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On Diffusion Processes and Medical Plants Processing

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Abstract

The paper concerns mathematical modeling of natural medical products processing. Fick's Laws are forming the core of our understanding of diffusion in solids, liquids, and gases. The drying models mainly Fick's Laws based are described in detail. The drying process of medical plants is relatively well studied. The extraction studies are not so well developed. The paper gives some insight in mathematical modeling for extraction. Phenomenological models of the extraction process consist of mass balance equations for solute in solid phase and in fluid phase. By integration of these differential equations time-dependent concentration profiles in both phases are obtained and the extraction curve is calculated from fluid-phase concentration at the extractor outlet. Phase equilibrium depends on extraction pressure and temperature and on the composition of solute, solvent, and matrix. Unfortunately, all these studies are yet very far from the ability to produce the practical recommendations for medical plants processing.

Keywords: Fick's Laws, medical plants processing, mathematical modeling, drying process, extraction process.

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О диффузионных процессах и переработке лекарственных растений

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Аннотация

Статья посвящена математическому моделированию процессов переработки натуральных лекарственных средств. Законы Фика формируют ядро нашего понимания диффузии в твердых телах, жидкостях и газах. Подробно описаны модели сушки, основанные главным образом на законах Фика. Процесс сушки лекарственных растений изучен относительно хорошо. Экстракционные исследования не так хорошо развиты. В статье дается представление о математическом моделировании экстракции. Феноменологические модели процесса экстракции состоят из уравнений баланса масс для растворенного вещества в твердой фазе и в жидкой фазе. Интегрированием этих дифференциальных уравнений получены зависящие от времени профили концентрации в обеих фазах и рассчитана кривая экстракции по концентрации жидкой фазы на выходе экстрактора. Фазовое равновесие зависит от давления и температуры экстракции, а также от состава растворенного вещества, растворителя и матрицы. К сожалению, все эти исследования еще очень далеки от возможности выработки практических рекомендаций по переработке лекарственных растений.

Ключевые слова: законы Фика, обработка лекарственных растений, математическое моделирование, процесс сушки, процесс экстракции.

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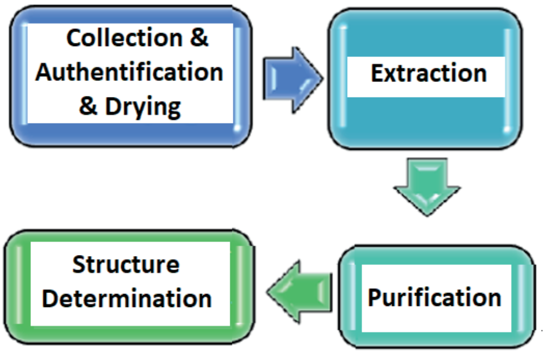
Introduction

Figure 1 shows the basic technologies for processing of natural products. All these technologies are diffusion phenomena based.



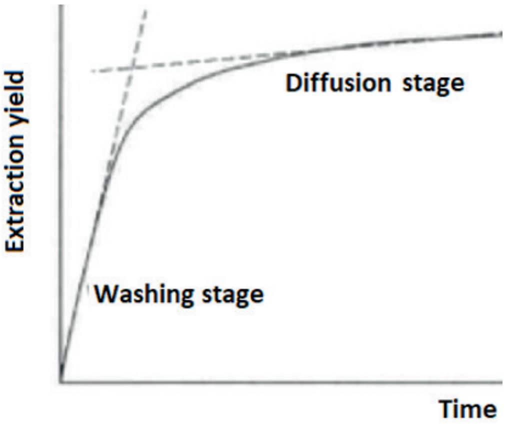
F i g. 1. A Summary in Extraction, Isolation and Determination of Natural Products from Medicinal Plants [1]

Drying. Drying is the most common and fundamental method for post–harvest preservation of medicinal plants. Drying research is an outstanding example of a very complex field, where it is necessary to look comprehensively on the simultaneous energy and mass transfer process that takes place within and on the surface of the material [2].

