Online Dynamic Optimization of Feed Flowrate and Temperature Trajectories in Semi Batch Esterification Reactor

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Abstract— **In semi batch esterification process, an operating feed flowrate and temperature reactor has a significant effect on conversion that determines final product quality. This work addresses the implementation of an online dynamic optimization cascaded with a dual mode control strategy for improving the product quality of an autocatalytic esterification of Propionic Anhydride with 2-Butanol. An orthogonal collocation method is implemented to re-optimize the feed rate and temperature trajectories to compensate the deviation of end product due to disturbance. The problem of dynamic optimization is formulated to maximize the conversion. The simulation results indicate that with the proposed strategy, large improvement in semi batch reactor performance can be achieved compared to the method where the optimal trajectories set point is pre-determined. Moreover, the online dynamic optimization of temperature and feed flowrate trajectories obtained able to sustain the limiting reactant concentration within active constraint. Meanwhile, the offline optimization failed to handle the effect disturbance thus the end concentration is off-spec.**

Keywords: semi batch esterification reactor; autocatalytic esterification; online dynamic optimization; orthogonal collocation method

I. INTRODUCTION

To improve the batch processes performance, the dynamic optimization is preferred to implement due to its ability to cover the dynamic behavior of the process. Dynamic batch system is very sensitive to uncertainties and disturbances in the process operation. Due to the existence of uncertainties and disturbances, the final product may significantly differ from the desired value. Under these circumstances it is suitable to implement the on-line dynamic optimization strategy to desired trajectories to reach the optimal performances. To evaluate the deviation of end product, it is required to re-optimize the optimal trajectory as an on-line optimization strategy whenever new feedback information is presented. This strategy could compensate the uncertainty and disturbances leading to process operation enhancement [1].

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The aim of online dynamic optimization for semi batch reactor is to generate the optimal trajectories of process variables such as flow rates of feeds, temperature, and heat duty which are typically adjusted to optimize the objective function [2]. Mostly, the optimal trajectories for semi batch reactor are executed by flowrates of feeds [3, 4]. There are a few works which implemented the flowrate of feed and temperature as control variables simultaneously. Both variables are used since they have significant improvement for the product quality. Rahman and Palanki [5] study the online dynamic optimization on a semi batch reactor where the reaction rate follows a Langmuir-Hinshelwood type of kinetics. The objective is to maximize the amount of product at the end of one hour by manipulating the flow rate of feed and the batch temperature. The optimal trajectories were obtained by using the modified classical variation method. Kadam et al. [6] optimize the feed rate and cooling temperature trajectories in Williams-Otto benchmark reactor using control vector parameterization technique via solution model in order to maximize the yield of the main product.

In this work, simultaneous (orthogonal collocation) method will be implemented for online dynamic optimization of the feed flowrate and temperature reactor trajectories in the presence of disturbance. The process considered is Catalyzed Esterification of Propionic Anhydride with 2-Butanol in semi batch. The optimal trajectories obtained are based on maximum conversion problem.

II. MODELING OF AUTOCATALYTIC ESTERIFICATION PROPIONIC ANHYDRIDE WITH 2-BUTANOL

Esterification of propionic anhydride with 2-butanol is producing sec-butyl propionate and propionic acid. The reaction kinetics of this esterification have been investigated and the data of parameters is depicted from [7].The reaction rate variable is a function of catalyst (strong acid, such as

sulphuric acid); exhibits a second-order kinetics when no strong acid is present and exhibits a kind of autocatalytic behaviour when sulphuric acid is introduced [8].

The model is developed based on the following assumptions: constant reacting heat capacity, effective overall heat transfer coefficient, transport properties of reaction mixture and density are exist; the heat losses with the ambient surroundings are negligible; homogeneous mixing and uniform distribution temperature: no heat accumulation in the reactor wall; no secondary heating effects such as power introduced by stirrer; no pressure effect; 2- butanol stated as limiting reactant. The scheme of esterification semi batch reactor is shown in Fig.1.

The mathematical model of semi batch esterification reactor:

$$\frac{dC_A}{dt} = -((k_1 + k_2 C_{cat1}) C_A C_B + k_3 C_{cat2} C_B) - \frac{F_0 C_A}{V} \quad (1)$$

$$\frac{dC_B}{dt} = -((k_1 + k_2 C_{cat1}) C_A C_B + k_3 C_{cat2} C_B) + \frac{F_o}{V}(C_{B0} - C_B) \quad (2)$$

$$\frac{dC_C}{dt} = \frac{dC_D}{dt} = ((k_1 + k_2 C_{cat1}) C_A C_B + k_3 C_{cat2} C_B) - \frac{F_0 C_C}{V} \quad (3)$$

$$\frac{dC_{cat1}}{dt} = -\frac{dC_{cat2}}{dt} = -(k_4 10^{-H} C_{cat1} C_A) - \frac{F_0 C_{cat1}}{V} \quad (4)$$

