EVALUATION OF OPTIMAL COOLING CONTROL FOR SEEDED BATCH CRYSTALLIZATION INCLUSIVE DISSOLUTION WITH UNCERTAINTIES

SITI ZUBAIDAH BINTI ADNAN

MASTER OF SCIENCE

UNIVERSITI MALAYSIA PAHANG

SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science in Chemical Engineering

_____

(Supervisor's Signature)

Full Name : DR NOOR ASMA FAZLI BIN ABDUL SAMAD

Position : SENIOR LECTURER

Date :

_____

(Co-supervisor's Signature)

Full Name : DR SITI KHOLIJAH BINTI ABDUL MUDALIP

Position : SENIOR LECTURER

Date :

STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

_____

(Student's Signature)

Full Name : SITI ZUBAIDAH BINTI ADNAN

ID Number : MKC16018

Date :

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ABSTRAK

Proses penghabluran biasanya dikawal untuk beroperasi di antara zon metastabil dimana zarah kristal akan membesar sehingga proses berakhir. Tahap supersaturasi yang tinggi disebabkan proses beroperasi terlalu hampir dengan had metastabil pada permulaan operasi penyejukan penghabluran telah mengakibatkan kadar nukleasi dan pembesaran kristal menjadi tinggi. Akibatnya target produk kristal yang berjaya diperoleh mempunyai sejumlah kristal halus yang tidak diperlukan apabila berakhirmya operasi. Nukleasi yang berlebihan ini boleh dikurangkan dengan memperkenalkan langkah melarutkan dalam model penghabluran di mana kristal-kristal halus yang terjadi akibat nukleasi larut kembali dan kesannya ia mengurangkan jumlah kristal halus dalam keseluruhan taburan saiz kristal (TSK). Oleh itu objektif kajian ini adalah untuk membangunkan strategi penyejukan optimum yang menggabungkan fenomena pelarutan bagi penghabluran secara kumpulan mengguna penghabluran kalium nitrat sebagai kes. Model matematik untuk fenomena pelarutan dalam penghabluran kalium nitrat dibangunkan dan disimulasikan menggunakan perisian Matlab. Simulasi secara kawalan terbuka untuk proses penyejukan nominal telah disahkan melalui data eksperimen. Beberapa strategi yang bersesuaian untuk mencapai target TSK dengan jumlah kristal halus yang minimum telah digunakan. Algoritma pengoptimuman telah dibangunkan untuk menentukan trajektori set titik yang optimal untuk kawalan secara tertutup. Berdasarkan trajektori ini, kawalan secara tertutup menggunakan pengawal perkadaran-pengamiran (PI) mampu untuk mengekalkan operasi penghabluran pada set titik dan telah berjaya mencapai sasaran produk kristal yang dikehendaki dengan jumlah kristal halus yang minimum. Proses penghabluran penyejukan optimum yang dicadangkan mampu menambah baik purata panjang karakter TSK dan jumlah kristal halus sebanyak 67% dan berkurang sebanyak 71% berbanding dengan strategi penyejukan nominal. 4% pembesaran purata panjang karakter TSK dan 25% pengurangan jumlah kristal halus telah berjaya dicapai jika dibandingkan dengan strategi ayunan suhu. Kemudian, penilaian ketahanan pengawal telah diuji dengan analisis ketidakpastian menggunakan simulasi Monte Carlo untuk menganalisis kesan ketidakpastian input terhadap variasi taburan produk. Variasi yang tinggi terhadap TSK akhir telah dilihat. Analisis kepekaan kemudiannya telah diaturkan untuk mengenalpasti korelasi ketidakpastian input dengan model output TSK. Penalaan untuk pengawal telah dipilih sebagai langkah untuk menambah baik ketahanan pengawal. TSK akhir telah menunjukkan pengurangan variasi setelah langkah penalaan terhadap pengawal dilakukan. Oleh itu pengawalan yang tahan dan optimum untuk proses penyejukan berkumpulan secara pembenihan bagi penghabluran kalium nitrat telah berjaya ditubuhkan.

