Created in COMSOL Multiphysics 6.0

Freeze-Drying

Introduction

Freeze-drying, or lyophilization, is a dehydration process that is often used to preserve a perishable food or material. It is also frequently used as a way to remove water from goods, making them lighter and easier to transport. Lyophilization is widely recognized as an important technique in many industries. It is used, for example, in the pharmaceutical industry for the preservation of antibiotics, in the manufacturing of semiconductor ceramics, and by preservation initiatives, such as in the restoration of water-damaged documents. And of course, the process is widely used in the food industry, to preserve tasty snacks that can last up to 30 years. The wet substance is frozen and ice (or some other frozen solvent) is removed through sublimation in the presence of a high vacuum.

Freeze-drying relies on sublimation, where a frozen liquid undergoes phase change directly from a frozen state and into a gaseous state. As can be shown in a phase diagram, at very low pressures and temperatures, a solid can pass directly into the gaseous stage without passing through the intermediary liquid phase.

This model demonstrates the process of ice sublimation in a vial under vacuum-chamber conditions, a test case for many freeze-drying setups.

Model Definition

In this example, you model the process of sublimation of ice through the pores of a porous medium in a vial. Figure 1 depicts the model geometry cut in half.

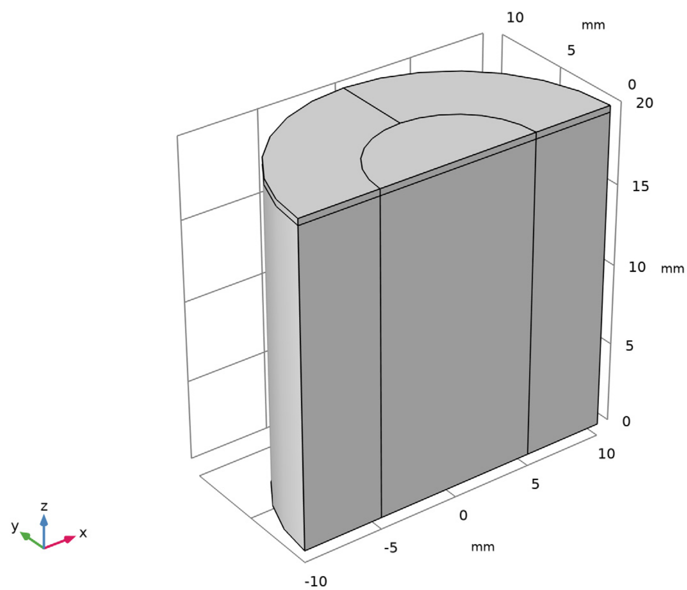

Figure 1: Vial model geometry.

Since the geometry and boundary conditions are axisymmetric, a 2D axisymmetric model would be sufficient. However, for demonstration purposes and to make the model more general, a good compromise considering the computation time is to solve the model in 3D with a symmetry on the $y = 0$ plane.

MASS TRANSFER

The frozen product (here skim milk) initially fills 98% of the vial.The remaining gap contains the dried product and the vapor generated by sublimation, and expands during the sublimation process.The inert gas is neglected in the simulation.

The vapor flow rate Q_m through the pores of the dried product is defined by the stationary Darcy's law:

$$\nabla \cdot \left(-\frac{\rho_\mathrm{v}\kappa}{\mu_\mathrm{v}}\nabla p_\mathrm{v}\right) = Q_\mathrm{m} \tag{1}$$

where ρ_v is the vapor density, μ_v is the vapor dynamic viscosity, p_v is the vapor partial pressure, and κ is the permeability of the dried product.

The top of the vial is close to vacuum, maintained at a vapor pressure p_0 of 24 Pa.

At the sublimation interface, the mass flux N_0 is:

$$N_0 = \varepsilon \rho_{ice} V_s \tag{2}$$

where ρ_{ice} is the ice density, ε is the porosity of the product, and V_s is the sublimation interface velocity.

HEAT TRANSFER

You solve the heat transfer equation for porous media, without convection in the frozen domain,

$$(\rho C_p)_{eff, fr} \frac{\partial T}{\partial t} + \nabla \cdot (-k_{eff, fr} \nabla T) = 0 \tag{3}$$

and with convection in the dried domain

$$(\rho C_p)_{eff, dr} \frac{\partial T}{\partial t} + \rho_v C_{p, v} \mathbf{u} \cdot \nabla T + \nabla \cdot (-k_{eff, dr} \nabla T) = 0 \tag{4}$$

Here, $(\rho C_p)_{eff,dr}$ and $k_{eff,dr}$ refer to the equivalent volumetric heat capacity and thermal conductivity of the dried region, $(\rho C_p)_{eff,fr}$ and $k_{eff,fr}$ refer to the equivalent volumetric heat capacity and thermal conductivity of the frozen region, $C_{p,v}$ is the specific heat capacity of vapor, and $\mathbf{u}$ is the velocity obtained from Darcy's law.

In the vapor domain, the density is calculated using the molar mass of vapor, M_v, and the ideal gas law:

$$\rho_v = \frac{M_v p_v}{RT} \tag{5}$$

where R is the universal gas constant.

Convective exchanges with the surrounding air occur all around the vial. At the bottom of the vial, convective exchanges with the shelf occur as well, and the heat transfer coefficient is lower on the center than on the exterior of the vial due to an air gap.

ICE-VAPOR INTERFACE

Assuming thermodynamic equilibrium at the phase change interface, the sublimation front temperature T_s is defined from the vapor pressure p_v at this boundary, using the Clausius–Clapeyron relation:

$$T_s = 2.19 \times 10^{-3} \frac{L_s}{(28.89 - \log(p_v))} \tag{6}$$

where L_s is the sublimation latent heat.

