Control Strategies of the 13C Cryogenic Separation Column

Cristina Ioana Pop, Eva Henrietta Dulf

Technical University of Cluj-Napoca, Department of Automatic Control,
26-28 Gh. Baritiu Str., 400027 Cluj-Napoca
(e-mail:Cristina.Pop@aut.utcluj.ro)

Abstract: The main requirement for an efficient carbon isotope separation in a cryogenic distillation column is to maintain the operating parameters at constant levels. This paper uses some simplified nonlinear equations of the carbon isotope separation process that model the isotope transfer rate, the top and bottom column temperatures. The control strategies proposed in this paper are an optimal, linear quadratic regulator that effectively rejects the disturbances in temperature variations, thus allowing only minor variations in the isotope transfer rate and a robust nonlinear controller, based on feedback linearization and a H_∞ controller that ensures the robust stability despite modeling uncertainties.

Keywords: cryogenic isotope separation column, linear quadratic regulator, robust nonlinear controller, nonlinear model

1. INTRODUCTION

The 13 carbon isotope, with a natural abundance of 1.1%, plays an important role in numerous applications, such as the study of human metabolism changes, molecular structure studies, non-invasive respiratory tests, Alzheimer tests, air pollution and global warming effects on plants. The main drawback with the (^{13}C) isotope is related to the difficulty of raising its natural concentration. The main methods available for the separation of (^{13}C) are: gaseous diffusion, chemical exchange, thermal diffusion, laser isotope separation by multi-photon dissociation, chromatography. One of the effective methods for separating the carbon isotopes was developed at the National Institute of Research and Development for Isotopes and Molecular Technologies (INCDTIM) Cluj Napoca and consists of a distillation column that operates at very low temperatures.

The column, with the simplified scheme presented in Fig.1, separates the carbon isotopes based on the cryogenic distillation of pure carbon monoxide, which is fed at a constant flow rate as a gas through the feeding system. At extremely low temperatures (about -192^{o}C), the vapour pressure (P_1) of ($^{12}C^{16}O$) is greater than the pressure (P_2) of ($^{13}C^{16}O$) and the separation coefficient is (Axente, et al., 1994):

$$\frac{P_1^o}{P_2^o} = a \approx 1.007 \qquad (1)$$

Due to the very-low operating temperature, an efficient thermal isolation vacuum jacket is necessary.

Since the "elementary separation ratio" (α) (Axente, et al., 1994) is very close to unity in order to raise the (^{13}C) isotope concentration up to a desired level, a permanent counter-current of the liquid-gaseous phases of the carbon monoxide is created by the main elements of the equipment: the *boiler* in the bottom-side of the column and the *condenser* in the

