Iterative Learning Control of Crystallisation Systems

by

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Abstract

Under the increasing pressure of issues like reducing the time to market, managing lower production costs, and improving the flexibility of operation, batch process industries thrive towards the production of high value added commodity, i.e. specialty chemicals, pharmaceuticals, agricultural, and biotechnology enabled products. For better design, consistent operation and improved control of batch chemical processes one cannot ignore the sensing and computational blessings provided by modern sensors, computers, algorithms, and software. In addition, there is a growing demand for modelling and control tools based on process operating data. This study is focused on developing process operation data-based iterative learning control (ILC) strategies for batch processes, more specifically for batch crystallisation systems.

In order to proceed, the research took a step backward to explore the existing control strategies, fundamentals, mechanisms, and various process analytical technology (PAT) tools used in batch crystallisation control. From the basics of the background study, an operating data-driven ILC approach was developed to improve the product quality from batch-to-batch. The concept of ILC is to exploit the repetitive nature of batch processes to automate recipe updating using process knowledge obtained from previous runs. The methodology stated here was based on the linear time varying (LTV) perturbation model in an ILC framework to provide a convergent batch-to-batch improvement of the process performance indicator. In an attempt to create uniqueness in the research, a novel hierarchical ILC (HILC) scheme was proposed for the systematic design of the supersaturation control (SSC) of a seeded batch cooling crystalliser. This model free control approach is implemented in a hierarchical structure by assigning data-driven supersaturation controller on the upper level and a simple temperature controller in the lower level.

In order to familiarise with other data based control of crystallisation processes, the study rehearsed the existing direct nucleation control (DNC) approach. However, this part was more committed to perform a detailed strategic investigation of different possible structures of DNC and to compare the results with that of a first principle

model based optimisation for the very first time. The DNC results in fact outperformed the model based optimisation approach and established an ultimate guideline to select the preferable DNC structure.

Batch chemical processes are distributed as well as nonlinear in nature which need to be operated over a wide range of operating conditions and often near the boundary of the admissible region. As the linear lumped model predictive controllers (MPCs) often subject to severe performance limitations, there is a growing demand of simple data driven nonlinear control strategy to control batch crystallisers that will consider the spatio-temporal aspects. In this study, an operating data-driven polynomial chaos expansion (PCE) based nonlinear surrogate modelling and optimisation strategy was presented for batch crystallisation processes. Model validation and optimisation results confirmed this approach as a promise to nonlinear control.

The evaluations of the proposed data based methodologies were carried out by simulation case studies, laboratory experiments and industrial pilot plant experiments. For all the simulation case studies a detailed mathematical models covering reaction kinetics and heat mass balances were developed for a batch cooling crystallisation system of Paracetamol in water. Based on these models, rigorous simulation programs were developed in MATLAB®, which was then treated as the real batch cooling crystallisation system. The laboratory experimental works were carried out using a lab scale system of Paracetamol and iso-Propyl alcohol (IPA). All the experimental works including the qualitative and quantitative monitoring of the crystallisation experiments and products demonstrated an inclusive application of various in situ process analytical technology (PAT) tools, such as focused beam reflectance measurement (FBRM), UV/Vis spectroscopy and particle vision measurement (PVM) as well. The industrial pilot scale study was carried out in GlaxoSmithKline Bangladesh Limited, Bangladesh, and the system of experiments was Paracetamol and other powdered excipients used to make paracetamol tablets.

The methodologies presented in this thesis provide a comprehensive framework for data-based dynamic optimisation and control of crystallisation processes. All the simulation and experimental evaluations of the proposed approaches emphasised the potential of the data-driven techniques to provide considerable advances in the current state-of-the-art in crystallisation control.