Simultaneous heat and mass balances at the sublimation interface lead to the Stefan condition for the interface velocity V_s:

$$V_s = \frac{Q_s}{\rho_{ice} L_s} \tag{7}$$

where Q_s is the jump in the normal heat flux at the interface.

Results

The process of primary drying takes about 10 hours to transform most of the initial amount of ice. The frozen product has the proportions shown in Figure 2.

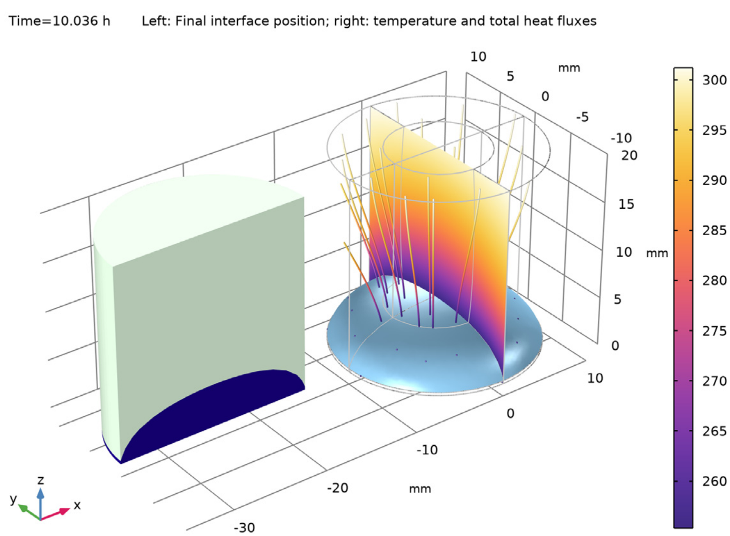

Figure 2: Temperature and heat flux at the end of the drying time period.

You can see from this visualization that the sublimation front has a concave curved shape that is lower around the outside edges of the vial wall. As the ice becomes a vapor, the mass of the solid decreases and therefore the mesh in the frozen domain squeezes as well.

Notes About the COMSOL Implementation

To set up the application in COMSOL Multiphysics, use the Deformed Geometry interface to track the ice surface, and then compute coupled mass and heat balances on the moving mesh. Figure 3 shows the initial mesh and Figure 4 shows the deformed mesh at the end of the simulation.

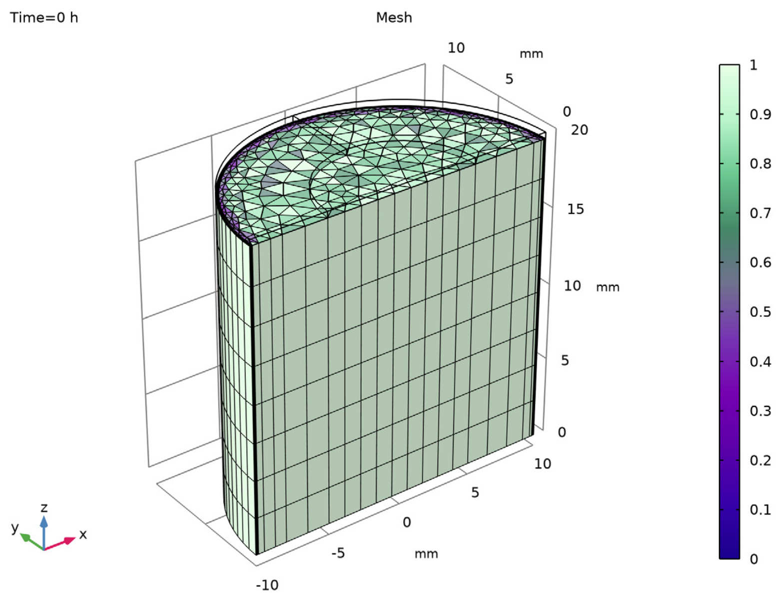

Figure 3: Initial swept mesh.

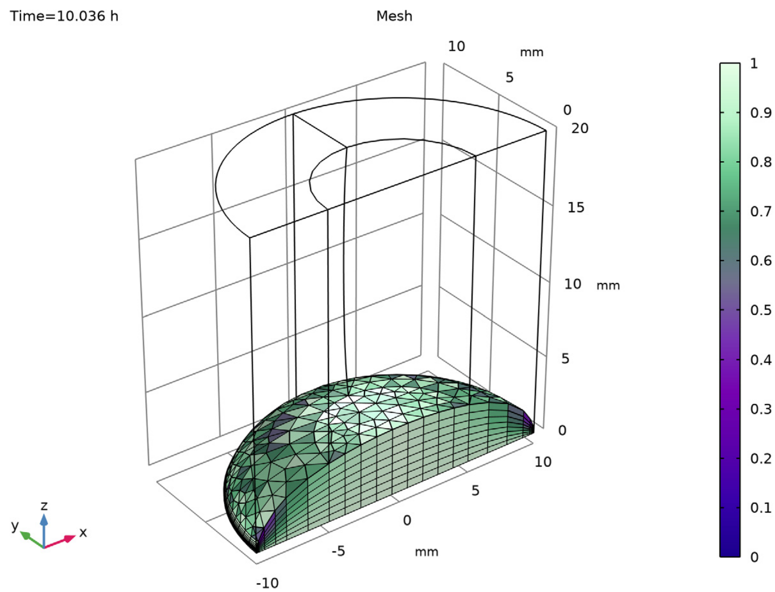

Figure 4: Deformed mesh at the end of the simulation.

