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Experimental and simulation studies of primary vacuum freeze-drying process of random solids at microwave heating ☆

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Abstract

This paper concerns experimental and theoretical studies of freeze-drying process at microwave heating. Two kinds of random solids were dried: material which are assumed to have no internal porosity (ground glass), as well as one containing internal porosity (Sorbonorit 4 activated carbon). Formulated one-dimensional two-region model of freeze-drying process at microwave heating takes into account unknown a priori sublimation temperature $T_s(t)$ and mass concentration of water vapor $C_s(t)$ at moving ice front. Steady capacity of internal heat source is correlated with electric field strength E and dissipation coefficient $K(T)$ in both regions of the material to be dried. Linear temperature dependency of dissipation coefficient is assumed and described by two regression parameters: μ_{1_i} and μ_{2_i} for dry (i=I) and frozen (i=II) bed, respectively. A correlation between both measured and calculated temperatures of the sample and actual electric field strength was observed. Fairly good agreement between experimental and simulated results was stated.

Keywords: Freeze-drying; Microwave heating; Internal heat source capacity; Random solids

1. Introduction

Application of microwaves in freeze-drying process results in energy generation directly in dried material. The ability of electromagnetic energy to selective heat affects process kinetics and considerably enhances mass and energy transfer in freeze-dried sample. For specific microwave system of constant frequency f, microwaves absorption and dissipation mechanism depends on dielectric constant ε' and loss factor ε'' of material. Electric field strength E around a sample reflects heating intensity and is the parameter that can be controlled [1–3].

In freeze-dried sample there exist two different dielectrics corresponding to frozen and dried layer of a bed separated by ice sublimation front being moving boundary. Both layers can be assumed to have constant composition. During the process the loss factor ε'' of material varies with its temperature therefore empirical correlation $\varepsilon''=f(T)$ should be applied for both sample regions [4]. In this paper temperature dependence of layer loss factor is directly incorporated into source term in equations of energy balance and assumed to have linear character.

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Nomenclature

a_e	effective thermal diffusivity, m^2/s
c_{p_e}	effective specific heat, J/(kgK)
c_{p_g}	specific heat of gas phase, J/(kgK)
c_{p_w}	specific heat of water vapor, J/(kgK)
D_{eII}	vapor diffusivity in dry layer, m^2/s
D_K	Knudsen diffusivity, m^2/s
D_M	molecular diffusivity, m^2/s
d_z	particle diameter, m
E	electric field strength, V/m
f	microwave frequency, Hz
I_{det}	current intensity of microwave detector, A
k_e	effective thermal conductivity, W/(mK)
K	dissipation coefficient, $W/(mV^2)$
L	material layer thickness, m
N_w	mass flux density diffusing from moving boundary, $kg/(m^2s)$
P	vacuum chamber total pressure, Pa
q	heat flux, W/m^2
Q_v	capacity of internal volumetric heat source, W/m^3
r_p	mean pore radius, m
t	time, s
T	temperature, K
T_{IIref}	arbitrary reference temperature, K
T_L	exposed surface temperature, K
T_0	temperature of bottom surface, K
T_s	temperature of sublimation at moving boundary, K
$T_{s,3}$	triple point temperature, K
$T_{s_{eq}}$	reference temperature (equilibrium temperature of ice sublimation), K
T_∞	surroundings temperature of the sample, K
W	average moisture content of dried bed, kg/kg
W_p	initial moisture content, kg/kg
W_{eq}	equilibrium moisture content, kg/kg
x	Cartesian position coordinate, m
X	position coordinate of moving boundary, m
X_p	initial position coordinate of moving boundary, m

Greek symbols

$\alpha_{II\infty}$	heat transfer coefficient at the surface of region II, $W/(m^2K)$
β_{II}	internal mass transfer coefficient in region II, m/s
Δh_s	enthalpy of sublimation, J/kg
ε	porosity, −
ε_0	permittivity of free space, F/m
ε'	relative dielectric constant, −
ε''	relative loss factor, −
μ_1, μ_2	parameters in Eq. (3), −
ρ_{eI}	effective density of region I, kg/m^3
$\rho_{bu_{II}}$	bulk density of region II, kg/m^3