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Real-time optimization using gradient adaptive selection and classification from infrared sensors measurement for esterification oleic acid with glycerol

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Abstract

Purpose – The production of glycerol derivatives by the esterification process is subject to many constraints related to the yield of the production target and the lack of process efficiency. An accurate monitoring and controlling of the process can improve production yield and efficiency. The purpose of this paper is to propose a real-time optimization (RTO) using gradient adaptive selection and classification from infrared sensor measurement to cover various disturbances and uncertainties in the reactor.

Design/methodology/approach – The integration of the esterification process optimization using self-optimization (SO) was developed with classification process was combined with necessary condition optimum (NCO) as gradient adaptive selection, supported with laboratory scaled medium wavelength infrared (mid-IR) sensors, and measured the proposed optimization system indicator in the batch process. Business Process Modeling and Notation (BPMN 2.0) was built to describe the tasks of SO workflow in collaboration with NCO as an abstraction for the conceptual phase. Next, Stateflow modeling was deployed to simulate the three states of gradient-based adaptive control combined with support vector machine (SVM) classification and Arduino microcontroller for implementation.

Findings – This new method shows that the real-time optimization responsiveness of control increased product yield up to 13 percent, lower error measurement with percentage error 1.11 percent, reduced the process duration up to 22 minutes, with an effective range of stirrer rotation set between 300 and 400 rpm and final temperature between 200 and 210°C which was more efficient, as it consumed less energy.

Research limitations/implications – In this research the authors just have an experiment for the esterification process using glycerol, but as a development concept of RTO, it would be possible to apply for another chemical reaction or system.

Practical implications – This research introduces new development of an RTO approach to optimal control and as such marks the starting point for more research of its properties. As the methodology is generic, it can be applied to different optimization problems for a batch system in chemical industries.



Originality/value – The paper presented is original as it presents the first application of adaptive selection based on the gradient value of mid-IR sensor data, applied to the real-time determining control state by classification with the SVM algorithm for esterification process control to increase the efficiency.

Keywords Gradient technique, Infrared sensor, Real-time optimization, Simulation and modelling, Support vector machine

Paper type Research paper

1. Introduction

Esterification products such as monoglyceride and diglyceride represent high needs as modifying agents and show steadily incremental demand for industrial consumption in the future, as they have various industrial applications. These products as a raw material are required in pharmaceutical, cosmetics and personal care industries, as well as ink manufacturing (Pagliaro and Rossi, 2008; Fernandez *et al.*, 2004; Mostafa *et al.*, 2013; Alstad *et al.*, 2009). Another use of a derivative product of glycerol in Indonesia, particularly in oil mining, is for mixing substances to produce oil-based mud and water-based mud. Production of monoglyceride from esterification of glycerol and synthesis of middle- or long-chain fatty acids offers promising industrial opportunities (Hui, 1996). To optimize the esterification reaction, it is essential for the process performed in real-time mode to cover various disturbances and uncertainties that occur all at the time of the process. In esterification, as previously researched (Srinivasan *et al.*, 2003), “there is influence of process variables as temperature and time requirement affected their efficiency in esterification.” A previous research by van Ast *et al.* (2008/2009) showed that the continuous model of the system to be controlled was transformed to a stochastic discrete automation after a variation was applied to derive the control policy. We use this model to accommodate the control process of esterification. The esterification process has several constraints such as the inconstant yield of the production target and the lack of efficiency for it has a synthesizing reaction that needs high-energy use in the reactor to achieve desired temperature and time for the process. Traditionally, this type of process was operated based on the experience of the operator.

Real-time optimization (RTO) was used to indicate the continuous re-evaluation of selecting variables in operation (Chachuat *et al.*, 2009) as a part of this research and in the next development of RTO for the chemical process based on Necessary Condition Optimum (NCO) (Srinivasan and Bonvin, 2007). With recent advances in digital hardware and optimization software, the RTO method can be connected to a computer control system (Bocker *et al.*, 2006). In recent years, spectroscopic methods by using infrared have gained popularity to be chosen in real-time industrial process control, especially for the esterification process (Blanco *et al.*, 2004). A systematic and rational approach was required in order to accommodate different sources of sensors and process fluctuations as dynamic conditions such as disturbance of a process parameter that can affect monitoring and classification performance from uncertainty of class imbalance and noisy attributes (Yusta, 2009). It is necessary to adjust the set point of basic control precisely to adapt the system requirements.

