A Multiphase Porous Medium Transport Model with Distributed Sublimation Front to Simulate Vacuum Freeze Drying

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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. The sublimation process in freeze drying has been modeled using two main approaches---a moving boundary (or sharp sublimation front) approach (3,4), and a distributed non-equilibrium sublimation front (1,2). ***Here, we show the efficacy of the first continuum finite element non-equilibrium sublimation front approach in modeling freeze drying.***

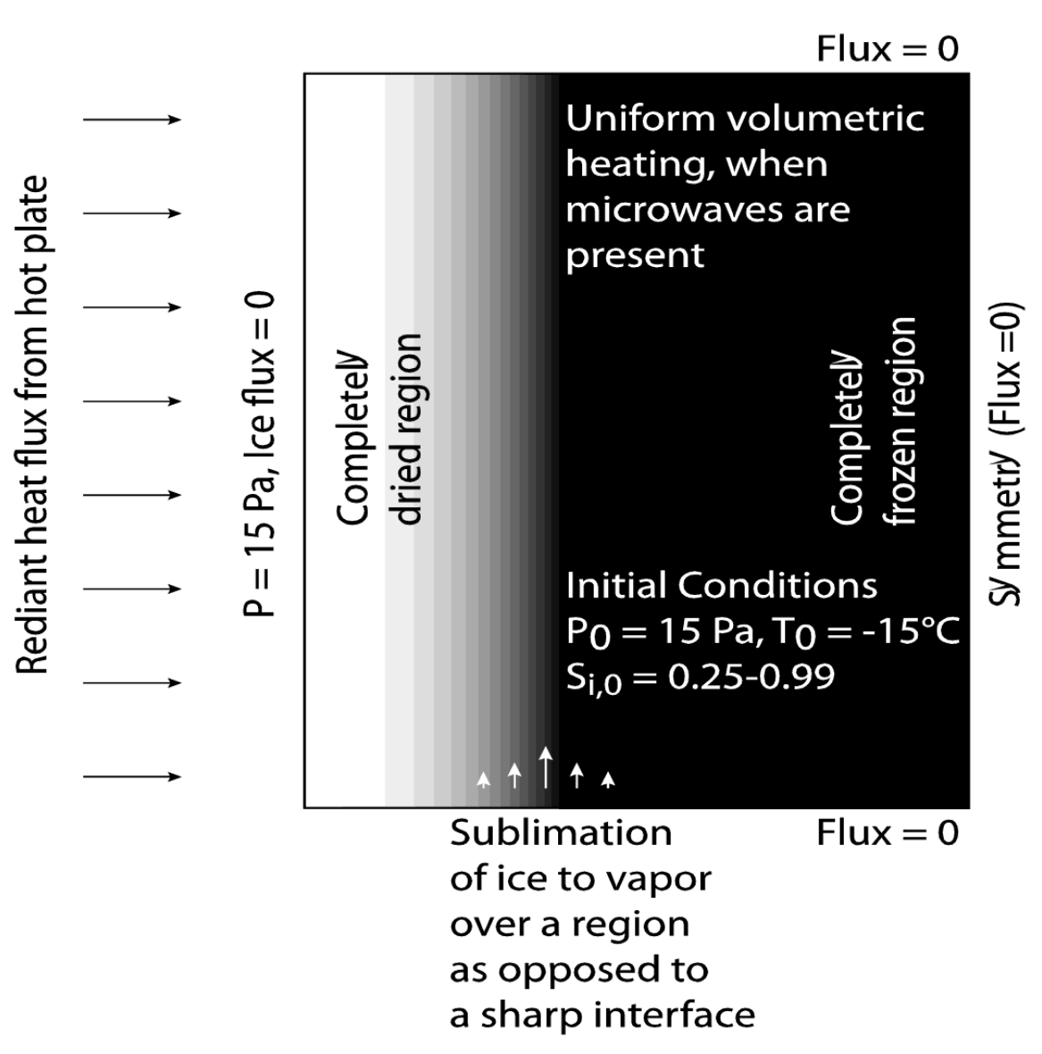

Figure 1. A schematic of the 1D freeze drying processes. There is zero flux of the three governing equations except on the left. On the left, radiation flux (between the hot plate and product), constant pressure, and zero flux for ice saturation is set.