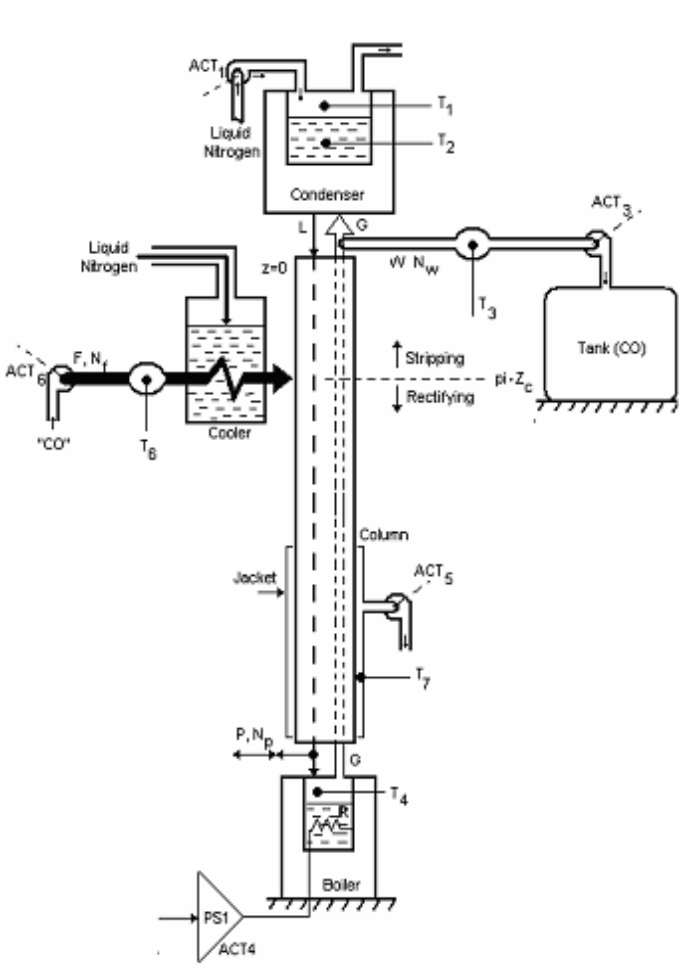

Fig. 1. Simplified cryogenic isotope separation column

top-side. The gaseous carbon monoxide upstream (from boiler and from the feeding system) condenses on the "cold-wall" of the condenser, cooled with liquid nitrogen by atmospheric pressure and falls, in small drops, downstream to the electrically heated boiler. The (^{13}C) isotope, slightly heavier than the predominant (^{12}C) isotope, accumulates in liquid phase and will be extracted as end product at the bottom of the column, while the (^{12}C) component accumulates in vapour phase and will be extracted as waste at the top of the column. The column operates with two zones: the stripping zone, from the feeding point to the top of the column and the enriching (rectifying) zone, in its lower part (Gligan, et.el., 2006).

The characteristics of the carbon cryogenic separation column, presented in what follows, suggest the necessity of modern control strategies. The paper focuses on a two different strategies: optimal control and nonlinear robust control, a problem that the authors have previously discussed (Pop, *et al*., 2009; Dulf, et al., 2009; Dulf, et al., 2008).

2. CONTROL PROBLEMS

The counter current cryogenic distillation column described is a highly complex plant, nonlinear, multivariable, with large time constants that overcomplicate the control solutions. The control strategy is to maintain the column operation parameters constant, by eliminating the effects of the disturbances while keeping the overall system stable, despite the uncertainties that may arise. The main parameters that need to be overseen are:

The liquid nitrogen level in the condenser. The drop of the liquid nitrogen level below a critical value would lead to the impossibility of efficiently condensing the vapour upstream and thus would compromise the entire separation process.

The electrical power supplied to the boiler. High variations would affect the separation by modifying the upward gaseous stream.

The vacuum pressure. Variations in the vacuum pressure bring about the loss of the efficient thermal isolation and cause the increment of the inner column temperature.

The column temperature. Considering the above presented isotope separation column, the objective is to keep the ^{13}C isotope transfer rate ( ) constant. The isotope transfer rate depends on the separation ratio and as seen in (Axente, et al., 1994) the latter is closely related to the temperature. The typical values of the separation factor (α) as a function of the column temperature are given in Table 1.

Table 1. The typical values of the separation factor (α**)**

Temperature	-191.3	-163.1	-205
(α)	1.0069	1.0052	1.012

Thus, by keeping the column temperature constant, the separation factor will also be kept constant and ultimately a properly designed controller would ensure the overall requirement of constant ^{13}C isotope transfer rate.

The paper proposes two control strategies, one that tackles the problem of rejecting temperature disturbances and one that deals with parameter uncertainties. For temperature disturbance rejection the proposed solution is a linear quadratic regulator, while for compensation of the uncertainties the solution consists of a nonlinear robust controller. Both controllers were designed tacking into account the linearized system, obtained in nominal conditions. The closed loop nonlinear system is then simulated considering a temperature disturbance for the optimal controller and a range of possible values for the uncertain parameters in the case of the robust controller. Based on experimental data, the paper considers the equilibrium operating point of the separation column to be: 79K and 77K temperature at the boiler and condenser, respectively. Under this assumption, considering nominal operating conditions (no disturbances), a 25W electrical power supplied to the boiler and around 20cm nitrogen level in the condenser, during 100 hours of column operation the maximum achievable ^{13}C isotope concentration would be approximately 2.8 % at. The aim of the controllers designed is to maintain the isotope transfer rate constant and thus maintain a proper separation process that would ensure the goal of 2.8% at concentration in the isotope of interest, during 100 hours, despite disturbances or uncertainties.