$$\frac{dV}{dt} = F_o \quad (5)$$

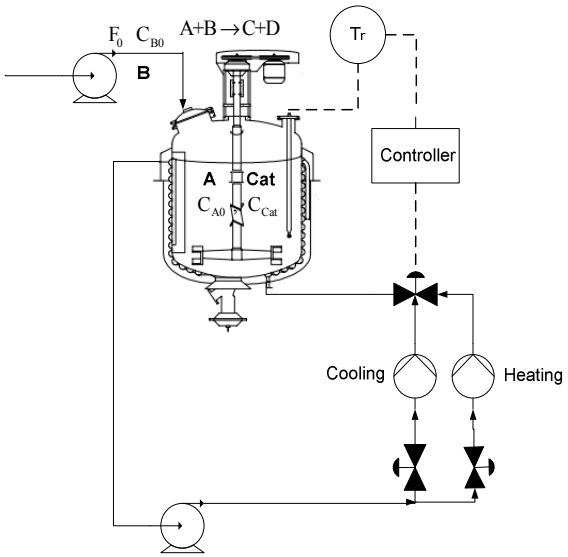
$$\frac{dT}{dt} = \frac{-\Delta Hr_{total}}{\rho C_p} + \frac{UA}{\rho C_p V}(T_j - T) + \frac{F_O}{V}(T_{feed} - T) \quad (6)$$

$$\frac{dT_j}{dt} = \frac{F_j}{V_j}(T_{jin} - T_j) + \frac{UA}{\rho_j V_j C_j}(T - T_j) \quad (7)$$

$$H = -(p_1 C_{cat1} + p_2 C_c)\left(p_3 + \frac{p_4}{T}\right) \quad (8)$$

where C_A, C_B,C_C, C_{Cat1}, and C_{cat2} are concentration of 2-butanol, propionic anhydride; propionic acid, sulphuric acid, mono-butyl sulphuric acid, respectively.

F_o , V is the feed rate and volume of solution within reactor. F_j is jacket flowrate, T_j is jacket temperature; T_{jin} is inlet jacket temperature; T_{feed} is feed temperature; A is the heat exchange area; V_j is volume of jacket; U is the heat exchange coefficient; C_p, C_j is heat capacity of solution in reactor and jacket, respectively; ΔHr is heat of reaction; ρ is density of solution in reactor; ρ_j is density of jacket solution. Those pertaining constant value is depicted from [8]. The initial value of C_A, C_B, C_C, C_D, C_{cat1}, C_{cat2} and V is 3.4M, 0M, 0M, 0M, 1.02x 10^{-2}M, 0M,1L, respectively.



Fiigure.1 : Scheme of esterification semi batch reactor;A: 2-butanol (limiting reactant), B: propionic anhydride,Cat: catalyst

III ONLINE DYNAMIC OPTIMIZATION PROCEDURE

In this study, cascade optimization is implemented where the overall problem is decomposed into two sub-problems (with consistent objectives) [2]. The set-point trajectory that guarantees performance is computed.

A `low level' tracking controller ensures that the system does not deviate from the optimal trajectory. In addition, a `high level' optimizer is invoked periodically to ensure the optimality.

Due to disturbance, pre-determined optimal trajectories need to be updated. This may not be necessary updated by solving a finite horizon optimization problem at each time step. It will update solution of the dynamic optimization problem only if deviation of parameter (limiting reactant concentration) is occurred which can generate the off-spec product. There is a trigger which acts like a switch for re-calling the optimization layer [9]. The spec concentration (±10% of limiting reactant) constraint is an active constraint which is applied as the trigger. When the deviation exceeds the active constraint as the presence of significance disturbance due to variation of feed concentration, the online dynamic optimization will be activated to generate the new re-optimize trajectories. The static optimal of temperature and feed flowrate, 323K and 8.3 x10^{-5}L/s are submitted as pre-determined setpoint. The end product desired is 97% [10].

The blocks in the cascade optimization framework scheme which assumed all the states can be measured (as shown in Fig.2), are described:

A. Dynamic Re-optimization

1. Procedures of dynamic optimization

In this work, orthogonal collocations of finite elements were implemented within the MATLAB® environment by using the dynopt code package developed by Cizniar and co-workers in which the algorithm was developed by Cuthrell and Biegler [11]. In this approach, state and control variables were parameterized and the model solution and the optimization problem were solved simultaneously.