As previous research, that the decision for model parameter adaptation is to select the parameters to be adapted (Chachuat *et al.*, 2009). Feature selection is generally used in machine learning when the learning task involves high-dimensional and noisy attribute data sets as a parameter, as observed in real-time sensor application. In this work, feature selection with gradient measurement as parameter adaptation was applied to select the type of appropriate sensor as a parameter related for measurement in this esterification process. These higher dimensional results increased the accuracy in monitoring the measurement, especially for the measurement of yield. Furthermore, our algorithm is very straightforward to apply as all of the parameters have been deployed with sensor operation.

In the current work, we set the combination of RTO in adaptive optimization approaches named gradient adaptation with sensor selection, combined with computational methods for classification. Thus, the objective of this research is to analyze and design the dynamic process condition of glycerol esterification in detail, in which Business Process Model and Notation version 2.0 was used and then supported with computational methods such as gradient measurement for the selection of sensors, and a classification task with a support vector machine (SVM) consecutively was set to determine the state condition in controlling and applying microcontroller simulation in Stateflow. As applied for the esterification process, the investigated system consisted of several performance indicators that have to be fulfilled such as yield, process time, stirring speed and temperature.

2. RTO

The RTO concept was developed based on self-optimization (SO), which is defined in Skogestad as how to find an acceptable loss with constant set point values for the controlled variables (CVs) without the need to re-optimize when disturbances occur. This method has to be combined with the NCO method (Srinivasan *et al.*, 2008). However, according to the research by Ye *et al.* (2012), it is necessary to measure all the NCO components in real time. RTO improves process performance iteratively by selecting optimization variables using measurement data. For this research, we claim that this new optimization combined method, supported with measurement by an adaptive selection sensor and real-time data acquisition system, is useful for chemical industry application that has a dynamic condition such as the esterification process. In sensor deployments, each sensor collected data at regular time intervals, captured a time series representing that the dynamic condition occurs and build the database that needs the classification approach (Alstad *et al.*, 2009).

Figure 1 shows the schematic diagram of the integration method of SO and NCO to set in the real-time control process. The concept of SO is a strategic aim to appropriately select the CVs so when they are maintained at constant set points, the overall plant operation is optimal or near optimal despite various disturbances (François *et al.*, 2005); this concept was related to an offline system. To develop a real-time system and improve the performance, the concept of SO was combined with NCO that was related to the CVs for controlling the variable (Halvorsen *et al.*, 2003). In this research, the developed active control variable was obtained in real time with the measurement from the IR sensor with adaptive selection and was computed for tracking the NCO to select the state with computational methods

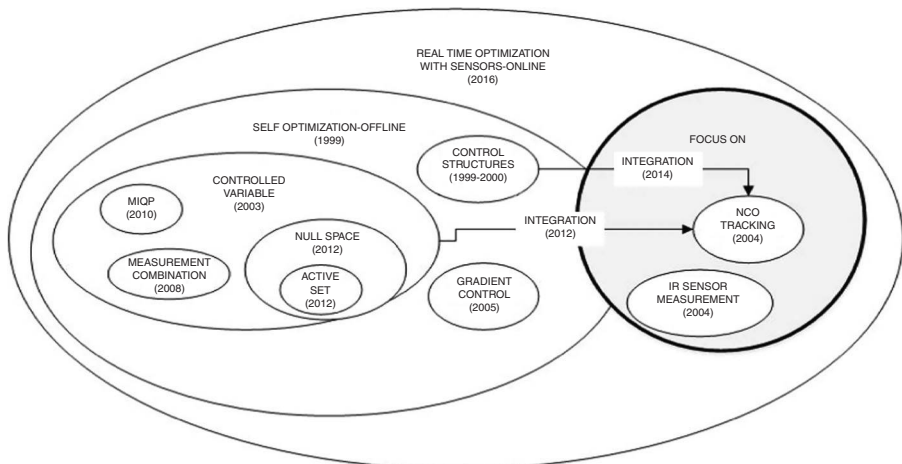


Figure 1.
Relationship between
SO and NCO in RTO

for classification. The real-time task had integral action to track the selected sensor using the adaptive method and to set the control at a necessary set point with state. The set point was determined as considered to be the best performance condition of apparatuses such as heater and agitation motor.

3. Implementation of the RTO method with gradient and state control to optimize process time

In industrial production, the run-time process variations need to be accounted for, especially in the industrial process with a chemical reaction, typically to cope with these uncertainties by adopting a conservative strategy that guarantees constraint satisfaction even in the worst-case situation. This measurement can be used in an optimization framework to compensate for the effects of uncertainty in the form of model mismatch or process disturbances. Nowadays, real-time optimization with necessary conditions of optimality (NCO) proposed by Jäschke and Skogestad (2011) and optimal operation is achieved by designing a “smart” control structure. As the comparison of the SOC method was combined with NCO tracking (Srinivasan *et al.*, 2003) or zone control MPC (Graciano *et al.*, 2015), this concept became real-time optimization using an online model in data acquisition. Among the various options for input adaptation, there is a promising approach consisting of directly enforcing the NCO that includes two parts, the active constraints and the sensitivities. In NCO, there is a two-level approach that does not require us to repeat the optimization: at the upper level, the constraints that are active in the optimal solution are identified from optimization of a nominal process model. At the lower level, feedback control is used to enforce the NCO to define the control problem in matched criteria as described in Figure 2. The use of measurements to compensate for the effect of uncertainty has recently gained attention in the context of RTO of dynamic systems.

