MODELLING AND SIMULATION OF THE NON-IDEAL MIXING BEHAVIOUR OF THE CRYSTALLIZATION STAGE IN A SUGAR FACTORY BATCH CRYSTALLIZER

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ABSTRACT

A first principles model of a semi-batch crystallizer is proposed, taking into account the non-ideal mixing behaviour of the process. For this purpose, a twocompartment model is developed in order to predict the evolution of the variables related to the sugar crystallization in a more realistic way. The model is based on mass and energy balance equations. It also includes population balance equations to keep track of the sugar crystal size distribution (CSD). Finally, the model is adjusted and validated with real factory data.

Keywords: Sugar crystallization; Dynamic modelling; Crystal Size Distribution (CSD); Process simulation

1. INTRODUCTION

The development of a suitable process simulation model is a difficult engineering task, due to the complexity of industrial processes, the uncertainty related to them and, in some cases, the lack of information about some important variables.

In the case of crystallization processes, one of the challenges is associated with sensor limitations, considering that super-saturation, for instance, is not directly measured. In this context, the design of model-based soft-sensors plays an important role (Damour et al, 2010).

This paper presents an extension of a previously developed model of a beet sugar crystallizer (Mazaeda, 2010). The model presented in this work considered perfect mixing, but this assumption is rarely true for an industrial-scale crystallizer. In fact, a significant source of uncertainty in industrial crystallizers is associated with mixing (Braatz, 2002). For this reason, the new approach in this contribution is oriented to capture the non-ideal mixing behaviour of the massecuite that takes place in actual industrial practice, in order to improve the fidelity of the model.

Besides, this model is adjusted and validated, in a practical operation range, from data obtained from a sugar factory in Spain. The aim of this model will be to perform an optimal control of the process.

This paper is organized as follows: Section 2 focuses on the description of the process. Then, the

improved model is presented in Section 3. The results obtained using the model comparing them against industrial data are presented in Section 4, ending with some conclusions in Section 5.

2. SUGAR CRYSTALLIZATION PROCESS

Crystallization process can be considered as the key stage of sugar production (Van der Poel, 1998). It takes place in evaporative crystallizers, called "tachas", operating in semi-batch mode, where each cycle is divided into several sequential stages.

As many batch processes, sugar crystallization is controlled by a recipe. In this paper, we are going to make reference to the specific way of controlling the strike of a particular factory in Spain.

Initially, a reference pressure of 0.20-0.25 bar is established in the syrup chamber in order to reduce the boiling point, thus preventing the thermal decomposition of the sucrose.

Once the vacuum pressure is established, the pan's chamber is filled with the sucrose technical solution, up to a pre-specified level. This initial charge of liquor is concentrated in the next stage, by evaporating the amount of water needed to achieve the required sucrose super-saturation, which is the driving force of the crystallization process. The energy required for evaporation is delivered by means of a heat exchanger or calandria, which uses, as heating source, saturated steam from other sections of the factory.

Once the specified concentration or Brix is reached, it is assumed that the correct super-saturation has been obtained, so that it is safe to introduce a small amount of seed crystals, with an average size of about 10 micrometres.

Then, the dissolved sucrose begins to crystallize around this initial population of crystals, resulting in their growth in the subsequent boiling stage. The crystallization will then proceed smoothly along this latter stage, the most important and lengthiest of the batch cycle, provided that the super-saturation of the solution is kept at correct values. Super-saturation, which is defined as the ratio of the actual concentration of sucrose to the one defining the solubility, is the key variable governing the growing of the grains. It depends, obviously, on the concentration and temperature, but also on the purity of the mother liquor. The objective of the boiling stage is to deliver a uniform population of sugar grains with an average size of around 550 μ m and a coefficient of variation (CV) that should, typically, be below 30%. This can be accomplished only if the mass, size and uniformity of the initial seed is carefully considered and the supersaturation of the mother liquor is kept in the meta-stable zone of the Ostwald-Miers Diagram (see Fig. 5), avoiding the spontaneous nucleation (formation of new crystals) or the dissolution of the existing grains that would occur otherwise.