In Equation 7, Q_s corresponds to the jump in the normal heat flux at the interface. This quantity can be precisely evaluated through the Lagrange multiplier for temperature, `T_lm`. This variable is computed in the **Phase Change Interface** feature, which sets a fixed temperature weak constraint on the sublimation front.

References

1. A. Alonso and others, “Time-scale modeling and optimal control of freeze-drying,” *Journal of Food Engineering*, vol. 111, pp. 655–666, 2012.

2. W.J. Mascarenhas, H.U. Akay, and M.J. Pikal, “A computational model for finite element analysis of the freeze-drying process,” *Computer Methods on Applied Mechanics and Engineering*, vol. 148, pp. 105–124, 1997.

3. Hriberšek and others, “Lyophilization model of mannitol water solution in a laboratory scale lyophilizer”, *Journal of Drug Delivery Science and Technology*, vol. 45, pp. 28–38, 2018.

Application Library path: `Heat_Transfer_Module/Phase_Change/freeze_drying`

Modeling Instructions

From the **File** menu, choose **New**.

NEW

In the **New** window, click **Model Wizard**.

MODEL WIZARD

1 In the **Model Wizard** window, click **3D**.

2 In the **Select Physics** tree, select **Fluid Flow>Porous Media and Subsurface Flow>Darcy's Law (dl)**.

3 Click **Add**.

4 In the **Select Physics** tree, select **Heat Transfer>Porous Media>Heat Transfer in Porous Media (ht)**.

5 Click **Add**.

6 In the **Select Physics** tree, select **Mathematics>Deformed Mesh>Legacy Deformed Mesh>Deformed Geometry (dg)**.

7 Click **Add**.

8 Click **Study**.

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

10 Click **Done**.

For this model, some parameters are needed. Start by loading all of them from a text file.

GLOBAL DEFINITIONS

Parameters 1

1 In the **Model Builder** window, under **Global Definitions** click **Parameters 1**.

2 In the **Settings** window for **Parameters**, locate the **Parameters** section.

3 Click **Load from File**.

4 Browse to the model's Application Libraries folder and double-click the file `freeze_drying_parameters.txt`.

Add empty materials that will be linked later to the porous materials.

Vapor

1 In the **Model Builder** window, under **Global Definitions** right-click **Materials** and choose **Blank Material**.

2 In the **Settings** window for **Material**, type `Vapor` in the **Label** text field.

Ice

1 Right-click **Materials** and choose **Blank Material**.

2 In the **Settings** window for **Material**, type `Ice` in the **Label** text field.

Product (Skim Milk)

1 Right-click **Materials** and choose **Blank Material**.

2 In the **Settings** window for **Material**, type `Product (Skim Milk)` in the **Label** text field.

GEOMETRY 1

1 In the **Model Builder** window, under **Component 1 (comp1)** click **Geometry 1**.

2 In the **Settings** window for **Geometry**, locate the **Units** section.

3 From the **Length unit** list, choose **mm**.

Cylinder 1 (cyl1)

1 In the **Geometry** toolbar, click **Cylinder**.

2 In the **Settings** window for **Cylinder**, locate the **Size and Shape** section.

3 In the **Radius** text field, type `R0`.

4 In the **Height** text field, type `Z0`.

5 Click to expand the **Layers** section. In the table, enter the following settings:

Layer name	Thickness (mm)
Layer 1	Zi

6 Clear the **Layers on side** check box.

7 Select the **Layers on top** check box.

Cylinder 2 (cyl2)

1 In the **Geometry** toolbar, click **Cylinder**.

2 In the **Settings** window for **Cylinder**, locate the **Size and Shape** section.

3 In the **Radius** text field, type `R0`.

4 In the **Height** text field, type `Z0`.

5 Locate the **Layers** section. In the table, enter the following settings:

Layer name	Thickness (mm)
`Layer 1`	`R0/2`

Block 1 (blk1)

1 In the **Geometry** toolbar, click **Block**.

2 In the **Settings** window for **Block**, locate the **Size and Shape** section.

3 In the **Width** text field, type `R0*5`.

4 In the **Depth** text field, type `R0*2`.

5 In the **Height** text field, type `Z0`.

6 Locate the **Position** section. In the **x** text field, type `-R0*2`.

7 In the **y** text field, type `-R0*2`.

Difference 1 (dif1)

1 In the **Geometry** toolbar, click **Booleans and Partitions** and choose **Difference**.

2 In the **Settings** window for **Difference**, locate the **Difference** section.

3 Click **Paste Selection**.

4 In the **Paste Selection** dialog box, type `cyl1 cyl2` in the **Selection** text field.

5 Click **OK**.

6 In the **Settings** window for **Difference**, locate the **Difference** section.

7 Find the **Objects to subtract** subsection. Click to select the **Activate Selection** toggle button.

8 Select the object **blk1** only.

9 Click **Build All Objects**.

10 Click the **Zoom Extents** button in the **Graphics** toolbar.

These steps create a geometry similar to that in Figure 1.

DEFINITIONS

Interface

1 In the **Model Builder** window, expand the **Component 1 (comp1)>Definitions** node.

2 Right-click **Definitions** and choose **Variables**.

Define the sublimation temperature at the phase change interface according to the Clausius-Clapeyron equation.