Based on the control problem in the case study of this research, the consideration of the esterification process as a reaction system of synthesized from glycerol with oleic acid was taken in the laboratory scale model:



where A is the glycerol; B the oleic acid; C the glycerol monooleate; and D the water. This reaction was related to the temperature reaction process that corresponded to k_1 as the kinetic parameter. The reaction rate was defined as:

$$-r_A = k_1 C_A C_B - k_1 C_C C_D \quad (2)$$

where C_A , C_B , C_C and C_D present the concentrations of oleic acid, glycerol, glycerol monooleate and water, respectively.

The commonly used approach consists of updating a process model and performing numerical optimization. In this research, because of the dynamic condition of the esterification process and slow control response, especially the control of temperature, we refined the model using IR sensors supported with the computational method as classification and implemented the new optimization model as shown in the flow diagram in Figure 3.

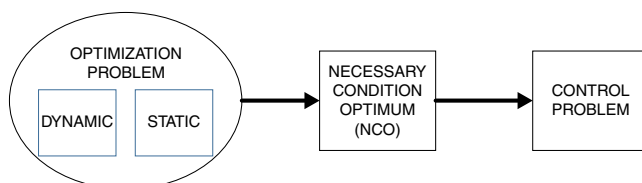


Figure 2.
Connection of NCO
between problems

As per Chachuat *et al.* (2009) the model of optimization was based on the formulation:

$$(k_1^*) = \underset{\theta}{\operatorname{argmin}} \left\{ \sum_{i \in \{C\}} \left(1 - \frac{c_i(t_f; \theta)}{c_{p,i}(t_f)} \right)_{\pi = \pi_k}^2 \right\} \quad (3)$$

with k_1^* the adjustable parameters which correspond to the kinetic coefficients such as the temperature of the reactor $\theta = (k_1)$. At the end of the k th RTO operation that was determined by the sensor, those values were updated by minimizing a weighted sum of square error, while $c_{p,i}(t_f)$ is the concentration of product i at the end of the batch, measured using gas chromatography-mass spectrometry (GCMS) and Fourier transform infrared spectroscopy (FTIR):

$$(k_1, k) = (1 - \kappa_\theta)(k_{1,k-1}) + \kappa_\theta(k_1^*) \quad (4)$$

The value of parameter θ_t is estimated using process output measurement y_s with adaptation parameter $\kappa_\theta = 0.5$.

We tried to develop the optimization system from previous research (Soenandi *et al.*, 2015). In this research, to select the appropriate sensor for data processing in the real-time condition within three wavelengths IR sensors, we have developed a real-time simulation model of the selection parameter in previous research in simulation (Soenandi and Djatna, 2014), and to make better optimization for esterification control in this research was operated in with formulation:

$$\theta_t = D_v(y_s) \quad (5)$$

where D_v is the gradient measurement from sensor data measurement.

Previous research by Mahassni (2013) shared the similar idea as a comparison to research which also used sensor selection regarding the operating environment. To simplify the selection method, we tried gradient measurement to select the appropriate sensors used for regression. As mathematical formulation, the gradient of f is defined as the unique vector field whose dot product with any vector v at each point x is the directional derivative of f along v (Korn and Theresa, 2000), as:

$$(\nabla f(x)) \cdot v = D_v f(x) \quad (6)$$

In this research, we measured the gradient by comparing sample data points from the two-dimensional field as identified by the sensors. The formulation can be seen in Equation (7), and by limiting the gradient value m_i we set the decision to use the data or pass it:

$$m_i = \frac{t_n - t_{n-1}}{x_n - x_{n-1}} \quad (7)$$

where t_n is the time interval sampling and x_n is the value from the sensor.