3. NONLINEAR MODEL OF THE ^{13}C ISOTOPE SEPARATION COLUMN

The paper considers a simplified nonlinear model for the cryogenic isotope separation column presented, in which the isotope transfer rate depends on the inner column temperature. The objective is to design a temperature control loop, by manipulating the level of liquid nitrogen in condenser (h_c) and the electrical power at the boiler (P_{el}).

The nonlinear system of the isotope separation process can be modeled as (Dulf, et al., 2008):

$$\begin{pmatrix} \dot{x}_1 \\ \dot{x}_2 \\ \dot{x}_3 \end{pmatrix} = \begin{pmatrix} f_1 \\ f_2 \\ f_3 \end{pmatrix} + \begin{pmatrix} 0 \\ k_{21} \\ 0 \end{pmatrix} u_1 + \begin{pmatrix} 0 \\ 0 \\ k_{31} \end{pmatrix} u_2 \tag{2}$$

with $f_1 = -k_{10}x_1 + k_{11}(x_2 - x_3) - k_{12}(x_2 + x_3)^2$,

$f_2 = -\frac{1}{T_B}x_2$, $f_3 = -\frac{1}{T_C}x_3$, where x_1 is the isotope concentration, x_2 is the temperature in the boiler zone, x_3 is the temperature in the condenser zone T_B and T_C are the boiler and condenser time constants and k_{10}, k_{11}, k_{12}, k_{21} and k_{31} are specific coefficients. The control vector is: $u = [u_1 \ u_2]^T = [P_{el} \ h_c]^T$.

The equations for the temperatures evolutions are approximated by first order transfer functions. The first state variable equation is derived based on experimental data

obtained at INCDTIM and on the assumptions that the vapour pressure difference is proportional to the column temperature and also inversely proportional to the square of the absolute temperature (Vasaru, 1968).

As it has been stated, the separation factor (α) depends upon the vapour pressure difference (equation (1)) and thus the isotope transfer rate would be modelled as depending on the column temperature, resulting in the final equation for the isotope transfer rate:

$$\dot{x}_1 = -k_{10}x_1 + k_{11}(x_2 - x_3) - k_{12}(x_2 + x_3)^2 .$$

To control the temperatures at the boiler and condenser, the nonlinear model of the isotope separation column is firstly linearized around a nominal operating point chosen as: $x_0 = [2.87;79;77]$. The linearized model of the column is:

$$\dot{x}_r = \begin{pmatrix} -k_{10} & k_{11} - 312k_{12} & -k_{11} - 312k_{12} \\ 0 & -\frac{1}{T_B} & 0 \\ 0 & 0 & -\frac{1}{T_C} \end{pmatrix} x_r + \begin{pmatrix} 0 & 0 \\ k_{21} & 0 \\ 0 & k_{31} \end{pmatrix} u \quad (3)$$

The simulations, performed in the programming environment Simulink, Matlab, of the open loop system, without considering any disturbances, are given in Fig. 2, which presents the evolution of the ^{13}C isotope concentration, for both the linear and the nonlinear closed loop system.

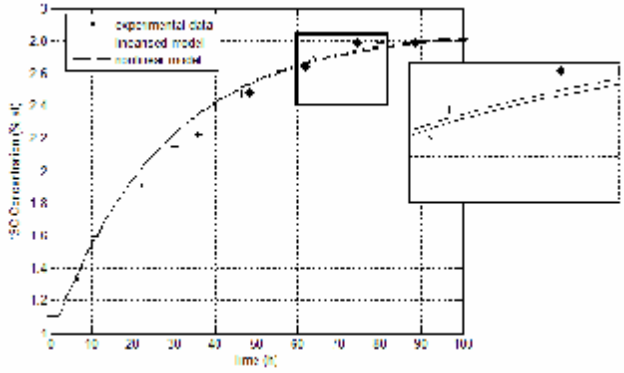

Fig. 2. ^{13}C isotope evolution for linear and nonlinear closed loop system

The simulations show a good agreement between the dynamics of the nonlinear system, given in (2) and the linearized system given in (3), both models behaving according to the experimental data provided at INCDTIM. The simulation values are compared to the experimental data obtained under the same operating parameter values: 77K for the condenser temperature, 79K for the boiler temperature, 25W electrical power and approximately 20cm liquid nitrogen level. .