Governing Equations

$$\frac{dc_i}{dt} = \dot{I} \qquad c_i = S_i\varphi\rho_i \qquad S_i + S_g = 1$$

Eqs. 1-3: Mass transfer eqs.

$$\frac{\partial(\rho_{eff}Cp_{eff}T)}{\partial t} + \frac{\partial}{\partial y}\cdot(\rho_g Cp_g v_g T) = \frac{\partial}{\partial y}\left(k_{eff}\frac{\partial T}{\partial y}\right) - \lambda\dot{I} + Q_{MW}$$

Eq. 4: Heat transfer

$$\frac{\partial(\rho_g S_g \varphi)}{\partial t} + \frac{\partial}{\partial y}\cdot(\rho_g v_g) = \frac{\partial}{\partial y}\left(D_g\frac{\partial \rho_g}{\partial y}\right) + \dot{I}$$

Eq. 5: Darcy's eq. (pressure)

$$v_g = -\frac{\kappa}{\mu_g}\frac{\partial p}{\partial y}$$

Eq. 6: Velocity

$$\dot{I} = K(p_{v,i} - p)\frac{m_w \varphi S_g}{RT}$$

Eq. 7: Sublimation

COMSOL Implementation

Three modules in COMSOL Multiphysics version 4.3a, *Transport of Diluted Species*, *Heat Transfer in Solids*, and *Darcy's Law* (weak form expression modified for diffusion term) were used to solve for concentration of ice (Eq. 1), temperature (Eq. 4), and pressure (Eq. 5), respectively. The weak form equation of *Darcy's Law* module was modified to include the diffusive term and a translational velocity term (Eq. 6) was added to the *Heat Transfer in Solids* module in order to account for convection.