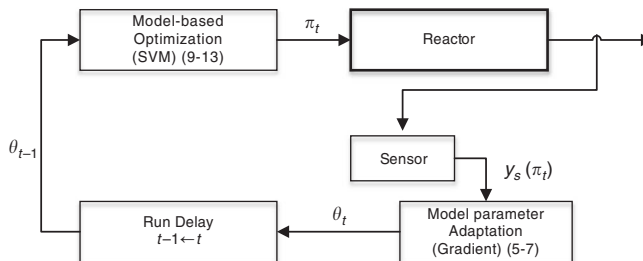


Figure 3.
Real-time optimization
with model parameter

We assumed that we have a plant measurement model from the sensor:

$$Y = f^y(\mathbf{u}, \mathbf{x}_n, D(t)) \quad (8)$$

The optimization algorithm uses the process model and the objective function to solve for the new optimum state for the process used the SVM for classification. SVM regression was performs linear regression in the high-dimensional feature space using ε -insensitive loss and, at the same time, the feature space $f(x, \omega)$ in this research we got Y value as plant measurement from sensor as f^y is the function mapping the variables \mathbf{u}, \mathbf{x}_n and $D(t)$ onto the measurement space, tries to reduce model complexity by minimizing $\|\omega^2\|$. This can be described by introducing (non-negative) slack variables $\xi_i, \xi_i^*, i = 1, \dots, n$, to measure the deviation of training data. Thus, SVM is formulated as minimization of the following function:

$$\text{minimize } \frac{1}{2} \|\omega^2\| + C \sum_{i=1}^n (\xi_i + \xi_i^*) \quad (9)$$

subject to:

$$y_i - f(x_i, \omega) - b \leq \varepsilon + \xi_i^* \quad (10)$$

$$f(x_i, \omega) + b - y_i \leq \varepsilon + \xi_i^* \quad (11)$$

$$\xi_i, \xi_i^* \geq 0 \quad (12)$$

with C the positive constant (regular parameter) being a user-specified misclassification penalty and x_i was from sensors used. This formulation can be transformed into the dual problem given as:

$$f(x) = \sum_{i=1}^n (\alpha_i - \alpha_i^*) K(x_i, x) + b \quad (13)$$

where the dual variables are subject to constraints $0 \leq \alpha_i, \alpha_i^* \leq C$. The sample points of data that have nonzero coefficients in Equation (12) are called support vectors.

Afterward, in the general optimization, the optimal operation can be formulated as minimizing a process time function T :

$$\min_{\mathbf{u}, \mathbf{x}} \bar{T}(\mathbf{u}, \mathbf{X}, d) \text{ s.t. } \begin{cases} h(\mathbf{u}, \mathbf{x}_n, D(t)) \\ g(\mathbf{u}, \mathbf{x}_n, D(t)) \end{cases} \quad (14)$$

where \mathbf{u} is the degree of freedom; \mathbf{x}_n the sensor output; $D(t)$ the disturbance as time function; and T the process time.

As previously investigated by Datskov *et al.* (2006), the optimal operating point can be found by solving Equation (14) that must be realized through a control system. To implement the control system, from the optimal operating point, vector must be specified and the identification process stage classified with the SVM algorithm (Soenandi *et al.*, 2015). This method can also be used as a general framework for constrained multivariable optimization problems under insufficient system information (Wong *et al.*, 2008) that is suitable in the chemical reaction process, and for the previous research we developed an adaptive control method for the optimization of an esterification reaction (Soenandi *et al.*, 2015).

In order to implement the formulation in the esterification process, we used a commercial infrared LED source as made from original growth of narrow gap semiconductor alloys onto the n^+ -InAs substrate, optical coupling through the use of chalcogenide glasses and Si lenses with antireflection coating. Three types mid-IR sensors of $3.4\ \mu\text{m}$ LED-34SR full thread body, $5.5\ \mu\text{m}$ LED-55SR full thread body and $7\ \mu\text{m}$ OPLED 70 full thread body are shown in Figure 4, with the specification in Tables I-III; also the thermopile detectors from Heimann HTIA Dx-Tx and thermocouple as temperature sensors were installed in the reactor.

Several reaction and variation conditions were tested in Surfactant and Bioenergy Research Center Bogor Agricultural University.

In order to test various conditions of the temperature and reaction time, the temperature and reaction time was varied between 200 and 230°C and 100 - 120 minutes using a laboratory scale reactor with apparatuses such as a four-neck round-bottom flask, condensation tube, and Arduino with sensors that interfaced to a computer as set up in Figure 5.

In the validation phase, to measure the performance of the new system, the RTO system was built as shown in Figure 6. Stable operation is generally assumed for modeling and validation in RTO. Real-time sensing and data acquisition was examined first to ensure that this assumption is not violated and streams well to the database. For RTO of the esterification process, real-time data and laboratory data were integrated and merged first, both in a steady-state manner. In measuring the performance, yield is one of the most important indexes for the esterification process and calculated for operation evaluations in each batch basis.

