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INFLUENCE OF EVAPORATION SPACE GEOMETRY ON RATE OF DISTILLATION IN HIGH-VACUUM EVAPORATOR

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ABSTRACT

The problem of calculation of a necessary evaporation surface area in a high-vacuum distillation apparatus with complicated geometry has not been fully resolved yet. Surface evaporation ranges (molecular distillation, the intermediate range, equilibrium distillation), in which different conditions of collisions between vapor particles exist, and their influences on effective evaporation still remain to be taken into account. An example of the apparatus, where the geometrical arrangement of the evaporator makes it difficult to estimate the evaporation rate, is a high-vacuum mechanical evaporator with rotating discs. A solution to this problem is proposed in this article.

INTRODUCTION

The quantity of the distillate stream in high-vacuum evaporators used in the gentle distillation of thermally-sensitive matter is small due to the low unit

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*Corresponding author. E-mail: kawala@iic.pwr.wroc.pl surface evaporation rates obtained at low temperatures—the latter being significantly lower than the boiling temperature of the distilled liquids in specified pressure conditions.

New apparatus that would design solutions, enable the development of a large evaporation area in a small volume, and thus possibly assure a high efficiency of distillation, are being constantly sought.

An apparatus in which evaporation proceeds from evaporator discs, covered with a film of hot liquid, rotating in the perpendicular plane in a common body belongs to such new designs. The apparatus has been built on a pilot and industrial scale (1,9). It has been used with success in the technology of producing high-quality oils for vacuum pumps and in the food and pharmaceutical industries (9).

To design such apparatus on any scale, methods of calculating the actual rate of superficial evaporation in specified physical and geometrical conditions need to be developed.

The authors carried out several experiments on the pilot-scale apparatus and decided to work out a theoretical model, which would describe experimental results and enable the calculation of the distillate stream obtained from devices of any size at similar geometrical parameters. The successive stages of model development are presented in this article.

EXPERIMENTAL TESTS

The high-vacuum evaporator with rotating discs is a horizontal cylindrical apparatus. It incorporates a rotor with several discs (usually 10) rotating in the vertical plane. The discs, partially plunged into the distilled liquid, have six baffles on both sides of each of them. There are semi-spherical cuts on the discs to facilitate the movement of vapor to an outlet located in the upper part of the apparatus. As the disc rotates, the liquid spreads over its uncovered part and forms a thin film. At the beginning of the process, the discs are usually plunged to two-thirds of their diameter. As the quantity of the feed in the apparatus decreases, more and more of the discs' surface is uncovered and the total evaporation area increases. This effect is advantageous because it may compensate a reduction in the vapor-flow caused by the evaporation of more volatile components from the distilled mixture.

Experiments were carried out with pure dibutylphthalate (DBF), which is a substance commonly used for testing this type of apparatus. The experimental equipment consisted of a high-vacuum evaporator of 0.400 m diameter and 0.410 m length, outfitted with 10 discs of 0.386 m diameter each (Figs. 1 and 2). The experimental results are shown in Fig. 3a and b. Figure 3a shows changes in the liquid level in the apparatus and Fig. 3b shows the variation of

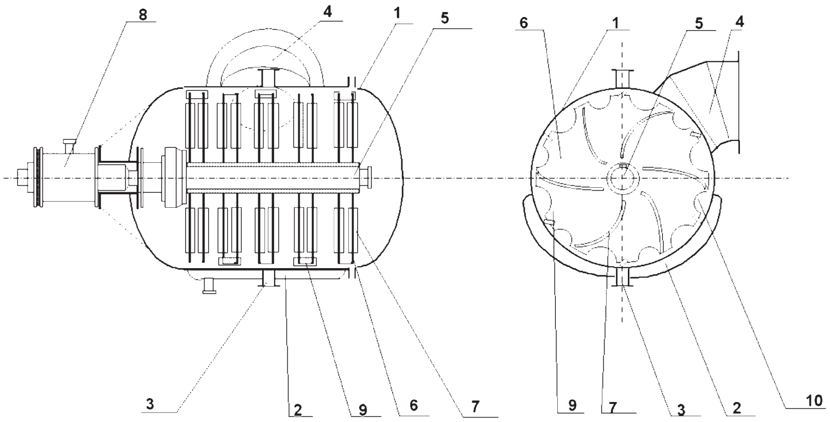

Figure 1. High-vacuum mechanical evaporator with rotating discs: 1—body, 2—heating jacket, 3—residue outlet, 4—vapor outlet, 5—shaft, 6—evaporator disc, 7—scoops, 8—shaft seal, 9—mixing strips, 10—cuts.

the vapor mass flow in time. The changes in the distillation rate are small despite the diminishing liquid level in the apparatus. The distillation rate increases slightly at the beginning of the process and decreases at its end, whereas one could expect the quantity of distillate to increase with the increasing evaporation area in the apparatus. The reasons are explained further on in the paper.

THEORY OF VACUUM SURFACE EVAPORATION

There are three major ranges of high-vacuum distillation. Evaporation proceeds at a different rate and the vapor phase has different physical properties in these ranges. The characteristic quantity for each of the ranges is the Knudsen number, $Kn = \lambda/h_o$, which expresses a ratio of the mean free path of vapor particles to the distance between the evaporator and the condenser.

