

Vacuum Drying

Introduction

Vacuum drying is a chemical process frequently used in the pharmaceutical and food industries to remove water or an organic solvent from a wet powder. When designing a vacuum drying system, engineers aim to minimize the drying time while maintaining high quality in the product. This model investigates vacuum drying in a Nutsche filter-dryer, which consists of a cylindrical drum filled with wet cake as seen in Figure 1. The top of the cake is exposed to a low pressure head space, and the side and bottom walls are exposed to heating fluid. By operating at a very low pressure and an elevated temperature, the evaporation rate of the liquid increases, thus accelerating the drying process of the powder. By modeling the heat transfer and evaporation of the solvent in the powder, the temperature and liquid phase profiles can be studied.

This example is based on the paper published by Murru and others for a powder dried with n-propanol under static conditions (Ref. 1).



Figure 1: Vacuum drying in an axisymmetric Nutsche filter dryer (Ref. 1).

Model Definition

The cylindrical cake can be modeled using a rectangular geometry in a 2D axisymmetric component. The cake radius is 40 cm and the cake height is 10 cm.

The vacuum drying process involves heat transfer and vapor transport in modeling the evaporation of the solvent. This example simulates the heat transfer through the cake using the Heat Transfer in Solids interface and the volume fraction of solvent using a Coefficient

Form PDE interface. A predefined moisture transport in porous media physics interface is available in the Heat Transfer Module.

Heat flux boundary conditions are defined on the side and bottom boundaries to account for the external heating fluid. The evaporation of solvent is captured in two ways: a heat sink term to account for the energy lost from the cake as the liquid solvent changes to vapor phase, and a mass sink term to account for the loss of solvent in the cake.

LIQUID PHASE EVAPORATION

The equation in the Coefficient Form PDE interface solves for the volume fraction of liquid, θ_L , in the cake:

$$\frac{\partial \theta_L}{\partial t} = \nabla \cdot (D_L \nabla \theta_L) - \frac{\dot{m}_{LG}}{\rho_L}$$

where D_L is the apparent liquid diffusion coefficient, \dot{m}_{LG} is the evaporation rate, and ρ_L is the liquid phase density.

The source term m_{LG}/ρ_L accounts for the loss of solvent as it evaporates. All boundaries are prescribed as no flux conditions.

The evaporation rate \dot{m}_{LG} is related to the difference in the equilibrium vapor pressure of the gas in the cake p^* and head space vapor pressure p_G with:

$$\dot{m}_{LG} = k_{\text{vap}} \rho_L (p^* - p_G) / p_G, \text{ if } \theta_L > 0$$

$$\dot{m}_{LG} = 0, \text{ if } \theta_I < 0 \text{ or } p^* \le p_G$$

where k_{vap} is the evaporation rate constant (1/s). In this case, the head space pressure, or the vacuum pressure surrounding the cake, is set to be 15 mbar.

Evaporation should cease once the value of liquid phase reaches zero (fully evaporated) or if the local vapor pressure does not exceed the head space vapor pressure (no driving force for the evaporation). In the model, step functions are used to smoothly ramp the evaporation rate down to zero under these conditions.

The equilibrium vapor pressure of n-propanol, which is related to the temperature (T), can be found using Antoine's equation: $p^* = 10^{A-B/(C+T)}$ where A, B and C are constants found in Ref. 2.

The solvent migrates in the cake due to capillary flow. Instead of directly solving for the velocity field of the liquid phase in the porous media, the transport of the solvent through the cake can be approximated as a diffusion process. Therefore, when there is a gradient

in the volume fraction of solvent, the liquid phase migrates from the region with a high volume fraction of solvent to the region with a low volume fraction of solvent. The liquid diffusion coefficient is calculated with:

$$D_L = \alpha(\theta_L - \theta_L^*), \text{ if } \theta_L \ge \theta_L^*$$
$$D_L = 0, \text{ if } \theta_L < \theta_L^*$$

where θ_L^* is the residual saturation and α is the proportionality constant, both of which are determined experimentally in Ref. 1. When the volume fraction of liquid phase present is below the critical value of θ_L^* , then diffusion of the solvent in the cake should no longer occur. In the model, a step function is used to smoothly ramp the diffusion coefficient down to zero under these conditions.

As the cake dries, the liquid phase evaporates and is replaced by gas between the solid particulates. The sum of the volume fractions of the three phases should equal unity. Therefore, the volume fraction of gas present (θ_G) can be calculated with respect to the constant volume fraction of solid powder (θ_S) and the variable liquid volume fraction (θ_L) with $\theta_G = 1 - (\theta_L + \theta_S)$.

