TEST-BASED PARAMETER ESTIMATION OF A BENCH-SCALE DISTILLATION COLUMN FOR PREDICTIVE CONTROL

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Abstract: A methodology to determine unmeasurable parameters of a bench-scale distillation column is proposed. This methodology includes time domain analysis, sensitivity analysis and finally a global parametrical estimation. Some of these parameters are: condenser heat transfer coefficient, Murphree efficiency and atmospheric heat-losses. For the estimation of the parameters mentioned above specific experiments are suggested. The aim of this dynamic model is to be useful for predictive control. *Copyright ©2007 IFAC*

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1. INTRODUCTION

Distillation is a very-common separation method. e.g. this method plays an important role in the petrochemical, chemical, pharmaceutical and veterinary industries. The distillation column is the principal tool in this kind of industries. Distillation process constitutes a very significative inversions ratio in chemical refineries around the world. As a result it is very important to obtain mathematical models that represent the dynamical properties of the process. The aim is the faithful representation of the dynamic behavior of the column. Also it is essential to develop efficient monitoring, prediction and control systems for this type of process. In real time control a fundamental model always is advantageous (Yip and Marlin 2004). Because distillation process is complex it is necessary to develop models based in

experimental data. These models should capture relevant information of the process and help to understand its dynamic behavior.

A series of tests are proposed in this paper to characterize a bench-scaled distillation column located of the National Center for Research and Technological Development (CENIDET). Previously (Torres-Ortiz 2005) developed a simplified model while (Valencia-Palomo 2006) applied model predictive control for the condenser cooling flow. (Rivas-Cruz 2006) built a graphic user interphase to manipulate the controllers.

The model described in this work is a fundamental model based in physical principles. This model takes in consideration important physical properties and parameters (geometric and mass transfer effects). These parameters can be identified with the tests proposed for this work.

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This work presents a series of tests for the estimation of these parameters. Section 2 present a description of the bench-scaled distillation column under study. Section 3 describe the dynamic mathematical model and unknown parameters. Section 4 described the different analysis and a series of test applied to bench-scaled distillation column. Section 5 present the general conclusions.

2. CENIDET BENCH-SCALED DISTILLATION COLUMN

The bench-scaled distillation column considered has 10 trays, one condenser and one reboiler. The instrumentation diagram is presented in the Fig. 1
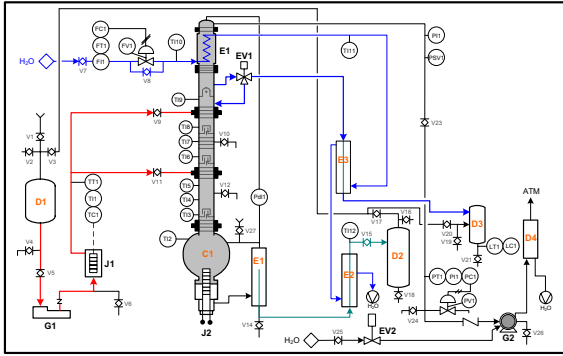

Fig. 1. Instrumentation diagram

The column has temperature sensors in each tray, differential and absolute pressure measurements. The geometric properties that describe the column are showed in Table 1.

Table 1. DPP Geometric Properties of Distillation Column

Property	Value	Unit
Condenser Heat Transfer Area	0.5	m^2
Condenser Volume	1	l
Numbers of Trays	10	-
Feeding Tray Volume	6	l
Weir Height	0.0061	m
Tray pitch	0.11	m
Tray Diameter	0.10	m
Reboiler Volume	6	l

3. DYNAMIC MATHEMATICAL MODEL AND UNKNOWN PARAMETERS

To model binary mixture Methanol-Ethanol the following assumptions are made.

- Vapor-liquid equilibrium with two mixing parameters can be represented by the Peng-Robinson-Strijek-Vera(Stryjek 1986) equation of state.
- Murphree efficiency represents the non ideal effects of mass transfer.

- Hydraulics characteristics can be described as a variation of Francis equation.
- Each plate contains two phases that affect the hydraulic behavior.
- The supplied heat is absorbed completely by the fluid in the boiler.