Ranges of High-Vacuum Distillation

Molecular Distillation

At $Kn > 10$, vapor is anisotropic and the degree of anisotropy depends on, among other things, the radius of curvature of the evaporation surface (4). Vapor

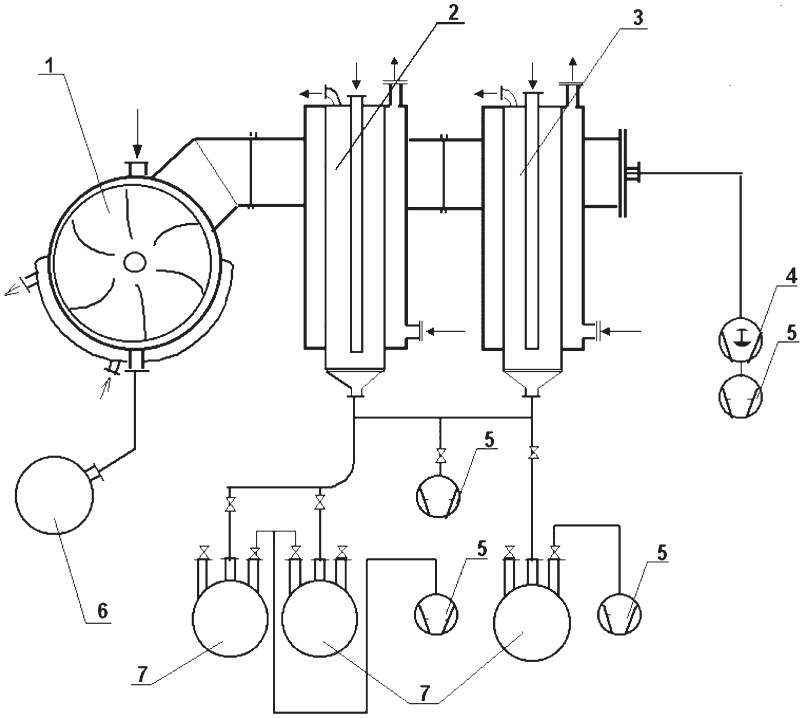

Figure 2. Scheme of experimental equipment: 1—high-vacuum bath still, 2—first condenser stage, 3—second condenser stage, 4—Roots pump, 5—mechanical vacuum pump, 6—residue tank, 7—product tanks.

molecules travel the distance between the evaporator and the condenser practically without a collision. In this range, evaporation proceeds at the maximal (total) rate given by the Langmuir–Knudsen equation (2–7):

$$\dot{G}_{\mathrm{A}} = p^{\mathrm{o}}\sqrt{\frac{M}{2\pi R_{\mathrm{g}}T}} \tag{1}$$

It should be noted that there is very good agreement between experimental and calculated results for this range of distillation, when evaporation proceeds from a falling liquid stream of small diameter or from a metallic rod (8).

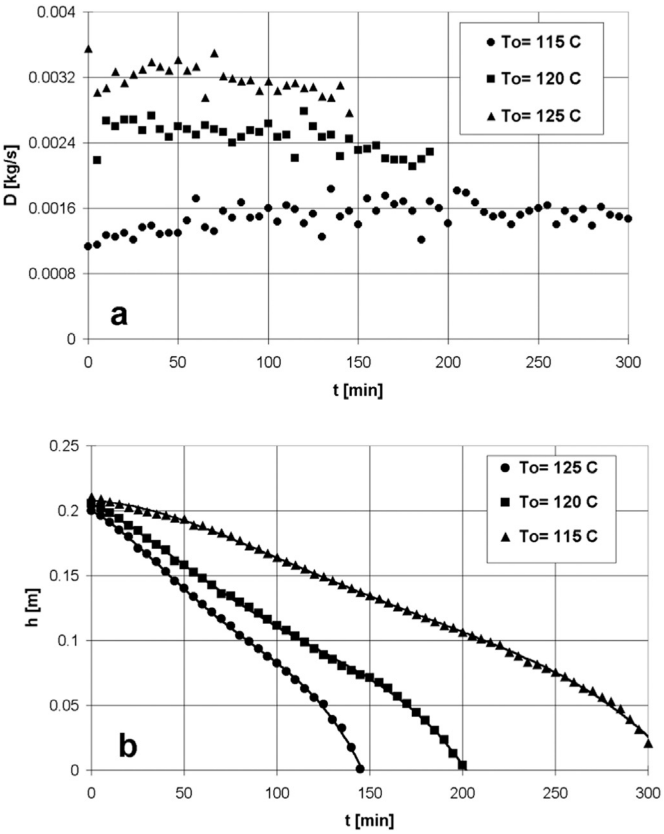

Figure 3. Results of experiments.

Intermediate Range

The range between molecular and equilibrium distillation ($0.05 < Kn < 10$) is the best to run the process because of the proper distillation rate and good protective properties (Fig. 4).