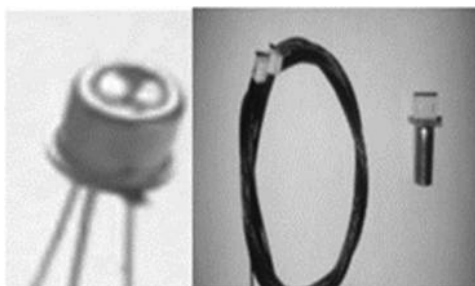
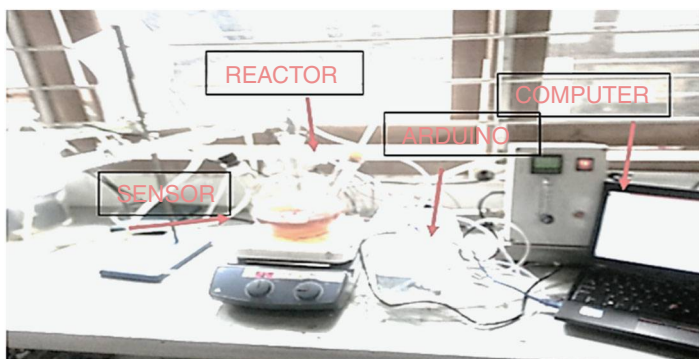


Figure 4.
LED MID-IR source
and detector

Table I. Specification of LED34Sr	Peak wavelength	μm	3.4 ± 0.05
	Pulse power	μW	$0.25 \div 0.35$
	CW voltage	V	Drive current $0.2\ \text{A}$ $0.26 \div 0.29$

Table II. Specification of LED55Sr	Peak wavelength	μm	$5.4 \div 5.5$
	Pulse power	μW	$5 \div 7$
	CW voltage	V	Drive current $0.2\ \text{A}$ $1.5 \div 2.5$

Table III. Specification of OPLED70	Peak wavelength	μm	$6.5 \div 7.0$
	Pulse power	μW	$5 \div 7$
	CW voltage	V	Drive current $0.2\ \text{A}$ $1.5 \div 2.5$



RTO using
gradient
adaptive
selection

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Figure 5.
Parts of apparatus
experiment in
laboratory

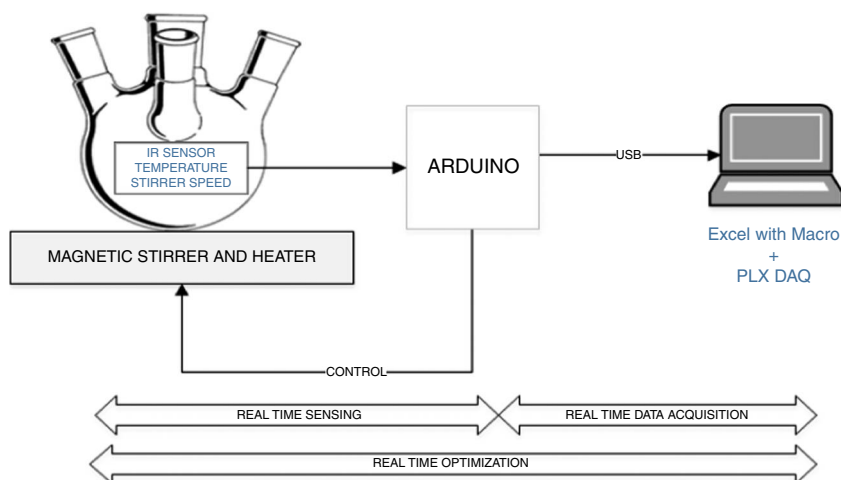


Figure 6.
Validation of
esterification process
diagram

4. Experimental results

For the first step, Business Process Modeling and Notation (BPMN) 2.0 (SAP, 2013) was used to analyze and design the system modeling for the esterification process by abstraction for conceptual design to describe the section of SO and NCO in detail; the diagram in Figure 7 describes the details of each task of digital apparatus and the communication between them, which will be the base structure for programming in Arduino to run the implementation step, such as data acquisition and controlling in the esterification process.

For the characterization tests, this study carried out several characterization tests for calibrating the signal from sensors, both for the identification and validation of the process results, such as FTIR and GCMS. The information (transmittance level) from the FTIR test would be used for selecting the wavelength. The selected wavelengths regarding the difference of transmittance level between before, during the process and after esterification were $3.4 \mu\text{m}$, $5.5 \mu\text{m}$ and $7 \mu\text{m}$, respectively. To confirm this selected wavelength a GCMS test was carried out to ascertain the level (yield) of glycerol that resulted from the end of esterification as the production target during the process span. Figures 8 and 9 display the spectrum from FTIR and GCMS generated by Sigmaplot 13.0, respectively (Systat, 2014).