But super-saturation is not a directly measured variable since it has to be inferred from other measurements. In the industrial crystallizer here taken as reference, the crystal growing process in conducted by carefully balancing the rate of water evaporation with the feeding of the right amount of syrup to compensate the sucrose that migrates from the mother liquor to the crystal phase (Mazaeda, 2010). This is achieved by controlling the Brix or concentration of the massecuite, implementing a so called boiling curve that relates the desired evolution of Brix to the level attained by the mass inside the pan, which is thus taken as a measure of the evolution of the process. The manipulated variable is the standard liquor inlet flow, through the valve that regulates this stream (see Fig. 1).



Figure 1: Crystallizer control scheme

The Brix vs. level curve is completely parameterized by specifying its endpoints and a parameter α that specifies its shape as shown in Figure 2. The curve is adjusted by the operator taking in consideration the purity of the feeding syrup.

The boiling phase is concluded when the level of the strike in the pan reaches the value that determines its maximum capacity.

After the growth phase, a tightening stage follows, with the purpose of increasing the concentration of the massecuite in preparation for its discharge.

Finally, the vacuum is broken and the massecuite is evacuated to the downstream centrifuges, where the sugar grain, which is the commercial product, gets separated from the mother liquor in which it was embedded.



Figure 2: Boiling Curve

3. THE MODEL

The concept of a mixed-suspension, mixed-product removal (MSMPR) reactor has been widely adopted for the modelling of many industrial processes. However, there are some cases where deviation from the ideal mixing can be significant (Lie-Ding and Tsan-Sheng, 2005). Frequently the MSMPR supposition is more difficult to justify for continuous processes: it is the one of two limiting simplifying cases: perfectly mixed or plug flow.

In general, it is common to assume a perfectly mixed volume for batch processes: the discharge is delayed to the end of the process and the contents of the agitated reactor have the same residence time. However in our case, for its particular characteristics, we are assuming that a two volume representation would best capture the problem.

In the sugar crystallization batch process, the assumption of a perfectly mixed mass is quite reasonable in the first minutes of the boiling stage, when the agitation produced by the mechanical stirrer and by the bubbles of the evaporated water flowing up through the volume of massecuite, is high enough to ensure a high degree of homogeneity. However, the progression of the process causes a considerable decrease of the agitation effect (see Fig. 3). Some studies have been carried out about the influence of the circulation in vacuum pans (Echeverri, 2005). The causes are multiple: the level of the mass keeps increasing and with it the pressure and thus the temperature in the neighborhood of the calandria (it is no longer realistic to assume the temperature is everywhere the same). A higher temperature of the mass in direct contact with the calandria implies a smaller temperature difference between the two sides, and thus a lesser heat flux and with it, less agitation provoked by the bubbles. Furthermore, we must take into account the increased viscosity of the massecuite due to the crystals growth, one of the main facts that explain the deterioration of the circulation intensity.

To tackle this issue, a two compartment model is proposed. The total volume of massecuite is divided into two regions:

The volume when the boiling phase is started (V1), which remains constant, essentially

corresponds to the region around the calandria where the circulation is somehow better due to the presence of the mechanical stirrer.

• The variable volume up to the final level (V2, as shown in Figure 3) is going to increase to absorb the syrup which is introduced after the initial charge.



Figure 3. Scheme of the circulation inside the pan

The V1 compartment is in direct contact with the calandria and so it receives the heat contribution from the heat exchanger. The V2 volume, on its turn, is heated only as the result of the convective interchange of mass between the two compartments (F1 and F2 as shown in Fig. 4).

This interchange flow (F1 = F2) decreases as the strike proceeds, and this behaviour models the aforementioned deterioration of massecuite circulation. The flow between compartments is then parameterized by a factor β_{NI} , which is defined as the ratio between the interchanged flow and the flow of standard liquor as follows,

$$\beta_{NI} = \frac{F_i}{F_{input}} = b_0 - b_1 \cdot h_2 \tag{1}$$

where h_2 denotes the height of massecuite in V2, while b_0 and b_1 are adjustable coefficients.



Figure 4: Schematic diagram of the model

The dynamic mass and energy balances are formulated for the two considered crystallization volumes, as well as the growth and nucleation rates. The population balance equation (PBE) is solved by discretising the original partial differential equation which allows to keep track of the shape of the crystal size distribution (CSD). For the sake of comparison, the PBE is also solved with the more computationally efficient method of moments (Hulburt and Katz, 1964). The model equations can be reviewed in the works of Mazaeda (2010) for a single compartment.