Results

Validation for varying half thickness

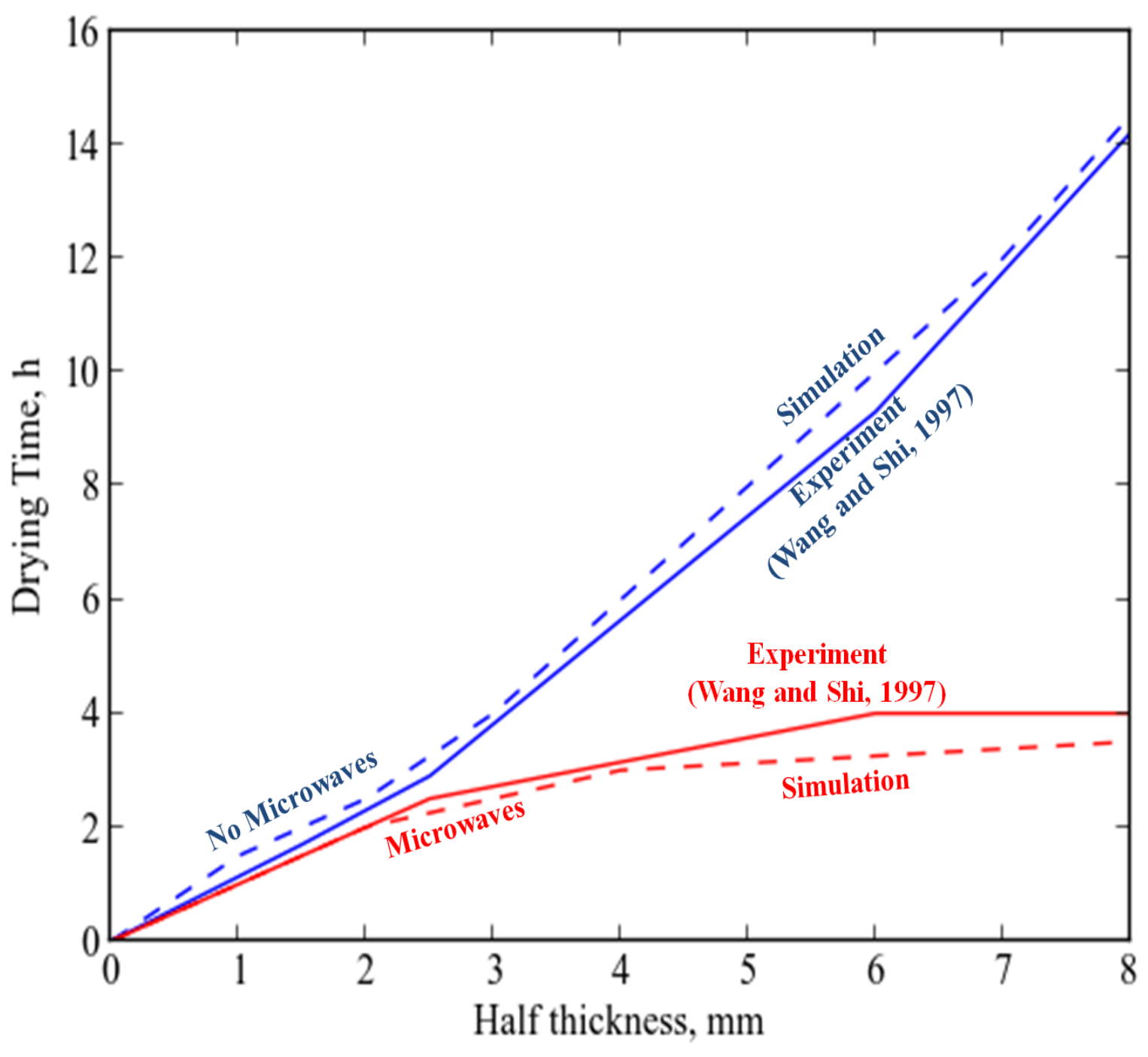

Figure 2. Total drying time versus sample half thickness.