The test of GCMS in a sample which has variance in temperature and process time, compared with the database of WILLEY09THL, it was detected as the reference number 587,486 with

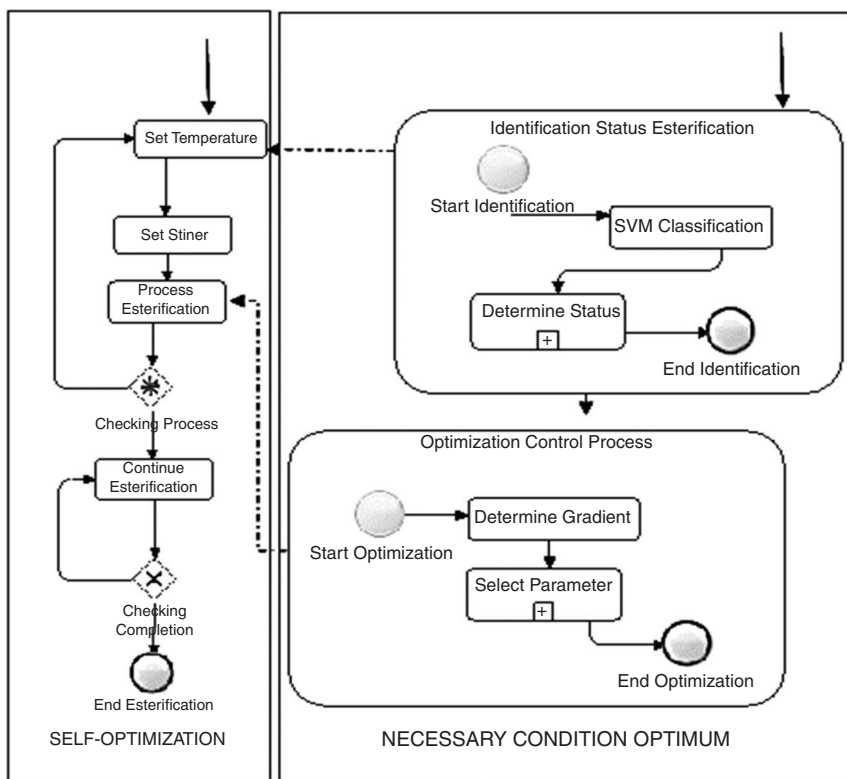


Figure 7.
BPMN diagram for
SO and NCO

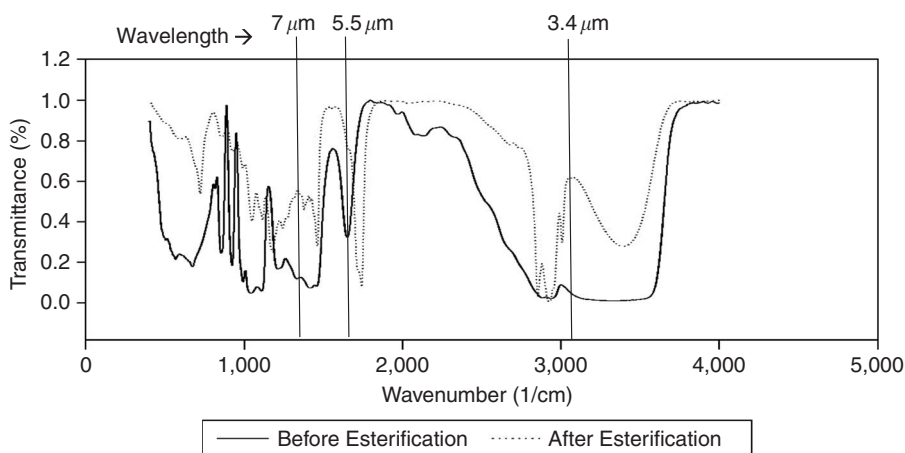


Figure 8.
Spectrum FTIR before
and after esterification

molecular weight = 356. The yield obtained at 200°C for the 100-minute process using raw material purity is 90 percent, which is the highest obtainable yield compared to another condition.

This model assisted in predicting and simulating the behavior of control with state, especially to control the heater to set the temperature in a reactor, using Stateflow from

MathWorks (2014). In this stage, we wanted to simulate a control logic tool used for modeling reactive systems via state machines and flow charts within a Simulink model with specific applications in mode logic, where each discrete mode of a system represented by a state was deployed as a control model in Stateflow (Figure 10).

