European Symposium on Computer Aided Process Engineering – 15
L. Puigjaner and A. Espuña (Editors)

Short-Path Evaporation for Chemical Product Modelling, Analysis and Design

Mauricio Sales-Cruz and Rafiqul Gani *
CAPEC, Department of Chemical Engineering
Technical University of Denmark, DK-2800 Lyngby, Denmark

Abstract

The last stage in the design process for a chemical product is its manufacture, where the purification has an important role. Short-path evaporation is a safe method suitable for separation and purification of thermally unstable materials. In this work the process modelling and a systematic strategy of solution is presented for a short-path evaporator to obtain the desired product specification. The model describes the influence of the evaporator design and the operational conditions for obtaining an efficient separation and improving yield of the desired chemical product. The model performance is illustrated with a case study (pharmaceutical product) using a computer-aided modelling framework for the model analysis and solution.

Keywords: computer-aided modelling, product and process design, short-path evaporation.

1. Introduction

Conventional distillation is one of the oldest methods to separate liquid or molten substances. However, it is not recommended for substances that can be degraded under distillation temperatures, such as vitamins, insecticides, drugs and flavours/fragrances. The short-path distillation is a separation technique used as an alternative in various processes of the chemical, pharmaceutical, fragrance and food industry. It is a safe method suitable for separation and purification of thermally unstable materials, through a small distance between the evaporator and the condenser, and characterized by low temperatures, short residence times of the distilled liquid on the thermally exposed surface and sufficiently low pressure in the distillation gap (space between evaporator and condenser). Therefore, the modelling, design and analysis of short-path evaporation (or molecular distillation) are important elements in many chemical product engineering problems.

Information about the film surface temperature on the condensation surface is important to determine yield and purity of the distilled product, as well as to define the evaporator design (i.e., the feed position and the evaporator geometry). However, direct measurement on the temperature profiles in the film of the condensate is extremely difficult, so that a key issue is the building of an appropriate model that can describe the

* Author to whom correspondence should be addressed: rag@kt.dtu.dk

separation process as a function of the (concentration, temperature and velocity) film profiles, and which can be useful for operation analysis and process design.
Thus, the aim of this work is to present a systematic strategy for process modelling with particular emphasis in analysis and design of short path distillation, to establish the operational conditions for obtaining an efficient separation, improving yield and purity of the desired chemical product. As starting point, a mathematical model is developed based on mass, heat and momentum balances for multi-component mixtures, resulting in a set of PDAEs (Partial Differential Algebraic Equations). The model analysis, model solution and model verification are done using a computer aided modelling system called *ICAS-MoT*. Through the model analysis, it is possible to understand how the feed concentration, the feed temperature, the heating surface, the system pressure and evaporator dimensions (length and gap) affect the film temperature profile, the film thickness, the evaporation rate and the evaporation yield. As a case study, the purification of an active pharmaceutical ingredient from a six-component mixture is presented.

2. Short-path evaporator

2.1 Process description

The short-path separator consists of a cylindrical body surrounded by a cylinder, one of them acts as evaporation surface and the other as condensation surface (see Figure 1a). The liquid material to be distilled is fed in the evaporation wall. The evaporation and condensation surfaces are kept at constant temperatures T_{w1} and T_{w2}, respectively. Due to the low pressure inside the separator, a falling film (without boiling) is formed and the concentration and temperature profiles (see Figure 1b) of the most volatile compounds decrease in the axial and radial directions.

