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DRYING OF THIN FILMS OF POLYMER SOLUTIONS COATED OVER IMPERMEABLE SUBSTRATES

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Abstract. Manufacturing of adhesive tapes, photographic films, magnetic media and many other products involve the coating and drying of thin films of polymeric solutions or emulsions over an impermeable substrate. Wet thickness may vary from 1 micron to 500 microns and solid content from 3% to 50% or more. Drying processes generally occur in convective ovens, where air temperature, air flow rate and solvent partial pressure may be adjusted. In many cases, it is the manufacturing step that controls the productivity, therefore it is very important to optimize the oven conditions to minimize the residence time inside the oven without causing defects on the coated film and keeping the residual solvent within the final product specification limits. A drying simulator was developed in order to solve the transient mass conservation and heat transfer equations that describe the drying process. This system of equations shows strong non-linearity due to the free surface and dependence of diffusion coeficients on solvent concentration and film temperature. An example of using the drying simulator to optimize an industrial adhesive tape line is discussed. The gain in productivity achieved by the analysis was larger than 40%.

***Keywords**. Drying, coated substrate, adhesive tape.*

1. Introduction

The production and sale of adhesive tapes is a large business worldwide. 3M, Avery Dennison, Tesa, Norton and Loma are companies playing in this market and working to improve their products and manufacturing process in order to get competitive advantage.

Adhesive tapes are applied in numerous market segments, ranging from domestic use to extremely complex applications like substitute of screws, clamps and other mechanical bonding systems.

The adhesive itself may be acrylic, rubber based or a mixture of both. Requirements of adhesion, shear resistance, temperature resistance, UV resistance, among others, define the formulation suitable to the application.

The manufacturing process of adhesive tapes includes the adhesive compound, coating over substrate, solidification of the liquid in a semi-solid material and slit of final rolls. The process of continuous liquid coating of substrates and final solidification is also widely used in the manufacturing of photographic and magnetic films, papers, and many other products.

Solidification may be achieved in different ways: polymerization of a reactive liquid monomer or oligomer, cooling of melt polymer, drying and crosslinking of a polymer solution or simply drying of a solution. Drying of a solution of polymer in organic solvents still is very much used in the manufacturing of adhesive tapes, although there are mounting incentives to switch to other types of formulations, notably water-borne and solventless (monomer or oligomer 100% polymerizable by thermal, UV and e-beam curing).

Usually, in modern drying processes, the solvents are removed in ovens with forced convection of heated air. Typically, the operating parameters that can be controlled are air temperature, solvent partial pressures, heat and mass transfer coefficients (depending on air delivery rate and how the air arrives at the coating and sometimes auxiliary heat delivery by radiation from infrared heaters).

A variety of defects can arise in the drying process. Blistering due to boiling of the solution is one of the most frequent in the manufacturing of adhesive tapes. Examples of other defects include formation of Bénard Cells, peeling and cracking of the coating surface. Both applying the liquid layer and drying it affect directly the quality of final product and so both should be considered together in optimization of the manufacturing process.

In the adhesive tape industry, where the solvent content of liquid is high and the coated liquid film is thick, the solvent removal is generally the controlling constraint of the speed of the overall process.

This paper presents a model of drying adhesive tapes and shows the optimization of the manufacturing process based on the simulation predictions. The partial differential equations that describe the heat and mass transfer are solved by the finite difference method. The resulting non-linear system of algebraic equations is solved by Newton's method. The predictions are compared to experimental data and the optimization analysis was able to increase the productivity of a production line by approximately 40%.

2. Theoretical Model

The drying process of a liquid layer can be modeled by heat and mass transfer equations, subjected to boundary conditions at the air-liquid interface and at the substrate. The set of equations is highly non-linear due to strong dependence of diffusion coefficients on liquid temperature and concentration of the different components and the presence of a moving free surface (air-liquid interface).

Figure 1 shows the geometry of the problem. Arrows inside the liquid represent diffusion of the components to the free surface and arrows at the gas phase represent mass transfer to the air stream. The curved arrows represent heated air sweeping the liquid free surface and the substrate side.

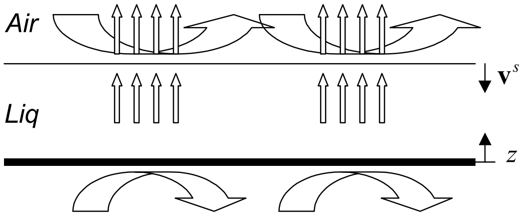

Figure 1: Sketch of dyring of a liquid layer over a substrate.