An orthogonal collocation on finite elements was used to parameterize both state and control variables, and a successive quadratic programming (SQP) was used to solve the resulting optimization problems

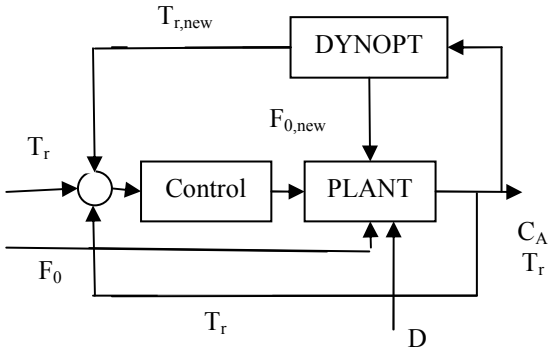


Figure 2. Scheme diagram of online dynamic optimization; DYNOPT: dynamic re-optimization; D:disturbance;

The general problem of the dynamic optimization can be expressed by applying the combination of the objective function, equality and inequality constraints, and bounds of the model.

2. Problem formulation

In this study, volume of solution, the reactant, catalyst and product concentration were considered as states variables. The control variables considered were feed flowrate and temperature reactor. The objective function was to maximize conversion (indication of the product purity). The inequality constraint associated was end concentration of limiting reactant and total volume reactor. The dynamic optimization formulations for both problems are shown as:

Problem:

$$\max_{T,F_0} \Im = \frac{C_{A0} - C_A}{C_{A0}}$$

Subject to semi batch dynamic model Eq.1-5
Inequality constraints: $C_A \leq 0.102$M, $V \leq 2.25$L
Bounds: $0 \leq F_0 \leq 3x\ 10^{-4}$L s^{-1} ; $303^0K \leq T \leq 343^0K$

B. Controller

The dual mode control (DM) strategy is implemented which combine the on-off and PID mode controller. Maximum heating is applied until the reactor temperature reaches within a specified percent of the set point and then maximum cooling is followed to bring the temperature to the set point smoothly. When the reactor temperature has reached its desired set point, a standard PID controller is switched on and used to maintain the reactor temperature. The details of DM control and its tuning can be found in Liptak [12].

IV. RESULTS AND DISCUSSION

The simulation of online dynamic re-optimization was carried out in the SIMULINK® environment. The initial optimal set point was introduced in process simulation for 200 min. The plant was disturbed by the step decrease (10%) of feed (propionic anhydride) concentration at 40- 60 min. The trigger will activate as the limiting reactant concentration deviate ±10%. The activated dynamic re-optimization generated one single optimization point of feed rate and temperature reactor trajectories with interval time of 30 min, and then the trajectories was switched back to the original set point for the remaining process time. The performance of the online dynamic optimization was compared to the offline optimization.

The temperature and feed flowrate trajectories obtained can be shown in Fig. 3 and 4, respectively. The profile of limiting reactant concentration in online, offline is shown in Fig. 5. The decline of feed concentration, as disturbance occurred, reduced the gradient of limiting reactant concentration. It caused the limiting reactant surpassed the active constraint, thus the trigger activates dynamic re-optimization. The optimal trajectories obtained switched the temperature and feed rate trajectories to 330 K and $1.2x\ 10^{-4}$ L/s, respectively, with duration of 30 minutes as shown in Fig. 3 and 4. The online dynamic optimization is able to tackle the disturbance effect. The re-optimized temperature and feed flowrate trajectories drove the limiting reactant concentration still within the active constraint. Fig.3 and 4 are also shown that the offline mode cannot capture the effect disturbance so that the set point of temperature reactor and feed flowrate applied was remain the same as the original profiles. Therefore, the application of online dynamic optimization preserves the quality of product purity.

The final concentration (conversion) obtained for online, and offline operation was 0.114 M (96.6%) and 0.243M (92.8%), respectively. Thus, the large improvement can be achieved by using the online dynamic optimization which able to maintain the concentration within the end point conversion requirement. Meanwhile, the end concentration of offline mode encountered an off-spec as beyond 10% of end product pre-determined. It is indicated that the offline optimization was failed to handle the effect disturbance towards the end concentration of limiting reactant.

From Fig.3, it is found that the action of DM controller to track temperature trajectory is acceptable due to the concentration of limiting reactant obtained was still within the active constraint. Furthermore, there is a chance to enhance

the performance of catalytic esterification operation by developing an effective controller which can track the trajectory more accurate.

