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Non-isothermal Convective Drying of Solids Wetted with Multicomponent Liquid Mixtures

The non-isothermal convective drying of an impermeable solid substrate covered with multicomponent solvent mixtures was studied. The aim was to simulate the nonsteady-state evaporation process under the control of both the gas side heat and mass transfer, and in the presence of diffusional resistances in the liquid layer. Diffusional interactions in both phases were accounted for by applying the generalized Fick's formulation of diffusion with a temperature and composition dependent matrix of multicomponent diffusivities. In the experimental part, a teflon substrate covered with the ternary mixture ethanol-methylethylketone-toluene was dried using air flowing parallel to the evaporation surface as drying agent. Good agreement was obtained between experiments and simulations. Calculations show that liquid composition, liquid temperature, gas composition and diffusional interactions have a significant influence on the instantaneous selectivity while the effect of gas temperature and gas velocity is less important. Selectivity can be controlled by using a gas preloaded with an appropriate composition.

INTRODUCTION

In the convective drying of solids, the rate of removal of vapors from the interface and their subsequent transport into the gas phase (external transfer) have a decisive influence on the appearance of internal resistances and on the extent to which these mechanisms are important. If the solid contains a single solvent, the only condition required to obtain a gas-phase-controlled process is that the solid be saturated. Under such circumstances, except for a short initial period required to bring the phases into thermal equilibrium, the process is isothermal and is characterized by a constant drying rate. If the solid contains a mixture of solvents, even a saturated one will, in general, evaporate non-isothermally. In addition, resistances in the liquid phase may appear during an intensive drying regime.

Multicomponent drying may also involve special interaction between the diffusing components that are not present in the drying of a single solvent.

Convective drying of solids containing binary mixtures has been extensively investigated by Turner and Schlunder [1,2], Gamayunov et al. [3], Schwarzbach and Schlunder [4]. Studies on ternary mixtures containing a non-volatile component have been performed by Reide and Schlunder [5]. In these papers, the selectivity of the process has been studied under isothermal conditions. Mixtures of three components, all of them volatile components, have been studied by Labutin et al. [6] and Migunov and Sadykov [7]. Interactions between diffusing vapors in the gas phase have not been taken into account in any of these studies.

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GAS-PHASE-CONTROLLED EVAPORATION

The convective evaporation from a saturated solid surface of a liquid mixture composed of n components is represented in Fig.1. The total number of component in the gas phase is n .

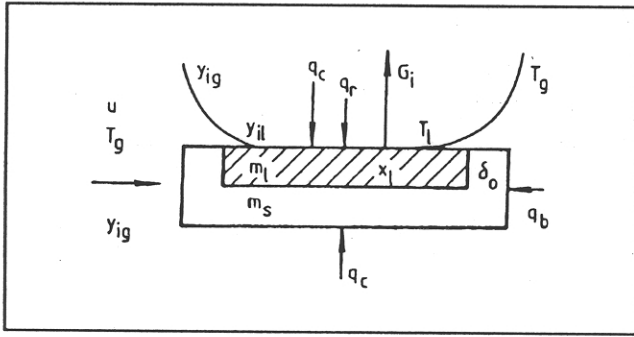


Fig.1. Schematic diagram of the evaporation of a liquid mixture by convection

Simplifying assumptions:

- 1) unchanged gas temperature and composition
- 2) no resistance to heat transfer in the liquid and the solid, and negligible resistance to mass transfer in the liquid film
- 3) equilibrium is reached instantaneously at every point in the interphase
- 4) negligible solubility of the non-condensing gas in the liquid mixture.

Mass balance

$$\frac{d(N)_1}{dt} = -A_d(G) \quad (1)$$

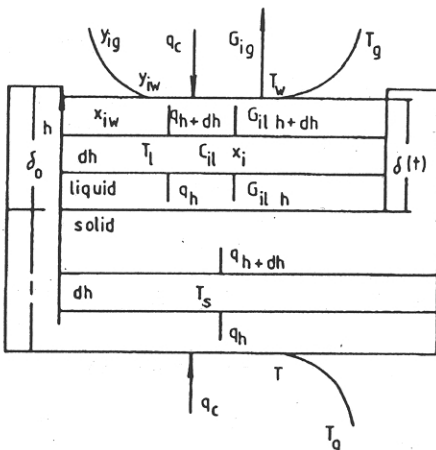
Energy balance

$$\frac{dT_1}{dt} = \frac{A_c q_c + A_r q_r + A_b q_b - A_d \sum_{i=1}^{n_c} G_i \{H_{ig} - H_{il}\}}{\sum_{i=1}^{n_c} N_{il} M_i c_{il} + m_s c_s} \quad (2)$$