3.1. Concepts related to the sugar production

Some of the most common concepts used in the field of sugar industry are:

- The dry substance content or Brix
- The purity
- The super-saturation
- Nucleation and growth kinetics

The Brix is a measure of total soluble solids content in sugar juice or syrup, defined as follows:

$$Brix = \frac{m_{sucrose} + m_{imp}}{m_{sucrose} + m_{imp} + m_{water}} \cdot 100$$
(2)

The purity is the sucrose content as a percent of the dry substance or dissolved solids content:

$$Purity = \frac{m_{sucrose}}{m_{sucrose} + m_{imp}} \cdot 100$$
(3)

Note that when these concepts are referred to the massecuite, the mass of crystals must be included in the equations (2) and (3):

$$Brix_{mc} = \frac{m_{sucrose} + m_{imp} + m_{crystal}}{m_{sucrose} + m_{imp} + m_{water} + m_{crystal}} \cdot 100$$
(4)

$$Purity_{mc} = \frac{m_{sucrose} + m_{crystal}}{m_{sucrose} + m_{imp} + m_{crystal}} \cdot 100$$
(5)

The super-saturation can be defined as the degree to which the sucrose content in the solution is greater than the sucrose content in a saturated solution, expressed as

$$s = \frac{q_{suc/W}}{q_{suc/W,sat} \cdot y_{sat}}$$
(6)

where y_{sat} denotes the super-saturation coefficient, which takes into account the presence of impurities in the syrup. Its value is estimated as follows:

$$y_{sat} = A \cdot q_{ns/W} + B + (1 - B) \cdot \exp(-C \cdot q_{ns/W})$$
(7)

where $q_{ns/W}$ is the concentration of non-sucrose substances, while A, B and C are adjusted parameters according to the relative amount and the kind of specific impurities whose values are quite uncertain. Nevertheless, the values suggested by Van der Poel (1998) are A = 0.178; B = 0.820; C = 2.1.

As shown in Figure 5, over the solubility line, where s = 1, crystallization takes place. For high values of super-saturation, the labile region is reached and the generation of new small crystals happens. This phenomenon is known as secondary nucleation.



Figure 5: Ostwald-Miers diagram

However, the frontier between the metastable region and the labile zone is not clear. In other words, nucleation is not modelled to be completely nonexistent all along the metastable zone width, but it will increase exponentially once super-saturation reaches the labile zone.

Several models for growth and nucleation kinetics have been developed (Garside, 1984; Nyvlt et al., 1985; Rawlings et al., 1993). The expression used for nucleation here is the one that follows:

$$B = \frac{k_b}{\rho_{ml}} \cdot (S-1)^{n_b} \tag{8}$$

where k_b and n_b are adjustable parameters and ρ_{ml} the density of the mother liquor. Anyway, as said before, because of the high uncertainty related to this phenomenon, it is a complex task to model it accurately.

With regard to the growth rate, the expression used is presented below

$$R_{g} = \frac{\Delta C_{eff}}{\frac{1}{\beta} + \frac{1}{\Gamma_{kd0}} \frac{C_{sal,i}}{\Delta C_{eff}} + \frac{1}{k_{R}}}$$
(9)

where ΔC_{eff} denotes the effective difference of sucrose concentration, while β and k_R are mass transfer coefficients (Ekelhof & Schliephake, 1995).

3.2. Mass balances

The dynamic mass and energy balances applicable to the boiling phase are formulated as usual, taking into account, in this case, the two volumes, as shown previously in Figure 4.

The mass balances are shown below.

Compartment V1

$$\frac{dM_1}{dt} = M_{syrup} - W_{evap\ 1} + F_2 - F_1 - F_3 \tag{10}$$

$$\frac{dM_{suc1}}{dt} = M_{syrup} \cdot w_{syrup}^{suc} + F_2 \cdot w_2^{suc} - [F_1 + F_3] \cdot w_1^{suc}$$
(11)
$$-W_{c1}$$

$$\frac{dM_{c1}}{dt} = W_{c1} + F_2 \cdot w_2^c - [F_1 + F_3] \cdot w_1^c$$
(12)

$$\frac{dM_{imp_1}}{dt} = M_{syrup} \cdot w_{syrup}^{imp} + F_2 \cdot w_2^{imp} - [F_1 + F_3] \cdot w_1^{imp} \quad (13)$$

$$M_{w_1} = M_1 - M_{suc_1} - M_{c_1} - M_{imp_1}$$
(14)