Validation for varying initial ice saturation

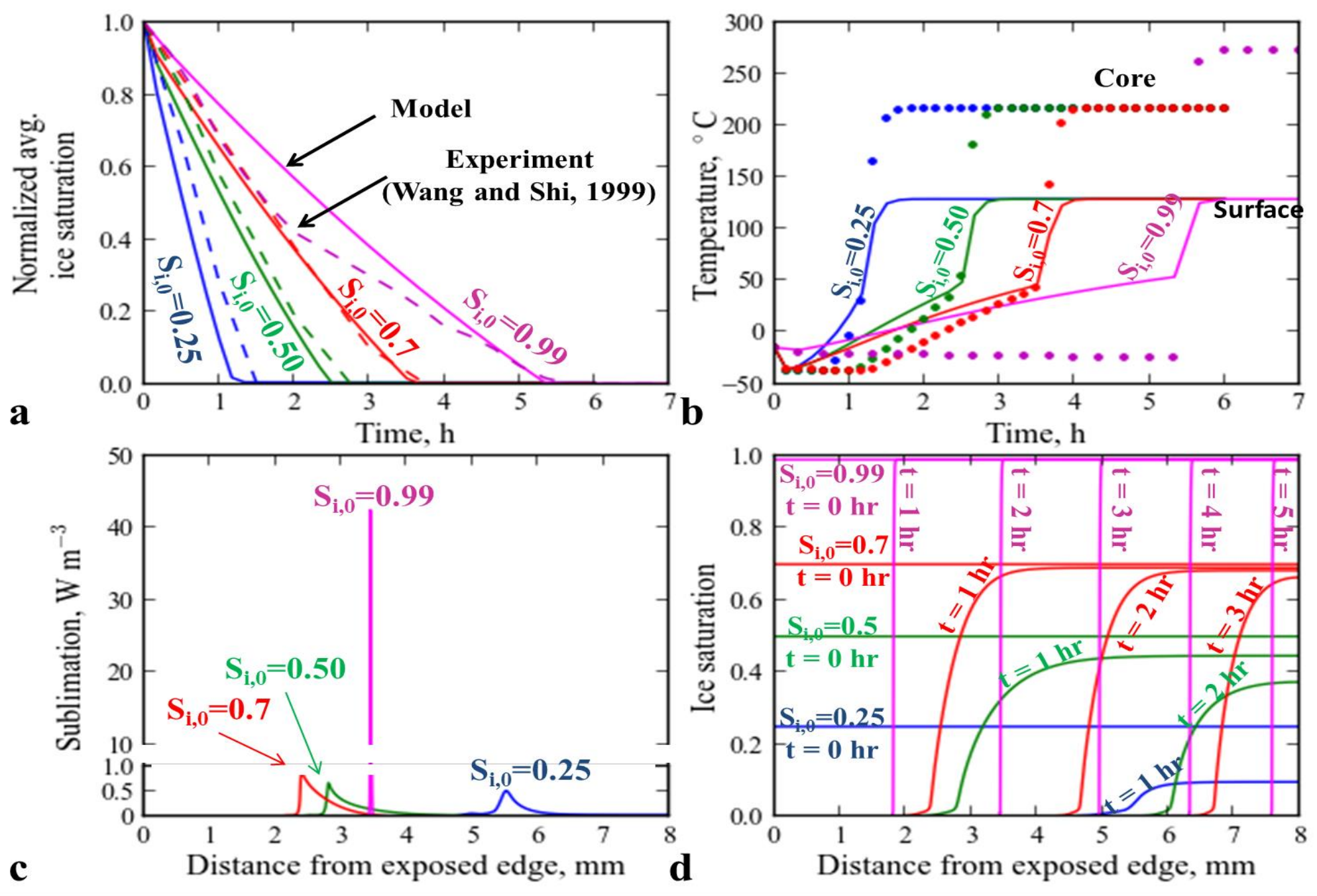

Figure 3. Sensitivity of initial saturation in sample with half thickness of 3 mm, hot plate temperature of 20 ° C, microwaves, and vacuum pressure 15 Pa.