As drying procedes, the volatile components transfer from liquid to gas phase and the liquid film thickness *l(t)* reduces at rate $v^s = {dl(t)}/{dt}$.

Two phases are present in the problem: the liquid phase defined by its temperature, pressure and composition and the gas phase determined by the same variables. Although these phases are not in equilibrium – otherwise there would be no mass transfer– it is common the use of this concept to simplify the problem.

2.1. Mass Transfer in the Liquid Phase

The liquid phase is formed by *n* well mixed components. As the liquid phase shrinks due solvent evaporation, it is convenient to choose a control volume that follows this movement. The equation of conservation of the chemical species in this moving frame of reference is given by:

$$\frac{d\rho_i}{dt} = -\nabla \bullet \rho_i \mathbf{v}_{ref} - \nabla \bullet \mathbf{j}_i + \mathbf{v}^p \bullet \nabla \rho_i + R_i ; \qquad 0 < z < l(t). \tag{1}$$

R_i is the rate of generation of component *i*, $\mathbf{v}^p$ is the control volume velocity, $\mathbf{v}_{ref}$ is the reference velocity conveniently chosen to represent the convective mass transfer in the liquid phase, and $\mathbf{j}_i = \rho_i(\mathbf{v}_i - \mathbf{v}_{ref})$ is the diffusive flux of component *i*. If (i) there is no chemical reaction, (ii) the liquid is an ideal solution, (iii) the substrate is impermeable, (iv) Fick's law is valid, and (v) the thickness is uniform and there is no gradient on the web direction, the mass transfer equation becomes:

$$\frac{d\rho_i}{dt} = \frac{\partial}{\partial z}\left(\sum_{j=1}^{n-1} D_{ij} \frac{\partial \rho_j}{\partial z}\right) + v^p \frac{\partial \rho_i}{\partial z} ; \qquad 0 < z < l\,(t) \tag{2}$$

The diffusion coefficients $D_{ij}(T, P, \rho_1, \ldots, \rho_{n-1})$ must be known and v^p defined as convenient. Here, it was chosen to be: $v^p = v^s \frac{z(p)}{l(t)}$, where z(p) is the position of the point p. All thermodynamic variables ($\hat{V}_i, \rho_i, \hat{H}_i$) and transfer coefficients (D_{ij}, k_i) are assumed independent of pressure and the gas phase is a *mixture of ideal gases.*

2.2. Boundary conditions to the equation of conservation of the chemical species

The substrate is considered impermeable, i.e. there is no mass flux through it. In this location, the appropriate boundary condition is:

$$j_i = -\sum_{k=1}^{n-1} D_{ik}\frac{\partial \rho_k}{\partial z} = 0 \Rightarrow \frac{\partial \rho_k}{\partial z} = 0; \qquad z = 0 \qquad k=1,...,n. \tag{3}$$

The boundary condition at the liquid-air interface is determined by a mass conservation equation at that location. It is important to consider the interface velocity on the mass flux of each component:

$$-\sum_{j=1}^{n-1} D_{ij}\frac{\partial \rho_j}{\partial z} = \rho_i v^s + k_i^G\left(P_{i,\text{int}}^G - P_{i,\infty}^G\right); \quad z = l(t) \quad i=1,...,n-1\ . \tag{4}$$

k_i^G [s/cm] is the mass transfer coefficient, ($P_{i,\text{int}}^G - P_{i,\infty}^G$) [g/cm.s²] is the difference between the partial pressures of component *i* just close to the interface and at the bulk of the gas phase.

The reference velocity in eq.(2), $\mathbf{v}_{ref}$, is assumed as the volume average velocity (that is zero, since the substrate is impermeable). The Chilton-Colburn (Chilton, 1934) analogy is used to estimate the mass transfer coefficients.

The velocity of the interface, v^s, is determined multiplying the equation (2) of each component by the specific volume of the respective component and add them all. Using the concept of ideal solution, it yields:

$$v^s = \frac{dl(t)}{dt} = \sum_1^n \hat{V}_i k_i^G\left(P_{i,\text{int}}^G - P_{i,\infty}^G\right) \tag{5}$$

The initial conditions are the concentration profile of each component and wet film thickness at the beginning of the process.