3 In the **Settings** window for **Variables**, type `Interface` in the **Label** text field.

4 Locate the **Variables** section. In the table, enter the following settings:

Name	Expression	Unit	Description
T_s	2.19e-3*DelHs*1[kg/J]/(28.89-log(p*1[1/Pa]))*1[K]	K	Interface temperature

Integration 1 (intop1)

1 In the **Definitions** toolbar, click **Nonlocal Couplings** and choose **Integration**.

2 In the **Settings** window for **Integration**, locate the **Source Selection** section.

3 Click **Paste Selection**.

4 In the **Paste Selection** dialog box, type `1 3 5` in the **Selection** text field.

5 Click **OK**.

Minimum 1 (minop1)

1 In the **Definitions** toolbar, click **Nonlocal Couplings** and choose **Minimum**.

2 In the **Settings** window for **Minimum**, locate the **Source Selection** section.

3 From the **Geometric entity level** list, choose **Boundary**.

4 Click **Paste Selection**.

5 In the **Paste Selection** dialog box, type `6 13 20` in the **Selection** text field.

6 Click **OK**.

Add porous materials to the materials node, but do not define their properties at this point. After setting up the physics interface, COMSOL Multiphysics automatically detects which material properties are needed.

MATERIALS

Porous Material 1 (pmat1)

In the **Model Builder** window, under **Component 1 (comp1)** right-click **Materials** and choose **More Materials>Porous Material**.

Porous Material 2 (pmat2)

1 Right-click **Materials** and choose **More Materials>Porous Material**.

2 Select Domains 1, 3, and 5 only.

Now, set up the physics interfaces. Start with the Darcy's law interface, that simulates the vapor flow through the pores of the dried layer.

3 Click the Show More Options button in the **Model Builder** toolbar.

4 In the **Show More Options** dialog box, in the tree, select the check box for the node **Physics>Advanced Physics Options**.

5 Click **OK**.

DARCY'S LAW (DL)

1 In the **Model Builder** window, under **Component 1 (comp1)** click **Darcy's Law (dl)**.

2 Select Domains 2, 4, and 6 only.

3 In the **Settings** window for **Darcy's Law**, click to expand the **Equation** section.

4 From the **Equation form** list, choose **Stationary**.

5 Locate the **Physical Model** section. In the p_{ref} text field, type `0`.

6 Click to expand the **Discretization** section. Select the **Compute boundary fluxes** check box.

7 Select Domains 2, 4, and 6 only.

Fluid 1

1 In the **Model Builder** window, under **Component 1 (comp1)>Darcy's Law (dl)> Porous Medium 1** click **Fluid 1**.

2 In the **Settings** window for **Fluid**, locate the **Fluid Properties** section.

3 From the **Fluid type** list, choose **Ideal gas**.

4 From the **Gas constant type** list, choose **Mean molar mass**.

Initial Values 1

1 In the **Model Builder** window, under **Component 1 (comp1)>Darcy's Law (dl)** click **Initial Values 1**.

2 In the **Settings** window for **Initial Values**, locate the **Initial Values** section.

3 In the p text field, type `Pc`.

Pressure 1

1 In the **Physics** toolbar, click **Boundaries** and choose **Pressure**.

2 Select Boundaries 7, 14, and 21 only.

3 In the **Settings** window for **Pressure**, locate the **Pressure** section.

4 In the p_0 text field, type `Pc`.

Mass Flux 1

1 In the **Physics** toolbar, click **Boundaries** and choose **Mass Flux**.

2 Select Boundaries 6, 13, and 20 only.

3 In the **Settings** window for **Mass Flux**, locate the **Mass Flux** section.

4 In the N_0 text field, type `-ht.pci1.vn*rho_ice*por_p`.

Symmetry 1

1 In the **Physics** toolbar, click **Boundaries** and choose **Symmetry**.

2 Select Boundaries 4, 12, and 25 only.

HEAT TRANSFER IN POROUS MEDIA (HT)

Now, set up the Heat Transfer in Porous Media interface, in both dried and frozen layers.

Dried Layer

1 In the **Model Builder** window, under **Component 1 (comp1)>** **Heat Transfer in Porous Media (ht)** right-click **Porous Medium 1** and choose **Rename**.

2 In the **Rename Porous Medium** dialog box, type `Dried Layer` in the **New label** text field.

3 Click **OK**.

Fluid 1

1 In the **Model Builder** window, click **Fluid 1**.

2 In the **Settings** window for **Fluid**, locate the **Heat Convection** section.

3 From the **u** list, choose **Darcy's velocity field (dl/porous1)**.

4 Locate the **Thermodynamics, Fluid** section. From the **Fluid type** list, choose **Ideal gas**.

5 From the **Gas constant type** list, choose **Mean molar mass**.

Porous Matrix 1

1 In the **Model Builder** window, click **Porous Matrix 1**.

2 In the **Settings** window for **Porous Matrix**, locate the **Matrix Properties** section.

3 From the **Define** list, choose **Solid phase properties**.

Initial Values 1

1 In the **Model Builder** window, under **Component 1 (comp1)>** **Heat Transfer in Porous Media (ht)** click **Initial Values 1**.