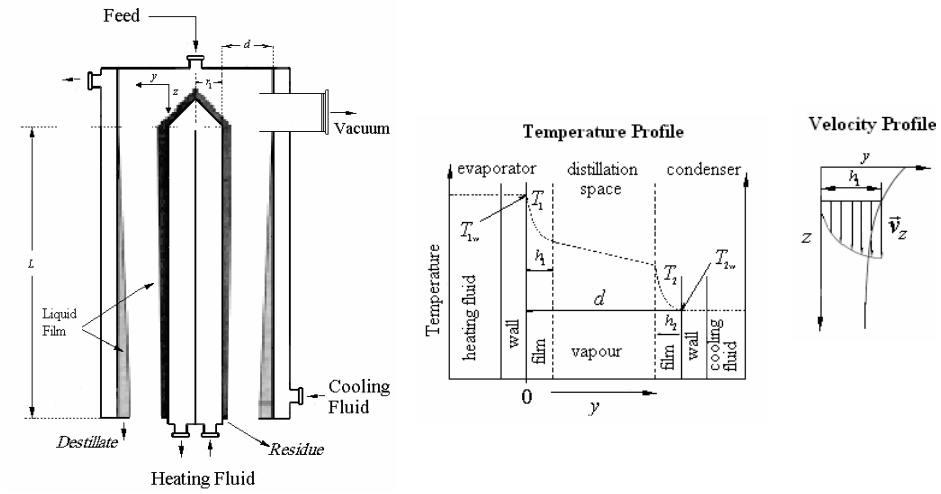

Figure 1. (a) The short-path evaporator, (b) temperature profile and (c) velocity profile.

2.2 Mathematical Model

Let consider the liquid films on the evaporation and condensation walls are much thinner than the corresponding cylinder diameters then rectangular coordinates can be used. The mathematical model for the short-path evaporator under steady state comes from momentum, energy and mass balances for both evaporation and condensation films as follows (Lutišan et al, 2002).

2.2.1 Momentum balance

In most cases of short-path evaporation, the evaporating liquid is highly viscous and hence the Reynolds numbers are small. The Navier-Stokes equation (at steady state) for laminar regimen describes the velocity profile (see Figure 1c) of falling film

$$v(z)\frac{\partial^2 v(y,z)}{\partial y^2} = -g \tag{1}$$

This has the following boundary conditions

$$v(0,z) = 0\,, \qquad v(y,z) = v_{\max} \tag{2}$$

Where v is the velocity, g is the gravity constant, and y and z is the radial and axial coordinates respectively.

2.2.2 Energy balance

The temperature (T) profile in the falling film is given by the equation

$$v(y,z)\frac{\partial T(y,z)}{\partial z} = \frac{\lambda}{\rho Cp}\left[\frac{\partial^2 T(y,z)}{\partial y^2} + \frac{\partial^2 T(y,z)}{\partial z^2}\right] \tag{3}$$

With boundary conditions

$$T(y,0) = T_F\,, \qquad T(0,z) = T_{w1}\,, \qquad \lambda\frac{\partial T(y,z)}{\partial y}\bigg|_{y=h_1} = \Delta H^{vap}\cdot k \tag{4}$$

Where λ, ρ, C_p, ΔH^{vap} are the thermal conductivity, density, thermal capacity and heat of evaporation of the multicomponent mixture respectively.

2.2.3 Mass balances

The composition (C_i) profiles for each component are calculated from the diffusion equation

$$v(y,z)\frac{\partial C_i(y,z)}{\partial z} = D_i\left[\frac{\partial^2 C_i(y,z)}{\partial y^2} + \frac{\partial^2 C_i(y,z)}{\partial z^2}\right], \qquad i = 1,\ldots,N \tag{5}$$

Where D_i is the (constant) diffusion coefficient for the i-th component. The boundary conditions for Eq. (5) are

$$C_i(y,0) = C_{i,o}\,, \qquad \frac{\partial C_i(0,z)}{\partial y} = 0\,, \qquad D_i\frac{\partial C_i(y,z)}{\partial y}\bigg|_{y=h_1} = I_i(z) \tag{6}$$

2.2.3 Rate of evaporation

The flow rate I_i for each component is describe by the continuity equation

$$\frac{\partial I_i(z)}{\partial z} = -2\pi \cdot R \cdot k_i, \qquad i = 1,\ldots,N \tag{7}$$

Where the effective rate of evaporation (k_i) is calculated through a modified Langmuir-Knudsen equation (Kawala and Stephan, 1989)

$$k_i = \frac{p_i^{vap} T_s(z)}{\sqrt{2\pi R_g M_i T_s(z)}} \left(\frac{P}{P_{ref}}\right) \left\{1 - (1-F)\left[1 - e^{h/(\kappa\beta)}\right]^n\right\}, \qquad i = 1,\ldots,N \tag{8}$$

It contains a factor (P/P_{ref}) for correcting the vacuum pressure, as well as a correction that takes into account the anisotropic properties of the vapour, where β is the mean path of vapour molecule, h is the distillation gap, n is the number of intermolecular collision, F is the surface ratio and κ is the anisotropy of the vapour phase given by

$$F = \frac{A_k}{A_k + A_V}, \qquad \log\kappa = 0.2F + 1.38(f + 0.1)^4 \tag{9}$$

A_k and A_v are the condensation and evaporation areas, respectively. The effective rate of evaporation [Eq. (7)] also depends on some mixture properties (the vapour pressure p_i^{vap} and molecular weight M_i of each compound) as well as on design parameters (the radius of the evaporator inside cylinder R and the surface temperature T_s).