2.3. Phase Equilibrium

When studying drying of thin liquid films, the concept of phase equilibrium is usually used. However, it is clear that in drying process there is no thermal, mechanical or phase equilibrium. But considering an infinitesimal control volume placed at interface between the two phases, it is a good approximation to neglect the temperature and pressure gradients. In addition, considering that although there is mass flux throw the interface, at infinitesimal shorter times the mass flux tends to zero and there is a situation of *instantaneous phase equilibrium*.

The partial pressure of component *i* at interface (in the liquid phase) is proportional to the vapor pressure of the respective pure component. The proportionality factor is the activity a_i of this component in solution:

$$P_{i,\text{int}}^G = y_i P = x_i \gamma_i P_{v,i} = a_i P_{v,i} \qquad i = 1,...,n \tag{6}$$

If the ideal solution hypothesis is used, the previous expression reduces to the Raoult's law, which has a narrow range of application, including only solution of small molecules with similar size and chemical nature. That is why it is not used here. In this work, the Flory-Huggins model (1953) has been used to determine the activity of components in solution. It is limited to the case of one polymer in one or two solvents. The Flory-Huggins model relates the activity of each solvent in the solution, $a_{i,}$ with the volume fraction of all components, ϕ_j , molar volume of each solvent, $\tilde{V}_j$, and the interaction factor between each pair of components of solution, $\chi_{jk}\left(T, x_1,..., x_{n-1}\right)$.

$$a_1 = \phi_1 \exp\left[(1-\phi_1) - \left(\frac{\tilde{V}_1}{\tilde{V}_2}\right)\phi_2 + \left(\chi_{12}\phi_2 + \chi_{13}\phi_p\right)\left(\phi_2 + \phi_p\right) - \chi_{23}\left(\frac{\tilde{V}_1}{\tilde{V}_2}\right)\phi_2\phi_p\right] \tag{7}$$

$$a_2 = \phi_2 \exp\left[(1-\phi_2) - \left(\frac{\tilde{V}_2}{\tilde{V}_1}\right)\phi_1 + \left(\chi_{12}\phi\left(\frac{\tilde{V}_2}{\tilde{V}_1}\right)_1 + \chi_{13}\phi_p\right)\left(\phi_2 + \phi_p\right) - \chi_{23}\left(\frac{\tilde{V}_1}{\tilde{V}_2}\right)\phi_2\phi_p\right] \tag{8}$$

Considering that the components do not change specific volume during mixing, $\overline{V}_i = V_i$, then:

$$\sum_1^3 \phi_i = \sum_1^3 \rho_i \overline{V}_i = \sum_1^3 \rho_i V_i = 1 \tag{9}$$

The molar volume of each component in solution will be the molar volume of the respective pure component, $\widetilde{V}_i = V_i MM_i$ [cm³/mol]. The interaction factors $\chi_{jk}\left(T, x_1, \ldots, x_{n-1}\right)$ shall be supplied to the model. They represent the affinity among the components in the solution and its value is small when the interaction is strong.

Basically some experiments are made with diluted solutions in order to get the interaction between polymer and solvent, χ_{13} and χ_{23}. Phase equilibrium data of a solution formed by the two solvents is used to get χ_{12}.

As in general the interaction factors show great dependence on temperature and composition, its determination is extremely hard. Some analysis have reported studies using constant interaction parameters χ_{jk} without expressive loss of accuracy (see Price, 1999; and Alsoy, 1999).

Favre (1996) reported studies made with ternary solutions using Flory-Huggins model (with constant interaction parameters) in order to get the activity of solvents and concluded that the experimental data were in accordance with the model only to elastomers in apolar solvents. When the polymer showed some cristalinity or the solvents were polars, the model did not show good results.

2.4. Bubble Point Temperature

Bubble formation in the coating layer is one of the main problems in drying. The mechanism of bubble formation is complex and not completely understood.

One condition proposed and used by many researchers to explain the bubble formation is that the vapor pressure in some point of solution must be higher than the pressure inside oven. (see Cairncross, 1995; Price, 1999; and Alsoy, 2001). An expression to solution vapor pressure, $P\,(T, x_1, \ldots, x_{n-1})$ is required.