Extraction methods. It is the crucial first step in the analysis of medicinal plants, because it is necessary to extract the desired chemical components from the plant materials for further separation and characterization Extraction methods include solvent extraction, distillation method, pressing and sublimation according to the extraction principle. Solvent extraction is the most widely used method. The extraction of natural products progresses through the following stages:

(1) The solvent penetrates into the solid matrix
(2) The solute dissolves in the solvents
(3) The solute diffuses out of the solid matrix
(4) The extracted solutes are collected.

For the extraction procedures, solvents such as water, ethanol, chloroform, dichloromethane, hexane, ethyl acetate, methanol, etc are most commonly used.



F i g. 2. Typical extraction curve of batch solvent extraction of active compounds from plants

Isolation and purification. The components in the extracts are complex mixture and contains various type of natural products with different polarities. To obtain pure bioactive compound involves further separation and purification. Many bioactive natural products have been isolated and purified by using different separation techniques such as Thin-layer chromatography (TLC), High Performance thin Layer Chromatography (HPTLC), Paper chromatography, Column chromatography, Gas chromatography, Optimum performance laminar chromatography (OPLC) and High Performance Liquid Chromatography (HPLC). Column chromatography and TLC have been used mostly due to their convenience, economy, and availability in various stationary phases.

Structure determination. Determination of the structure of natural products uses data from a wide range of spectroscopic techniques such as UV-Visible, Infrared (IR), Nuclear Magnetic Resonance (NMR) and Mass spectroscopy. The basic principle of spectroscopy is passing electromagnetic radiation through an organic compound that absorbs some of the radiation, but not all. By measuring the amount of absorption of electromagnetic radiation, a spectrum can be produced. The spectra are specific to certain bonds in a compound. For example, the mass spectrometry technique is based on measurements of diffusion in solution.

The rest of paper is as follows. Section 2 deals Fick's Laws forming the core of our understanding of diffusion in solids, liquids, and gases. In Sections 3 and 4, the drying models are described. Section 5 gives some insight in mathematical modeling for extraction.

Fick's Laws

In 1855, physiologist Adolf Fick first reported his now well-known laws governing the transport of mass through diffusive means. Today, Fick's Laws form the core of our understanding of diffusion in solids, liquids, and gases.

Fick's first law relates the diffusive flux to the concentration under the assumption of steady state. It postulates that the flux goes from regions of high concentration to regions of low concentration, with a magnitude that is proportional to the concentration gradient (spatial derivative), or in simplistic terms the concept that a solute will move from a region of high concentration to a region of low concentration across a concentration gradient. In one (spatial) dimension, the law most common form is:

$$J = -D\frac{d\varphi}{dx}$$

- *J* is the diffusion flux, of which the dimension is amount of substance per unit area per unit time. *J* measures the amount of substance that will flow through a unit area during a unit time interval.
- *D* is the diffusion coefficient or diffusivity. Its dimension is area per unit time.
- *φ* (for ideal mixtures) is the concentration, of which the dimension is amount of substance per unit volume.
- *x* is position, the dimension of which is length.

D is proportional to the squared velocity of the diffusing particles, which depends on the temperature, viscosity of the fluid and the size of the particles.

Fick's second law predicts how diffusion causes the concentration to change with respect to time. It is a partial differential equation, which in one dimension reads:

$$\frac{\partial \varphi}{\partial t} = D\frac{\partial^2 \varphi}{\partial x^2}$$

where

- φ is the concentration in dimensions of [(amount of substance) length^{-3}], example mol/m^3
- t is time, example s
- D is the diffusion coefficient in dimensions of [length^{2}time^{-1}], example m^2/s
- X is the position [length], example m.