The coupling between the phases is provided by the equilibrium assumption

$$y_{il} = x_i \gamma_i \{T_l(x)\} p_{il} \{T_l\} / P_i \quad i = 1, \dots, n_c \quad (3)$$

Fig.2. Schematic diagram of liquid-phase-controlled evaporation of a liquid mixture by convection



where $(N)_1$ is the vector of number of moles in the liquid mixture, (G) is the vector of molar fluxes and A is an area. The subscripts s, l, g denote solid, liquid and gas phases while the subscripts c, r, b and d denote convection, radiation, conduction and wetted, respectively. M is the molecular weight, H is molar enthalpy, c is the heat capacity, p is the vapour pressure of pure components and γ is the activity coefficient.

DIFFUSIONAL RESISTANCES IN LIQUID PHASE

Referred to Fig.2, the following dimensionless equations govern the evaporation process:

Single component mass balances

$$\frac{\partial(x)}{\partial\tau} = \frac{1}{C\delta^*} \frac{\partial\{C[D^*] \frac{\partial(x)}{\partial\xi}\}}{\partial\xi} - \frac{v\Xi}{C\delta^*} \frac{\partial(x)}{\partial\xi} \quad (4)$$

Total mass balance

$$\frac{\partial\Xi}{\partial\xi} + \frac{\delta^*}{v} \frac{\partial C}{\partial\tau} + \frac{C}{v} \frac{d\delta^*}{d\tau} = 0 \quad (5)$$

Energy balance in the solid

$$\frac{\partial\Theta_s}{\partial\tau_s} = \frac{\partial^2\Theta_s}{\partial\xi_s^2} \quad (6)$$

Energy balance in the liquid

$$\frac{\partial\Theta_l}{\partial\tau} = \frac{a_{l0}}{D_{l0}} \frac{1}{CL\delta^*} \frac{\partial\{\Lambda \frac{\partial\Theta_l}{\partial\xi}\}}{\partial\xi} - \frac{v\Xi}{C\delta^*} \frac{\partial\Theta_l}{\partial\xi} - \frac{\Theta_l}{L} \left\{ \frac{v\Xi}{C\delta^*} \frac{\partial L}{\partial\xi} + \frac{\partial L}{\partial\tau} \right\} \quad (7)$$

Interphase dimensionless velocity

$$\frac{d\delta^*}{d\tau} = f(\tau) \quad (8)$$

With the dimensionless variables related to initial values

$$\xi = \frac{h}{\delta(t)}, \quad \tau = \frac{tD_{l0}}{\delta_0^2}, \quad \xi_s = \frac{h}{l}, \quad \tau_s = \frac{t\lambda_s}{c_s \rho_s l^2}$$

$$C = \frac{C_l}{C_{l0}}, \quad \Xi = \frac{G_l}{G_{l0}}, \quad \Theta_1 = \frac{T_l}{T_{l0}}, \quad \Theta_s = \frac{T_s}{T_{s0}}$$

$$\delta^* = \frac{\delta(t)}{\delta_0}, \quad D_{ij}^* = \frac{D_{ij}}{D_{l0}}, \quad \Lambda = \frac{\lambda_l}{\lambda_{l0}}, \quad L = \frac{c_l}{c_{l0}} \quad (9)$$

$$a_{l0} = \frac{\lambda_{l0}}{\rho_{l0} c_{l0}}, \quad v = \frac{G_{l0} \delta_0}{D_{l0} C_{l0}} \quad (10)$$

where the heat capacity, the total concentration, the thermal conductivity λ , and the multicomponent diffusion coefficients D_{ij} in the liquid phase are

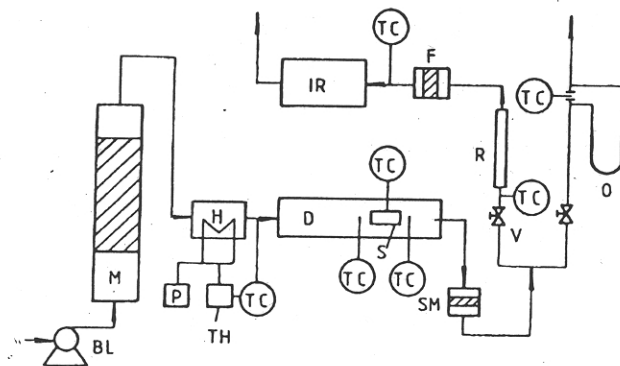


Fig.3. Experimental set-up
BL - blower, D - drying channel, F - filter, H - resistance, IR - spectrophotometer, M - molecular sieve, O - orifice plate flowmeter, P - power regulator, R - rotameter, S - sample, SM - static mixer, TC - thermocouples, TH - thyristor, V - valves

functions of the local temperature and composition while the physical properties of the solid are considered constant. The function f in Eq.(8) includes the effects of evaporation and density changes on the liquid film thickness.