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LIST OF ACRONYMS

2D-FM	Two Dimensional Fornasini-Marchsini
AC	Adaptive Control
ADNC	Automated Direct Nucleation Control
AI	Active Ingradients
ANN	Artificial Neural Networks
ARMAX	Auto Regressive Moving Average Models with eXogenous
ATR	Attenuated Total Reflection
BMPC	Batch Model Predictive Control
CBMPC	Characteristic Based Model Predictive Control
CDPS	Control of Distributed Parameter Systems
CLD	Chord Length Distributions
CryPRINS	Crystallisation Process Informatics System
CSD	Crystal Size Distribution
CSS	Constant Supersaturation
DNC	Direct Nucleation Control
DPS	Distributed Parameter Systems
D-RTO	Dynamic Real-Time Optimisation
ESC	Expert Systems Based Control
FBD	Fluid-Bed Dryer
FBRM	Focused Beam Reflectance Measurement
HILC	Hierarchical ILC
ILC	Iterative Learning Control
ILRC	Iterative Learning Reliable Control
IPA	isoPropyl Alcohol
KL	Karhunen-Loéve
LMIs	Linear Matrix Inequalities
LOD	Loss on Drying

LP	Linear Program
LPS	Lumped Parameter Systems
LSM	Least Square Minimisation
LTV	Linear Time Varying
MIMO	Multiple Input Multiple Output
MOCH	Method of Characteristics
MPC	Model Predicted Control
MSZ	Metastable Zone
MSZW	Metastable Zone Width
NMPC	Nonlinear Model Predictive Control
NIR	Near Infrared
NN	Neural Network
OC	Operating Curve
ODE	Ordinary Differential Equation
OLE	Object Linking and Embedding
OPC	Object Linking and Embedding for Process Control
PAT	Process Analytical Technology
PCM	Probabilistic Collocation Method
PDEs	Partial Differential Equations
PDF	Population Density Function
PHBP	Polyhydroxybenzophenone
PID	Proportional Integral Derivative
PML	Product Managed Locally
PTFE	Polytetrafluoroethylene
PVM®	Particle Vision Measurement
QBMPC	Quality Control-Combined Batch Model Predictive Control
Q-ILC	Quadratic Criterion Based ILC
QMOM	Quadratic Method of Moments
RNN	Recurrent Neural Network

RPM	Rotation Per Minute
SC	Solubility Curve
SEM	Scanning Electron Microscope
SHMPC	Shrinking Horizon Model Predictive Control
SISO	Single Input Single Output
SNARMAX	Spatial Nonlinear Autoregressive Moving Average Exogenous
SQP	Sequential Quadratic Programming
SSC	Supersaturation Control
SVD	Singular Value Decomposition
SWCLD	Square Weighted Chord Length Distribution
SWMCL	Square Weighted Mean Chord Length
UV/Vis	Ultra-violet/Visible

NOMENCLATURE

$\boldsymbol{\Omega}$	Historical data sets
$\boldsymbol{\Phi}$	Coefficient matrix
$\boldsymbol{\Gamma}$	Coefficient matrix
$\boldsymbol{\Upsilon}$	Coefficient matrix
$\boldsymbol{\Psi}$	Nonlinear static function
α	The bias correction parameter in Equation 2.3
β	Forgetting factor
$\boldsymbol{\varepsilon}$	Model prediction
η	Discrete linear systems models
Δ	Change
μ_0	Zeroth moment (total number of crystals)
μ_1	First moment (total length of crystals)
μ_2	Second moment (total area of crystals)
μ_3	Third moment (total volume of crystals)
$\mathbf{A}$, $\mathbf{B}$, $\mathbf{C}$	Coefficient matrices in Equation 2.11
B	Nucleation rate
C	Concentration
C_S	Solubility
DT	Performance index in Equation 2.4
$F(.)$	Nonlinear function in a matrix form
G	Growth rate
$\mathbf{H}$	Learning gain matrix
$\mathbf{K}$	Controller gain
L	Nonlinear learning gain in Equation 2.9
Ln	Mean crystal size
$\mathbf{L}_s$	LTV perturbation model

M	Midpoint of an individual FBRM channel
N	Number of observations
$\mathbf{O}$, $\mathbf{P}$	Weighting matrices
S	Supersaturation
T	Temperature
b_o, b_1, b_2, b_3	Regression coefficients
d	Derivative of absorbance
e	Error between desired output and actual output
k	Batch index
$\mathbf{m}$	Sequence of model errors
$\mathbf{n}$	Vector of measurement noice
m	Control horizon
n	Model horizon
p	Model prediction horizon
r_0	Size of the nuclei
t	Time
x	Input to the batch process
y	Output from the batch process
y_d	Desired output from the process
z	State variable

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