The apparent rate of distillation in this range, where complicated conditions in vapor phase above liquid film have great influence on the effective mean free path of molecules (7), is described by the following expression:

$$\dot{G}_{\mathrm{E}} = \dot{G}_{\mathrm{A}} f \tag{2}$$

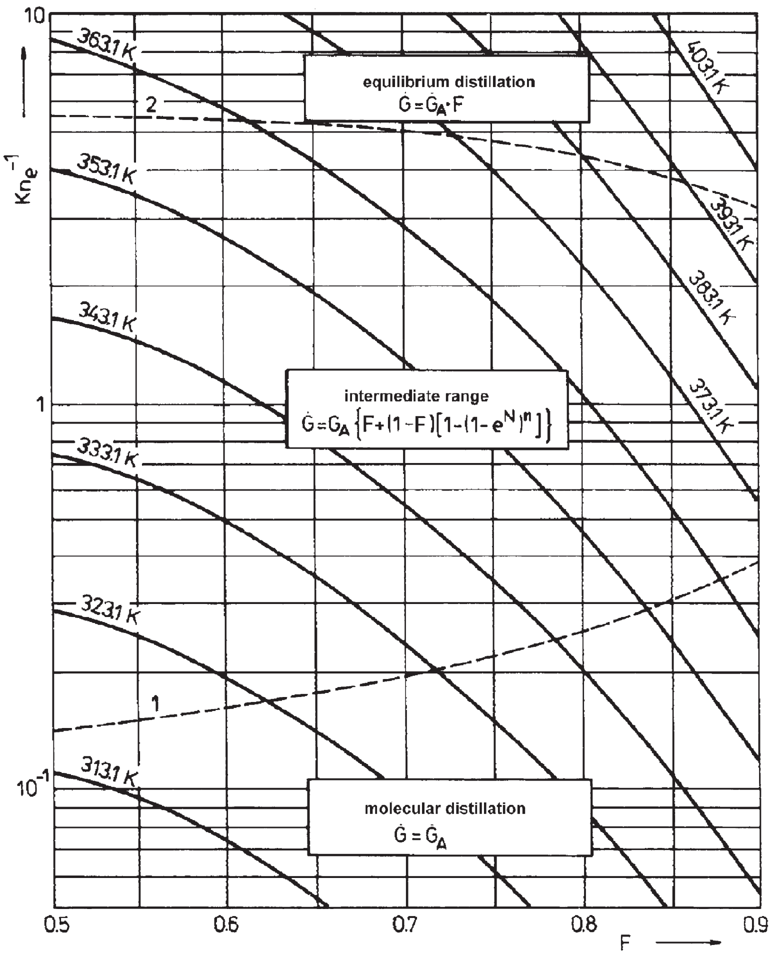

Figure 4. Evaporation limits in high-vacuum distillation regions.

where

$$f = [1 - (1 - F)(1 - e^{kKn})^n] \tag{3}$$

k, *n* are the experimental coefficients (depending on grade of anisotropy of vapor phase).

Parameter *f*, called an evaporation efficiency factor (4,7), depends on the surface phenomena occurring in the evaporated liquid film and on surface ratio *F*, which describes the curvature of the evaporation surface:

$$F = \frac{d_e - 2h_o}{2d_e - 2h_o} \tag{4}$$

where d_e is the diameter of evaporation surface curvature and h_o is the distance between the evaporator and the condenser.

Equilibrium Distillation

If the Knudsen number is less then 0.05 ($Kn < 0.05$), then the vapor has isotropic properties and it behaves as a continuous matter. The number of collisions between particles under these conditions is very high. A particle can reach the condenser but it can also return to the evaporation surface. One can say that then there is a thermodynamic equilibrium at the liquid–vapor phase boundary and hence this range is referred to as equilibrium distillation.

Under equilibrium distillation conditions, when all the directions of motion of particles after multiple collisions are equally probable, parameter f tends toward the value of the surface ratio and the evaporation rate is given by the following equation:

$$\dot{G}_E = \dot{G}_A F \tag{5}$$

For apparatuses with a plane evaporation surface, where the evaporation and condensation surfaces are equal, the surface ratio is 0.5 according to:

$$\lim_{d_e \to \infty} \frac{d_e - 2h_o}{2d_e - 2h_o} = \lim_{d_e \to \infty} \frac{1 - \frac{2h_o}{d_e}}{2 - \frac{2h_o}{d_e}} = \frac{1}{2} \tag{6}$$

This means that due to intermolecular collisions, the probability of a gas particle reaching the condenser is equal to the probability of its return to the evaporation surface. For example, in a gravitational apparatus with the evaporation surface in the shape of a vertical cylinder, when $d_e \to 0$, the value of F tends toward one:

$$\lim_{d_e \to 0} \frac{d_e - 2h_o}{2d_e - 2h_o} = \lim_{d_e \to 0} \left(\frac{d_e}{2d_e - 2h_o} - \frac{2h_o}{2d_e - 2h_o} \right) = 0 + 1 = 1 \tag{7}$$

Although it is hard to predict precisely the value of parameter F for an apparatus with a more complicated geometry, it can be assumed (to make the calculations easier) that the effective rate of evaporation can be calculated as in the case of equilibrium distillation (Eq. (5)).

The quantity, which determines the total rate of the process, is the total mass flow rate of distillate. The latter can be calculated from this relationship:

$$\dot{D} = \sum_{i=1}^{n} \dot{G}_{\mathrm{Ei}} A_{\mathrm{e}i} \tag{8}$$

where: $\sum_{i=1}^{n} A_{\mathrm{e}i} = A_{\mathrm{e}}$ is the total evaporation area in an evaporator.

