_0$ $[10^{-10}m]$
0.05	189900	0.8	2.35	125	225	3
0.1	84250	0.9	1.3	29	36	7
0.2	33026	0.9	1.24	71	90	3

Table 1 JCAL parameters estimated by RokCell software via three microphone impedance tube measurements and additionally measured flow resistivity and porosity.

Feature extraction from SEM images

- Comparison of extracted pore area size ratios with viscous and thermal characteristic length.
- Second biggest/smallest clusters are analyzed since segmentation is prone to over and under segmentation.
- As NaCl concentration increases, the neck-belly ratio and viscous-thermal characteristic length ratio, both quantifying the modulation, decreases.

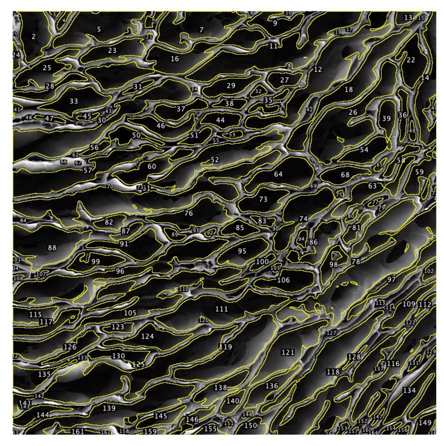

Figure 3 Segmented SEM image of pectin cryogel cross-section.

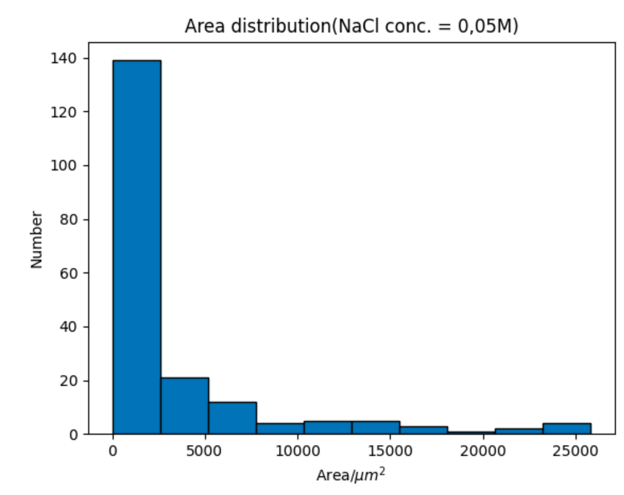

Figure 4 Histogram of pore mean area for salt concentration 0.05M.

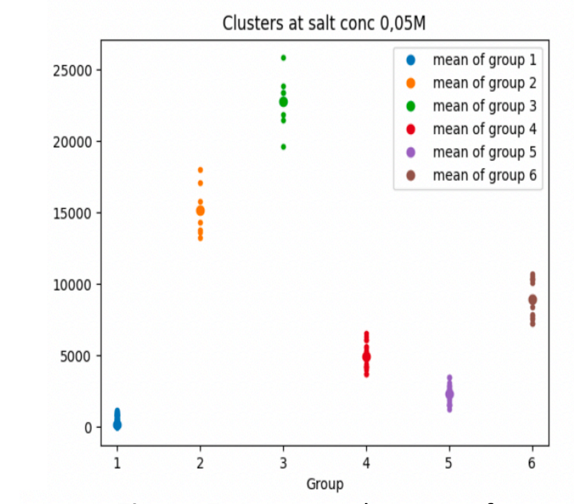

Figure 5 K-means clustering from segmented pore area.

Pore geometry features

NaCl concentration $[M]$	Mean area of second smallest cluster $A_1[\mu m]$	Mean area of second largest cluster $A_2[\mu m]$	Neck-belly ratio $\sqrt{A_2/A_1}$
0.05	2900	15000	2.3
0.1	6900	26000	1.9
0.2	5400	18000	1.8

Table 2 Extracted features from SEM images

Parameters from characterisation

NaCl concentration $[M]$	Measured flow resistivity σ $[Nsm^{-4}]$	Viscous characteristic length Λ $[\mu m]$	Thermal characteristic length Λ' $[\mu m]$	Ratio $[\Lambda'/\Lambda]$
0.05	189900	125	225	1.8
0.1	84250	29	36	1.24
0.2	33026	71	90	1.26

Table 3 Excerpt of acoustic characterization

CFD modelling

- Flow resistivity and tortuosity decrease when holes are added on walls of sinusoidal pore model especially for higher neck-belly ratios.
- Fluid can divert to more direct paths and those with less resistance.
- The decrease of tortuosity and flow resistivity will be due to all the three factors described, the strength of modulation, micropore size and number.