HEAT TRANSFER

The equations in the Heat Transfer in Solids interface solve for the temperature, T, in the cake:

$$\rho_{\mathrm{eff}} c_{p,\,\mathrm{eff}} \frac{\partial T}{\partial t} \,=\, \nabla \cdot (\lambda_{\mathrm{eff}} \nabla T) + Q$$

where ρ_{eff} is the density (SI unit: kg/m^3), C_p is the heat capacity (SI unit: J/(kg*K)), λ_{eff} is the thermal conductivity (SI unit: W/(m*K)), and Q is the heat source (SI unit: W).

Energy is required for evaporation to occur, which is characterized in a heat source domain condition with $Q = -\dot{m}_{LG}\Delta H_{vap}$, where ΔH is the latent heat of vaporization (SI unit: J/kg) found in Ref. 3.

The side and bottom boundaries of the filter-dryer are exposed to a heating fluid. In this model, heat flux boundary conditions account for the energy transferred to the cake from $60^{\circ}C$ heating fluid given a heat transfer coefficient of 10 W/(m^2*K) .

The cake consists of solid powder particulates, a liquid solvent, and a gas that fills the voids between the solid powder particulates. Therefore, the material properties of the cake must take into account the properties of the 3 phases in proportion to their presence in the cake. In this case, the effective density ρ_{eff} and effective heat capacity $c_{p,eff}$ are calculated with:

$$\begin{split} \rho_{\text{eff}} &= \theta_L \rho_L + \theta_S \rho_S + \theta_G \rho_G \\ c_{p,\,\text{eff}} &= (\theta_L \rho_L c_{p,\,L} + \theta_S \rho_S c_{p,\,S} + \theta_G \rho_G c_{p,\,G}) / \rho_{eff} \end{split}$$

where θ_L , θ_S , and θ_G represent the volume fraction of the liquid, solid, and gas phases in the cake, respectively. The material properties of the liquid phase (ρ_L and $c_{p,L}$) are taken from Ref. 4 for n-propanol at a temperature of 40°*C*. Solid powder properties are found in Ref. 1.

The effective thermal conductivity is calculated with:

$$\lambda_{\text{eff}} = \lambda_{\text{dry}} + \frac{\theta_L}{1 - \theta_S} (\lambda_{\text{wet}} - \lambda_{\text{dry}})$$

where λ_{dry} and λ_{wet} are the thermal conductivities of the dry and fully saturated cake, respectively, found in Ref. 1. Instead of writing user defined expressions for the effective material properties of the cake, the **Multiphase Material** is used.

Results and Discussion

The following figures show the temperature (Figure 2), volume fraction of liquid phase (Figure 3), and the apparent moisture diffusivity (Figure 4) in the cake after 30 hours. The temperature of the cake approaches the temperature of the heating fluid ($60^{\circ}C$ or 333.15 K) at the bottom and side boundaries, as seen in Figure 2. Because the liquid phase evaporates in the heated region, the volume fraction of the liquid phase is lowest near the heated side and bottom boundaries and highest in the center of the cake, as seen in Figure 3. The apparent moisture diffusivity is also highest in the center of the cake where liquid phase has not yet evaporated and approaches zero in areas where the liquid phase has already evaporated, as seen in Figure 4.

The evaporation rate after 10, 20, and 30 hours is shown in Figure 5, Figure 6, and Figure 7. The evaporation front starts near the side and bottom walls where the cake is heated. As the amount of solvent present near these boundaries decreases, the evaporation rate decreases in turn, shifting the evaporation front toward the center of the cake.



Figure 2: Temperature in the cake after 30 hours.



Figure 3: Volume fraction of liquid phase in the cake after 30 hours.



Figure 4: Apparent moisture diffusivity in the cake after 30 hours.



Figure 5: Evaporation rate after 10 hours.



Figure 6: Evaporation rate after 20 hours.



Figure 7: Evaporation rate after 30 hours.

References

1. M. Murru et al., "Model-based scale-up of vacuum contact drying of pharmaceutical compounds", *Chemical Engineering Science*, 66, pp. 5045–5054, 2011.

2. H.R. Kemme and S.I. Kreps, "Vapor pressure of primary n-alkyl chlorides and alcohols", *Journal of Chemical Engineering Data*, 14 (1), pp. 98–102. 1969.

3. I.M. Smallwood, Handbook of Organic Solvent Properties, Arnold, 1996.

4. "n-Propanol", BASF, Technical Leaflet, March 2008.

Application Library path: COMSOL_Multiphysics/Chemical_Engineering/ vacuum_drying

Modeling Instructions

From the File menu, choose New.

NEW

In the New window, click 🙆 Model Wizard.

MODEL WIZARD

- I In the Model Wizard window, click 👘 2D Axisymmetric.
- 2 In the Select Physics tree, select Heat Transfer>Heat Transfer in Solids (ht).
- 3 Click Add.