One must also determine the variation of the apparent evaporation rate in time. The extent of the variation depends on the evaporation surface temperature, which can be easily determined for gravitational static apparatus and centrifugal apparatus with one disc rotating in the horizontal plane. In this kind of apparatus, the flow of a thin film of evaporated liquid, produced by a gravitational or centrifugal force, is laminar and it is possible to estimate the distribution of temperature and concentration on the film's surface (5,10). In the simplest case, when isothermal conditions are assumed, the temperature of the liquid on the entire evaporation surface is equal and constant. As a result, evaporation proceeds at a constant rate. Of course, this is true only for one-component feed. When a multi-component mixture is distilled, the more volatile components evaporate faster. As their quantities in the feed diminish, the total evaporation rate decreases as follows:

$$\dot{G}_i = x_i p_i^{\mathrm{o}} \sqrt{\frac{M_i}{2\pi R_{\mathrm{g}} T}} \tag{9}$$

$$\dot{G} = \sum_{i=1}^{n} \dot{G}_i \tag{10}$$

Changes in the composition of the evaporated liquid will cause changes in the composition of the distillate. To estimate the changes, one can assume a differential distillation model in which the concentration of a more volatile component in the vapor phase can be calculated from this relationship:

$$y_{\mathrm{A}} = \frac{\alpha_{\mathrm{AB}} x_{\mathrm{A}}}{1 + (\alpha_{\mathrm{AB}} - 1) x_{\mathrm{A}}} \tag{11}$$

The coefficient of relative volatility under conditions of high vacuum is calculated from the following relationship:

$$\alpha_{\mathrm{AB}} = \frac{p_{\mathrm{A}}^{\mathrm{o}}}{p_{\mathrm{B}}^{\mathrm{o}}} \sqrt{\frac{M_{\mathrm{B}}}{M_{\mathrm{A}}}} \tag{12}$$

Changes in the liquid's composition are described by Rayleigh's equation, a fundamental equation of differential distillation, which for the equilibrium curve described by expression, Eq. (11) has the following solution:

$$\ln\left(\frac{W_{\mathrm{o}}}{W_{\mathrm{K}}}\right) = \int_{x_{\mathrm{AK}}}^{x_{\mathrm{A0}}} \frac{\mathrm{d}x}{y - x} = \frac{1}{\alpha_{\mathrm{AB}} - 1} \ln\left(\frac{x_{\mathrm{A0}}(1 - x_{\mathrm{AK}})}{x_{\mathrm{AK}}(1 - x_{\mathrm{A0}})}\right) + \ln\left(\frac{1 - x_{\mathrm{AK}}}{1 - x_{\mathrm{A0}}}\right) \tag{13}$$

CALCULATIONS AND DEVELOPMENT OF THEORETICAL MODEL

As mentioned before, the basic quantities, which determine the rate of the process, are the total evaporation area and the temperature of the film of evaporating liquid. The total evaporation area consists of the evaporating liquid surface area and the total surface area of the rotating discs' free regions. Changes in these quantities during the process can be described by this relationship (Fig. 5):

$$A_{\mathrm{d}} = 2N\left[\left(\frac{\pi}{2} + \operatorname{arctg}\left(\frac{H - R_{\mathrm{d}}}{\sqrt{R_{\mathrm{d}}^2 - (H - R_{\mathrm{d}})^2}}\right)\right)R_{\mathrm{d}}^2 + (H - R_{\mathrm{d}})\sqrt{R_{\mathrm{d}}^2 - (H - R_{\mathrm{d}})^2}\right] \tag{14}$$

$A_{\mathrm{l}} = 2L_{\mathrm{a}}\sqrt{2R_{\mathrm{a}}H - H^2}$, where N is the number of rotating discs. (15)

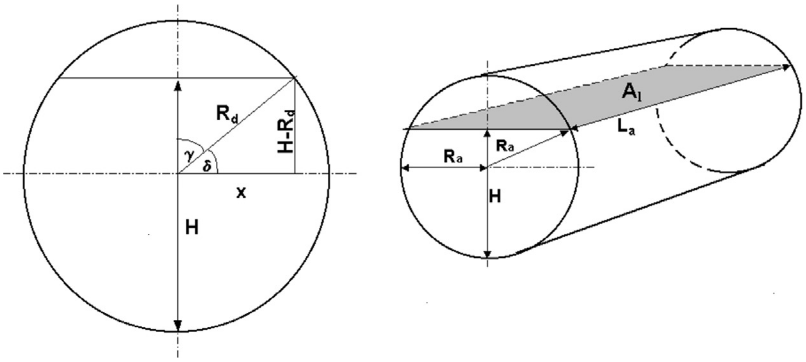

Figure 5. Total evaporation surface in a high-vacuum evaporator.

For the initial calculations, the apparatus was assumed to be isothermal, i.e., the temperature of the liquid's surface and that of the liquid film on the rotating discs are the same and constant. It was additionally assumed that the whole vapor produced in the evaporator reaches the condenser and on condensing on its surface becomes a distillate. Furthermore, it was assumed that *F* (the surface ratio) equals 0.5 as for an apparatus with a flat evaporation surface.