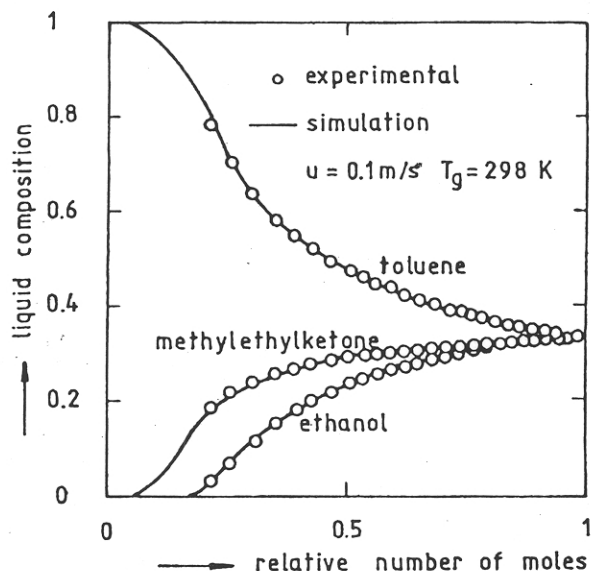
Beside proper initial and boundary conditions, expressions for mass and heat transfer rates are required in order to integrate Eqs.(1) and (2) or Eqs.(5) to (8).

MASS AND HEAT TRANSFER RATES

There are two main relationships for the calculation of evaporation rates considering diffusional interactions in multicomponent mass transfer literature. The first comes from the solution of the continuity equation using the generalized Fick's law to relate diffusion fluxes to concentration gradients. The resulting equations are uncoupled by linearization [8]. The second relationship is obtained from the exact solution of the Maxwell-Stefan equations [9]. The results of these methods may be summarized in the following general formula:

$$(G) = [\beta][k^*]\{(y\{t\})_w - (y)_g\} \quad (11)$$

Fig.4. Gas-phase-controlled evaporation into dry air. Comparison between experiments and simulations
 $m_l = 12.1$ g, $m_s = 145$ g, $\delta_0 = 3$ mm, $l = 0.03$ m



where $[k^*]$ is the matrix of finite mass transfer coefficients and $[\beta]$ permits calculation of the molar fluxes from diffusion fluxes. The manner in which these parameters are calculated in the different methods may be found in [9,10].

The convective heat flux is calculated from the heat transfer coefficients at finite mass transfer rates and temperature driving force

$$q_h = \alpha^*(T_g - T_w). \quad (12)$$

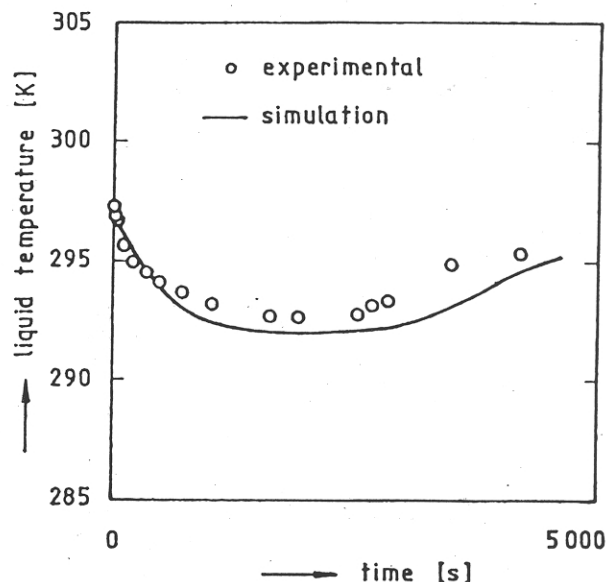
Radiation heat may be neglected because of the low temperatures involved. The possible contribution of conduction from the walls may be taken into account by assessing the heat transfer coefficient in the contact points between the walls and the solid.