Add also a PDE interface to solve for the volume fraction of liquid in the cake.

- 4 In the Select Physics tree, select Mathematics>PDE Interfaces>Coefficient Form PDE (c).
- 5 Click Add.
- 6 In the Field name text field, type theta.
- 7 In the **Dependent variables** table, enter the following settings:

thetaL

8 In the Source term quantity table, enter the following settings:

Source term quantity	Unit
Custom unit	1/s

9 Click 🔿 Study.

IO In the Select Study tree, select General Studies>Time Dependent.

II Click **M** Done.

The list of parameters and variables are imported from files available in the Application Libraries folder.

GLOBAL DEFINITIONS

Parameters 1

- I In the Model Builder window, under Global Definitions click Parameters I.
- 2 In the Settings window for Parameters, locate the Parameters section.
- **3** Click **b** Load from File.
- 4 Browse to the model's Application Libraries folder and double-click the file vacuum_drying_parameters.txt.

DEFINITIONS

Variables I

- I In the Model Builder window, under Component I (compl) right-click Definitions and choose Variables.
- 2 In the Settings window for Variables, locate the Variables section.
- 3 Click 📂 Load from File.
- **4** Browse to the model's Application Libraries folder and double-click the file vacuum_drying_variables.txt.

Next define two step functions to smoothly decrease the evaporation rate and apparent moisture diffusivity to zero as the solvent evaporates.

Step I (step I)

- I In the Home toolbar, click f(X) Functions and choose Global>Step.
- 2 In the Settings window for Step, locate the Parameters section.
- **3** In the **Location** text field, type **1.005**.
- 4 Click to expand the **Smoothing** section. In the **Size of transition zone** text field, type 0.01.

Step 2 (step 2)

- I In the Home toolbar, click f(X) Functions and choose Global>Step.
- 2 In the Settings window for Step, locate the Parameters section.

- **3** In the **Location** text field, type **0.005**.
- 4 Locate the Smoothing section. In the Size of transition zone text field, type 0.01.

GEOMETRY I

Rectangle 1 (r1)

- I In the **Geometry** toolbar, click **Rectangle**.
- 2 In the Settings window for Rectangle, locate the Size and Shape section.
- 3 In the Width text field, type R0.
- 4 In the **Height** text field, type H0.



MATERIALS

Multiphase Material I (mpmat1)

In the Model Builder window, under Component I (compl) right-click Materials and choose More Materials>Multiphase Material.

Constrained Gas Phase

- I In the Model Builder window, under Component I (compl)>Materials> Multiphase Material I (mpmatl) click Phase I (mpmatl.phasel).
- 2 In the Settings window for Phase, type Constrained Gas Phase in the Label text field.

3 Locate the Material Contents section. In the table, enter the following settings:

Property	Variable	Value	Unit	Property group
Thermal conductivity	k_iso ; kii = k_iso, kij = 0	lambda_dr y/(1- thetaS)	W/(m·K)	Basic
Density	rho	rhoG	kg/m³	Basic
Heat capacity at constant pressure	Ср	CpG	J/(kg·K)	Basic

Liquid Phase

- I In the Model Builder window, right-click Multiphase Material I (mpmat1) and choose Phase.
- 2 In the Settings window for Phase, type Liquid Phase in the Label text field.
- ${\bf 3}$ Locate the ${\bf Volume\ Fraction\ section.}$ In the $V_{\rm f}$ text field, type thetaL.
- 4 Locate the Material Contents section. In the table, enter the following settings:

Property	Variable	Value	Unit	P roperty group
Thermal conductivity	k_iso ; kii = k_iso, kij = 0	lambda_we t/(1- thetaS)	W/(m·K)	Basic
Density	rho	rhoL	kg/m³	Basic
Heat capacity at constant pressure	Ср	CpL	J/(kg·K)	Basic

Solid Phase

- I Right-click Multiphase Material I (mpmat I) and choose Phase.
- 2 In the Settings window for Phase, type Solid Phase in the Label text field.
- ${\bf 3}$ Locate the Volume Fraction section. In the $V_{\rm f}$ text field, type thetaS.
- **4** Locate the **Material Contents** section. In the table, enter the following settings:

Property	Variable	Value	Unit	Property group
Thermal conductivity	k_iso ; kii = k_iso, kij = 0	0	W/(m·K)	Basic

Property	Variable	Value	Unit	Property group
Density	rho	rhoS	kg/m³	Basic
Heat capacity at constant pressure	Ср	CpS	J/(kg·K)	Basic

HEAT TRANSFER IN SOLIDS (HT)

Heat Source 1

- I In the Model Builder window, under Component I (compl) right-click Heat Transfer in Solids (ht) and choose Heat Source.
- **2** Select Domain 1 only.
- 3 In the Settings window for Heat Source, locate the Heat Source section.
- **4** In the Q_0 text field, type -mdot*deltaH.