The calculations results for the distillation of the model substances: DBF and dibuthylsebacate (DBS) are shown in Fig. 6. It appears that the distillation rate calculated in this way is several times higher than the one measured during

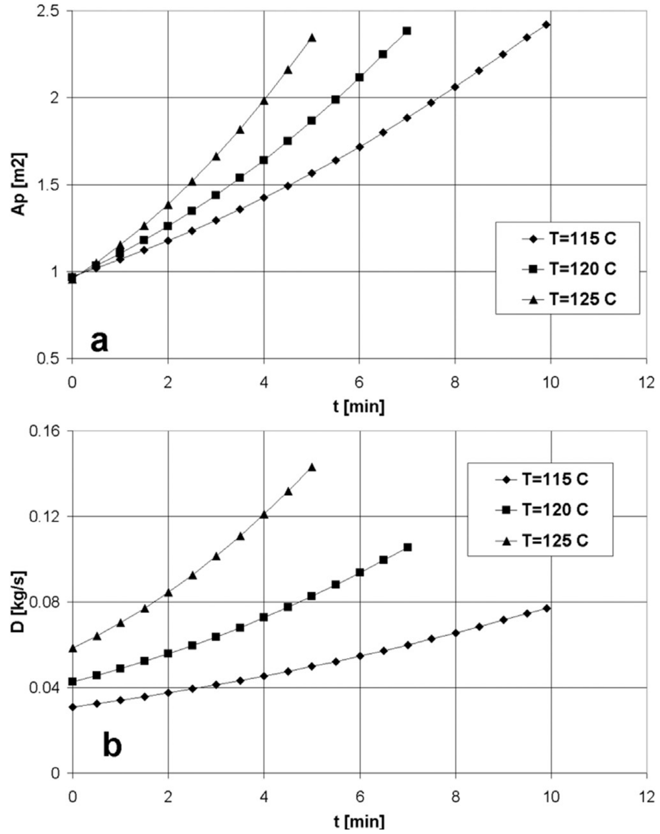

Figure 6. Results of calculation: (a) changes of total evaporation area, (b) total mass flow rate for evaporation of pure DBF, (c) total mass flow rate for evaporation of mixture of DBF–DBS at $T_o = 120°C$, (d) changes in composition of vapor in time, and (e) changes of effective evaporation rate for mixture of DBF–DBS.

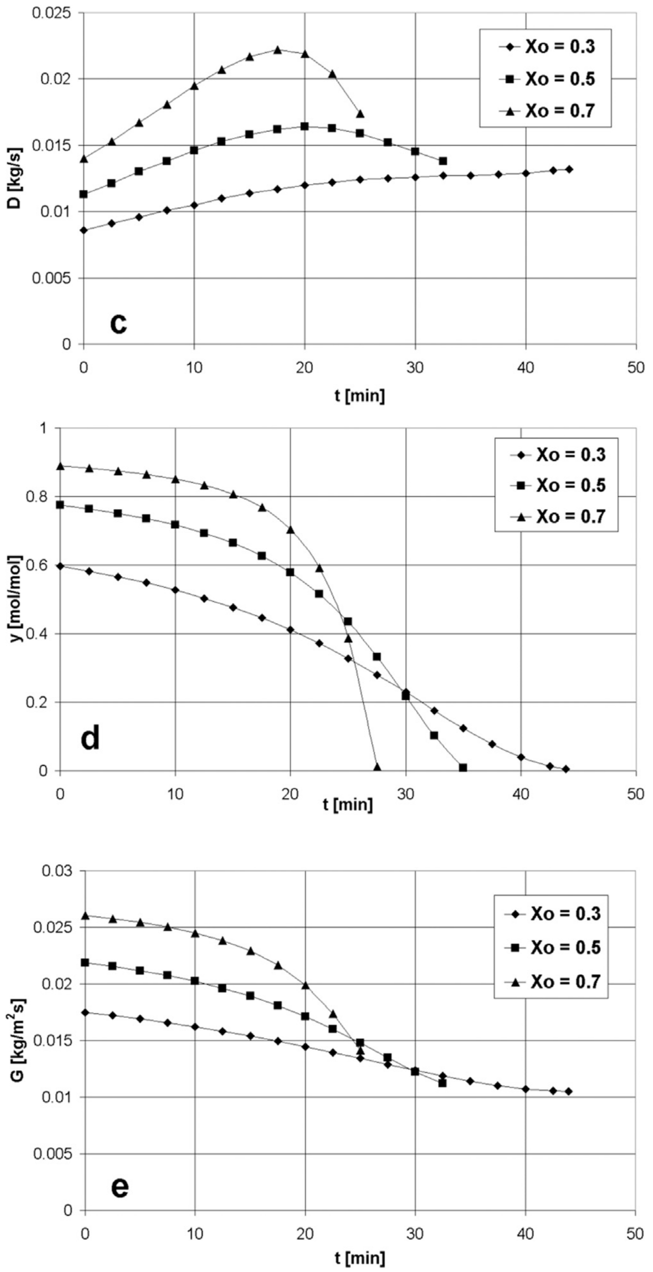

Figure 6. Continued.

the experiments. From the theoretical model, it follows that the distillation rate should increase with the progress of the process because of the increasing total evaporation area. However, since the calculation results did not agree with the experimental results, we gave up the isothermal apparatus assumption and referred to earlier treatments in which the temperature profile in a liquid film was calculated (5,6). The results of those experiments were used in the following way: the typical radial temperature distributions on the disc then determined for different initial liquid temperatures T_o were approximated by polynomial of third degree (Fig. 7).