Fick's second law has the same mathematical form as Heat equation, and its fundamental solution is the same as Heat kernel:

$$\varphi(x,t) = \frac{1}{\sqrt{4\pi Dt}}\exp\left(-\frac{x^2}{4Dt}\right).$$

Fick's second law can be derived from Fick's first law and the mass conservation in absence of any chemical reactions:

$$\frac{\partial \varphi}{\partial t} + \frac{\partial}{\partial x}J = 0 \Rightarrow \frac{\partial \varphi}{\partial t} - \frac{\partial}{\partial x}\left(D\frac{\partial}{\partial x}\varphi\right) = 0$$

Assuming the diffusion coefficient D to be a constant, one can exchange the orders of the differentiation and multiply by the constant:

$$\frac{\partial}{\partial x}\left(D\frac{\partial}{\partial x}\varphi\right) = D\frac{\partial}{\partial x}\frac{\partial}{\partial x}\varphi = D\frac{\partial^2 \varphi}{\partial x^2}$$

Thus, we receive the form of the Fick's equations as was stated above.

Several mathematical models have been used for the description of extraction process; among them, models based on the Fick's law and semi-theoretical or 'pure' empirical models.

Mathematical Basics for Food Drying

The main mechanisms of drying are surface diffusion or liquid diffusion on the pore surfaces, liquid or vapor diffusion due to moisture concentration differences, and capillary action in granular and porous foods due to surface forces. The dominant diffusion mechanism is a function of the moisture content and the structure of the food material and it determines the drying rate. The dominant mechanism can change during the process and, the determination of the dominant mechanism of drying is important in modeling the process.

Drying processes consider simultaneous heat and mass transfer. They take into consideration both the internal and external heat and mass transfer, and predict the temperature and the moisture gradient in the product better.

If we do not pay attention to the temperature gradient in the product and assume an uniform temperature distribution that equals to the drying air temperature in the product then from Fick's second law of diffusion for planar geometries we get equations

$$\frac{\partial M}{\partial t} = D_{eff}\left[\frac{\partial^2 M}{\partial x^2} + \frac{a_1}{x}\frac{\partial M}{\partial x}\right] \qquad (1)$$

$$\frac{\partial T}{\partial t} = \alpha\frac{\partial^2 T}{\partial x^2}$$

The constant coefficients: *Deff* - effective moisture diffusivity and α – thermal diffusivity.

Assumptions:

(i) The particle is homogenous and isotropic;

(ii) The material characteristics are constant, and the shrinkage is neglected;

(iii) The pressure variations are neglected;

(iv) Evaporation occurs only at the surface;

(v) Initially moisture distribution is uniform and symmetrical during process;

(vi) Surface diffusion is ended, so the moisture equilibrium arises on the surface;

(vii) Temperature distribution is uniform and equals to the ambient drying air temperature, namely the lumped system;

(viii) The heat transfer is done by conduction within the product, and by convection outside of the product;

(ix) Effective moisture diffusivity is constant versus moisture content during drying.

Then analytical solution of (1) one get by (2) [3]:

$$MR = A_1\sum_{i=1}^{\infty}\frac{1}{J_i^2}\exp\left[-\frac{J_i^2 D_{eff}t}{A_2}\right] \qquad (2)$$

where D_{eff} is the effective moisture diffusivity (m^2/s), t is time (s), MR is the fractional moisture ratio, J_i are the roots of the Bessel function, and A_1, A_2 are geometric constants.

Drying Models

The review [4] contains a list of 15 drying models, namely: three semi-theoretical models, nine models derived from Fick's second law of diffusion and three empirical models. We name a few of them.

Semi-theoretical model: Lewis (Newton) Model. In 1921, Lewis [5] suggested that during the drying of porous hygroscopic materials, the change of moisture content of material in the falling rate period is proportional to the instantaneous difference between the moisture content and the expected moisture content when it comes into equilibrium with drying air. This concept assumed that the material is thin enough, or the air velocity is high, and the drying air conditions such as the temperature and the relative humidity are kept constant.

$$\frac{dM}{dt} = -k\,(M - M_e) \qquad (3)$$

where k is the drying constant (s^{-1}).

If K is independent from M, then (3) can be rewritten as:

$$MR = \frac{(M_t - M_e)}{(M_i - M_e)} = \exp(-kt) \qquad (4)$$

where the drying constant k can be obtained from the experimental data and (4) is known as the Lewis (Newton) model.