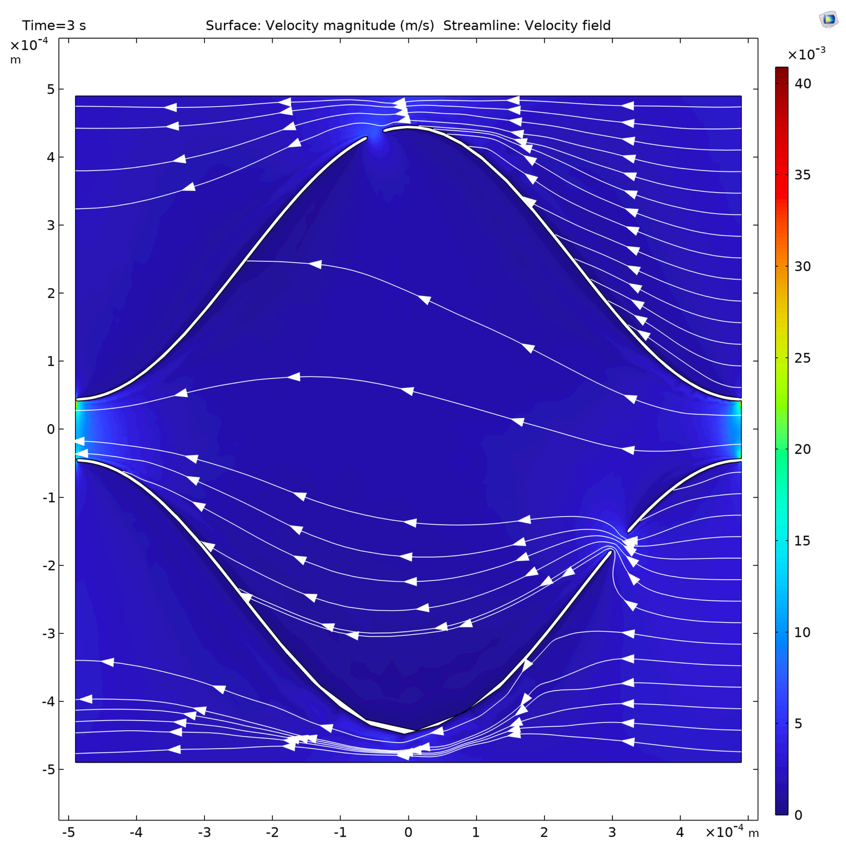

Figure 6 Inviscid fluid simulation for sinusoidal pore of neck-belly ratio 10.

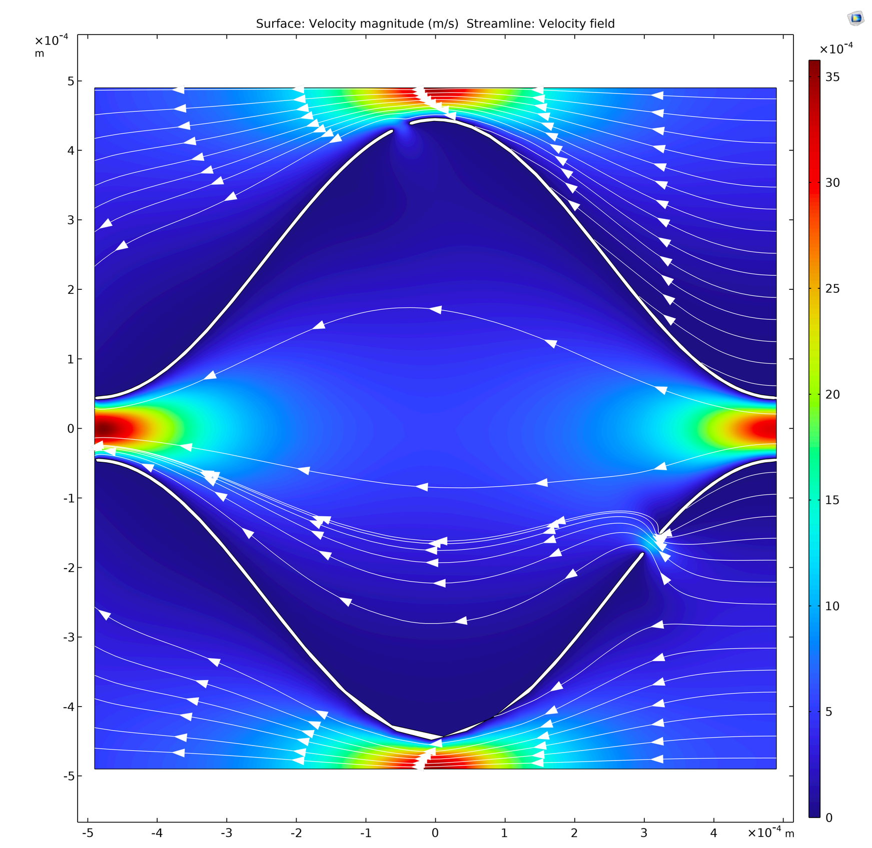

Figure 7 Viscid fluid simulation for sinusoidal pore of neck-belly ratio 10.

		Neck-belly ratio					
		1		3		10	
Micro pore number	Micro pore size	Flow resistivity $\sigma[Nsm^{-4}]$	Tortuosity α_∞	Flow resistivity $\sigma[Nsm^{-4}]$	Tortuosity α_∞	Flow resistivity $\sigma[Nsm^{-4}]$	Tortuosity α_∞
0	0	922	1.08	2580	1.36	24600	2.25
2	0.5	897	1.07	2500	1.36	23500	1.96
	1	817	1.06	2290	1.16	20700	1.93
	3	546	1.06	114	1.16	17500	1.86
4	0.5	684	1.07	2410	1.20	21600	1.69
	1	529	1.06	1990	1.17	17800	1.75
	3	-	-	991	1.14	4620	1.38

Table 4 Flow resistivity and tortuosity for various neck-belly ratios as well as micropore numbers and size. Micropore size is set relative to the size of the neck.

Aknowledgements

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Aalto University

KAUTEsäätiö

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[1] F. Zou, J. Cucharero, Y. Dong, P. Kangas, Y. Zhu, J. Kaskirinne, G. C. Tewari, T. Hänninen, T. Lokki, H. Li, and J. Vapaavuori, "Maximizing sound absorption, thermal insulation, and mechanical strength of anisotropic pectin cryogels," Chemical Engineering Journal, vol. 462, 2023.