Then the polynomial was averaged and the following equation, which can be used to calculate the approximate temperature in any place of the disc (Fig. 8), was obtained:

$$T_d = 4395.9r^3 - 547.74r^2 + 74.54r + d_f \tag{16}$$

In this equation, parameter d_f, which is a function of initial process temperature T_o (corresponding to the temperature of the liquid's surface), was approximated by means of this binomial:

$$d_f = 0.0052T_o^2 - 3.056T_o + 773.59 \tag{17}$$

For the calculations, each disc was divided into 50 rings of equal width and the average temperature of each disc was calculated. It was assumed that the

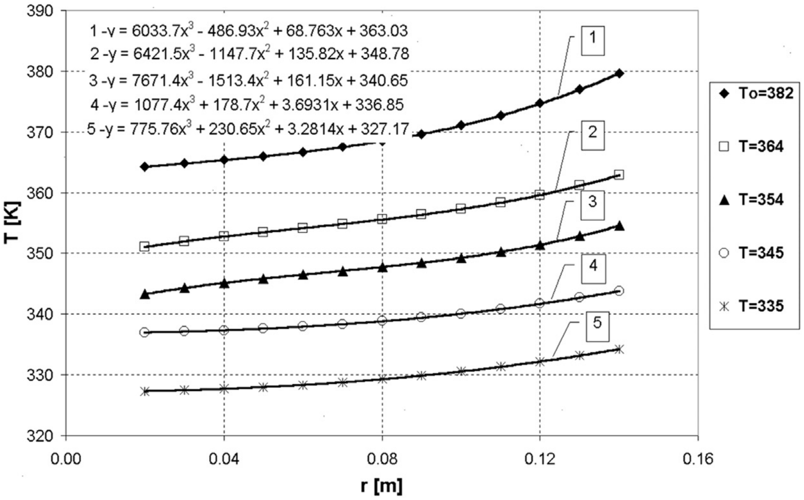

Figure 7. Approximation of experimental points by $T_d = ar^3 + br^2 + cr + d$, where $0 < r < R_d$.

temperature of each ring on the disc is constant during the process and equal to the integral average of the temperatures at the ring's edges. Hence, the temperature of the i-th ring can be calculated from this relationship:

$$T_{\mathrm{ring},i} = \frac{\int_{r_i}^{r_{i+1}} (ar^3 + br^2 + cr + d_{\mathrm{f}})\mathrm{d}r}{r_{i+1} - r_i}, \quad \text{where} \quad r_{i+1} - r_i = r_{\mathrm{ring}} \tag{18}$$

After several transformations and the insertion of appropriate values, we obtain:

$$T_{\mathrm{ring},i} = \frac{1098.96(r_{i+1}^4 - r_i^4) - 182.58(r_{i+1}^3 - r_i^3)}{r_{i+1} - r_i} + 37.271(r_{i+1}^2 + r_i^2) + d_{\mathrm{f}} \tag{19}$$

If the disc liquid temperature distribution is taken into account in the calculations, the total evaporation rate will be 3.5 times lower (about 2.5 times in the initial phase and over 5.0 times in the final phase) in comparison with the results obtained under the isothermal conditions assumption.

At the beginning of the process, the rate of distillation increases, after some time, it reaches the maximal value, and then it decreases. But this assumption still did not lead to a good agreement between the experiments and the calculations. There were large differences probably due to the fact that in the theoretical model, only the effects of collisions between particles are taken into account. The model does not take into account the influence of the geometrical arrangement on the effective rate of evaporation. It was assumed that the vapor particles do not encounter any obstacles on the way between the evaporator and the condenser and to simplify the calculations, the same value of f as for the apparatus with a flat evaporation surface equal to the condensation surface was assumed. These assumptions are fully reasonable for a centrifugal apparatus with one horizontal rotating disc and for a gravitational apparatus, but in the case of an evaporator with vertical rotating discs, they lead to disappointing results because of the more complicated geometrical arrangement: the mutual position of evaporation and condensation surfaces and the complexity of the evaporation surface, consisting of the surfaces of the perpendicular discs and the liquid's surface, with small distances between the discs on the shaft. As a result, many vapor particles, which despite collisions could reach the condenser, hit the next disc or the evaporator's body where they often condense. The effect of the spatial configuration of evaporation and condensation surfaces on the distillate mass flow can be estimated by analogy with radiation processes (8). Using this analogy, one can calculate surface configuration coefficient β accounting for this portion of vapor, produced in a time unit, which reaches the condenser and becomes a distillate.

Hence the effective rate of evaporation can be described as follows:

$$\dot{G}_{\mathrm{E}} = \dot{G}_{\mathrm{A}} f \beta \tag{20}$$

To calculate the fraction of radiation which after leaving one surface (A_1) will reach another arbitrarily-configured surface (A_2), one can use Lambert's law:

$$\beta = \frac{1}{A_1} \int_{A_1} \int_{A_2} \frac{\cos \psi_1 \cos \psi_2}{\pi h^2} \mathrm{d}A_1 \mathrm{d}A_2 \tag{21}$$