The partial pressure of solvent at interface between liquid and gas phase can be modeled by eq.(6). The partial pressure of each component at interface is equal to the vapor pressure of this component times the activity of it at solution.

Extending this idea, the solution vapor pressure is assumed to be equal the sum of product of vapor pressures of each component times its activity in the solution, i.e.

$$\mathrm{P} = \sum_{1}^{\mathrm{n}} \mathrm{a_i P_{v,i}} \tag{10}$$

Considering the pressure inside the oven as atmospheric, P_{atm}, and one ternary solution of two solvents and one polymer, an estimation of the bubble temperature is got by solving the non-linear equation (11).

$$\sum_{1}^{\mathrm{n-1}} \mathrm{a_i P_{v,i}} - \mathrm{P_{atm}} = 0 \tag{11}$$

One important factor that can cause big deviation between model and real data is the presence of air dispersed in solution. The dispersed air tends to increase the vapor pressure of solution, so reducing the bubble temperature. The problem is that the concentration of dispersed air is difficult to be determined and included in the equation (11). That is the main reason why, in practice, it is always recommended to reduce air-liquid interface during the liquid handling processes.

2.5. Heat Transfer

In general, the necessary energy to promote drying comes from the convection of heated air on the liquid-gas interface and substrate side. Infrared radiation also can be used to increase energy transfer, but it will not be considered in this analysis. Depending on the type of oven, there is the possibility to set independent temperatures and airflow at each side of coating.

Yapel (1989) and Alsoy and Duda (1999) have shown that the temperature gradient in the coating layer and substrate thickness is not representative. Uniform temperature along coating and substrate thickness is assumed.

The heat transfer equation considering lumped parameters is given by:

$$\frac{dT}{dt} = -\left[\frac{h^G\left(T - T^G\right) + k_1^G \Delta\hat{H}_{v,1}\left(P^G_{1,\mathrm{int}} - P^G_{1,\infty}\right) + k_2^G \Delta\hat{H}_{v,2}\left(P^G_{2,\mathrm{int}} - P^G_{2,\infty}\right) + h^g\,(T - T^g)}{\rho^L Cp^L l(t) + \rho^s Cp^s H}\right] \tag{12}$$

The initial condition is given by: $T(0) = T_0$

2.5. Diffusion Coefficient

The diffusion coefficients represent the velocity that concentration gradients are dissipated within the solution. The mass transfer in binary solutions is described by one mutual diffusion coefficient, D, while ternary solutions need four mutual diffusion coefficients to describe the diffusion process: D_{11}, D_{12}, D_{21}, D_{22}. Each Dij represents the diffusion of component i due to concentration gradient of component j.

In solutions of low molecular weight, the coefficient Dij is a weak function of temperature and solution concentration. Considering polymeric solutions, Dij can vary several orders of magnitude due to changes in temperature and concentration of each component. The coefficient Dij also can be a function of pressure, but this dependence is not considered here.

There is no general theory that precisely gives the diffusion coefficients in the whole range of concentration, and the existing models use the self-diffusion coefficients of polymer and solvents – Free Volume Theory (Vrentas and Duda, 1976) – and a thermodynamic factor as parameters in the expression to get the mutual diffusion coefficients.

There are several models to determine the mutual diffusion coefficients of a ternary solution (Alsoy and Duda, 1999; Zielinsky and Hanley, 1999; and Price and Romdhane, 2003). All these models came from the theory proposed by Bearman (1961) that relates the self-diffusion coefficient of each component in solution with friction factors among them.

The self-diffusion coefficient of a component measures the diffusion velocity of this component in the absence of concentration gradients and is intimately related with chemical nature of the component.

The Free Volume Theory by Vrentas and Duda (1976) allows the calculation of self diffusion coefficients of each component given some solution parameters. There are tables with the parameters of the refered models to different binary solutions polymer/solvent.

2.6. Solution Method

The set of partial differential equations was solved by the finite difference method. A second-order, central difference scheeme was used to descritize the spacial derivatives, and a fully implicit formulation was used to descritize the time derivative. The unknowns of the discretized problem at each time step are the concentration of both solvents at each node, the coordinate of each node of the mesh and the solution temperature. With the implicit time integration, a set of non-linear algebraic equations is obtained at each time step. The system was solved by Newton's method, which requires the evaluation of the Jacobian matrix, i.e. the sensitivity of each equation to every unknown of the problem. The evaluation of all the Jacobian entries was extremely complex, because the diffusion coefficient of the mass transfer equations are a strong function of concentration and temperature. Details of the solution methods are presented in Perez (2004).