Henderson and Pabis (Single term) Model. In 1961, Henderson and Pabis [6] improved a model for drying by using Fick's second law of diffusion and applied the new model on drying of corns. As the derivation was shown in the previous section, they use (2). For sufficiently long drying times, only the first term (i = 1) of the general series solution of (2) can be used with small error. According to this assumption, (2) can be written as:

$$MR = \frac{(M_t - M_e)}{(M_i - M_e)} = A_1 \exp\left(-\frac{\pi^2 D_{eff}}{A_2} t\right) \qquad (5)$$

If D_{eff} is constant during drying, then (5) can be rearranged by using the drying constant k as:

$$MR = \frac{(M_t - M_e)}{(M_i - M_e)} = a \exp(-kt)$$

where a is defined as the indication of shape and generally named as model constant (dimensionless). These constants are obtained from experimental data.

Two-Term Model. In 1974, Henderson [7] proposed to use the first two term of the general series solution of Fick's second law of diffusion (1) for correcting the shortcomings of the Henderson and PabisModel. With this argument, the new model derived as:

$$MR = \frac{(M_t - M_e)}{(M_i - M_e)} = a \exp(-k_0 t) + b \exp(-k_1 t)$$

where a, b are defined as the indication of shape and generally named as model constants (dimensionless), and k_0, k_1 are the drying constants (s^{-1}). These constants are obtained from experimental data.

Thompson Model as Empirical one. Thompson [8] developed a model with the experimental results of drying of shelled corns

$$t = a \ln(MR) + b[\ln(MR)]^2$$

where a and b are dimensionless constants obtained from experimental data:

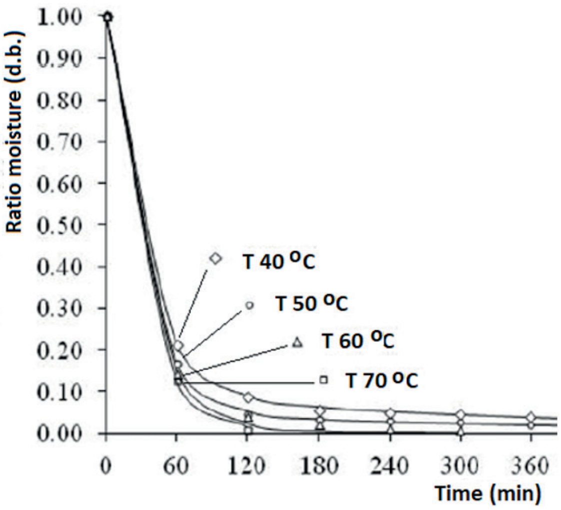
$a = -1.862 + 0.00488\, T$,

$b = 427.4\, e^{-0.033T}$,

t time to dry to MR with drying temperature T, h,

T in Fahrenheit scale.

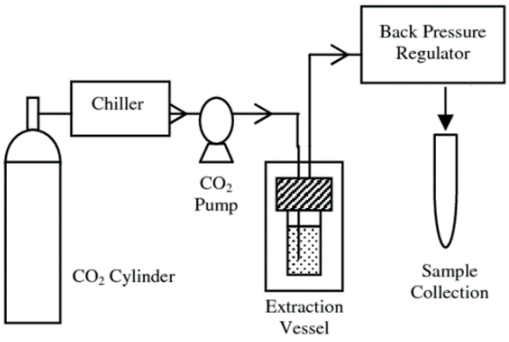
Example 1. In [9], the drying curves were fitted to the experimental data using thirteen different semi-empirical and empirical equations and the conclusion was done: Two Term model is the best one.



