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A multiphase porous medium transport model with distributed sublimation front to simulate vacuum freeze drying

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ABSTRACT

A porous medium transport model with a distributed sublimation front is developed for low pressure freeze drying of beef by radiant surface heating and volumetric microwave heating. The model incorporates the importance of Knudsen flow in porous materials during low pressure freeze drying. This effort is part of fundamental physics-based framework building for simulating food and biomaterial processes involving rapid evaporation/sublimation. Temperature, pressure and ice saturation histories were computed. Drying rates and spatial temperature profiles showed excellent agreement with literature experimental data. Sublimation front width, a novel result, is seen to increase as ice saturation decreases, justifying the importance of this distributed sublimation formulation in contrast with the sharp sublimation front commonly employed in literature. The insulation effect of the gas fraction in the pores is observed by the slow movement of the sublimation front in 'thick' samples. Effects of porosity, initial ice saturation and microwave heating are illustrated.

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1. Introduction

In freeze drying, a moist product is frozen, and then placed in a low temperature vacuum chamber. As the chamber pressure is lowered, rapid sublimation occurs once the pressure is below frozen product's vapor pressure, leading to dehydration of the product. One of the main reasons for freeze drying is that it is generally considered the optimal method by which to dehydrate high quality and heat sensitive materials, such as foods, pharmaceuticals, and biomedical products (Millman et al., 1985; Liapis, 1987; Ratti, 2001). Freeze drying is also used in the preparation of aerogels (Wang et al., 2013; Pojanavaraphan et al., 2010), hydrogels (Cho et al., 2012), and nanoparticle formulations (Chung et al., 2012). In all cases, the precise control of operating conditions is a necessity in order to prevent such complications as cracking or product degradation. The main processing challenges in freeze drying are to reduce the long drying times and minimize energy use in its inherent energy intensive operation (Nam and Song, 2007). Surface heating, using hot plates or heated air or volumetric heating, using microwaves or radiofrequency, are used to speed up the sublimation process in freeze drying. Higher rates of heating can cause the product to melt, severely degrading the quality. This tradeoff between quality and productivity necessitates development of a physics-based model and optimization of freeze drying to reduce the trial-and-error experiments for process design. With a physics based model, the freeze drying process can be precisely controlled to design and better understand the product. Such measureables are the pore pressure or the distribution and magnitude of the sublimation to study their effects on the resulting dried product.

The sublimation process in freeze drying has been modeled using two main approaches – a moving boundary (or sharp sublimation front) approach (Ma and Peltre, 1975; Wang and

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Nomenclature

c	concentration ($\mathrm{kg\,m^{-3}}$)
Cp	specific heat capacity ($\mathrm{J\,kg^{-1}\,K^{-1}}$)
D	diffusivity ($\mathrm{m^2\,s^{-1}}$)
d_p	molecule diameter (m)
f	frequency (MHz)
F	view factor
$\dot{I}$	volumetric evaporation ($\mathrm{kg\,m^{-3}\,s^{-1}}$)
k	thermal conductivity ($\mathrm{W\,m^{-2}\,K^{-1}}$)
k_B	Boltzmann constant ($\mathrm{m^2\,kg\,K^{-1}\,s^{-2}}$)
Kn	Knudsen number
K	non-equilibrium evaporation function ($\mathrm{s^{-1}}$)
L_c	characteristic length (m)
m_w	molecular weight of water
p	pressure (Pa)
p_v	vapor pressure (Pa)
R	gas constant ($\mathrm{m^3\,Pa\,K^{-1}\,mol^{-1}}$)
S	saturation
t	time (s)
T	temperature (K)
Q	volumetric microwave source term ($\mathrm{J\,m^{-3}\,s^{-1}}$)
v	velocity ($\mathrm{m\,s^{-1}}$)
x	mass fraction