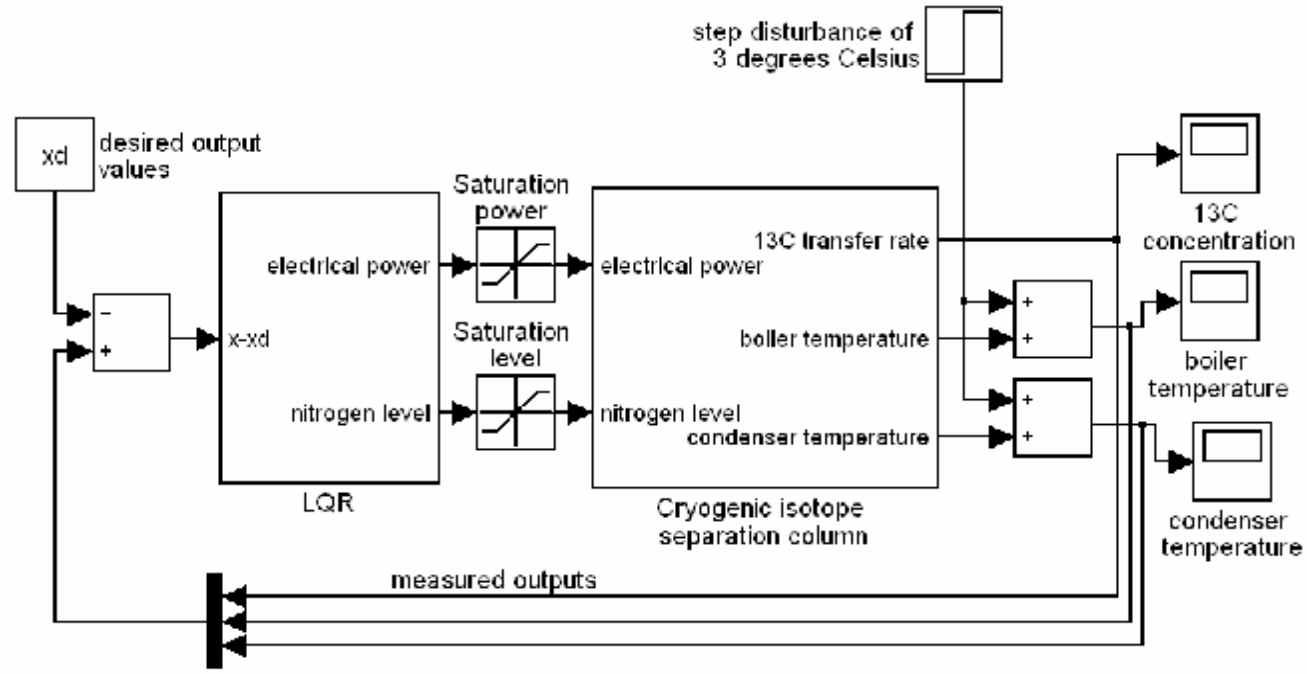

Fig. 3. Closed loop control scheme of the cryogenic isotope separation column using a linear quadratic regulator

4. LINEAR QUADTRATIC REGULATOR FOR TEMPERATURE CONTROL

For the linearized state space representation given in (3), a linear quadratic regulator was designed. The cost function used in this paper is translated from classic disturbance rejection to a tracking problem (Budiyono, et al., 2007). The authors propose a modified cost function as given below:

$$J = \int_0^{\infty} \left[(x - x_d)^T Q (x - x_d) + u^T R u \right] dt \tag{4}$$

with x_d being the desired values for the isotope transfer rate, boiler and condenser temperatures and Q and R, the weighting matrices for the states and the input vector, respectively.

The weighting matrices in (4) were chosen to be:

$$R = \begin{pmatrix} 2.7 & 0 \\ 0 & 1 \end{pmatrix} \tag{5}$$

for specifying input constraints, that is constraints regarding the electrical power supplied to the boiler and the liquid nitrogen level and:

$$Q = \begin{pmatrix} 9 & 0 & 0 \\ 0 & 18000 & 0 \\ 0 & 0 & 11000 \end{pmatrix} \tag{6}$$

for specifying state constraints, meaning restrictions on the isotope transfer rate, the boiler and condenser temperatures.