F i g. 3. Experimental and estimated values of the moisture ratio by estimating the parameters of the equation of Two Terms (d.b. = dry basis) [9]

The insight in Mathematical Modeling for Extraction

The extraction process of plant substances is much more complex then drying. Let us give a short insight by the reviewing of Sovova's paper [10]. In [10], a mathematical model for extraction of natural products is based on the concept of broken and intact cells. It is particularly suited to fit experimental data as it almost independently simulates two extraction periods, the first one governed by phase equilibrium and the second one governed by internal diffusion in particles. Fig. 2 serves as an illustration to this reviewed paper: the washing stage relates to extraction from broken cells and the diffusion stage relates in great extent to extraction from intact cells. Extraction conditions for supercritical carbon dioxide are considered (Fig. 4), namely: above the critical temperature of 31 C and critical pressure of 75 atm (but these conditions have no influence to the below given mathematical equations).



F i g. 4. Flow diagram of supercritical fluid extraction system

Assumptions: The solute is assumed to be homogeneously distributed in untreated solid particles placed in the extractor. The particles contain broken cells near the surface and intact cells in the core. The volumetric fraction of broken cells in the particles, called grinding efficiency, is r. The easily accessible solute from broken cells is transferred directly to the fluid-phase, while the solute from intact cells diffuses first to broken cells and then to the fluid-phase. Mass transfer from broken cells to the solvent is characterized by fluid-phase mass transfer coefficient that is by several orders of magnitude larger than the solid-phase mass transfer coefficient related to the diffusion from intact cells to broken cells. The specific surface area per unit volume of extraction bed, a_0, is directly proportional to the solid-phase volumetric fraction in the extraction bed and inversely proportional to particle size. For spherical particles, it is equal to $6(1-\varepsilon)/d$. The specific area between the regions of broken and intact cells, a_s, is equal or lower than the specific surface area in dependence on particle shape; e.g. for spherical particles, the ratio a_s/a_0 is equal to $(1-r)^{2/3}$ and for slabs it is equal to 1.

The above notations used:

a_0 specific surface area per unit volume of extraction bed (m^{-1})

a_s specific area between the regions of intact and broken cells (m^{-1})

d particle diameter (m)

r grinding efficiency (fraction of broken cells)

ε bed void fraction

Mass balance per unit volume of extraction bed is written for plug flow. It consists of equations for the solute in fluid phase, solid phase with broken cells, and solid phase with intact cells:

$$\rho_f \varepsilon \left(\frac{\partial y}{\partial t} + U\frac{\partial y}{\partial h}\right) = j_f$$

$$r\rho_s(1-\varepsilon)\frac{\partial x_1}{\partial t} = j_s - j_f$$

$$(1-r)\rho_s(1-\varepsilon)\frac{\partial x_2}{\partial t} = -j_s$$

Initial and boundary conditions are

$$y|_{t=0} = y_0; \quad x_1|_{t=0} = x_{1,0;}$$
$$x_2|_{t=0} = x_{2,0;} \quad Y|_{h=0} = 0.$$

The extraction curve is calculated as

$$E = \dot{Q}\int_0^t y|_{h=H}\,\mathrm{d}t$$

The additional notations used here:
E extract (kg)
h axial co-ordinate (m)
H extraction bed length (m)
j_f flux from broken cells to solvent (kgm^{-3} s^{-1})
j_s flux from intact cells to broken cells (kgm^{-3} s^{-1})
$Q^{\cdot}$ solvent flow rate (kg s^{-1})
U interstitial fluid velocity (m s^{-1})
ρ_f solvent density (kg m^{-3})
ρ_s solid density (kg(insoluble solid) m(solid phase)$^{-3}$)
x_1 concentration in broken cells (kg(solute) kg(insoluble solid)$^{-1}$)
$x_{1,0}$ initial concentration in broken cells (kg(solute) kg(insoluble solid)$^{-1}$)
x_2 concentration in intact cells (kg(solute) kg(insoluble solid)$^{-1}$)
$x_{2,0}$ initial concentration in intact cells (kg(solute) kg(insoluble solid)$^{-1}$)
y fluid-phase concentration (kg(solute) kg(solvent)$^{-1}$)
y_0 initial fluid-phase concentration (kg(solute) kg(solvent)$^{-1}$)
Y (=y/y_0) dimensionless fluid-phase concentration
Phase equilibrium between the fluid-phase and the solid phase with broken cells is given by the discontinuous equilibrium function depicted in Fig. 5:

$$y^*(x_1) = y_s \quad \text{for} \quad x_1 > x_t;$$
$$y^*(x_1) = Kx_1 \quad \text{for} \quad x_1 \le x_{t;} \quad Kx_t < y_s,$$