• Compartment V2

$$\frac{dM_2}{dt} = F_3 - W_{evap\ 2} + F_1 - F_2 \tag{15}$$

$$\frac{dM_{suc2}}{dt} = [F_3 + F_1] \cdot w_1^{suc} - F_2 \cdot w_2^{suc} - W_{c2}$$
(16)

$$\frac{dM_{c2}}{dt} = W_{c2} + [F_3 + F_1] \cdot w_1^c - F_2 \cdot w_2^c$$
(17)

$$\frac{dM_{imp_2}}{dt} = [F_1 + F_3] \cdot w_1^{imp} - F_2 \cdot w_2^{imp}$$
(18)

$$M_{w2} = M_2 - M_{suc2} - M_{c2} - M_{imp_2}$$
(19)

The global mass balances to the total amount of massecuite in V1 and V2 are performed in the equations (10) and (15) respectively, while the individual balances are shown in the equations (11) and (16) to the sucrose, (12) and (17) to the crystals and (13) and (18) to the impurities. The mass of water is calculated algebraically by means of (14) and (19) for both considered volumes.

The terms W _{evap 1} and W _{evap 2} (from the equations 10 and 15 respectively) denote the flow of evaporating water that leaves the solution phase. The mass balance to the steam space is shown in (20):

$$\frac{dM_{steam}}{dt} = W_{evap\ 1} + W_{evap\ 2} - W_{steam\ out}$$
(20)

On the other hand, the terms W $_{c i}$ represent the mass flow of sucrose that leaves the mother liquor to be incorporated to the growing crystals:

$$W_{cris_{i}} = R_{g_{i}} \cdot A_{c_{i}}; \ i \in (1, 2)$$
(21)

where A_c represents the total surface of the crystals and R_g the growth rate, previously mentioned (equation 9).

3.3. Energy balances

To formulate the dynamic energy balances to the massecuite and to the steam space, it is assumed the thermal equilibrium between the two phases, which implies that the existing pressure in the steam space P_{cham} is equal to the equilibrium pressure. Nevertheless, for the compartment V1, the hydrostatic head of the column on top (V2) is taken into account as follows:

$$P_{eq_1} = P_{cham} + \rho \cdot g \cdot h_2 \tag{22}$$

The pressure in the steam space of the crystallizer is calculated using the ideal gas law:

$$P_{cham} = \frac{\left[\frac{M_{steam}}{MM_{H2O}}\right] \cdot R \cdot T_{cham}}{V_{cham}}$$
(23)

As V_{cham} decreases along the process, it is estimated as follows:

$$V_{cham} = V_{tot} - V_{mc} = V_{tot} - \frac{M_{mc}}{\rho_{mc}}$$
(24)

where V_{mc} denotes the volume occupied by the massecuite and V_{tot} is the total volume of the crystallizer.

The energy balances to both considered volumes are expressed in (25) and (26), where H denotes the enthalpy:

$$\frac{dH_{1}}{dt} = \frac{\begin{bmatrix} M_{syrup} \cdot H_{syrup} + F_{2} \cdot H_{2} - [F_{1} + F_{3}] \cdot H_{1} \\ -W_{evap \ 1} \cdot H_{vap \ 1} + Q_{cal} - H_{1} \cdot \frac{dM_{1}}{dt} \end{bmatrix}}{M_{1}}$$
(25)

$$\frac{dH_2}{dt} = \frac{\begin{bmatrix} [F_1 + F_3] \cdot H_1 - F_2 \cdot H_2 \\ -W_{evap \ 2} \cdot H_{vap \ 2} - H_2 \cdot \frac{dM_2}{dt} \end{bmatrix}}{M_2}$$
(26)

The temperature of the massecuite is calculated as a function of the enthalpy, the Brix of the massecuite and the Brix and purity of the mother liquor (Bubnik et al., 1995).

Note that, as represented in Figure 4, it is assumed that only the compartment V1 is heated from the calandria (Q_{cal} in the equation 25) and heat losses are neglected. This flow of heat is calculated using the expression that follows

$$Q_{cal} = U \cdot A_{cal} \cdot \left(T_{cal} - T_{mc1}\right) \tag{27}$$

where T_{cal} is the temperature of the saturated steam circulating through the shell of the calandria, T_{mc1} is the temperature of the massecuite in V1, A_{cal} denotes the heating surface and U represents the overall heat transfer coefficient, estimated as:

$$U = \frac{K_U}{Brix_{mc1}}$$
(28)

The heat transfer coefficient is modelled to drop along the boiling phase, due to the increase of the Brix of the massecuite and, therefore, of the viscosity.