The weighting matrix for the states was chosen significantly larger than the weighting matrix for the input vector in order to penalize more strictly the evolution of the system states as compared to the evolution of the input signals (Zhang, et al., 2007). The resulting linear quadratic regulator is:

$$K = \begin{bmatrix} 0.029232 & 81.3338 & -5.5744 \cdot 10^{-5} \\ -0.06144 & -1.19073 \cdot 10^{-4} & 1.0462 \cdot 10^{2} \end{bmatrix} \tag{7}$$

The nonlinear closed loop system control scheme is presented in Fig. 3, the linearized closed loop system control scheme being similar.

To test the ability of the optimal controller designed, a fail of the vacuum is considered, acting as a disturbance. The effect of the vacuum loss is modelled as a 3 degrees increment in the boiler and condenser temperature. Such increment, in an open loop case- without the optimal controller considered- implies a modification of the separation coefficient and thus would cause significant changes in the isotope transfer rate.

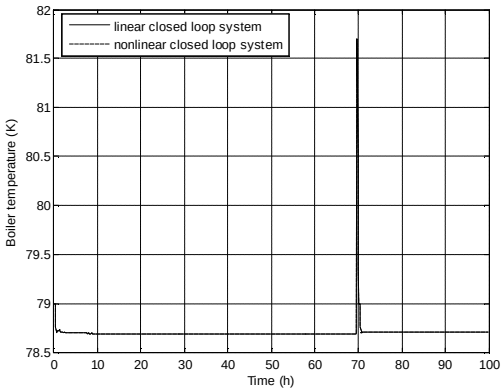

Fig. 4. Evolution of the boiler temperature, considering a vacuum fail

Fig. 4-8 present the closed loop simulations for the linear and nonlinear system, considering the disturbance mentioned previously.

Fig. 4 presents the evolution of the boiler temperature, while Fig. 5 presents the evolution of the condenser temperature.

The input control signals evolutions are given in Fig. 7 and Fig. 8. The electrical power and the liquid nitrogen level are maintained around their nominal operating values.

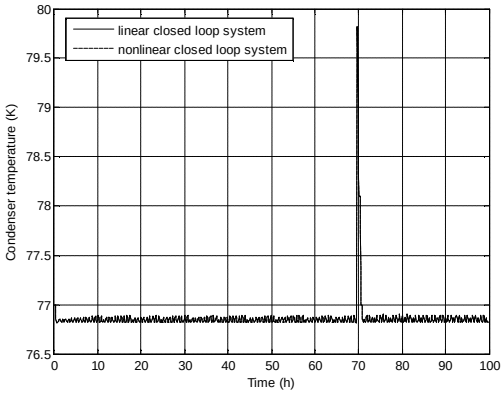

Fig. 5. Evolution of the condenser temperature, considering a vacuum fail

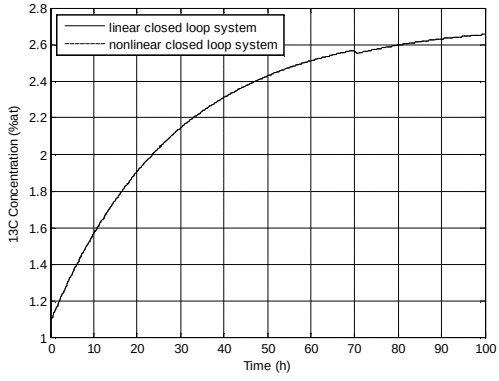

Fig. 6. Evolution of the ^{13}C isotope concentration, considering a vacuum fail

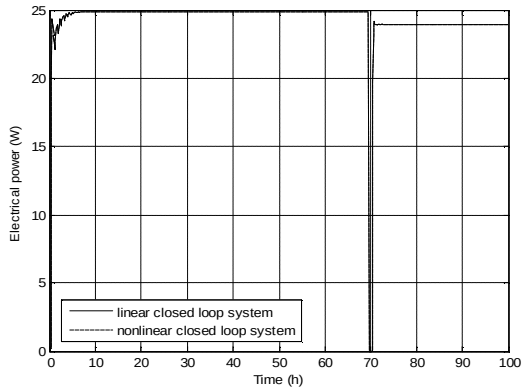

Fig. 7. Evolution of the electrical power supplied to the boiler, considering a vacuum fail

The simulations, given in Fig. 4 and 5, show that the optimal controller can eliminate the disturbances caused by the vacuum fail within less than an hour. However, it cannot maintain the exact set-point values – 79K and 77K, respectively - for the boiler and condenser temperatures. The isotope transfer rate is then slightly altered and a final isotope concentration of approximately 2.8% at cannot be achieved. Due to the disturbance affecting the separation process, the isotope transfer rate is again slightly changed, for a short period of time. Nevertheless, the final isotope concentration value is not significantly altered, the maximum concentration reached being around 2.68% at (Fig. 6).