2.2.4 Thickness film

Finally, an important variable of interest is the thickness film (h_1) along the evaporator height that is calculated as follows (Kawala and Stephan, 1989)

$$h_1(z) = \sqrt[3]{\frac{3\nu}{2\pi \cdot R \cdot g \cdot c} I(z)}, \qquad I(z) = \sum_{i=1}^{N} I_i(z), \qquad c = \sum_{i=1}^{N} C_i(z) \tag{10}$$

where ν is the kinematic viscosity of the multicomponent mixture.

3. Strategy of Solution

3.1 Computer-aided modelling framework

It is useful to take advantage of Computer-Aided Modelling Systems (CAMS) and tools for integrated process analysis, to reduce the time to market and investment costs, and to achieve a successful integrated product and process design through in a fast, reliable and efficient way. In particular, *ICAS-MoT* is used in this work that is an integrated modelling environment to build, analyse, manipulate, solve and visualise mathematical models. An important feature of *ICAS-MoT* is that the model developer does not need to write any programming codes to enter the model equations. Models are entered (imported) as text-files or XML-files, which are then internally translated. In model analysis step *ICAS-MoT* orders the equations into lower triangular form (if feasible), generates the incidence matrix, verifies the degrees of freedom, and checks for singularity. After this interactive model analysis, the appropriate solver for the model equations is selected together with a corresponding strategy of solution. As solver options, *ICAS-MoT* provides several solvers for AEs (algebraic equations), DAEs (differential algebraic equations) and numerical optimisation methods. More details can be found in Sales-Cruz and Gani (2003).

3.2 Model discretisation

In order to solve the evaporator model that involves PDAEs [Eqs. (1)-(10)], method of lines using centered finite difference is applied considering an *M*-point discretisation scheme for the radial coordinate "*y*" as shown in Figure 2 (Cvengroš et al, 2000). Good performances can be achieved with a minimum value of $M = 10$. Afterwards, the resulting DAE system is solved through *ICAS-MoT* using the Backward Difference Formula method. One particular advantage of the integrated *ICAS* is that all physicochemical properties are recalled from modules that can be easily integrated to the short-path evaporator model. Therefore a large number of chemical products can be studied very fast and with minimum effort.

4. Model evaluation

4.1 Case study: a pharmaceutical mixture

Consider the process production of a drug where after the reaction stage, the active molecule of the active pharmaceutical ingredient (API) is formed. Then the resulting liquid mixture composed of six heat sensitive compounds (that are called *A*, *B*, *C*, *D*, *E* and *F* for confidentially reasons) needs to be purified. *A* is the lightest and more volatile compound and *F* is the one with the highest boiling point. The role of the short-path evaporator is to separate the active molecule (form mainly by *C*, *D* and *E*) together with the inert component *F* from the feed multicomponent mixture coming from the reactor.

4.2 Model analysis

The classification of variables and model equations was done through *ICAS-MoT* as follows: (a) there are 128 equations sorted as 17 ODEs, 1 implicit and 110 explicit AEs, and (b) 306 variables sorted as 17 dependent, 1 unknown, 4 known, 174 parameters, and 110 explicit. Afterwards, the incidence matrix, degrees of freedom and non-singularity were verified to ensure that the problem was not ill-posed before going to the solution step.

4.3 Simulation results

The feed flows as well as experimental and calculated flows of the distillate and residual are reported in Table 1, where it can be seen that the model is able to predict quite satisfactory the exit flow rates.

Table 1. Experimental and calculated flow rates of distillate and residual.