Greek symbols

ρ	density ($\mathrm{kg\,m^{-3}}$)
ϵ	emissivity
σ	electrical conductivity ($\mathrm{S\,m^{-1}}$)
Σ	Stefan–Boltzmann constant ($\mathrm{W\,m^{-2}\,K^{-4}}$)
κ	permeability
λ_{MFP}	mean free path (m)
λ	latent heat of sublimation ($\mathrm{J\,kg^{-1}}$)
Λ	non-equilibrium evaporation coefficient ($\mathrm{s^{-1}}$)
ϕ	porosity
μ	dynamic viscosity (Pa s)
δ_p	penetration depth (m)
ω	angular frequency ($\mathrm{rad\,s^{-1}}$)
ε_0	permittivity of free space ($\mathrm{F\,m^{-1}}$)
ε''	dielectric loss constant

Subscripts

ch	chamber
eff	effective
HP	hot plate
g	gas
i	ice
in	intrinsic
MW	microwave
r	relative
s	solid
0	initial

Shi, 1997; Millman et al., 1985; Sheehan and Liapis, 1998; Sandall et al., 1967) and a distributed non-equilibrium sublimation front (Nam and Song, 2007, 2005). The two main approaches are similar except for two major differences – the number of phases and the way in which sublimation is modeled. The sharp sublimation front approach includes two phases (frozen and unfrozen) while the distributed front approach incorporates a third phase, a partially frozen region, where only a fraction of the original ice remains (the rest of the ice has sublimated). The sharp sublimation front approach obtains the frontal velocity by relating it to the difference in heat flux between the frozen and dried side of the front while the distributed sublimation front uses the pressure difference between the gas in the pore and ice vapor pressure as the driving mechanism for sublimation. The rest of the model in these two approaches is similar, having mass conservation equations for gas and ice to obtain moisture content, and an energy conservation equation for the combined phases to obtain temperature. Microwave heating has been introduced in freeze drying models by using simple approximations of electromagnetic field variations such as constant value and exponential decay or Lambert's law (Dibben, 2001). Since porosity changes little in freeze drying, especially when compared to air-drying (Ratti, 2001), shrinkage is generally ignored, making solution of solid mechanics equations unnecessary.

Comprehensive physics-based models provide increased understanding and predictive capabilities that can increase efficiency in food product, process, and equipment design, and improve quality and safety. Development of a concise modeling framework, as opposed to a custom model for each process, as it mostly exists today, can greatly accelerate computer-aided food process engineering. For solid and semi-solid foods, a modeling framework (Dhall and Datta, 2011) that considers homogenized macroscale multiphase transport in the food as a deformable/swellable porous medium has been successful in modeling a number of important processes including drying, rehydration (Weerts et al., 2003), baking (Zhang et al., 2005), frying (Yamsaengsung and Moreira, 2002; Halder et al., 2007), meat cooking (Dhall and Datta, 2011), microwave heating (Ni et al., 1999) and microwave puffing (Rakesh and Datta, 2013). The distributed sublimation (or evaporation in the case of certain aforementioned processes) front does not require the solution of the additional equation for a sharp moving sublimation front location, therefore there is no need to continually remesh the geometry as the front moves with time and the distributed front is more physically accurate, especially in the case of atmospheric freeze-drying (Bralsford, 1967). Two main limitations for the distributed front modeling approach are that ice saturations of zero or one create computational instabilities that can create unphysical saturations. Computation time increases due to the number of elements needed to capture the front along with the sharp transition between phases. Freeze drying has significant differences with the other aforementioned processes and our goal is to be able to infer the extent to which a distributed phase change modeling framework succeeds in freeze drying with the associated numerical benefits and challenges.

The manuscript is organized by first introducing the theory behind vacuum freeze drying with an emphasis on the transport at high Knudsen numbers in porous media. Then, continuum equations are developed for a multiphase, porous media based transport model with a distributed, non-equilibrium sublimation front that calculates pore ice fraction, pressure, and temperature. Results are validated against literature and experimental data. Then, the results are presented for various operating conditions including hot plate temperature, sample thickness, microwave heating. Finally, optimization of freeze drying as a function of drying time, energy usage, and product quality is discussed.

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