2 In the **Settings** window for **Initial Values**, locate the **Initial Values** section.

3 In the T text field, type `Ti`.

Frozen Layer

1 In the **Physics** toolbar, click **Domains** and choose **Porous Medium**.

2 In the **Settings** window for **Porous Medium**, type `Frozen Layer` in the **Label** text field.

3 Select Domains 1, 3, and 5 only.

Porous Matrix 1

1 In the **Model Builder** window, click **Porous Matrix 1**.

2 In the **Settings** window for **Porous Matrix**, locate the **Matrix Properties** section.

3 From the **Define** list, choose **Solid phase properties**.

Ambiant Heat Flux

1 In the **Physics** toolbar, click **Boundaries** and choose **Heat Flux**.

2 In the **Settings** window for **Heat Flux**, type `Ambiant Heat Flux` in the **Label** text field.

3 Locate the **Heat Flux** section. From the **Flux type** list, choose **Convective heat flux**.

4 In the h text field, type `3.6`.

5 In the T_{ext} text field, type `Ta`.

6 Locate the **Boundary Selection** section. Click **Paste Selection**.

7 In the **Paste Selection** dialog box, type `2 5 7 14 21 22 23` in the **Selection** text field.

8 Click **OK**.

Surface-to-Ambient Radiation 1

1 In the **Physics** toolbar, click **Boundaries** and choose **Surface-to-Ambient Radiation**.

2 Select Boundaries 7, 14, and 21 only.

3 In the **Settings** window for **Surface-to-Ambient Radiation**, locate the **Surface-to-Ambient Radiation** section.

4 From the ε list, choose **User defined**. In the associated text field, type `0.9`.

5 In the T_{amb} text field, type `Ta`.

Shelf Heat Flux (Center)

1 In the **Physics** toolbar, click **Boundaries** and choose **Heat Flux**.

2 In the **Settings** window for **Heat Flux**, type `Shelf Heat Flux (Center)` in the **Label** text field.

3 Locate the **Boundary Selection** section. Click **Paste Selection**.

4 In the **Paste Selection** dialog box, type `10` in the **Selection** text field.

5 Click **OK**.

6 In the **Settings** window for **Heat Flux**, locate the **Heat Flux** section.

7 From the **Flux type** list, choose **Convective heat flux**.

8 In the h text field, type `11`.

9 In the T_{ext} text field, type `Ts`.

Shelf Heat Flux (Exterior)

1 In the **Physics** toolbar, click **Boundaries** and choose **Heat Flux**.

2 In the **Settings** window for **Heat Flux**, type `Shelf Heat Flux (Exterior)` in the **Label** text field.

3 Locate the **Boundary Selection** section. Click **Paste Selection**.

4 In the **Paste Selection** dialog box, type `3 17` in the **Selection** text field.

5 Click **OK**.

6 In the **Settings** window for **Heat Flux**, locate the **Heat Flux** section.

7 From the **Flux type** list, choose **Convective heat flux**.

8 In the h text field, type `62.3`.

9 In the T_{ext} text field, type `Ts`.

Symmetry 1

1 In the **Physics** toolbar, click **Boundaries** and choose **Symmetry**.

2 Select Boundaries 1, 4, 9, 12, 24, and 25 only.

Phase Change Interface 1

1 In the **Physics** toolbar, click **Boundaries** and choose **Phase Change Interface**.

2 In the **Settings** window for **Phase Change Interface**, locate the **Boundary Selection** section.

3 Click **Paste Selection**.

4 In the **Paste Selection** dialog box, type `6 13 20` in the **Selection** text field.

5 Click **OK**.

6 In the **Settings** window for **Phase Change Interface**, locate the **Phase Change Interface** section.

7 In the T_{pc} text field, type `T_s`.

8 In the $L_{s\to f}$ text field, type `DelHs`.

9 From the **Solid side** list, choose **Downside**.

DEFORMED GEOMETRY (DG)

Finish with the Deformed Geometry interface under the Definitions node. Roller boundaries are set up along the vial and the symmetry axis. The **Phase Change Interface** feature in the Heat transfer interface already specifies a prescribed normal mesh velocity.

1 In the **Model Builder** window, under **Component 1 (comp1)** click **Deformed Geometry (dg)**.

2 In the **Settings** window for **Deformed Geometry**, locate the **Frame Settings** section.

3 From the **Geometry shape function** list, choose **1**.

4 Locate the **Free Deformation Settings** section. From the **Mesh smoothing type** list, choose **Hyperelastic**.

Free Deformation 1

1 In the **Physics** toolbar, click **Domains** and choose **Free Deformation**.

2 In the **Settings** window for **Free Deformation**, locate the **Domain Selection** section.

3 From the **Selection** list, choose **All domains**.

Prescribed Mesh Displacement 2

1 In the **Physics** toolbar, click **Boundaries** and choose **Prescribed Mesh Displacement**.

2 In the **Settings** window for **Prescribed Mesh Displacement**, locate the **Boundary Selection** section.

3 Click **Clear Selection**.

4 Select Boundaries 1, 2, 4, 5, 9, 12, and 22–25 only.

5 Locate the **Prescribed Mesh Displacement** section. Clear the **Prescribed Z displacement** check box.

MATERIALS

The material properties can now be specified.

Porous Material 1 (pmat1)

1 In the **Model Builder** window, under **Component 1 (comp1)>Materials** click **Porous Material 1 (pmat1)**.

2 In the **Settings** window for **Porous Material**, locate the **Homogenized Properties** section.

3 In the table, enter the following settings:

Property	Variable	Value	Unit	Property group
Permeability	kappa_iso ; kappaii = kappa_iso, kappaij = 0	kappa_p	m²	Basic

4 Locate the **Phase-Specific Properties** section. Click **Add Required Phase Nodes**.

Solid 1 (pmat1.solid1)

1 In the **Model Builder** window, click **Solid 1 (pmat1.solid1)**.

2 In the **Settings** window for **Solid**, locate the **Solid Properties** section.

3 From the **Material** list, choose **Product (Skim Milk) (mat3)**.

4 In the θ_s text field, type `1-por_p`.

Porous Material 2 (pmat2)

1 In the **Model Builder** window, under **Component 1 (comp1)>Materials** click **Porous Material 2 (pmat2)**.

2 In the **Settings** window for **Porous Material**, locate the **Phase-Specific Properties** section.

3 Click **Add Required Phase Nodes**.

Fluid 1 (pmat2.fluid1)