No microwaves

With microwaves

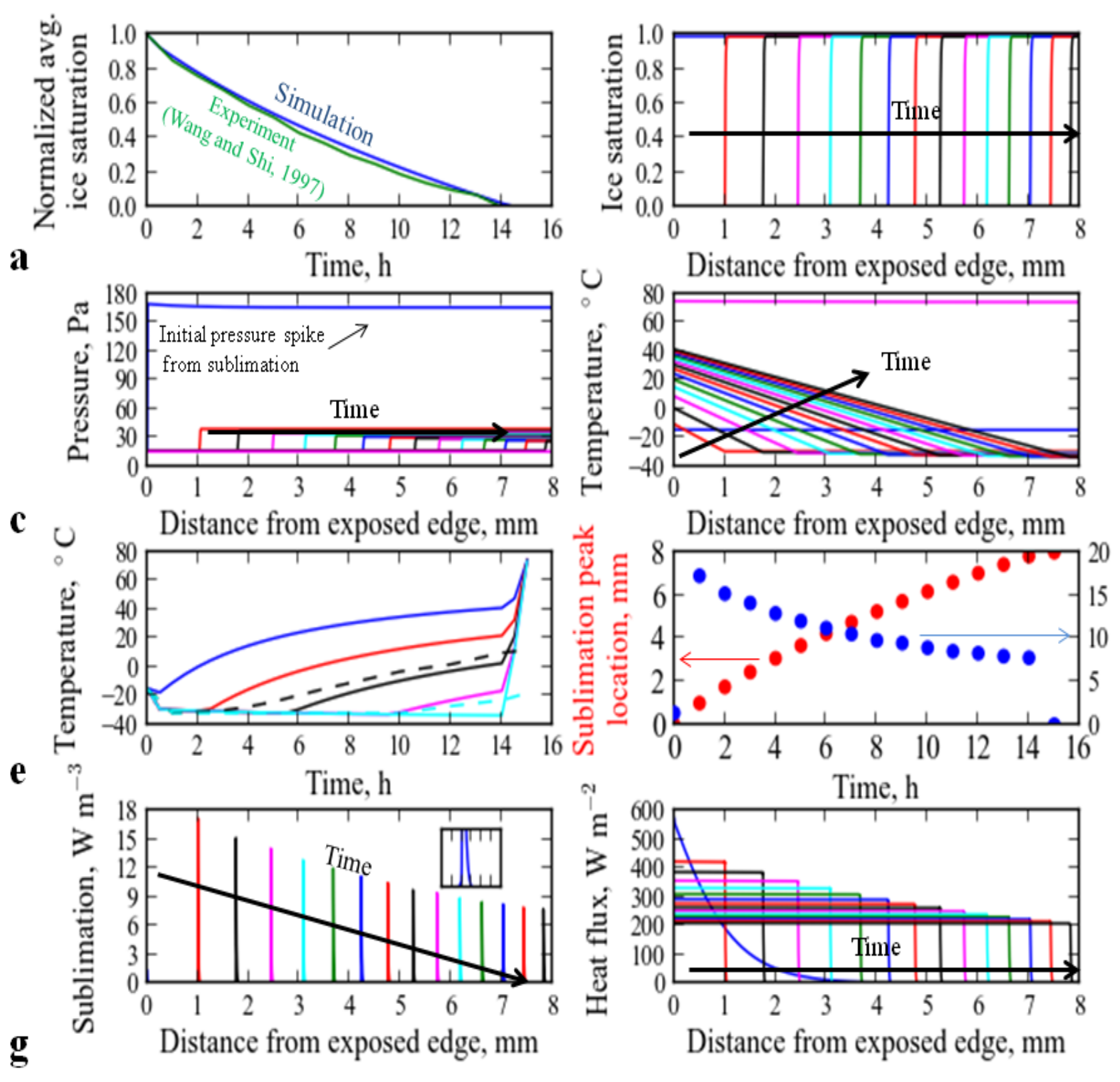

Figure 4. Results and their validation against experiment from literature (4). All results presented are for zero microwaves, hot plate temperature of 75 ° C, half thickness of 8 mm, $S_{i,0}$=0.99 and vacuum pressure 15 Pa. In figures B, C, G, and H, each line represents one hour from time equals zero to 15 hours.