3. Results

Results of the application of a computer-aided simulation in the case of manufacturing of adhesive tapes at 3M Brazil are presented. The coating liquid is an adhesive formed by an acrylic polymer in ethyl acetate (solvent 1) and toluene (solvent 2). The substrate is paper. The drying oven is divided in three zones. At each zone, the operating variables that can be controled are the air temperature and velocity (heat transfer coefficient). Table 1 shows the input variables for the drying simulator. They can be divided in two general categories: solution properties (including the parameters for the diffusion model) and process parameters. The parameters for the diffusion model were obtained experimentaly and supplied by Price (2004).

The flow inside impingment ovens is complex and the heat transfer is not easily determined. There was no available data on the heat transfer coefficients of the industrial oven considered in the analysis. The heat transfer coefficient of each of the three zones of the oven was estimated by determining the value such that the predicted coating temperature along the oven matched the measured liquid layer temperature. This measurement was done using an infrared sensor with the correct emissivity adjustment. Figure 2 shows the set temperature in each zone, the predicted and measured coating temperature, as well as the estimated heat transfer coefficient. The adjusted heat transfer coefficients are in the range of heat transfer coefficients of convection ovens, as the one used in this work.

Once the heat transfer coefficients are estimated, the model can be used to analyze the current drying conditions of the particular line been studied. The predicted solvent content during the drying process is presented in Figure 3. In the first zone, the drying rate is almost constant, and it is determined by the mass transfer rate from the surface of the liquid layer to the air in the oven. In the second and third zones, the drying rate falls with time, and it is determined by the diffusion of the solvent from the bulk of the liquid film to the surface. The predicted residual solvent is approximately $12\times10^{-5}\, g/cm^2$. The residual solvent is critical to the quality of the adhesives, and it has to be below a specified value. It tends to act as a plasticizer to adhesive, making it too soft and less resistant to stress. The measured residual solvent in a production run is $8\times10^{-5}\, g/cm^2$. Considering all assumptions made, the agreement is good.

Table 1 – Input data to simulation

Pure components	
Specific volume solvent 1 [cm3/g]	1,111
Specific volume solvent 2 [cm3/g]	1,154
Specific volume polymer [cm3/g]	1,111
Molar mass solvent 1 [g/mol]	88,10
Molar mass solvente 2 [g/mol]	92,11
Heat of vaporization solvent 1 [J/g]	364,8
Heat of vaporization solvent 2 [J/g]	361,1

Substrate	
Thickness [cm]	0,005
Density [g/cm3]	1,54
Specific heat [J/g.K]	1,90

Air properties	
Density[g/cm3]	0,001
Specific heat [J/g.K]	1,005

Solution properties	
Average density [g/cm3]	0,9
Average specific heat [J/g.K]	1,83
Interaction factor between polymer and solvent	-2,00
Interaction factor between polymer and solvent	0,30
Interaction factor between solvent 1 and solvent	0,20

Diffusion parameters (Vrentas)	
K_{11}/gama [cm^3/g.K]	0,002
K_{12}/gama [cm^3/g.K]	0,002
K_{13}/gama [cm^3/g.K]	0,001
K_{21}-Tg_1 [K]	-
K_{22}-Tg_2 [K]	-
K_{23}-Tg_3 [K]	-
V_1* [cm^3/g]	0,305
V_2* [cm^3/g]	0,817
V_3* [cm^3/g]	0,983
epsilon$_{13}$	0,843
epsilon$_{23}$	0,580
E_1	-
E_2	-

Initial conditions	
Wet thickness [cm]	0,027
Coating temperature [C]	25

Process parameters	
Zone 1	
Residence time [s]	49
Heat transfer coef. coating side [W/cm^2.K]	0,003
Heat transfer coef. substrate side [W/cm^2.K]	0,003
Air temperature coating side [C]	65
Air temperature substrate side [C]	77
Partial pressure solvent 1 in the oven [Bar]	0
Partial pressure solvent 2 in the oven [Bar]	0
Zone 2	
Residence time [s]	49
Heat transfer coef. coating side [W/cm^2.K]	0,001
Heat transfer coef. substrate side [W/cm^2.K]	0,001