Heat Flux 1

- I In the Physics toolbar, click Boundaries and choose Heat Flux.
- **2** Select Boundaries 2 and 4 only.
- 3 In the Settings window for Heat Flux, locate the Heat Flux section.
- 4 From the Flux type list, choose Convective heat flux.
- **5** In the *h* text field, type hq.
- **6** In the T_{ext} text field, type Th.

Initial Values 1

- I In the Model Builder window, click Initial Values I.
- 2 In the Settings window for Initial Values, locate the Initial Values section.
- **3** In the T text field, type T0.

Next, define the Coefficient Form PDE interface for the liquid phase evaporation.

COEFFICIENT FORM PDE (C)

Coefficient Form PDE 1

- I In the Model Builder window, under Component I (compl)>Coefficient Form PDE (c) click Coefficient Form PDE I.
- 2 In the Settings window for Coefficient Form PDE, locate the Diffusion Coefficient section.
- **3** In the *c* text field, type DL.
- **4** Locate the **Source Term** section. In the *f* text field, type -mdot/rhoL.

Initial Values 1

- I In the Model Builder window, click Initial Values I.
- 2 In the Settings window for Initial Values, locate the Initial Values section.
- **3** In the *thetaL* text field, type thetaL0.

Set the mesh to extremely fine to better resolve the evaporation front in the domain.

MESH I

- I In the Model Builder window, under Component I (compl) click Mesh I.
- 2 In the Settings window for Mesh, locate the Physics-Controlled Mesh section.
- 3 From the Element size list, choose Extremely fine.



4 Click 📗 Build All.

STUDY I

Solution 1 (soll)

I In the Study toolbar, click **Show Default Solver**.

The dependent variable scaling for thetaL is manually set to 1. This provides a better convergence.

2 In the Model Builder window, expand the Solution I (soll) node.

- 3 In the Model Builder window, expand the Study I>Solver Configurations>
 Solution I (soll)>Dependent Variables I node, then click
 Dependent variable thetaL (compl.thetaL).
- 4 In the Settings window for Field, locate the Scaling section.
- 5 From the Method list, choose Manual.

Step 1: Time Dependent

- I In the Model Builder window, under Study I click Step I: Time Dependent.
- 2 In the Settings window for Time Dependent, locate the Study Settings section.
- **3** From the **Time unit** list, choose **h**.
- 4 In the **Output times** text field, type range(0,1,70).
- **5** In the **Study** toolbar, click **= Compute**.

RESULTS

2D Plot Group 3

- I In the Model Builder window, under Results click 2D Plot Group 3.
- 2 In the Settings window for 2D Plot Group, locate the Data section.
- 3 From the Time (h) list, choose 30.
- 4 In the 2D Plot Group 3 toolbar, click 💿 Plot.

Temperature

- I In the Home toolbar, click 🚛 Add Plot Group and choose 2D Plot Group.
- 2 In the Settings window for 2D Plot Group, type Temperature in the Label text field.
- 3 Locate the Data section. From the Time (h) list, choose 30.

Surface 1

- I Right-click Temperature and choose Surface.
- 2 In the Settings window for Surface, locate the Coloring and Style section.
- 3 Click Change Color Table.
- 4 In the Color Table dialog box, select Thermal>HeatCameraLight in the tree.
- 5 Click OK.
- 6 In the **Temperature** toolbar, click **I** Plot.

Apparent moisture diffusivity

I In the Home toolbar, click 🚛 Add Plot Group and choose 2D Plot Group.

- **2** In the **Settings** window for **2D Plot Group**, type Apparent moisture diffusivity in the **Label** text field.
- 3 Locate the Data section. From the Time (h) list, choose 30.

Surface 1

- I Right-click Apparent moisture diffusivity and choose Surface.
- 2 In the Settings window for Surface, locate the Expression section.
- **3** In the **Expression** text field, type DL.
- **4** In the **Apparent moisture diffusivity** toolbar, click **I** Plot.

Evaporation rate

- I In the Home toolbar, click 🚛 Add Plot Group and choose 2D Plot Group.
- 2 In the Settings window for 2D Plot Group, type Evaporation rate in the Label text field.
- 3 Locate the Data section. From the Time (h) list, choose 30.

Surface 1

- I Right-click Evaporation rate and choose Surface.
- 2 In the Settings window for Surface, locate the Expression section.
- **3** In the **Expression** text field, type mdot.
- **4** In the **Evaporation rate** toolbar, click **I** Plot.