In this work a recent structure called "spotted box" is used (Astorga-Zaragoza 2006). In this kind of structure the energy and mass balances are known. There are well known parameters (equilibrium relationships), parameters partially know whose uncertainly we would like to reduce(Murphree efficiency), and unknown parameters like times constants and valves coefficient. Some of the known parameters are related to hydrodynamics equations and heat transference parameters, among others.

The conservation equations balances for the p-th tray are:

Mass balance:
$$\frac{dM}{dt} = L_{p+1} + F_p - L_p - V_p + V_{p-1} \quad (1)$$

Composition balance:
$$\frac{d(Mx)}{dt} = L_{p+1}x_{p+1} + F_p z_p - L_p x_p - V_p y_p + V_{p-1}y_{p-1} \quad (2)$$

Energy balance:
$$\frac{d(M_p H_p)}{dt} = L_{p+1}H_{L,p+1} + F_p H_{F,p} - L_p H_{L,p} \quad (3)$$
$$-V_p H_{V,p} + V_{p-1}H_{V,p-1} + Q_p - Q_{Amb}$$

Hydraulics equations:
$$V = \sqrt{\frac{\Delta P \rho_V}{K_{vV}}} \quad (4)$$

$$\Delta P = P_{P+1} + \varphi g h \rho_L - P_P \quad (5)$$

$$L_P = \rho_L \frac{\sqrt{h_{eff}^3}}{K_{vL}} \quad (6)$$

$$h_{eff} = \max(0, h - h_{wer}) \quad (7)$$

$$h = \frac{M}{A \rho_L} \quad (8)$$

3.1 Unknown Parameters

The unknown parameters are:

Param.	Description
τ_M	Mass time constant by tray.
τ_H	Energy time constant by tray.
τ_x	Composition time constant by tray
K_{vL}	Valve liquid coefficient in each tray.
K_{vV}	Valve vapor coefficient in each tray.
Q_{Amb}	Atmospheric heat-losses.
U	Condenser heat transfer coefficient.
η	Murphree efficiency by tray.
φ	Liquid phase affectivity factor which considers effervescence.

The parameters: τ_M, τ_H, τ_x, K_{vL}, K_{vV}, U and η have a direct relation with column geometrical properties. The dynamics of the column depends on these parameters.

Q_{Amb} is related to the atmospheric interaction that surrounds the column. φ depends on effervescence of the mixture. Some of these parameters have strong interaction between them, reason why the following methodology for parameters estimation is proposed.

4. ANALYSIS APPLIED FOR COLUMN CHARACTERIZATION

The System Identification is a technique to obtain in experimental way, the parameters of a model that reproduces with sufficient exactitude is it behavior. An advantage of the models obtained from fundamental principles, is that they are globally valid and therefore adapted to optimization and control tasks. This requires extrapolation outside of the data rank used to fit to the model (Lee 1998).

Our strategy in this work is to design experiments to isolate as much as possible the effect of every parameter. These experiments are similar to the scheme proposed by Luyben (1990) for control analysis: time domain analysis evaluates steady state parameters and time constants, while frequency domain analysis applied with a set of different frequencies evaluates time delays in input-output. Additionally sensitivity analysis helps us to find the range of the parameters and the propagation of the uncertainty to the output variables. Finally a global parameter estimation is applied to the remaining parameters based on the minimization of the sum of the squared errors (predicted- experimental) of the controlled variables with respect to the unknown parameters. The benefit of using these selective tests previous a global parameter estimation is that the problem dimension is reduced, or give at least adequate starting values to tune of the global parameters. The strategy to obtain the unknown parameters in the distillation column is to insolate effects. Based in the steady state, several types of tests are possible.

4.1 Test 1: Time Domain analysis

The linear analysis allows us to obtain the hydraulic and concentrations time constants. When the column is in steady state, if an input step-signal is applied possibly it will arrive at a new steady state. The balance of mass in each plate follows by equation (1).