Air temperature coating side [C]	84
Air temperature substrate side [C]	93
Partial pressure solvent 1 in the oven [Bar]	0
Partial pressure solvent 2 in the oven [Bar]	0
Zone 3	
Residence time [s]	49
Heat transfer coef. coating side [W/cm².K]	0,002
Heat transfer coef. substrate side [W/cm².K]	0,002
Air temperature coating side [C]	115
Air temperature substrate side [C]	115
Partial pressure solvent 1 in the oven [Bar]	0
Partial pressure solvent 2 in the oven [Bar]	0

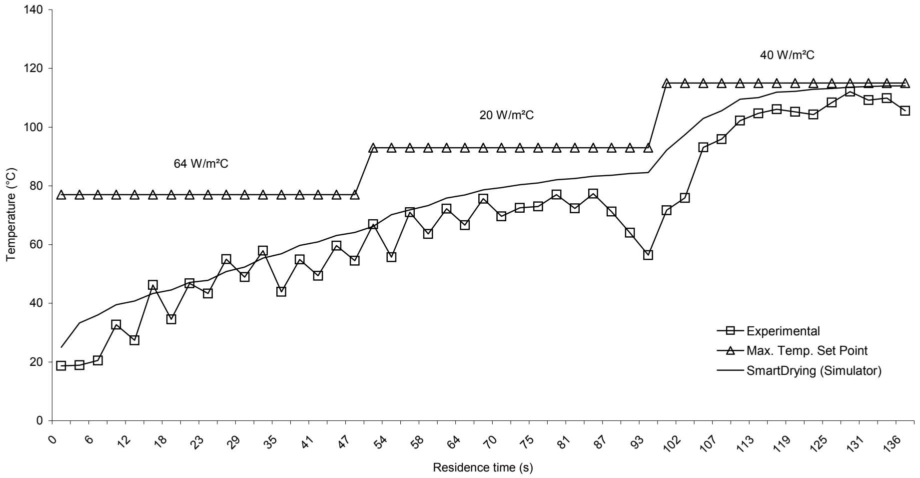

Figure 2 – Adjustment procedure to determine the heat transfer coefficients.

The drying simulator was used to *optimize the process parameters* in order to reduce the residence time of the web in the oven (larger web speed) without changing the residual solvent of the coating. That is equivalent to raising the line speed.

If the residence time is reduced from 45 s to 37 s in each zone (which correspond to a increase in line speed of approximately 40%), and all process variables remained the same, the residual solvent would rise to 28×10^{-5} g/cm². This value is out of the product specifications.

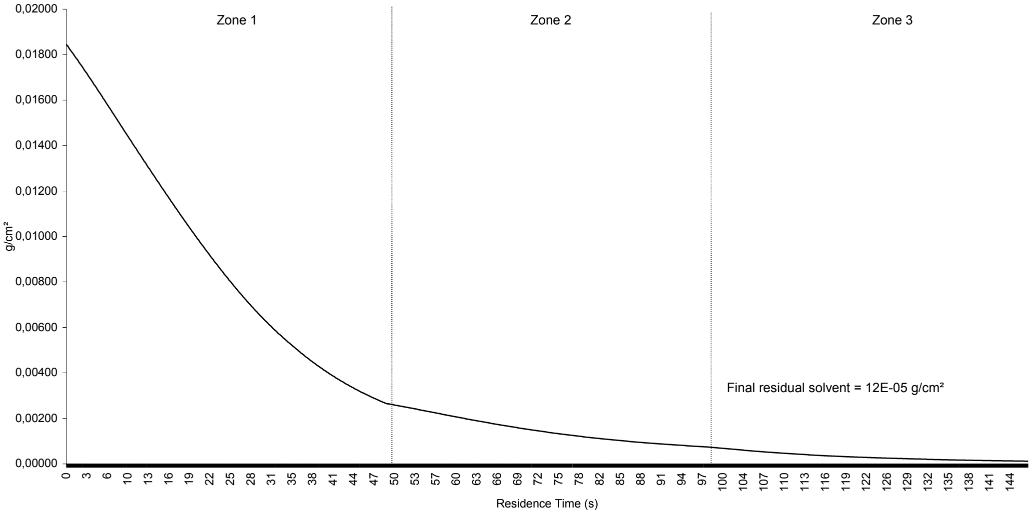

Figure 3 – Solvent content during the drying process.