1 In the **Model Builder** window, click **Fluid 1 (pmat2.fluid1)**.

2 In the **Settings** window for **Fluid**, locate the **Fluid Properties** section.

3 From the **Material** list, choose **Ice (mat2)**.

Solid 1 (pmat2.solid1)

1 In the **Model Builder** window, click **Solid 1 (pmat2.solid1)**.

2 In the **Settings** window for **Solid**, locate the **Solid Properties** section.

3 From the **Material** list, choose **Product (Skim Milk) (mat3)**.

4 In the θ_s text field, type `1-por_p`.

GLOBAL DEFINITIONS

Vapor (mat1)

1 In the **Model Builder** window, under **Global Definitions>Materials** click **Vapor (mat1)**.

2 In the **Settings** window for **Material**, locate the **Material Contents** section.

3 In the table, enter the following settings:

Property	Variable	Value	Unit	Property group
Mean molar mass	Mn	M_v	kg/mol	Basic
Dynamic viscosity	mu	mu_v	Pa·s	Basic
Thermal conductivity	k_iso ; kii = k_iso, kij = 0	k_v	W/(m·K)	Basic
Heat capacity at constant pressure	Cp	Cp_v	J/(kg·K)	Basic

Ice (mat2)

1 In the **Model Builder** window, click **Ice (mat2)**.
2 In the **Settings** window for **Material**, locate the **Material Contents** section.
3 In the table, enter the following settings:

Property	**Variable**	**Value**	**Unit**	**Property group**
Thermal conductivity	k_iso ; kii = k_iso, kij = 0	`k_ice`	W/(m·K)	Basic
Density	rho	`rho_ice`	kg/m³	Basic
Heat capacity at constant pressure	Cp	`Cp_ice`	J/(kg·K)	Basic

Product (Skim Milk) (mat3)

1 In the **Model Builder** window, click **Product (Skim Milk) (mat3)**.
2 In the **Settings** window for **Material**, locate the **Material Contents** section.
3 In the table, enter the following settings:

Property	**Variable**	**Value**	**Unit**	**Property group**
Thermal conductivity	k_iso ; kii = k_iso, kij = 0	`k_p`	W/(m·K)	Basic
Heat capacity at constant pressure	Cp	`Cp_p`	J/(kg·K)	Basic
Density	rho	`rho_p`	kg/m³	Basic

MESH 1

Set up a uniform mesh in the vertical direction in order to ease the mesh displacement and to avoid remeshing during the computation.

1 In the **Model Builder** window, under **Component 1 (comp1)** click **Mesh 1**.
2 In the **Settings** window for **Mesh**, locate the **Sequence Type** section.
3 From the list, choose **User-controlled mesh**.

Size

1 In the **Model Builder** window, under **Component 1 (comp1)>Mesh 1** click **Size**.
2 In the **Settings** window for **Size**, locate the **Element Size** section.
3 From the **Predefined** list, choose **Finer**.

Free Tetrahedral 1

In the **Model Builder** window, right-click **Free Tetrahedral 1** and choose **Disable**.

Free Triangular 1

1. In the **Mesh** toolbar, click **Boundary** and choose **Free Triangular**.
2. Select Boundaries 7, 14, and 21 only.

Size 1

1. Right-click **Free Triangular 1** and choose **Size**.
2. In the **Settings** window for **Size**, locate the **Element Size** section.
3. From the **Predefined** list, choose **Extra fine**.
4. Locate the **Geometric Entity Selection** section. From the **Geometric entity level** list, choose **Edge**.
5. Select Edges 8 and 29 only.

Boundary Layers 1

1. In the **Mesh** toolbar, click **Boundary Layers**.
2. In the **Settings** window for **Boundary Layers**, locate the **Geometric Entity Selection** section.
3. From the **Geometric entity level** list, choose **Boundary**.
4. Select Boundaries 7 and 21 only.

Boundary Layer Properties

1. In the **Model Builder** window, click **Boundary Layer Properties**.
2. Select Edges 8 and 29 only.
3. In the **Settings** window for **Boundary Layer Properties**, locate the **Layers** section.
4. In the **Number of layers** text field, type 4.

Swept 1

1. In the **Mesh** toolbar, click **Swept**.
2. In the **Settings** window for **Swept**, click to expand the **Source Faces** section.
3. Select Boundaries 7, 14, and 21 only.
4. Click to expand the **Destination Faces** section. Select Boundaries 3, 10, and 17 only.

Distribution 1

1. Right-click **Swept 1** and choose **Distribution**.
2. In the **Settings** window for **Distribution**, locate the **Domain Selection** section.
3. Click **Clear Selection**.

4 Select Domains 2, 4, and 6 only.

5 Locate the **Distribution** section. In the **Number of elements** text field, type 16.

Distribution 2

1 In the **Model Builder** window, right-click **Swept 1** and choose **Distribution**.

2 In the **Settings** window for **Distribution**, locate the **Domain Selection** section.

3 Click **Clear Selection**.

4 Select Domains 1, 3, and 5 only.

5 Locate the **Distribution** section. In the **Number of elements** text field, type 8.

STUDY 1

The necessary time to dry the product is unknown. An initial guess of less than 24 hours is made.

Step 1: Time Dependent

1 In the **Model Builder** window, under **Study 1** click **Step 1: 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(0,0.1,0.5) range(0.5,0.5,24)`.

Solution 1 (sol1)

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

2 In the **Model Builder** window, expand the **Solution 1 (sol1)** node.

Add a stop condition to stop the simulation when the interface is close to the bottom of the vial.