SELECTIVITY

The variable which reflects the ability of each component to be removed preferentially from the mixture is called selectivity. By generalizing the definition of Schlunder [11]

$$s_i = \frac{G_i}{G_t} - x_i. \quad (13)$$

Fig.5. Gas-phase-controlled evaporation into dry air. Comparison between experiments and simulations
 $m_l = 12.1$ g, $m_s = 145$ g, $\delta_0 = 3$ mm, $l = 0.03$ m



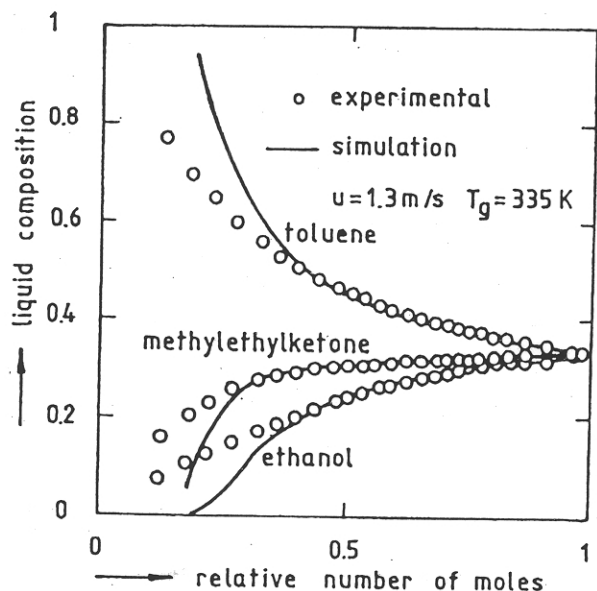


Fig. 6. Liquid-phase-controlled evaporation into dry air. Comparison between experiments and simulations $m_l = 11.9$ g, $m_s = 145$ g, $\delta_0 = 3$ mm, $l = 0.03$ m

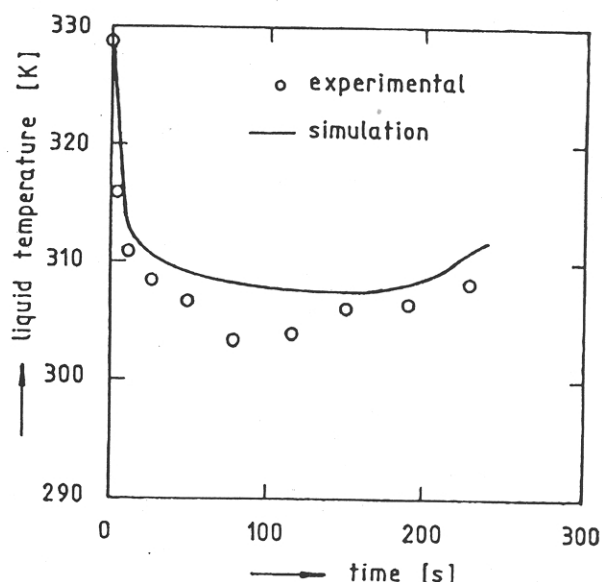


Fig. 7. Liquid-phase-controlled evaporation into dry air. Comparison between experiments and simulations $m_l = 11.9$ g, $m_s = 145$ g, $\delta_0 = 3$ mm, $l = 0.03$ m

Depending on the value of selectivity the following cases may arise:

- 1) $s_i > 0$: component i is removed preferentially
- 2) $s_i = 0$: the molar fraction of component i remains unchanged
- 3) $s_i < 0$: the molar fraction of component i in the liquid increases.

EXPERIMENTAL PART

In order to verify consistency of the model, evaporation experiments with ternary mixtures were performed at different drying rates. The experimental set-up is shown in Fig. 3.

Experimental evaluation

The composition of the gas stream was calculated from the nonlinear equation system provided by the measured absorbances and the calibration curves of the vapours. The evaporation fluxes and the composition of the liquid mixture can then be calculated at any time t_k according to

$$G_i(t_k) = \frac{V}{A_d} \{C_i(t_k) - C_{i0}\} \quad (14)$$

$$N_i(t_k) = N_{i0} - A_d \int_{t=0}^{t_k} G_i(t) dt \quad (15)$$

where V is the gas volume rate of flow and the subscript 0 denotes inlet conditions. The molar fractions x_i may be then calculated directly from the number of moles N_i .

The accuracy of the measurements was checked by determining the concentrations of different mixtures of known composition, obtaining an average deviation of 5% and a maximum deviation of 10% occurred in some mixtures of low concentration.

RESULTS

The results of evaporation experiments for the ternary mixture ethanol-methylethylketone-toluene at two different drying rates are compared with numerical simulations in Figs. (4) to (7). The evaporation fluxes were calculated according to the linearized theory. A modified algorithm of [10] with a first estimate of the corrected fluxes calculated according to an explicit expression of [12] was used. Equilibrium composition was computed by means of Wilson equation with constants from [13]. Mass and heat transfer coefficients at zero mass transfer rate were calculated assuming a flat geometry. For runs at low gas velocities, new constants were determined from experimental mass transfer coefficients. Binary diffusivities in the gas phase were calculated according to the method of Fuller et al. [14]. The liquid phase diffusion coefficient matrix was calculated using the method of Bandrowski and Kubaczka [15]. Other physical properties of pure components and mixtures were estimated using methods described in [16].