Fig. 4-8 also show that the designed optimal controller can achieve the same performance when applied to the linearized model or the nonlinear one, since the evolution of the state variables and the control inputs remain similar, with the corresponding curves overlapping.

5. ROBUST NONLINEAR CONTROL OF THE SEPARATION PROCESS

To account for the uncertainties that may exist considering the nonlinear model in (2), the authors propose a control strategy based on an inner feedback linearization method and a robust H_∞ controller. Since the feedback linearization method leads to a Brunovsky form for the linearized system, which has been stated to be a non robust form with a dynamic that is completely different from that of the original system and which is highly vulnerable to uncertainties (Franco, *et al.*, 2006), the authors have chosen a different approach (Franco, et al., 2006, Guillard, et al., 2000) to the classical feedback linearization technique, that leads not to the classical chain of integrators but to the linearized system given in (3).

In classical feedback linearization, we consider a multi input- multi output (MIMO) nonlinear system having n states and m inputs being given by:

$$\dot{x} = f(x) + g(x)u \tag{8}$$

with:

$$g(x)u = \sum_{i=1}^{m} g(x)u_i \tag{9}$$

where $x \in \Re^n$ is the state and $u \in \Re^m$ is the control input vector. We assume that f and g are smooth vector fields defined on $\Re^n$. In what follows, we assume the feedback linearization conditions (Isidori, 1989) are satisfied and that the output of the nonlinear system given in (1) can be chosen as: $y(x) = l(x)$, where $l(x) = [l_1(x).....l_m(x)]$ is a vector formed by functions $l_i(x)$, such that the sum of the relative degrees of each function $l_i(x)$ is:

$$r_1 + r_2 + ... + r_m = n \tag{10}$$

We also assume that the decoupling matrix of the system in (8) is invertible, being given by the following equation:

$$M(x) = \begin{pmatrix} L_{g_1} L_f^{r_1-1} l_1(x) & ..... & L_{g_m} L_f^{r_1-1} l_1(x) \\ . & & . \\ . & & . \\ . & & . \\ L_{g_1} L_f^{r_m-1} l_m(x) & ..... & L_{g_m} L_f^{r_m-1} l_m(x) \end{pmatrix} \tag{11}$$

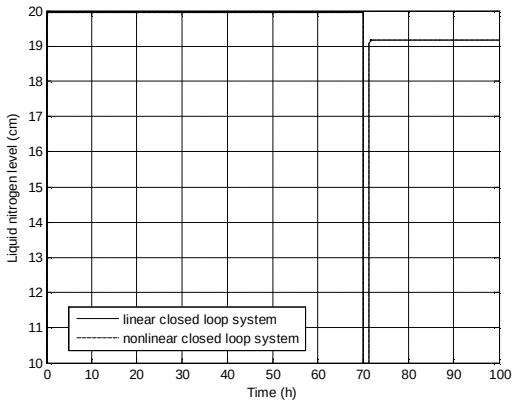

Fig. 8. Evolution of the liquid nitrogen level in condenser, considering a vacuum fail

In a classical approach, the feedback linearization leads to a linearized system of the form:

$$\dot{x}_f = A_f x_f + B_f w \tag{12}$$

with w a linear control. The system in (12) is obtained using a linearizing control law given by:

$$u = a_f(x) + b_f(x)w \tag{13}$$

and a state transformation given by (Franco, *et al.*, 2006):

$$x_f = \Phi_f(x) \tag{14}$$

With

$$\Phi_{f_i}^{T}(x) = [\lambda_i(x) \quad L_f\lambda_i(x) \quad ..... \quad L_f^{r_i-1}\lambda_i(x)] \tag{15}$$