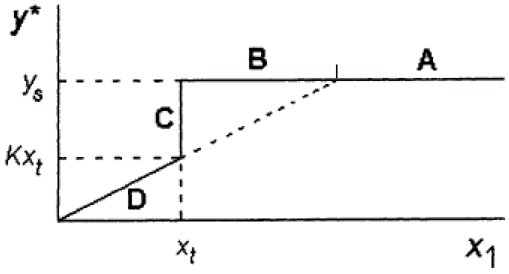


Fig. 5. Equilibrium curve. Letters A–D indicate the regions of initial equilibrium concentrations that determine four types of extraction curves

Here:
y_s solubility (kg(solute) kg(solvent)$^{-1}$)
$y^*(x_1)$ equilibrium fluid-phase concentration (kg(solute) kg(solvent)$^{-1}$)
x_t transition concentration (kg(solute) kg(insoluble solid)$^{-1}$)
K partition coefficient

In the following sections of Sovova's paper [10], the study of extraction model is carried on in much more detail brightly illustrating the complexity of the extraction mathematical models. Let us apply to the concluding example.

Example 2: essential oil from leaves and flowers. Reis-Vasco et al. [11] extracted pennyroyal essential oil at three mean particle sizes (0.3, 0.5 and 0.7 mm); the untreated material contained x_u = 0.026 g g^{-1} oil, where x_u - concentration in the untreated solid. The solvent was supercritical CO_2 at 100 atm and 50 °C and at three flow rates (Fig. 6). The first parts of extraction curves measured at the maximum flow rate of 0.62 g s^{-1} for different particle sizes were overlapping straight lines of the slope y_0 = 0.0015 continuing up to the yield of about 70%.

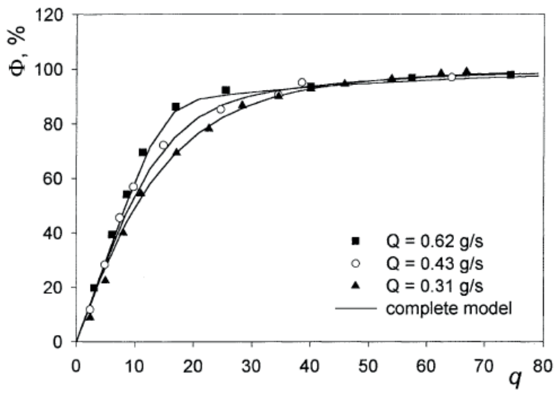


Fig. 6. Extraction of pennyroyal essential oil from leaves and flowers, particle size 0.5mm

Here:
Φ (=$E/(Nc_u)$) dimensionless extraction yield
c_u solute content in the untreated solid (kg(solute) kg(solid)$^{-1}$)
N solid charge in the extractor (kg)
q relative amount of the passed solvent (kg(solvent) kg(insoluble solid)$^{-1}$)
The Sovova's paper [10] could serve, in our opinion, as an example for medical plants extraction research.

Discussion and future work

The drying process of medical plants is relatively well studied. The extraction studies are not so well developed. Phenomenological models of the extraction process consist of mass balance equations for solute in solid phase and in fluid phase. By integration of these differential equations time-dependent concentration profiles in both phases are obtained and the extraction curve is calculated from fluid-phase concentration at the extractor outlet. Phase equilibrium depends on extraction pressure and temperature and on the composition of solute, solvent, and matrix. Unfortunately, all these studies are yet very far from the ability to produce the practical recommendations for medical plants processing. This important task is one exciting topic for future work.

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