The ordinary set of differential Eqs. (1) and (2) was solved by using a fifth-order Runge-Kutta method with adaptive stepsize control of local truncation error. The liquid-phase-controlled case was solved by an implicit finite difference method [17].

The composition curves in Figs. 4 and 6 reveal that the evaporation is selective in both cases. The initial variation of liquid temperature shows that mass transfer is faster than heat transfer and a part of the energy required to maintain the evaporation rate is supplied by the liquid. Note that simulations and experiments are not really comparable in the last stage, where the evaporation surface is reduced by the appearance of dry patches and thermocouples can no longer to be immersed in the liquid.

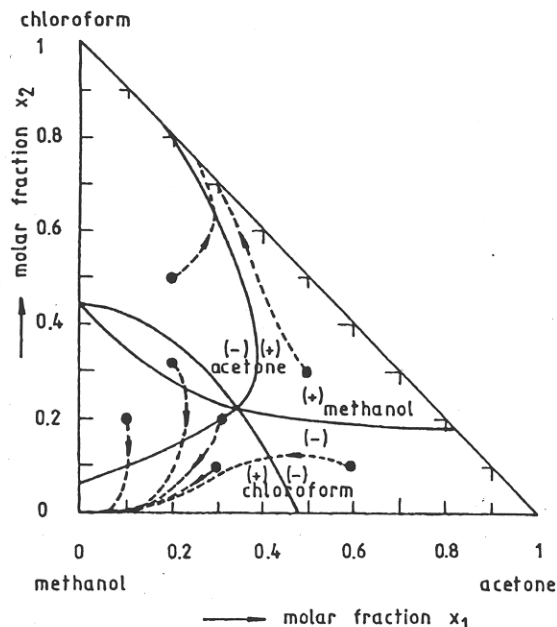


Fig.8. Zero selectivity curves as a function of liquid composition

INFLUENCE OF OPERATIONAL PARAMETERS ON THE SELECTIVITY

The dependence of selectivity on operational parameters may be written as follows:

$$s_i = s_i(x, T_w, (y)_g, T_g, u, P, \text{geometry}) \quad i = 1, \dots, n_c \quad (16)$$

Since the selectivities add up to zero, only $n_c - 1$ of Eqs.(16) are linearly independent.

Influence of liquid composition

Of particular interest is the occurrence of non-selective evaporation at liquid compositions different from a thermodynamic azeotrope. By generalizing the analysis of [2], the following conditions must be satisfied by the relative volatilities and effective mass transfer coefficients to obtain non-selective evaporation during gas-phase-controlled evaporation of a multicomponent liquid mixture into a pure inert gas

$$v_{ij} \frac{k_{i,eff}}{k_{j,eff}} = 1, \quad i = 1, 2, \dots, n_c, \quad j = i+1, \dots, n_c \quad (17)$$

or if the diffusional interactions are considered

$$x_i \sum_{j=1}^{n_c} \sum_{k=1}^{n_c} f_{jk} \gamma_k P_{kw} x_k - \sum_k f_{ik} \gamma_k P_{kw} x_k = 0 \quad (18)$$

$i = 1, 2, \dots, n_c$

As expected from the severe requirements expressed by Eqs.(17) or (18), the occurrence of dynamic azeotropes in higher order systems evaporating into a pure inert gas is rare. Of seven ternary systems examined, only the ternary system acetone-chloroform-methanol exhibits a dynamic azeotrope. This is shown in Fig.8 where the zero selectivity curves are represented by whole lines. The intersec-

Table 1. Thermodynamic and diffusional contributions to selectivity for the system acetone(1)-chloroform(2)-methanol(3) at the azeotropic conditions shown in Fig.8
 $u_g = 0.1 \text{ m/s}$, $T_g = 323 \text{ K}$, $T_l = 0.03 \text{ m}$

i	$k_{i,ef} 10^4$	$k_{i,ef}/k_{j,ef}$	$v_{i,j}$	Eq.(15)	x_i
		$j = 2,3$	$j = 2,3$	$j = 2,3$	
1	1.39	1.11 0.81	0.89 1.23	0.99 1.00	0.34
2	1.25	-0.73	-1.38	-1.00	0.22
3	1.72	—	—	—	0.44