In this research real-time data acquisition was developed. Data acquisition which is processing of sampling signals that measure real-world physical conditions (usually using a sensor) and converting the result into digital numeric values that can be manipulated by a computer. In this research, real-time data acquisition was operated in Arduino Mega 2560, connected via USB 2.0. The database was built in a personal computer with the specification of Core i5 2.2 GHz CPU, 4GB RAM, using Microsoft Excel with the add-in program named Parallax-Data Acquisition (PLX-DAQ), to collect the data from the sensor model listed in Table IV. Then, the task of selecting the sensor and the adaptive control was sequentially run in. The task of data acquisition from the sensors was collected in the one-second interval during 130 minutes in each batch process as implemented the formulation for optimization. Hence, the total data collected in the database was approximately 7,200-item data for each sensor while sensors were selected by using a gradient value minimum of 1.6 in 10 seconds' interval.

After the sensor selection step, we developed regression analysis to measure the relationships between variables. In this research, the regression was variables of sensor measurement to measure the yield of the esterification reaction, as the result in Table V.

We illustrate in Figure 11 a real-time data plot from data acquisition using three mid-IR sensors within 7,200-second time length; the value is digital bit number output related for the transmittance level in the esterification process for each wavelength by tracking of data. We implemented the classification on an Arduino microcontroller and compared the yield measurement using the regression method.

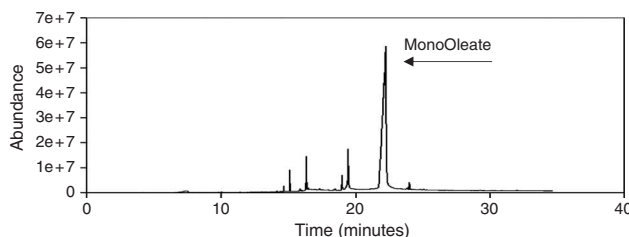


Figure 9.
GCMS Spectrum for
product testing with
setting variable 200°C
and 100 minutes

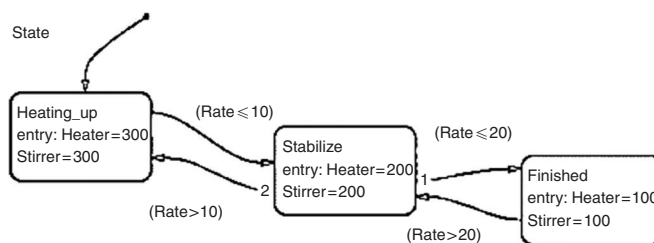


Figure 10.
Stateflow model of
esterification control

Symbol	Description	Availability	Sampling period	Number of samples
x_1	The temperature of the reactor	Real-time	1 s	7,200
x_2	The composition of the target yield	Real-time	1 s	7,200
x_3	The speed of agitation	Real-time	1 s	7,200

Table IV.
List of sensor model

The measurement of yield from regression was resulted by collected data input from sensors. We compared it by setting in a non-adaptive and adaptive mode using two samples of purity. To find the different yield measurement from each mode, as for validation, there was a slight difference in yield measurement between adaptive and non-adaptive methods as shown in the plot diagram in Figure 12.

For validation purpose, it is necessary to indicate related information of adaptive selection sensors' performance using gradient with the regression to measure the yield measurement between non-adaptive and adaptive sensor selection using two samples of raw material as in

Table V.

Yield regression for adaptive sensor selected in each state

State	Sensor selection			Regression
	3.4 μm (X) ₁	5.4 μm(X) ₂	7 μm(X) ₃	
1	ON	ON	ON	$Y = -33.62390 + 1.04874(X)_1 + 0.8362(X)_2 - 0.29288(X)_3$
2	OFF	ON	ON	$Y = 59.85565 - 0.04523(X)_2 + 0.062339(X)_3$
3	ON	ON	OFF	$Y = 26.77324 + 0.039855(X)_1 + 0.709494(X)_2$