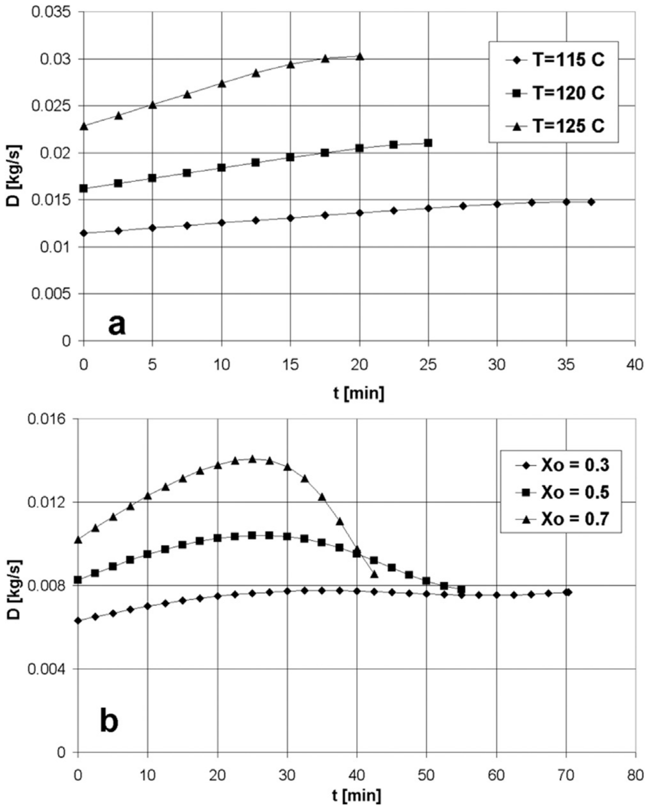

Figure 8. Calculation results with consideration of distribution of liquid temperature on disc: (a) changes of total mass flow for evaporation of pure DBF, (b) for mixture of DBF–DBS at $T_o = 120$°C, (c) changes of effective rate of evaporation, (d) changes of composition of vapor, and (e) comparison between experimental and calculation results.

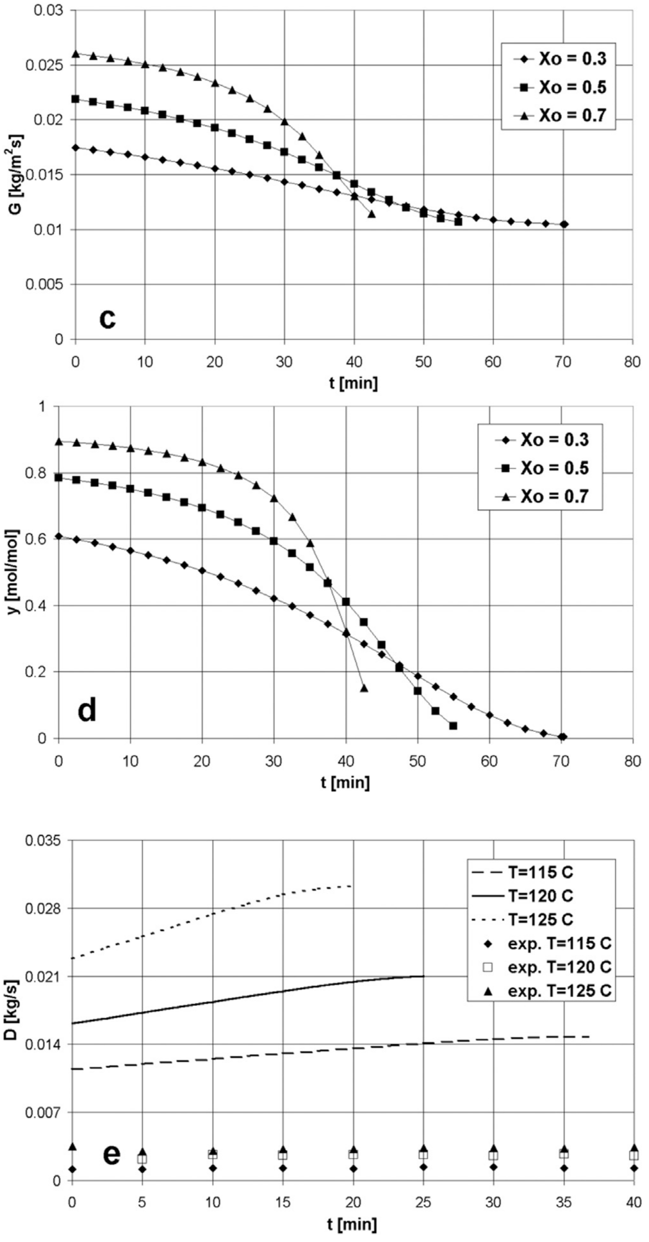

Figure 8. Continued.

where: h is the distance between A_1 and A_2, ψ_1, ψ_2, the angles between h and normal to A_1 and A_2, respectively.

But because of the system's geometrical complexity, it is hard to apply the above equation in practice. It would be difficult, both to attempt to define the two surfaces and to find an analytical solution of the equation. But one can use another approach to the problem. An analysis of the possible paths, which any particle inside the apparatus can take, shows that the probability of its reaching the condenser can be estimated from a simple ratio. Assuming that the particle will surely reach the condenser if it manages to reach the vapor outlet, this will be the ratio of the outlet cross-sectional area to the concise area, which the particle could hit:

$$\beta = \frac{A_{\mathrm{outlet}}}{A_{\mathrm{c}}} = \frac{A_{\mathrm{outlet}}}{A_{\mathrm{outlet}} + \frac{A_{\mathrm{l}}}{11} + \frac{A_{\mathrm{d}}}{10} + nA_{\mathrm{body}}} \tag{22}$$

The total area can be described using the diagram shown in Fig. 9. The diagram represents the space between any two discs in the apparatus. A vapor particle situated in this space may move in any direction as a result of numerous collisions. It can reach the vapor outlet but it can also hit one of the two discs, the liquid's surface, or the apparatus's body. Since there are 10 discs in the apparatus and they divide its interior into 11 identical parts, A_{c} will be equal to the sum of 1/10 of the total disc area, 1/11 of the total liquid surface area, the vapor outlet cross-sectional area, and a part of the body area. The size of the latter is the most difficult to determine. The best agreement between experimental and theoretical results is obtained when the share of the body area amounts to 3/10.