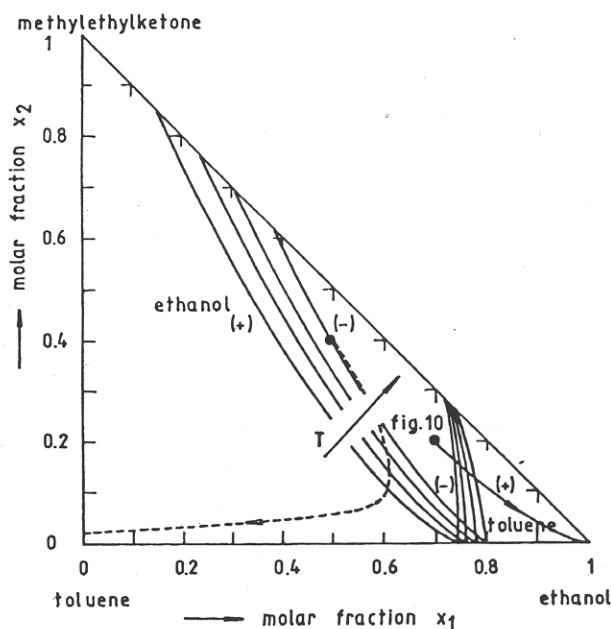
tion point represents a ternary dynamic azeotrope. The conditions prevailing there are shown in Table 1.

No restrictive assumption concerning the magnitude of the evaporation rates and the existence of liquid resistance underlines the calculation of the azeotropic composition and zero selectivity curves. The path followed by the different evaporation process plotted in dash lines, on the other hand, will be greatly influenced by any liquid resistances. In these simulations, liquid resistance has been neglected. The simulation of the process starting at the dynamic azeotrope reveals that the azeotrope is not stable. The condition for stability is

$$\left\{ \frac{dT_l}{dt} \right\}_{t=0} = 0 \quad (19)$$

which in an adiabatic process is equivalent to start the evaporation from a state of balanced energy fluxes. Under the conditions of the simulation the derivative is initially non zero, thus the dynamic azeotrope is not stable.

Fig.9. Influence of liquid temperature on selectivity
 $u = 0.1 \text{ m/s}$, $T_g = 323 \text{ K}$, $283 \leq T_l \leq 303 \text{ K}$, $l = 0.03 \text{ m}$



A special feature of the evaporation of mixtures containing more than two condensing components is the preferential evaporation of species that initially have negative selectivity. In such systems, the coexistence of several components with negative selectivity may force process evolution towards zones of positive selectivity for some of the components of initial unfavorable selectivity. See Figs. 9 and 10.

Influence of liquid temperature

The influence of liquid temperature on zero selectivity curves is of great importance, owing to its ability to displace the boundaries between zones of negative and positive selectivity during an evaporation process. The direction of the displacement of zero selectivity curves depends on the initial sign of (dT_l/dr) . In the case shown in Fig. 10, the derivative is negative. Temperature changes then lead to an enlargement of the positive selectivity zone for toluene, reinforcing its tendency to be removed preferentially.

Influence of gas composition

The particular effect of gas composition on dynamic azeotropes may be observed in the analogous expression to Eq. (17) for a preloaded gas in a binary system

$$\frac{f_{1eff}(\gamma_1 P_{1w} x_1 - P_r y_{1g}) x_2}{f_{2eff}(\gamma_2 P_{2w} x_2 - P_r y_{2g}) x_1} = 1. \quad (20)$$

On considering Eq. (20), it becomes apparent that the preloading of gas with condensing components may lead to the existence of not one but an infinite number of dynamic azeotropes, irrespective of the relationship between volatilities and diffusivities in the system. Furthermore, not only a pseudo-azeotrope but any desired selectivity may be achieved by choosing a gas mixture of appropriate composition. By rewriting Eq. (13)

$$\begin{aligned} (s_i + x_i) \sum_{j=1}^{n_c} \sum_{k=1}^{n_c} f_{jk} \{ \gamma_k P_{kw} x_k - P_r y_{kg} \} - \\ - \sum_{k=1}^{n_c} f_{ik} \{ \gamma_k P_{kw} x_k - P_r y_{kg} \} = 0 \end{aligned} \quad (21)$$

$i = 1, 2, \dots, n_c$

For a set of chosen initial selectivities s_i , the only unknowns are the molar fractions in the bulk y_{kg} . Since Eqs. (21) are linearly dependent a complementary relationship between molar fractions in the bulk is required to obtain a unique solution. If a gas preloading producing non-selective gas-phase-controlled evaporation is chosen, the dynamic azeotrope will be, in general, unstable unless Eq. (19) is satisfied. An initial state of balanced energy fluxes may be reached by choosing a gas temperature which satisfied simultaneously Eqs. (19) and (21) (Fig. 11).