3 In the **Model Builder** window, expand the **Study 1>Solver Configurations> Solution 1 (sol1)>Time-Dependent Solver 1** node.

4 Right-click **Study 1>Solver Configurations>Solution 1 (sol1)>Time-Dependent Solver 1** and choose **Stop Condition**.

5 In the **Settings** window for **Stop Condition**, locate the **Stop Expressions** section.

6 Click **Add**.

7 In the table, enter the following settings:

Stop expression	Stop if	Active	Description
`comp1.minop1(z)/Z0-0.03`	Negative (<0)	√	`Interface close to the vial bottom`

8 Locate the **Output at Stop** section. From the **Add solution** list, choose **Step after stop**.

9 Clear the **Add warning** check box.

Switch from segregated solver to a fully coupled one to ease the convergence.

10 Right-click **Time-Dependent Solver 1** and choose **Fully Coupled**.

11 In the **Settings** window for **Fully Coupled**, click to expand the **Method and Termination** section.

12 In the **Damping factor** text field, type `0.9`.

13 From the **Jacobian update** list, choose **Once per time step**.

14 From the **Stabilization and acceleration** list, choose **Anderson acceleration**.

15 In the **Study** toolbar, click **= Compute**.

RESULTS

Only half of the vial geometry has been built for the calculations. In the next steps, mirror plots are defined to visualize the entire geometry in postprocessing plots.

Mirror 3D 1

1 In the **Model Builder** window, expand the **Results>Datasets** node.

2 Right-click **Results>Datasets** and choose **More 3D Datasets>Mirror 3D**.

3 In the **Settings** window for **Mirror 3D**, locate the **Plane Data** section.

4 From the **Plane** list, choose **XZ-planes**.

Cut Point 3D 1

1 In the **Results** toolbar, click **Cut Point 3D**.

2 In the **Settings** window for **Cut Point 3D**, locate the **Point Data** section.

3 In the **X** text field, type `0`.

4 In the **Y** text field, type `0`.

5 In the **Z** text field, type `range(Z0,-Z0/6,0)`.

6 From the **Snapping** list, choose **Snap to closest boundary**.

To create the plots shown in Figure 3 and Figure 4, reproduce the following steps.

Mesh

1 In the **Results** toolbar, click **3D Plot Group**.

2 In the **Settings** window for **3D Plot Group**, type `Mesh` in the **Label** text field.

Mesh 1

1 Right-click **Mesh** and choose **Mesh**.

2 In the **Settings** window for **Mesh**, locate the **Coloring and Style** section.

3 From the **Color table** list, choose **AuroraBorealis**.

Selection 1

1 Right-click **Mesh 1** and choose **Selection**.

2 Select Boundaries 1–3, 6, 9, 10, 13, 17, 20, 22, and 24 only.

3 In the **Mesh** toolbar, click **Plot**.

4 Click the **Zoom Extents** button in the **Graphics** toolbar.

Initial Mesh

1 In the **Model Builder** window, right-click **Mesh** and choose **Duplicate**.

2 In the **Settings** window for **3D Plot Group**, type `Initial Mesh` in the **Label** text field.

3 Locate the **Data** section. From the **Time (h)** list, choose **0**.

4 In the **Initial Mesh** toolbar, click **Plot**.

5 Click the **Zoom Extents** button in the **Graphics** toolbar.

Sublimation Interface

To create the plot shown in Figure 2, follow these steps.

1 In the **Home** toolbar, click **Add Plot Group** and choose **3D Plot Group**.

2 In the **Settings** window for **3D Plot Group**, type `Sublimation Interface` in the **Label** text field.

3 Locate the **Data** section. From the **Dataset** list, choose **Mirror 3D 1**.

4 Click to expand the **Title** section. From the **Title type** list, choose **Manual**.

5 In the **Title** text area, type `Left: Final interface position; right: temperature and total heat fluxes`.

Slice 1

1 Right-click **Sublimation Interface** and choose **Slice**.

2 In the **Settings** window for **Slice**, locate the **Expression** section.

3 In the **Expression** text field, type `T`.

4 Locate the **Plane Data** section. In the **Planes** text field, type `1`.

5 Locate the **Coloring and Style** section. From the **Color table** list, choose **HeatCameraLight**.

Isosurface 1

1 In the **Model Builder** window, right-click **Sublimation Interface** and choose **Isosurface**.

2 In the **Settings** window for **Isosurface**, locate the **Expression** section.

3 In the **Expression** text field, type Zg.

4 Locate the **Levels** section. From the **Entry method** list, choose **Levels**.

5 In the **Levels** text field, type Z0-Zi.

6 Locate the **Coloring and Style** section. From the **Color table** list, choose **JupiterAuroraBorealis**.

7 Clear the **Color legend** check box.

8 In the **Sublimation Interface** toolbar, click **Plot**.

Streamline 1

1 Right-click **Sublimation Interface** and choose **Streamline**.

2 In the **Settings** window for **Streamline**, click **Replace Expression** in the upper-right corner of the **Expression** section. From the menu, choose **Component 1 (comp1)> Heat Transfer in Porous Media>Domain fluxes>ht.tfluxx,...,ht.tfluxz - Total heat flux (spatial and material frames)**.

3 Locate the **Streamline Positioning** section. From the **Positioning** list, choose **Magnitude controlled**.

4 Locate the **Coloring and Style** section. Find the **Line style** subsection. From the **Type** list, choose **Tube**.