For heat transfer the main elements are condenser and boiler, then we have:

- $L_C = V_C$ Total reflow thus

$$\frac{dM_B}{dt} = F + L - V - B \qquad (9)$$

also

$$V = \frac{Q_B}{\lambda_B} \qquad (10)$$

$$L = \frac{Q_C}{\lambda_C}$$

$$B = \sqrt{\frac{\rho_B\, g M_B}{K_{vL} A_B}}$$

$$\frac{dM_B}{dt} = F + \frac{Q_C}{\lambda_C} - \frac{Q_B}{\lambda_B} - \sqrt{\frac{\rho_B\, g M_B}{Kv A_B}} \qquad (11)$$

$$\frac{dM_C}{dt} = \frac{Q_B}{\lambda_B} - \frac{Q_C}{\lambda_C} \qquad (12)$$

As noticed earlier, distillation process has a first order dynamics (Skogestad 1987), from a linearized model is possible to obtain the time constants $\frac{dX}{dt} = \lambda x$

4.2 Test 2: Condenser

The condenser acts as an inherent regulator for the column. Since the actual column is made of glass, the operation should avoid vibrations. As a result it operates with an excess of cooling fluid. Thus, cooling water and condensing mixture temperature has little sensitivity.

Since, there is not any temperature sensor for the condenser outlet and his temperature it is not possible to know condenser heat transfer coefficient. But using other physical properties this parameter could be estimated (show Fig. 2).
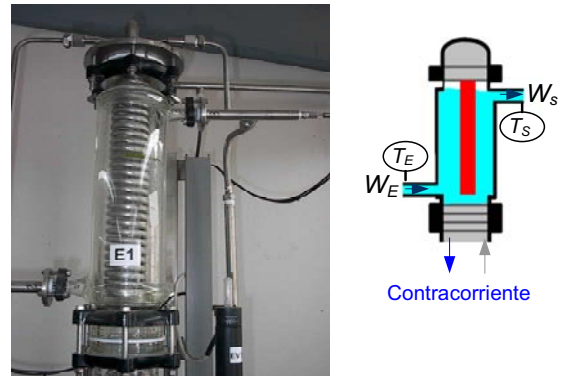

Fig. 2. Condenser heat exchanger

Taking in consideration that cooling flow across the condenser has a temperature variation of $\Delta T = 0.7^oC$ (the operations conditions are the showed in the table 2), the next analysis is possible to estimate the heat transfer coefficient.

$$Q = (H_{LI} - H_{LO})\, W_W \qquad (13)$$

$$Q = (H_{VI} - H_{VO})\, V_C$$

$$V_C = \frac{(H_{LI} - H_{LO})\, W_W}{(H_{VI} - H_{VO})} \qquad (14)$$

Table 2. Operations Conditions

Property	Value	Unit
x_{reb}	0.3339	-
Q_{reb}	100-2500	$Watts$
P_{reb}	96.15	kPa
T_{reb}	67.18	^oC
V_{reb}	1.6862	mol/min
B	0	mol/min
P	87.00	kPa
T	32	^oC
ΔP_{total}	3.63	kPa
T_D	63.02	^oC
L_D	1.41058	mol/min
D	0.35265	mol/min

when:

$$Q = UALm\Delta T \qquad (15)$$

$$U = K_u W^\gamma \qquad (16)$$

This relation at different condenser initial conditions and cooling flows can be estimated U.

$$U = K_u W^\gamma \qquad (17)$$

Another relevant effect is that condenser can present sub-cooling. The quantity $\frac{dX_D}{dQ}$ is small, whereas dT/dQ variation is severe. For this reason a control of the composition based on temperature can deteriorate its performance. (Archambault *et al*, 1973) showed that a methanol-water mixture cooled with water with a copper condenser by a cooling- flow with a reflux relation of 1:6 presents sub-cooling. The distillation column in this work presents a relation of 1:60 thus, sub-cooling is expected. For that reason it is necessary to obtain empirically the heat transference coefficient.

4.3 Test 3: Heat Losses Along the Column

The distillation column is built with a pyrex crystal material. The column is not insulated, thus the glass walls are open to the environment. The heat losses along the column are important. From condenser and boiler heat differences we can evaluate the heat transferred to the environment Q_{Amb}. This heat affects the equations of energy balance. In this context the column body according to Fig. 3 and taking in consideration the operations conditions:

- $L_F = 0$
- $L_B = 0$

The main heat losses are along the column body, thus:

$$L_C H_{L,C} + V_B H_{V,B} = V_C H_V + L_B H_{L,B} + Q_{Amb} \qquad (18)$$

With the Q_{Amb} approximated by Newton's cooling law for the column body.