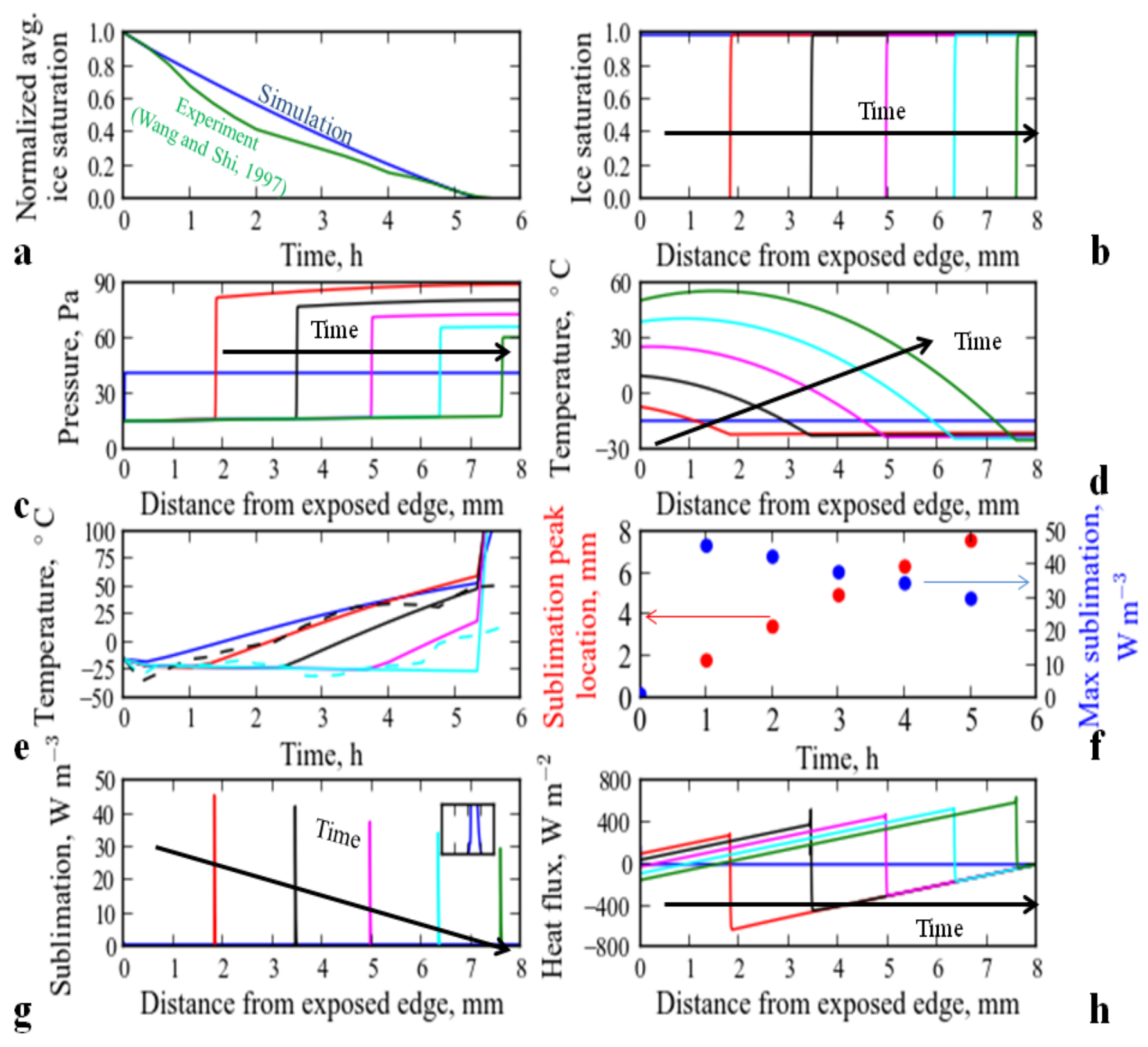

Figure 5. Results and their validation against experiment from literature (4). All results presented are for sample half thickness of 8 mm, $S_{i,0}$=0.99, hot plate temperature of 20 ° C, 120 V/cm microwaves, 2.45 GHz frequency, and vacuum pressure 15 Pa. In figures B, C, G, and H, each line represents one hour from time equals zero to 5 hours