Color Expression 1

1 Right-click **Streamline 1** and choose **Color Expression**.

2 In the **Settings** window for **Color Expression**, locate the **Expression** section.

3 In the **Expression** text field, type T.

4 Locate the **Coloring and Style** section. Clear the **Color legend** check box.

5 From the **Color table** list, choose **HeatCameraLight**.

Line 1

1 In the **Model Builder** window, right-click **Sublimation Interface** and choose **Line**.

2 In the **Settings** window for **Line**, locate the **Expression** section.

3 In the **Expression** text field, type 1.

4 Locate the **Coloring and Style** section. From the **Coloring** list, choose **Uniform**.

5 From the **Color** list, choose **Gray**.

6 In the **Sublimation Interface** toolbar, click **Plot**.

Volume 1

1 Right-click **Sublimation Interface** and choose **Volume**.

2 In the **Settings** window for **Volume**, locate the **Data** section.

3 From the **Dataset** list, choose **Study 1/Solution 1 (sol1)**.

4 Locate the **Expression** section. In the **Expression** text field, type `dom==1||dom==3||dom==5`.

5 Locate the **Coloring and Style** section. From the **Color table** list, choose **AuroraBorealis**.

6 Clear the **Color legend** check box.

Translation 1

1 Right-click **Volume 1** and choose **Translation**.

2 In the **Settings** window for **Translation**, locate the **Translation** section.

3 In the **x** text field, type `-2.5*R0`.

4 In the **y** text field, type `0`.

5 In the **z** text field, type `0`.

6 Clear the **Apply to dataset edges** check box.

7 In the **Sublimation Interface** toolbar, click **Plot**.

8 Click the **Zoom Extents** button in the **Graphics** toolbar.

Temperature history

Plot the temperature variation during the primary drying at seven locations equally spread along the vial.

1 In the **Home** toolbar, click **Add Plot Group** and choose **1D Plot Group**.

2 In the **Settings** window for **1D Plot Group**, type `Temperature history` in the **Label** text field.

3 Locate the **Data** section. From the **Dataset** list, choose **None**.

4 Click to expand the **Title** section. From the **Title type** list, choose **Manual**.

5 In the **Title** text area, type `Temperature history at seven different heights at the center of the vial`.

6 Locate the **Plot Settings** section. Select the **x-axis label** check box.

7 In the associated text field, type `Time (h)`.

8 Select the **y-axis label** check box.

9 In the associated text field, type `Temperature (K)`.

10 Locate the **Legend** section. From the **Position** list, choose **Upper left**.

Point Graph 1

1 Right-click **Temperature history** and choose **Point Graph**.

2 In the **Settings** window for **Point Graph**, locate the **Data** section.
3 From the **Dataset** list, choose **Cut Point 3D 1**.
4 Locate the **y-Axis Data** section. In the **Expression** text field, type `T`.
5 In the **Temperature history** toolbar, click **Plot**.
6 Click to expand the **Quality** section. Click to expand the **Coloring and Style** section. In the **Width** text field, type `2`.
7 Click to expand the **Legends** section. Select the **Show legends** check box.
8 From the **Legends** list, choose **Evaluated**.
9 In the **Legend** text field, type `z/Z0=eval(z/Z0)`.

Ice mass

Then, plot the ice mass decreasing during the primary drying.

1 In the **Home** toolbar, click **Add Plot Group** and choose **1D Plot Group**.
2 In the **Settings** window for **1D Plot Group**, type `Ice mass` in the **Label** text field.
3 Locate the **Title** section. From the **Title type** list, choose **Manual**.
4 In the **Title** text area, type `Ice mass relative to initial amount`.
5 Locate the **Plot Settings** section. Select the **x-axis label** check box.
6 In the associated text field, type `Time (h)`.
7 Select the **y-axis label** check box.
8 In the associated text field, type `(%)`.
9 Locate the **Legend** section. Clear the **Show legends** check box.

Global 1

1 Right-click **Ice mass** and choose **Global**.
2 In the **Settings** window for **Global**, locate the **y-Axis Data** section.
3 In the table, enter the following settings:

Expression	Unit	Description
comp1.intop1(1)*2/(pi*R0^2*(Z0-Zi))	1	

4 In the **Ice mass** toolbar, click **Plot**.
5 Click to expand the **Coloring and Style** section. In the **Width** text field, type `2`.

Initial Ice Mass

Finally, check the mass conservation: the difference between the initial and the final ice mass should be equal to the amount of vapor that has left the vial.

1 In the **Results** toolbar, click **Global Evaluation**.

2 In the **Settings** window for **Global Evaluation**, type `Initial Ice Mass` in the **Label** text field.

3 Locate the **Expressions** section. In the table, enter the following settings:

Expression	Unit	Description
comp1.intop1(rho_ice)*2*por_p	mg	Ice mass

4 Locate the **Data Series Operation** section. From the **Transformation** list, choose **Maximum**.

5 Click **Evaluate**.

Final Ice Mass

1 Right-click **Initial Ice Mass** and choose **Duplicate**.

2 In the **Settings** window for **Global Evaluation**, type `Final Ice Mass` in the **Label** text field.

3 Locate the **Data Series Operation** section. From the **Transformation** list, choose **Minimum**.

4 Click **Evaluate**.

Vapor Flux

1 In the **Results** toolbar, click **More Derived Values** and choose **Integration>Surface Integration**.

2 In the **Settings** window for **Surface Integration**, type `Vapor Flux` in the **Label** text field.

3 Select Boundaries 7, 14, and 21 only.

4 Locate the **Expressions** section. In the table, enter the following settings:

Expression	Unit	Description
dl.bndflux*2	mg/h	Vapor flux leaving the vial

5 Locate the **Integration Settings** section. Select the **Integration order** check box.

6 Locate the **Data Series Operation** section. From the **Transformation** list, choose **Integral**.

7 Click **Evaluate**.