A selective evaporation into a pure inert gas was transformed into a completely non-selective process by preloading the gas. When the initial state is not

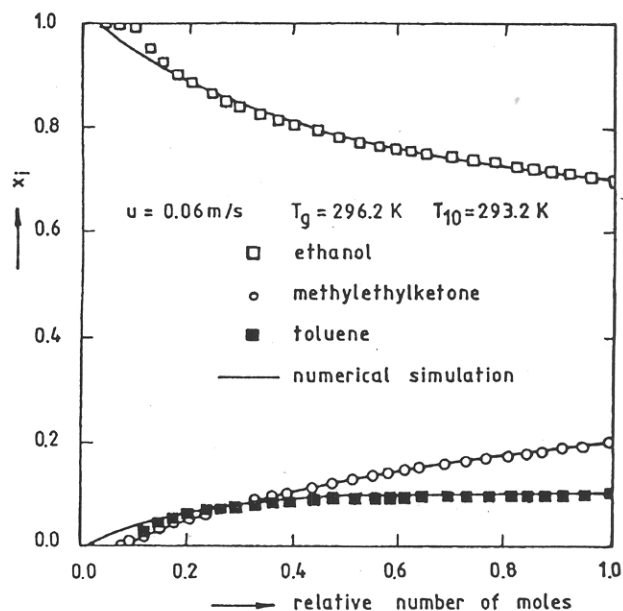


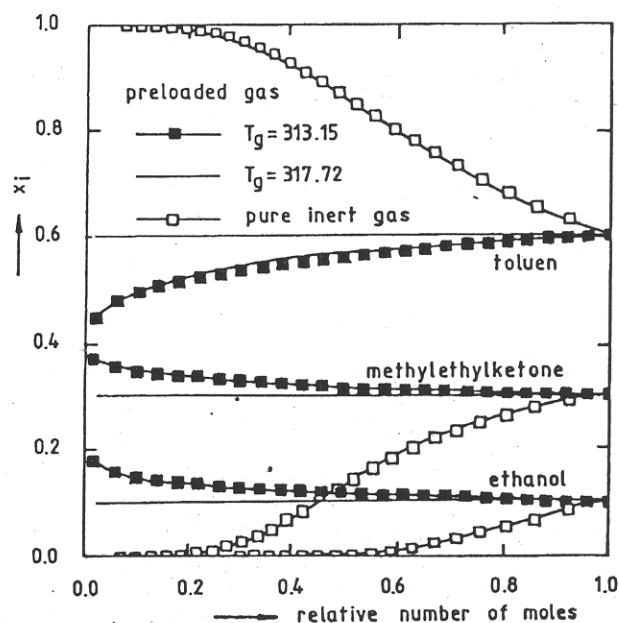
Fig.10. Gas-phase-controlled evaporation into dry air. Comparison between experiments and simulations $m_l = 4$ g, $m_s = 50$ g, $\delta_0 = 1$ mm, $l = 0.03$ m

thermally balanced, liquid temperature varies and non-selectivity is partially lost, although it never reaches the levels obtained with a pure inert gas. A constant gas preloading would not be enough to obtain a non-selective process if the evaporation is liquid-phase-controlled owing to the disturbing action of the temperature and composition gradients in the liquid.

Influence of diffusional interactions

Another implication of the use of preloaded gas is that diffusional interactions in the gas phase may

Fig.11. Influence of gas preloading on selectivity $u = 0.1$ m/s, $T_{l0} = 293$ K, $m_l = 12$ g, $m_s = 50$ g, $\delta_0 = 3$ mm, $l = 0.03$ m, $y_{10} = 0.024$, $y_{20} = 0.033$, $y_{30} = 0$. ethanol(1), methylethylketone(2), toluene(3)



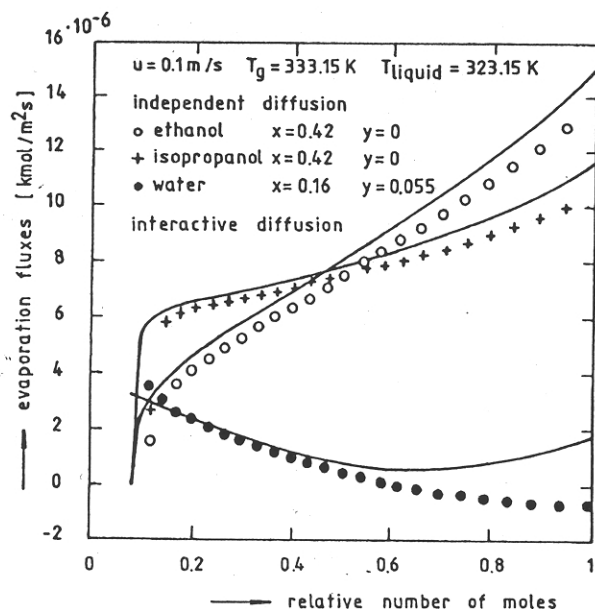


Fig.12. Influence of the diffusional interactions in gas phase on evaporation
 $m_l = 12$ g, $m_s = 50$ g, $\delta_0 = 3$ mm, $l = 0.03$ m