3.4. Population Balance Equation (PBE)

Considering a single growth direction with one characteristic length L, the Crystal Size Distribution (CSD) is given by the population balance equation (Nagy, 2009) with the form

$$\frac{\partial n}{\partial t} = \frac{Q_e n_e - Q_s n - n \frac{dV}{dt}}{V} - \frac{\partial (G(L, x) \cdot n)}{\partial L} + B$$
(29)

where the first term of the right hand side of (29) represents the convective term, the second is the growth rate of the characteristic size, and B and D denote the spontaneous nucleation and the dissolution. The growth rate G can be put into correspondence with the mass transfer rate R_g by means of (30), where k_s and k_v are the surface and the volume shape factors of the particles.

$$G = \frac{k_s}{3 \cdot k_v \cdot \rho_c} \cdot R_g \tag{30}$$

As (29) is a partial differential equation, it is necessary to convert it to a set of ODEs by the method of moments (Hulburt and Katz, 1964):

$$\frac{d\mu_k}{dt} = \frac{Q_e \mu_k^e - Q_s \mu_k - \frac{dV}{dt} \mu_k}{V} + k \cdot G \cdot \mu_{k-1} + B \cdot L_{nuc}^k \quad (31)$$

Note that the zero moment is proportional to the total number of particles per unit of volume, while the first, second and third moments are proportional to the aggregate values of length, area and volume of the particles per unit of volume, respectively (Mazaeda, 2007). From the moments, the mass based average size and the coefficient of variation of the crystals are estimated as follows (Randolph & Larson, 1988).

$$\bar{L} = \frac{\mu_4}{\mu_3} \tag{32}$$

$$CV = \sqrt{\frac{\mu_5 \mu_3}{(\mu_4)^2} - 1}$$
(33)

Note that the equations (29)-(33) are solved for both volumes.

4. MODEL ADJUSTMENT TO REAL DATA

The model has been adjusted and validated from data obtained from a sugar factory in Spain.

An optimisation problem is formulated to minimise the error between the real measured data and those predicted by the model, selecting the most sensitive parameters as decision variables. Among the large number of parameters, the ones that are adjusted, bounded in a reasonable interval, are:

- The super-saturation coefficients (A, B and C from the equation 7), used to take into account the effect of the relative amount of impurities on the super-saturation value (Bubnik et al, 1995).
- The spontaneous nucleation parameters (k_b and n_b from the equation 8), which relate the supersaturation to the nucleation rate.
- A factor used to estimate the value of the overall heat transfer coefficient along the process (k_U from the equation 28).
- The mass and the population characteristics of the seed, which are important sources of uncertainty.

The less sensitive parameters, that are not adjusted, have been set according to the typical values compiled in technical literature.

Measurements obtained directly from factory floor instrumentation include pressure in the pan, pressure of the steam in the calandria, temperature, level and Brix of the massecuite. There are also laboratory results, gathered at a much lower frequency, for the purity of the feeding syrup and the characteristics of the sugar grain population: average size and CV.

The set point of the pressure controllers are considered as inputs of the system, as well as the Brix and the purity of the standard liquor; while the temperature, level and Brix of the massecuite, as well as the final conditions of the crystals (average size, coefficient of variation and crystal production) are considered as the outputs to adjust.

Thus, the optimisation problem is defined in (34) and solved using a reduced SQP algorithm (Gill et al., 2002).

$$\begin{split} \min_{p} J &= \sum \gamma_{l} \cdot (level(t)_{mod} - level(t)_{plant})^{2} + \\ \gamma_{B} \cdot (Brix_{mc}(t)_{mod} - Brix_{mc}(t)_{plant})^{2} + \\ \gamma_{T} \cdot (T_{mc}(t)_{mod} - T_{mc}(t)_{plant})^{2} + \\ \gamma_{L} \cdot (\bar{L}_{mod} - \bar{L}_{plant})^{2} + \\ \gamma_{CV} \cdot (CV_{mod} - CV_{plant})^{2} + \\ \gamma_{CC} \cdot (CC_{mod} - CC_{plant})^{2} + \\ \gamma_{CC} \cdot (CC_{p$$

Crystallizer geometry

$$p_{low} \le p \le p_{upp} \tag{34}$$

The cost function is the sum of the errors between the measured and the model-predicted data weighted with a factor γ , which is greater for the less uncertain variables. The vector of parameters to be estimated is denoted as p, bounded in a reasonable interval.