$$\Phi_f^T(x) = [\Phi_{f_1}^T(x) \quad \Phi_{f_2}^T(x) \quad ..... \quad \Phi_{f_m}^T(x)] \tag{16}$$

$$a_f(x) = -M^{-1}(x)[L_f^{r_1} l_1(x).....L_f^{r_m} l_m(x)]^T \tag{17}$$

$$b_f(x) = M^{-1}(x) \tag{18}$$

The feedback linearization used in this paper for the nonlinear system (2) is performed in a neighbourhood of an operating point, x_0. The linearized system is obtained in a similar manner as with the classical approach. The linearizing control law has the form:

$$u = a_r(x) + b_r(x)v \tag{19}$$

with *v* a linear control and a state transformation given by:

$$x_r = \Phi_r(x) \tag{20}$$

The linearized system has the form:

$$\dot{x}_r = A_r x_r + B_r v \tag{21}$$

with $A_r = \partial_x f(x_0)$ and $B_r = g(x_0)$.

In the robust feedback linearization approach the computation steps to determine the linearized system in (14) are as follows (Franco, *et al.*, 2006), (Guillard, *et al.*, 2000): $\Phi_r(x) = T^{-1}\Phi_f(x)$, $L = -M(x_0)\partial_x a_f(x_0)$

$a_r(x) = a_f(x) + b_f(x)LT^{-1}\Phi_f(x)$, $T = \partial_x\Phi_f(x_0)$,

$R = M^{-1}(x_0)$ and $b_r(x) = b_f(x)R^{-1}$.

Thus, by choosing a feedback of the form given in (19) the nonlinear system in (8) will be transformed in its tangent linearized system (21) around the operating point x_0.

In our situation, in order to meet the feedback linearization requirements, the output of the nonlinear system given in (2) can be chosen as: $y(x) = l(x) = [x_1 \quad x_2]^T$. Given such choice for the controlled outputs, the sum of the relative degrees of each function $l_i(x)$ is $r_1 + r_2 = 3$, equal to the number of states. The decoupling matrix of the nonlinear system is:

$$M(s) = \begin{bmatrix} k_{21}\left(k_{11} - 2k_{12}\left(x_2 + x_3\right)\right) & k_{21} \\ -k_{31}\left(k_{11} + 2k_{12}\left(x_2 + x_{34}\right)\right) & 0 \end{bmatrix}^T \tag{22}$$

In a classical approach, the control law for the nonlinear system in (2) would be given by :

$$a_f = \begin{bmatrix} -\dfrac{1}{k_{21}x_2} \\ -\dfrac{k_{10}x_1 + k_{11}x_3 - k_{12}x_2^2 + k_{12}x_3^2}{k_{31}\left(k_{11} + 2k_{12}x_2 + 2k_{12}x_3\right)} \end{bmatrix} \tag{23}$$

$$b_f = \begin{bmatrix} 0 & \dfrac{1}{k_{21}} \\ \dfrac{1}{k_{31}\left(k_{11} + 2k_{12}x_2 + 2k_{12}x_3\right)} & -\dfrac{-k_{11} + 2k_{12}x_2 + 2k_{12}x_3}{k_{31}\left(k_{11} + 2k_{12}x_2 + 2k_{12}x_3\right)} \end{bmatrix} \tag{24}$$

while the state transformation is:

$$\Phi_f(x) = [x_1 \quad f_1 \quad x_2]^T \tag{25}$$

Using the mathematical background presented above, the linearized system obtained is in the Brunovsky form. In order to obtain the linearized system given in (3), the linearizing control law has the form:

$$u = a_r(x) + b_r(x)v \tag{26}$$

with v a linear control and a state transformation given by the mathematical equation, $x_r = \Phi_r(x)$. Some of the matrices required to derive the nonlinear control law are given below (Pop, et al., 2009):

$$R = \begin{bmatrix} 0 & \dfrac{1}{k_{21}} \\ -\dfrac{1}{k_{31}\left(k_{11} + 312k_{12}\right)} & \dfrac{k_{11} - 312k_{12}}{k_{31}\left(k_{11} + 312k_{12}\right)} \end{bmatrix}$$

$$T = \begin{bmatrix} 1 & 0 & 0 \\ -k_{11} & k_{11} - 312k_{12} & -k_{11} - 312k_{12} \\ 0 & 1 & 0 \end{bmatrix}$$