Compound	Experimental flows (*kmole*/*h*)			Calculated flows (*kmole*/*h*)	
	Feed I_0	Residual I_R	Distillate I_D	Residual I_R	Distillate I_D
A	6.11×10^{-5}	0.0	6.11×10^{-5}	0.0	6.11×10^{-5}
B	1.22×10^{-5}	0.0	1.22×10^{-5}	0.0	1.22×10^{-5}
C	4.72×10^{-2}	4.46×10^{-2}	2.61×10^{-3}	4.46×10^{-2}	2.61×10^{-3}
D	1.90×10^{-4}	1.88×10^{-4}	2.09×10^{-6}	1.87×10^{-4}	3.07×10^{-6}
E	2.17×10^{-3}	2.16×10^{-3}	6.79×10^{-6}	2.14×10^{-3}	2.46×10^{-5}
F	6.66×10^{-4}	6.66×10^{-4}	0.0	6.66×10^{-4}	0.0

As the chemical product (*C*, *D*, *E* and *F*) is obtained as the residual in the short-path evaporator, then the surface velocity, temperature, thickness and some flow rates are shown for the evaporating film in Figures 2. The rise in temperature (Figure 2a) is related to the evaporation of compounds *A* and *B* as can be seen in Figure 2b. In fact, *A* and *B* are the main compounds that are evaporated from the mixture and are obtained as distillate product. The surface velocity (Figure 2a) has a rapid increase at the first part of the evaporator axial position achieving a maximum point, and then decreasing slightly due to the decrease of the total evaporation rate. Figure 2b also shows the dependence of the film thickness throughout evaporator cylinder axis. Film thickness decreases with the increasing surface temperature due to evaporation. Both film surface temperature and film thickness turn asymptotic as soon as a constant film thickness has formed.

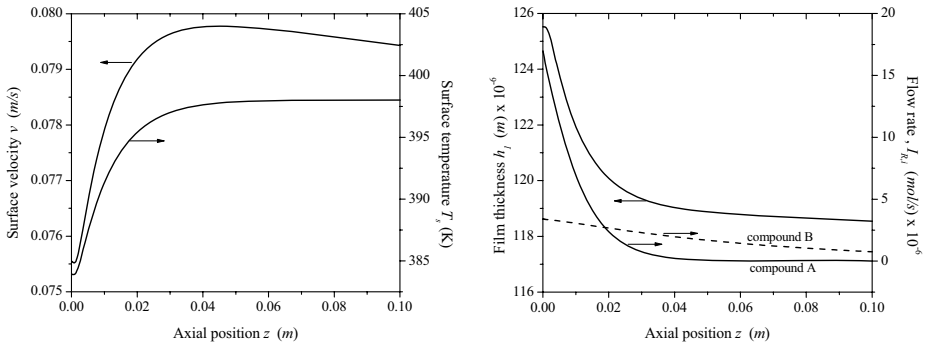

Figure 2. (a) Surface velocity and temperature, (b) Film thickness and flow rate for compound A.

5. Conclusions

A short-path evaporator model has been presented for the purification of multicomponent mixtures. The modelling methodology presented illustrates how the design and analysis aspects of a short-path evaporator are related to the purity and stability of the chemical product and the corresponding conditions of operation. The process flexibility allows designing the separator in such way that the mixture to be separated can be fed either in the cylindrical body or in the outside cylinder wall, depending on whether the chemical product need to be recovered as a distilled product or concentrated as the heavy residue. On the other hand, the results also highlight the importance of a general-purpose and easy to use modelling toolbox for computer-aided design and analysis of complex process operations.

References

Cvengroš J., J. Lutišan and M. Micova, 2000, Feed temperature influence on the efficiency of a molecular evaporator, Chemical Engineering Journal, 78, 61–67.

Lutišan J., J. Cvengroš and M. Micov, 2002, Heat and mass transfer in the evaporating film of a molecular evaporator, Chemical Engineering Journal, 85, 225–234.

Kawala Z. and K. Stephan, 1989, Evaporation Rate and Separation Factor of Molecular Distillation in a Falling Film Apparatus, Chem. Eng. Technology, 12, 406-413.

Sales-Cruz, M. and R. Gani, 2003, Computer-Aided Chemical Engineering, vol. 16: Dynamic Model Development, Eds. S.P. Asprey and S. Macchietto, Elsevier, Amsterdam.