ABSTRACT

Crystallization process is generally controlled to be operated within metastable zone where the crystal particles will grow until the end of the operation. High supersaturation level resulted from the process operated too close with metastable limit at the beginning of cooling crystallization operation causing high nucleation and crystal growth rate. As a consequence the target crystal product is able to achieve but there is unnecessary amount of fine crystals by the end of the operation. This excessive nucleation can be reduced by introducing dissolution step in the crystallization operation where the fine crystals generated by nucleation is dissolved back into solution which in return, reducing the amount of fine crystals in the overall CSD. Therefore the objective of this study is to develop the optimal cooling control inclusive dissolution phenomena for batch seeded crystallization using potassium nitrate crystallization as a case study. The mathematical model for potassium nitrate crystallization inclusive dissolution phenomena was developed and simulated in Matlab software. The open-loop simulation for nominal cooling of the process was validated against experimental data. Several other strategies pertaining to achieve desired CSD with minimum amount of fine crystals were deployed. The optimization algorithm was employed in order to determine the optimal set-point trajectory for closed-loop control. Based on this trajectory, optimal closed-loop control of cooling crystallization process using Proportional-Integral (PI) controller were able to maintain the crystallization operation at its set-point and successfully achieved the desired target of crystal product with minimum amount of fine crystals. The proposed optimal cooling crystallization process was able to improve mean characteristic length and amount of crystal fines by 67% and reduced by 71% respectively to compare with nominal cooling strategy. 4% larger crystal characteristic length and 25% lesser amount of crystal fines is successfully accomplished if compared with temperature swing strategy. Then the robustness evaluation of controller was tested through uncertainty analysis using Monte Carlo simulations in order to analyse the effect of input uncertainties towards the variation of the product distribution. Large variation on final CSD was observed. Sensitivity analysis was then deployed using Standardized Regression Coefficients (SRC) method in order to analyse the correlation of the input uncertainties with the model output CSD. Retuning of the controller was chosen as the action taken to improve the robustness. The final CSD show minimum variation after controller retuning step. Therefore robust optimal control for seeded batch cooling of potassium nitrate crystallization was successfully established.

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LIST OF SYMBOLS

A_1	Crystallizer's internal area (cm^2)
A_2	Crystallizer's external area (cm^2)
A_c	Total area of particles (cm^2)
a_{i1}	Polynomial coefficient for saturation concentration
B	Birth rate (number of particles/ cm^3.min)
B_{agg}	Birth rate of agglomeration (number of particles/ cm^3.min)
B_{br}	Birth rate of breakage (number of particles/ cm^3.min)
B_{nuc}	Birth rate of nucleation (number of particles/ cm^3.min)
b	Nucleation order
b_{i1}	Polynomial coefficient for saturation concentration
C	Solute concentration (g solute/g solvent)
C*	Saturation concentration (g solute/g solvent)
C_f	Acceptable solute concentration at the end of batch to achieve required yield (g solute/g solvent)
C_t	Expected solute concentration at the end of the batch (g solute/g solvent)
C_s	Seed loading ratio (%)
c_{i1}	Polynomial coefficient for saturation concentration
c_p	Heat capacity (J/g.°C)
c_{pw}	Water heat capacity (J/g.°C)
D	Crystal dissolution rate (µm/s)
D_{br}	Death rate due to breakage (number of particles/ cm^3.min)
D_{agg}	Death rate due to agglomeration (number of particles/ cm^3.min)
D_x	Crystal dissolution rate in length direction (µm/s)
D_y	Crystal dissolution rate in width direction (µm/s)
d	Dissolution order
d_{i1}	Polynomial coefficient for saturation concentration
F_{obj}	Objective function
F_{win}	Cooling water flow rate (cm^3/min)
f	Relative shape function of crystals
f_n	Crystal size distribution
f_v	Volume distribution function in length direction
G	Crystal growth rate (µm/s)
$G_{1/x}$	Crystal growth rate in length direction (µm/s)