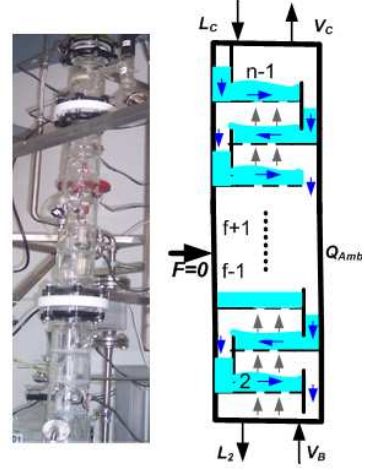$$\frac{dQ_{Amb}}{dt} = \sum_2^{Np-1} K_{Amb} * (T_p - T_{Amb})$$

Fig. 3. Q_{Amb} analysis along the column

using this equation it is possible to obtain the heat losses along the column. These losses differ according the time when the test is carried out, day of year and season.

4.4 Test 4: Reboiler

The reboiler has two tanks (Fig. 4). The smaller tank (s) has an electrical resistance, the big-tank is a mixing-tank (b). In the boiler, the mixture is heated by means of a coil. Since liquid density is
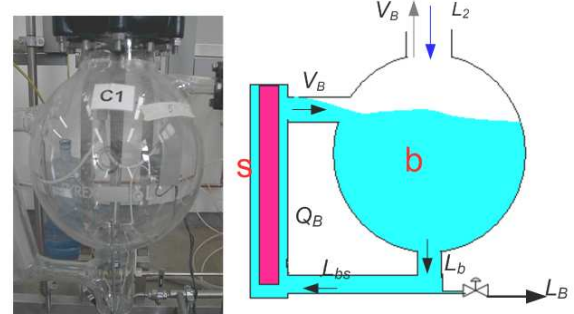

Fig. 4. boiler

greater than vapor density, the following balances are possible:

mass balance in the big-tank:

$$\frac{dM_b}{dt} = L_2 - L_{bs} - L_B \qquad (19)$$

mass balance in small-tank:

$$\frac{dM_s}{dt} = L_{bs} - V_B \qquad (20)$$

by communicating vessels

$$L_{bs} = \frac{\rho_b A_b V_s + \rho_s A_s (L_2 - L_B)}{\rho_s A_s + \rho_b A_b} \qquad (21)$$

Another important point in the modeled reboiler is that the enthalpies are different in every tank. And the energy balances are:

$$M_s \frac{dH_s}{dt} + M_b \frac{dH_b}{dt} = \quad L_2\left(H_2 - H_b\right)$$
$$+L_{bs}\left(H_b - H_s\right)$$
$$-V_B\left(h_{vB} - H_b\right) + Q_B$$
$$(22)$$

This model produces a closer behavior to the observed experimental startup time, since using a single tank produces a startup time of 20 min, while the observed time is 5-7min Fig. 5.
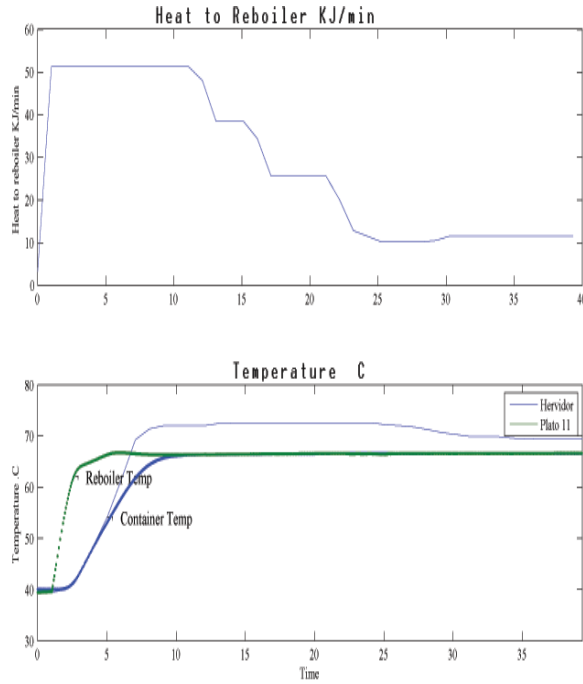

Fig. 5. Changes heat effects in the temperature of the re-boiler, a) (Up) Profile of provided heat, b) Measured temperature and predicted temperatures

When the different forms from particular analysis have been proven, then a global parametric adjustment can be used by model inversion.