The modelling and simulation software used to perform the simulation of the model is EcosimPro, capable of modelling dynamic systems represented by differential-algebraic equations (DAE) or ordinarydifferential equations (ODE) and discrete events (EcosimPro / PROOSIS by EA Internacional).

Table 1: Values of the parameters after the optimisation

Parameters				
Super-saturation coefficient parameters	А	0.12		
	В	1.07		
	С	2.08		
Nucleation parameters	n _b	5.0		
	k _b	$2.0^{-}10^{12}$		
Gain of the overall heat transfer coefficient	K_{U}	5.45 ⁻ 10 ⁴		
Mass and the population characteristics of the seed	M _{c seed} (kg)	0.18		
	L_{seed} (µm)	15		
	CV _{seed} (%)	25		

The Figures 6 and 7 show the crystallization stage model identification and validation respectively, for one of the factory crystallizers' taken as reference. Due to the high uncertainty and the inherent process nonlinearities, not forgetting the unmeasured disturbances and the limited available data, a perfect fit is impossible. Nevertheless, a robust model is validated in a practical operation range.





Figure 6: Model adjustment. (a) Pressures (calandria and vacuum); (b) Pan level; (c) Brix of the massecuite; (d) Temperature of the massecuite; (e) Crystal average size; (f) CSD.

Table 3: Final average size, CV and crystal content

Crystal average size and CV		
L average	CV	CC
529 µm	21.8 %	50.9%

The Figure 6-(a) shows the evolution of the pressure in the calandria (red line) and the vacuum established in the crystallizer (blue line), which are inputs to the model. These pressures are controlled by means of PIDs. The profile of set points is determined by the recipe based sequential controller, as parameter that can be modified by the operators.

The evolution of level and Brix of massecuite are shown in the Figures 6-(b) and 6-(c) respectively, where the blue line represents the measured data and the green one the values predicted by the model. The parameter of the boiling curve α was set to 1.2.

In Figure 6-(d) the dispersion between the temperature of the massecuite in the compartment V1 (where heat is directly received from the calandria) and the temperature in V2 can be observed. In this way, the average temperature (black line) fits well to the measured temperature data (blue line), which depend on the particular position where it is placed.

The evolution of the crystal size in V1, V2, as well as the average size, is shown in Figure 6-(e), where the dispersion between the grain size in V1 and V2 grows as the boiling stage proceeds.

Finally, the crystal size distribution is represented in Figure 6-(f), where we can see that the final average crystal size is near $500 - 550 \ \mu m$ and coefficient of variation is between 20 and 30% (Table 3).

|--|

Brix and purity of standard liquor	
Brix = 67.8	Purity = 95.2%

In this case, as the Brix of the standard liquor is lower than before (see Tables 3 and 4), logically, the duration of the boiling stage is larger (almost 2 hours). Concerning the rest of the inputs, their values are very similar to the ones used in the previous batch.





Figure 7: Model validation. (a) Pressures (calandria and vacuum); (b) Pan level; (c) Brix of the massecuite; (d) Temperature of the massecuite; (e) Crystal average size; (f) CSD.

Table 5: Final average size, CV and CC (validation)

Crystal average size and CV		
L average	CV	CC
552 µm	21.2 %	51.1%

5. CONCLUSIONS

A two-MSMPR-crystallizers-with-interchange model has been developed to capture the deviations from ideal behaviour caused by the existence of non-homogeneous agitated regions in a sugar batch crystallizer, in order to improve the fidelity of the previously developed model (Mazaeda, 2010). The model adjustment and validation are presented in the previous section, where a good fit is achieved, despite the unmeasured disturbances and the limited available data.

ACKNOWLEDGMENTS

The authors would like to express their gratitude to ACOR (Spain) for their financial support and technical assistance, as well as projects DPI2012-37859 of Spanish MINECO and the EU FP7 NoE HYCON2.

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