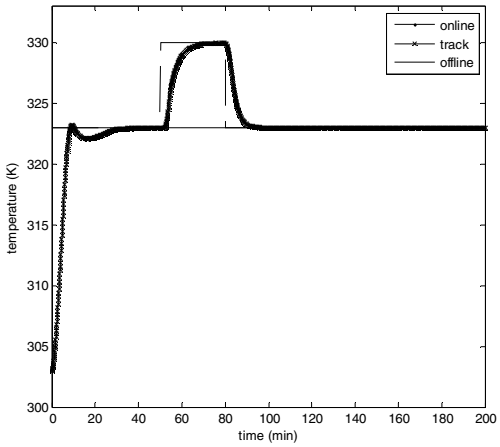


Figure 3. Temperature trajectory

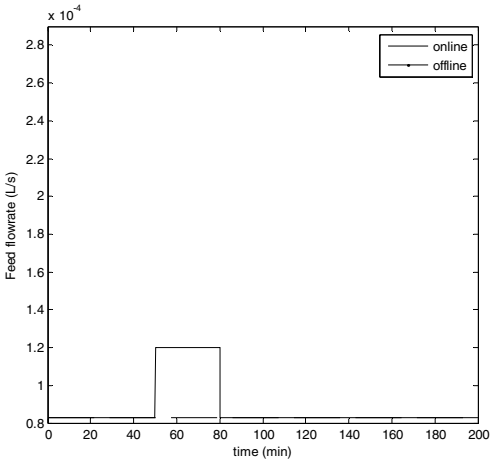


Figure 4. Feed flowrate trajectory

V. CONCLUSION

The online dynamic optimization of Catalyzed Esterification of Propionic Anhydride with 2-Butanol in semi batch have been carried out. Cascade optimization strategy was implemented to update the optimal trajectories conditionally which assumed all the states can be measured. The online dynamic optimization was able to tackle the disturbance effect. The re-optimized temperature and feed flowrate trajectories drove the limiting reactant concentration within the active constraint. Meanwhile, the end concentration of offline mode cannot capture the effect disturbance so that encountered an off-spec of the end product.

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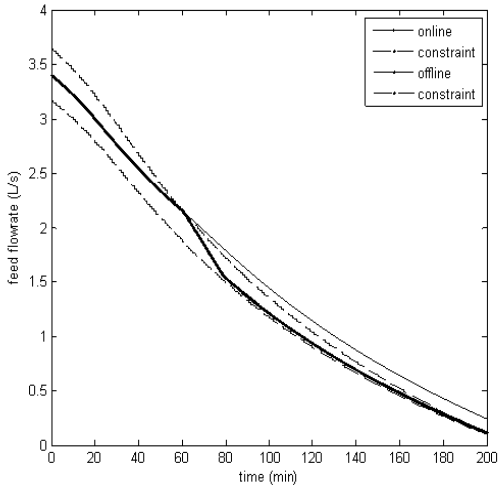

Figure 5. The profile of limiting reactant concentration in online: (+), offline:(-), active constraint (- -)

REFERENCES

[1] Tosukhowong, T (2006) Dynamic Real-Time Optimization and Control of an Integrated PlantGeorgia Institute of Technology, PhD thesis

[2] Hua, X, Rohani, S, Jutan, A (2004) Cascade closed-loop optimization and control of batch reactors, *Chemical Engineering Science* **59**: 5695 – 5708

[3] Perea L.F.T , Okorafo, O.E , Hutchinson, R.A , Guay , M(2010) Development of on-line optimization-based control strategies for a starved-feed semibatch copolymerization reactor , *Control Engineering Practice* **18**:131–139

[4] Maria, G, Dan, A (2010)Derivation of optimal operating policies under safety and technological constraints for the acetoacetylation of pyrrole in a semi-batch catalytic reactor, *Computer and Chemical Engineering*, in press

[5] Rahman, S and Palanki, S(1998)State feedback synthesis for on-line optimization of batch reactors with multiple manipulated inputs *Computers Chem. Engng* **22**(101):429-1439

[6] Kadam, J.V., Schlegel, M, Srinivasan, B, Bonvin, D, Marquardt, W (2007) Dynamic optimization in the presence of uncertainty: From off-line nominal solution to measurement-based implementation *Journal of Process Control* **17**: 389–398

[7] Zaldivar, J. M., Hernandez, H., Molga, E., Galvan, I. M., & Panetsos, F. (1993). The use of neural networks for the identification of kinetic functions of complex reactions. In Proceedings of the third European symposium on computer aided process engineering, ESCAPE 3

[8] Ubrich, O, Srinivasan, B, Bonvin, D, Stoessel, F (1999) Optimal Feed Profile for a Second Order Reaction in a Semi-Batch Reactor under Safety Constraints. Experimental Study, Journal *of Loss Prevention in the Process Industries* **12** (6): 485-493

[9] Ochoa, S, Repke, J.U, Wozny, G (2010) Integrating real-time optimization and control for optimal operation: Application to the bio-ethanol process Biochemical Engineering Journal **53**:18–25

[10] Rohman, F.S, Sata, S.A, and Aziz, N (2010) Simulation and optimization catalyzed esterification of propionic anhydride with 2-butanol: batch versus semi, PSE Asia, The 5th, Singapore

[11] Cizniar, M (2005) Dynamic Optimisation of Processes, Diploma Work, Slovak technical University

[12] Liptak, B.G (1999) Optimization of Industrial Unit Processes: 2nd edition, CRC Press, Florida