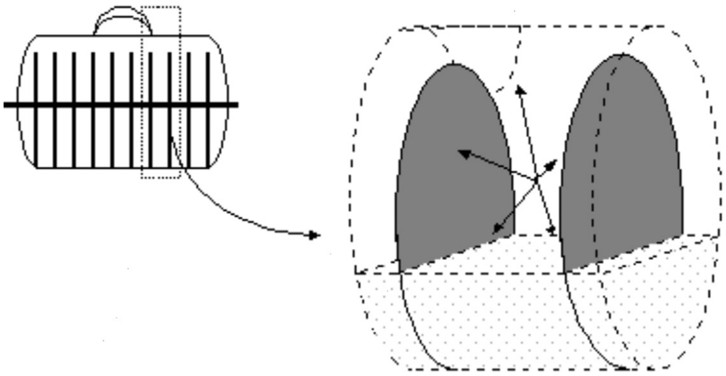

Figure 9. Possible path of particle in space between any two discs inside evaporator bath.

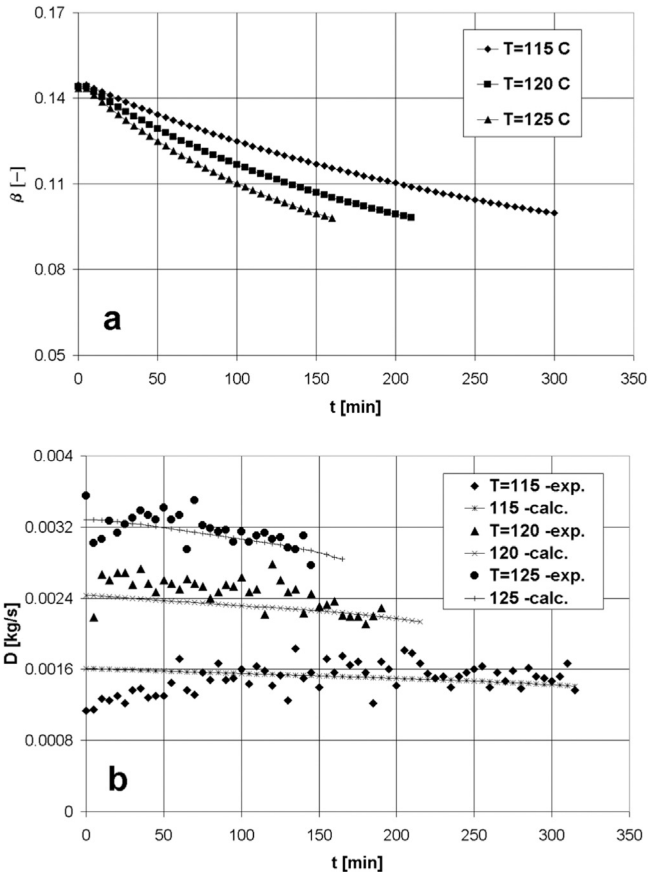

Figure 10. Results of calculation with allowing for β: (a) changes of configuration coefficient in a time progress, (b) changes of total mass flow rate for pure DBF.

Configuration coefficient β defined in this way is not constant during the whole process: it decreases as the liquid in the evaporator diminishes and the areas, which a vapor particle on its way to the condenser can hit, increase (Fig. 10).

CONCLUSIONS

The development of a theoretical model describing high-vacuum distillation in an evaporator with rotating discs has been described. Starting from Langmuir's equations for the total evaporation rate in high-vacuum conditions, taking into account the actual distribution of temperature in the distillate in the apparatus and adopting the β value in the form presented above, it is possible to obtain equations which describe, in good agreement with the experimental results, the rate and progress of the whole process. They can be used to calculate this type of apparatus. Furthermore, on this basis, several important conclusions about the design of bath evaporators with rotating discs can be drawn. The β value, which depends on (among others) the size of the vapor outlet cross-sectional area, has the greatest influence on the effective rate of evaporation. The larger this area, the larger the value of the configuration coefficient and the more vapor reaches the condenser. Therefore, the outlet should have the largest possible diameter. One of the most important quantities is the distance between the discs. In the tested evaporator, this distance was about 3–4 cm, but it seems that it should be somewhat larger in order to facilitate the flow of vapor.

NOTATION

A	area [m^2]
β	surface configuration coefficient
D	total mass flow rate (mass flow of distillate) [kg/s]
F	surface rate
G	total rate of evaporation [kg/m^2 sec]
H	height of liquid [m]
h_o	distance between evaporator and condenser [m]
Kn	Knudsen number
L	length [m]
λ	mean free path [m]
M	molar mass [kmol/kg]
p^o	saturation pressure [Pa]
R, r	radii [m]
R_g	gas constant
T	temperature [K, °C]
t	time [min]
W	mass of liquid
x	mole fraction in a liquid phase [kmol/kmol]
y	mole fraction in a vapor phase [kmol/kmol]

Indices

A	more volatile component, absolute
a	apparatus
B	less volatile component

c	concise
d	disc
E	effective
e	evaporation
i	number of component
0	initial
K	final
l	liquid surface

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