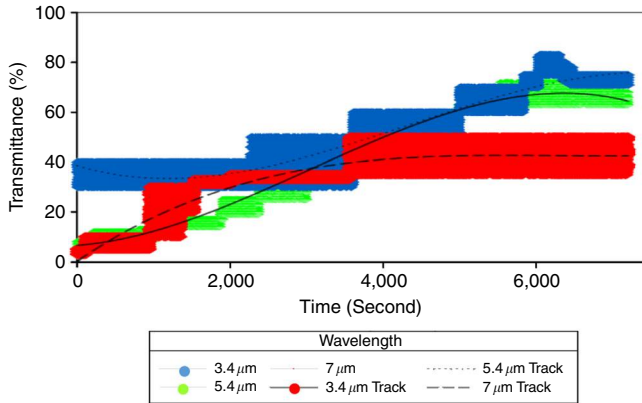


Figure 11.
Real time data sensor plot

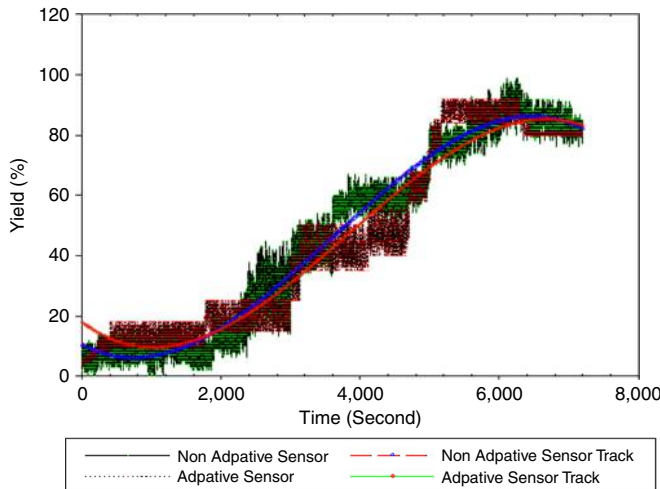


Figure 12.
Comparison of yield measurement from sensors

Table VI, showing that using the adaptive method leads to the lowest percentage error. From our model, the output example of SVM in state control is listed partially in Table VII.

The comparison of temperature in the reactor is as shown in Figure 13. From this data acquisition plot of temperature, we can compare the optimization with parameter adaptation operated with a maximum temperature at 250°C, to RTO with classification and gradient which was operated with a maximum temperature at 200-210°C, showing that RTO was more efficient as it consumed less energy. Finally, optimization indicators in real time compared with non-controlled methods as operated manually by the experience of the operator and by using RTO methods are summarized in Table VIII.

Table VI.
Comparison yields
from model parameter
adaptation,
non-adaptive and
adaptive methods

Sample	Model parameter adaptation	Yield (%)		GCMS test
		Non-adaptive sensor	Adaptive sensor	
80% purity glycerol Processed in 210°C and 105 minutes	70	75 Percentage error 6.25%	79 Percentage error 1.25%	80
90% purity glycerol processed in 200°C and 100 minutes	85	85 Percentage error 5.55%	89 Percentage error 1.11%	90

Table VII.
Example output for
SVM in state control

Sampling time (min)	Selecting sensor			Variable controlled	
	Sensor 1 (value)	Sensor 2 (value)	Sensor 3 (value)	State of temperature	State of agitation
0	On (10)	On (15)	On (16)	1	1
45	On (20)	Off	On (40)	1	2
60	On (27)	Off	On (46)	2	2
90	Off	On (60)	On (64)	2	3
120	Off	On (73)	On (78)	3	3

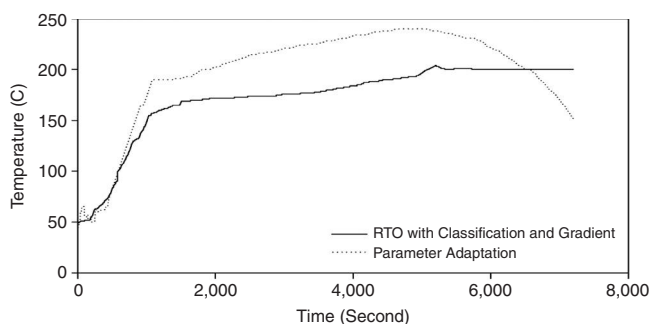


Figure 13.
Temperature
difference in reactor
using RTO and
parameter adaptation
method

Table VIII.
Comparison of
optimization
indicators

Indicators	Existing (non-controlled)		Comparison RTO with raw material 80%	
	Parameter adaptation		RTO with raw material 90%	
Process time (minutes)	120	120	105	100
%Yield	76	79	79	89
Agitation speed (RPM)	200-500	200-500	300-400	300-400
Max temperature (°C)	250	240	210	200
Avg. temperature (°C)	192	195	175	172

5. Conclusion

This research presents a new RTO system using adaptive selection and classification from infrared sensor measurement for esterification oleic acid with glycerol. As a result of this research, we have presented an abstraction for a conceptual model of the optimization of glycerol esterification in real time with BPMN 2.0 to ensure the implementation of RTO. Adaptive selection sensors' works using a gradient value minimum at 1.6 with a 10-second time interval, followed by SVM and state control with three states, had achieved a good performance. For system validation, an RTO using gradient adaptive selection and classification from the sensor measurement approach to optimize the esterification process shows that the responsiveness of control increased product yield up to 13 percent and reduced the required process duration up to 22 minutes, with an effective range of stirrer rotation set between 300 and 400 rpm and final temperature between 200 and 210°C which was more efficient as consuming less energy. For future research, we still need the development of a high-temperature resistance sensor for the reactor and implementation of RTO for the continuous system of production.

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Further reading

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