The goal of the study is to find the possible changes on process parameters that bring back the residual solvent value to the target value. The process conditions that can be adjusted are the temperature and heat transfer coefficient of each zone (air speed). It is important to notice that during most of the drying, the transfer rate is controlled by the diffusion process of the solvent from the bulk of the film to its surface. Therefore, raising the heat transfer coefficient, and consequently the mass transfer coefficient, will not produce any significant change to the process. Moreover, if the solvend adjacent to the liquid surface is dried very fast, the diffusion coefficient in that region is going to be very small (the diffusion coefficient decreases as the concentration falls) and a diffusional barrier is going to be formed, blocking the remaining solvent inside the film. On the other hand, the diffusion coefficient of the solvents can be raised by increasing the liquid temperature. As mentioned before, this has to be done inside certain limits, because if the film temperature is larger than the bubble temperature of the solution, the solvents may boil and bubbles would appear, affecting the quality of the final product. To evaluate the opportunities to increase air temperature in each zone, the coating temperature and the bubble temperature are calculated and presented in Figure 4.

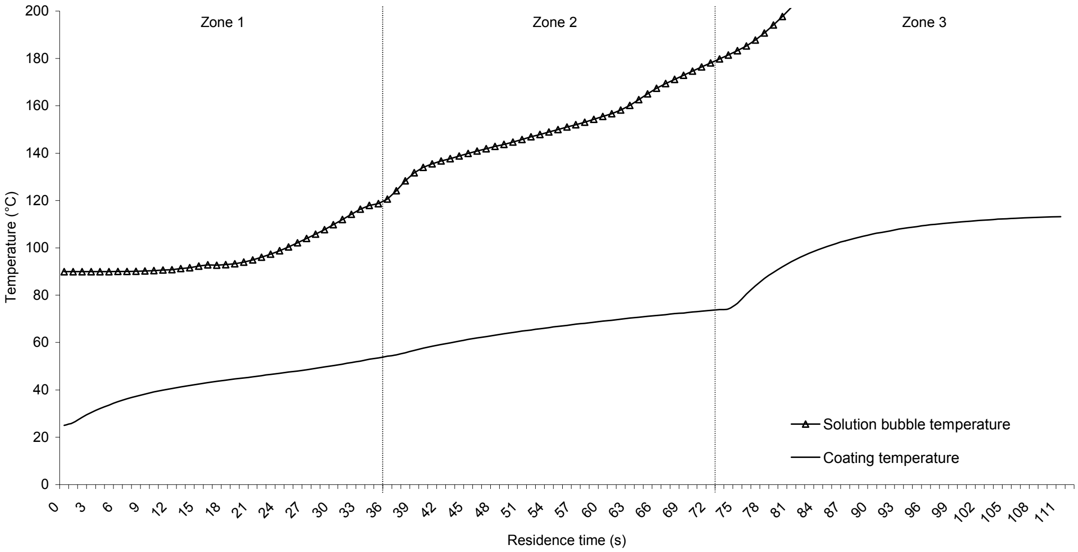

Figure 4 – Solution bubble temperature and coating temperature along the oven.

Because the coating temperature is below the bubble temperature in all the three zones, all the set temperatures were adjusted. It is clear from the plot, that the larger potential to increase the drying rates are in the second and third zones. After several trials, the set temperature of each zone is raised (zone 1, from 65 to 73 ^{o}C; zone 2, from 84 to 105 ^{o}C; and zone 3, from 115 to 140 ^{o}C). The new predicted value of residual solvent is $12x10^{-5}$ g/cm², same value obtained with the previous oven setting and smaller web speed.

This new operating conditions were tested in a real coating line. The validation at production occurred with success in 01/07/2004 at 3M Brazil with a batch size of 1000 square meters. This example illustrated the application of computer-aided simulation resulting in a productivity increase of about 40%, without any capital investment.

4. Conclusion

Drying process of liquids coated on substrates has been modeled with the goal of increasing the productivity of an adhesive coating line. The theory applies to a homogeneous system (phase), solution or emulsion. Several assumptions have been made to derive the set of equations with known parameters. This shows the complexity of the subject and the lack of more general models to get the mutual diffusion coefficients and the interaction parameters in a solution.

Application of a methodology to optimization to an industrial case has shown the usefulness of the computer-aided simulation to improve drying process.

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