4.5 Test 5: Sensitivity Analysis

The sensitivity analysis helps to obtain information about the rank of the parameters and it allows to identify the sensitivity of the bench-scaled column distillation to a change of these. By sensitivity analysis The effect of the uncertainty in some parameters can be reduced, such as the Murphree efficiency. Some recent studies (Gunter 2003) take the Murphree efficiency as 0.7 for this type of column. in Fig. 6 shows the sensitivity of x_D, x_B against this efficiency and the coefficient of heat transference of the condenser. Whereas the sensitivity against the Murphree efficiency has a

linear behavior, the sensitivity of both compositions against the coefficient of heat transference becomes important as the magnitude of this parameter diminishes.
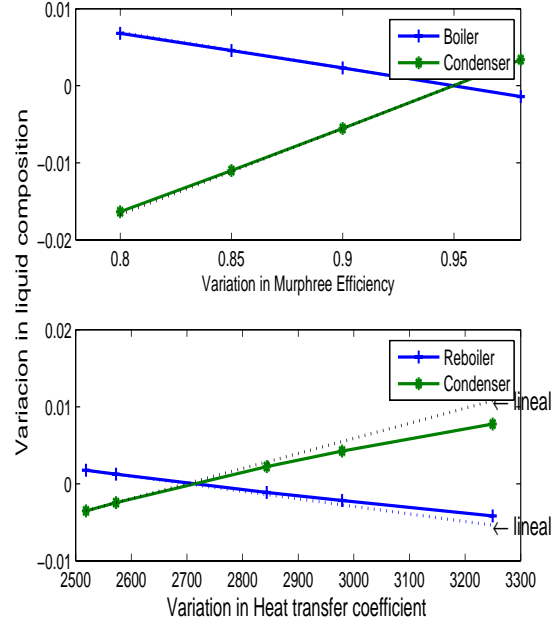

Fig. 6. Composition sensitivity

When the parameters are varying with time, in a system of the form:

$$\frac{dx}{dt} = f(t, x, p) \qquad (23)$$

the parameters sensitivity with relation time in p is given by (Cao 2006):

$$\frac{d}{dp}\left(\frac{dx}{dt}\right) = \frac{df}{dx}\frac{dx}{dp} + \frac{df}{dp}$$
$$\frac{dx(t_0)}{dp} = \frac{dx_0}{dp} \qquad (24)$$

when $\frac{dx(t_0)}{dp}$ is the initial sensitivity.

5. CONCLUSIONS

A set of test to evaluate different parameters required in a dynamic model of a distillation column has been presented. This strategy considers careful modeling of the relevant geometry, and definition of a set of tests. Some of this test are redundant but they help to verify the value of the parameters, thus reducing their uncertainty.

Appendix A. NOTATION

A	Area (m²).
B	Bottom product molar-flow (mol/min).
Cp_c	Heat capacity (kj/molC).
D	Distilled product molar flow (mol/min).
F	Feeding molar flow (mol/min).
F_V	Feeding volumetric flow (mol/min).
W_W	Condenser cooling flow (mol/min)
L	Liquid molar flow (moles/min).
g	Gravity.
H	Enthalpy.(J)
H_L	Liquid Enthalpy.
H_V	Vapor Enthalpy.
K_V	Equilibrium constant.
M	Molecular mass (Kg).
P	Atmospheric pressure (KPa).
Q_{Amb}	Atmospherics heat-losses.
Q_B	Power heating in the boiler (Watts).
T	Temperature (C).
U	Condenser heat-transfer coefficient.
x_{ci}	Liquid molar concentrations.
y_{ci}	Vapor molar concentrations.

Subscript

c	Compound.
p	Tray
b	big
s	small
B	Boiler
C	Condenser
wer	weir
eff	Effective
I	Input
O	Output

Greeks

δ	Exponent of the expression of variation of U
Δ	Increment
λ	Relative gain array element
ρ	Density
ϕ	holdup Effectiveness
φ	Liquid Phase affectivity factor

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