G_2	Crystal growth rate in width direction (µm/s)
g	Crystal growth order
k	Number of considered variables/ factors
k_b	Kinetic coefficient for nucleation (number of particles/ cm^3.min)
k_d	Kinetic coefficient for dissolution (number of particles/ cm^3.min)
k_g	Kinetic coefficient for crystal growth (number of particles/ cm^3.min)
k_v	Crystal shape factor
$L_{1/x}$	Length of crystal particles (µm)
L_2	Width of crystal particles (µm)
L_c	Total length of particles (µm)
L_s	Average mean size (µm)
M_c	Total crystal mass (g)
m	Molal concentration of solute (moles solute/ g solvent)
m_w	Mass of solvent (g)
N_c	Total number of particles (number of particles)
N_d	Number of experimental size bins
N_i	Number of crystals per unit of suspension for class i (number of particles/ cm^3)
p	Agitation order of nucleation
q	Agitation order of crystal growth
R	Ideal gas constant (J/ mol.K)
R^2	Coefficient of model determination
S	Supersaturation
$S^{b/,}$	Supersaturation for nucleation
S^d	Supersaturation for dissolution
S^g	Supersaturation for crystal growth
S_i	Mean size of class i (µm)
S_r	Supersaturation ratio
S_x	Supersaturation in length direction
T	Solution crystallizer temperature (°C)
$\dot{T}$	Rate of crystallizer temperature change (°C/ min)
T_{ex}	Exterior temperature (°C)
T_{feed}	Saturation temperature of feed(°C)
T_{fin}	Final temperature (°C)
T_s	Seeding temperature (°C)
T_w	Cooling water temperature (°C)

T_{win}	Inlet cooling water temperature (°C)
t	Crystallization time (min)
t_{batch}	Batch time (min)
t_c	Total crystallization time (min)
t_{cool}	Cooling duration (min)
t_f	Total batch time (min)
U_1	Heat transfer coefficient for internal crystallizer (J/ °C.min.cm^2)
U_2	Heat transfer coefficient for external crystallizer (J/ °C.min.cm^2)
V	Volume of solution (cm^3)
V	Mean crystal volume (cm^3)
V_c	Crystal volume (cm^3)
V_w	Cooling water volume (cm^3)
v	Kinematic viscosity (m^2/s)
x	Mole fraction of solute

Greek letters

α	Production-reduction term (number of particles/ cm^3.min)
α_d	Dissolution parameter
α_g	Crystal growth parameter
β_d	Dissolution parameter
β_g	Crystal growth parameter
μ_0	Zeroth moment of one-dimensional PBE
μ_m	mth moment of one-dimensional PBE
ΔCl_i	Size of classes for *i*th classes (µm)
ΔE_b	Activation energy for nucleation rate (kJ/mol)
ΔE_d	Activation energy for dissolution rate (kJ/mol)
ΔE_g	Activation energy for crystal growth rate (kJ/mol)
ΔH_c	Heat of crystallization (J/g)
ΔC	Concentration difference (g solute/g solvent)
σ	Relative supersaturation
ζ	Activity coefficient
ρ	Solution density (g/ cm^3)
ρ_c	Crystal density (g/ cm^3)
ρ_w	Water density (g/ cm^3)
γ	Size dependent growth constant
ω_c	Temperature weightage

ω_t	Concentration weightage
Φ	Design parameter
θ	Estimated parameter

LIST OF ABBREVIATIONS

CSD	Crystal Size Distribution
CI	Confidence Interval
CV	Coeffiecient Variation
FPI	Fast Probability Integration
IMC	Internal Model Control
LHS	Latin-Hypercube Sampling
MPC	Model Predictive Control
MV	Manipulated Variable
NMPC	Non-Linear Model Predictive Control
NMS	Number Mean Size
ODE	Ordinary Differential Equation
P	Proportional
PBE	Population Balance Equation
PDE	Partial Differential Equation
PI	Proportional-Integral
PID	Proportional-Integral-Derivative
SQP	Sequantial Quadratic Programming
SRC	Standardized Regression Coefficients

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