become important. A simulation of the system ethanol-isopropanol-water evaporating into nitrogen containing water vapour at a concentration slightly higher than at the interphase is shown in Fig. 12. Despite its negative driving force, water still evaporates due to the diffusion of the other species. Clearly, the effects of the driving forces of ethanol and isopropanol counteract the influence of the negative gradient of water resulting in evaporation.

CONCLUSIONS

The drying of solids wetted with solvent mixtures is almost always non-isothermal. One exception is represented by the case where the initial conditions correspond to a state of balanced energy fluxes and a dynamic azeotrope.

The occurrence of dynamic azeotropes during evaporation of ternary mixtures into pure inert gas is a rare event. Dynamic azeotropes in a binary system, on other hand, are common. The number of conditions that must be satisfied in order to obtain a dynamic azeotrope increases with the number of solvents. Such severe requirements mean that dynamic azeotropes are even scarcer in higher-order systems.

In systems containing more than two condensing components, an initial negative selectivity for some species may be displaced to positive zones owing to the effect of other component selectivities during the process, resulting in preferential evaporation. This situation is not possible in a binary system.

Gas composition, liquid temperature and composition have the greatest influence on instantaneous selectivity. Although gas temperature and gas velocity exert the least influence on instantaneous selectivity, their great effects on process dynamic and in determining the process controlling step make

them very useful when combined with other variables to indirectly control selectivity.

Simulations show that it is, in principle, possible to achieve any desirable set of selectivities which do not violate their dependency, by preloading the gas adequately. The chosen selectivities, however, will not remain unchanged throughout the evaporation, unless the gas preloading is continuously changed or the chosen selectivities correspond to a dynamic azeotrope evaporating from a state of balanced energy fluxes in a gas-phase-controlled process.

According to the simulations, diffusional interactions may have a significant influence on selectivity. Further experimental work should be necessary, however, to elucidate the influence of such effects from direct observations.

NOTATIONS

A	area [m^2]
c	heat capacity [$\text{kJ kg}^{-1} \text{K}^{-1}$]
C	concentration [kmol m^{-3}]
$[D]$	matrix of multicomponent diffusivities [$\text{m}^2 \text{s}^{-1}$]
$[J]$	matrix defined as $[\beta][k^*]$ [$\text{kmol m}^{-2} \text{s}^{-1}$]
G	molar flux [$\text{kmol m}^{-2} \text{s}^{-1}$]
h	length coordinate [m]
H	molar enthalpy [kJ kmol^{-1}]
$[k^*]$	matrix of mass transfer coefficients [$\text{kmol m}^{-2} \text{s}^{-1}$]
l	solid thickness, sample length [m]
m	mass [kg]
M	molecular weight [kg kmol^{-1}]
n	number of components
N	number of moles [kmol]
p	partial pressure [N m^{-2}]
P	total pressure [N m^{-2}]
q	heat flux [$\text{kJ m}^{-2} \text{s}^{-1}$]
s	selectivity
t	time [s]
T	temperature [K]
u	gas velocity [m s^{-1}]
V	gas volume rate of flow [$\text{m}^3 \text{s}^{-1}$]
y	molar fraction in gas phase [kmol kmol^{-1}]
x	molar fraction in liquid phase [kmol kmol^{-1}]

Greek letters

α^*	heat transfer coefficient [$\text{kW m}^{-2} \text{K}^{-1}$]
$[\beta]$	Bootstrap matrix
γ	activity coefficients
δ	liquid film thickness [m]
λ	thermal conductivity [$\text{kW m}^{-1} \text{K}^{-1}$]
ρ	density [kg m^{-3}]
v_{ij}	relative volatility

Brackets

$[]$	square matrix
$()$	column vector

Subscripts

0	inlet or initial
b	conduction
c	condensable or convection
d	mass transfer
eff	effective value
g	gas
l	liquid
r	radiation
s	solid
t	total
w	interphase

Superscripts

*	dimensionless
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