For the linearized system in (3) a robust controller is designed based on the McFarlane-Glover method (Glover, *et al.*, 1989) with loop-shaping that ensures the robust stabilization problem of uncertain linear plants, given by a normalized left coprime factorization. The loop-shaping $P_s(s) = W(s)P(s)$, with *P(s)* the transfer matrix of the linearized system in (3), is done with the weighting matrix *W* chosen as:

$$W = diag\left(\frac{8}{s}, 50, 60\right) \tag{27}$$

The weighting matrix *W* adds an integrator to the first row of the transfer matrix $P_s(s)$, which is related to the isotope concentration, to avoid steady state errors. To the other lines of the transfer matrix, related to the boiler and condenser temperatures, only gains are added. The resulting controller is simple and has a reduced order.

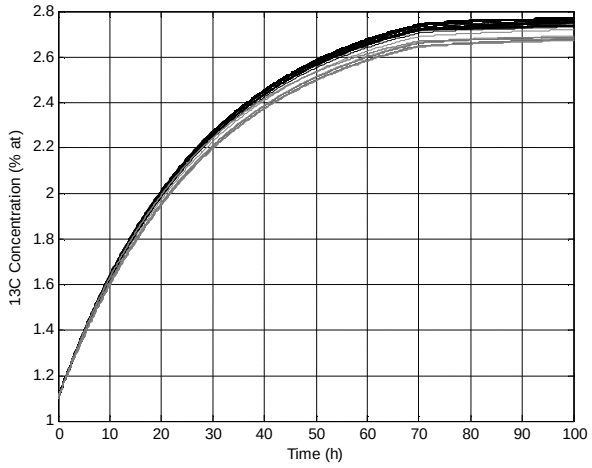

Fig. 9. Evolution of the ^{13}C isotope concentration, considering a vacuum fail

The simulations of the closed loop nonlinear system under nominal parameter values are given in Fig. 9, as well as the simulations considering the uncertain family of nonlinear closed loop system. The variations considered for the uncertain parameters, for simulation purposes, are: k_{10}, k_{11} and k_{12} ranging ±10% from nominal value, T_B and T_C ranging ±15% from nominal value.

The results in Fig. 9 show that with all the considered parameter variations (simulations marked with a black "-" line) the closed loop system controlled by the McFarlane-Glover regulator associated with the feedback linearization presented, behaves as desired, since the performance of the entire family of nonlinear systems tested remains close to the nominal case. However, for uncertainties outside the range considered (simulations marked with grey "-" line) the nonlinear robust controller can maintain the robust stability of the system, but the performances in terms of final isotope concentration are no longer met.

6. CONCLUSIONS

Figures 4 and 5 above show that the optimal controller designed can eliminate the disturbances in temperature variations, while keeping the control inputs around their nominal values. Variations in the electrical power and the liquid nitrogen level are within the acceptable range. All in all, the simulations show that the optimal controller designed for the linearized system still maintains its characteristics when applied to the initial nonlinear model of the column.

A comparison between Fig. 2 and Fig. 6, plotting the evolution of the ^{13}C isotope concentration in no disturbance and active disturbance situations, shows that the variation of the isotope transfer rate is only slightly affected by the disturbances in temperature variations.

As it has been previously demonstrated theoretically through mathematical computations (Guillard, *et al.*, 2000), the results in this paper prove that by combining the method detailed for feedback linearization with a robust linear controller, the robustness properties are kept when simulating the closed loop nonlinear uncertain system. As opposed to a Brunovsky form obtained classically by feedback linearization, the linearized system in (3) will have the same physical meaning as the initial nonlinear one. Thus, the choice of the weighting matrix is easier and will have the same meaning for the linearized system (3) as for the initial nonlinear system (Pop, *et al.*, 2009). The simulations demonstrate that the method described in this paper leads to a nonlinear controller that is robust with respect to model uncertainties.

The two control strategies proposed for the separation process maintain a proper isotope transfer rate under the conditions considered, despite some modelling uncertainties and disturbances. A comparison between Fig. 6 and 9 shows that robust controller can achieve a more desirable isotope transfer rate, than the optimal controller, since it ensures the maximum achievable ^{13}C isotope concentration.

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