Sensitivity of non-equilibrium constant

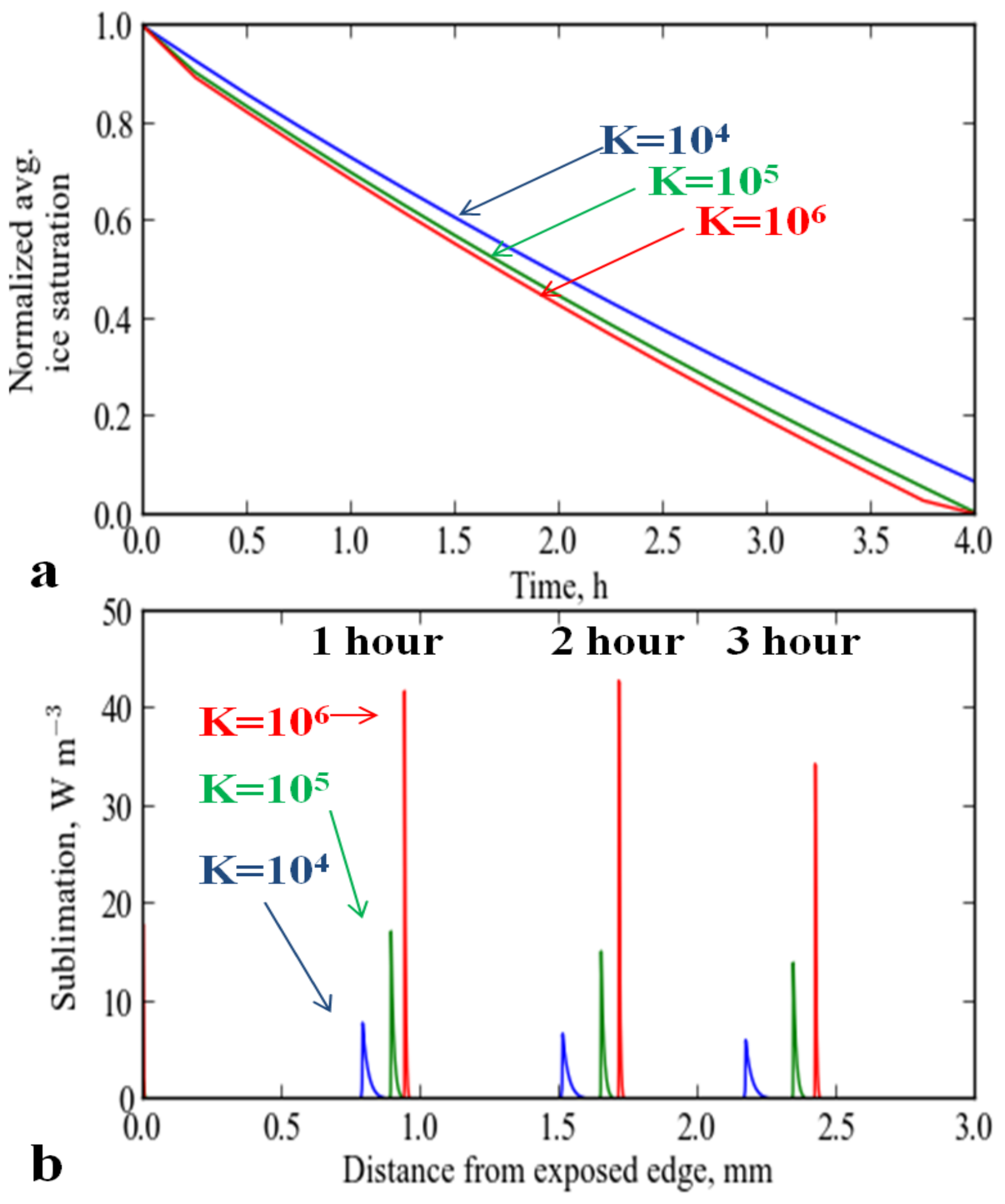

Figure 6. Sensitivity of non-equilibrium constant

Sensitivity of porosity

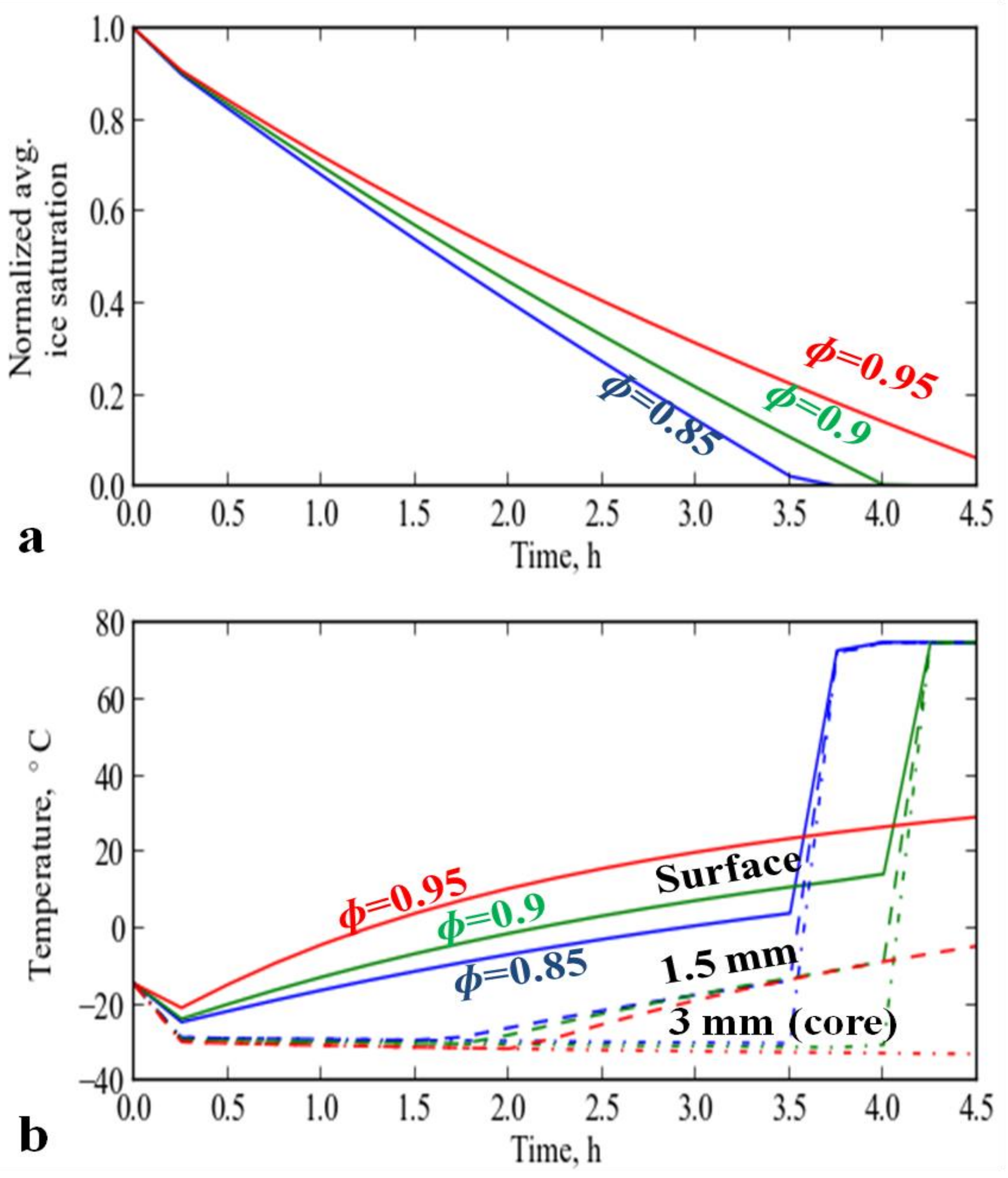

Figure 7. Sensitivity of porosity

Conclusions:

A continuum, porous medium formulation with non-equilibrium sublimation was developed and validated for freeze drying with and without uniform microwave volumetric heating. Excellent agreement was observed with experimental drying curves and spatial temperature data. The model incorporates the effect of Knudsen flow at low pressure and low permeability freeze drying.

This freeze drying model therefore brings together previous work with non-equilibrium evaporation formulations in porous media and demonstrates that this approach can be extended to sublimation, ultimately creating a general framework for modeling phase change in porous media for application to many different processing situations for food and biomaterials.

References:

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