Jurnal Metris, 16 (2015): 63 – 68

Jurnal
Metris
ISSN: 1411 - 3287

Estimation Quality Monitoring Glycerol Esterification Process with IR Sensors Using K Nearest Neighbours Classification

Iwan Aang Soenandi, Johansah Liman, Budi Harsono

Faculty of Engineering and Computer Science
Krida Wacana Christian University, Jakarta
Email: Iwan.as@ukrida.ac.id;johansah@ukrida.ac.id;
budi.harsono@ukrida.ac.id

Received 1 April 2015; Accepted 1 October 2015

Abstract.

The commercial synthesis of fatty acid esters of glycerol is important because it can be used for other derivative production varieties. This research aims to construct the quality monitoring system for esterification condition faster and more efficient for the production of esterification glycerol. The monitoring systems were based on the measurement parameters from two inputs LED mid IR 3,4 and 5,5 µm sensors that using the data acquisition with computer database via USB 2.0 using Arduino Leonardo microcontroller and classifying the esterification quality condition using the classification method K-Nearest Neighborhood (KNN) The purpose of KNN method is to classify the variations of parameter inputs from the LED mid IR sensors in quality monitoring. In this research the condition of esterification was divided into three conditions: not good, fair,good., these classification was trained and tested in Orange Software for data mining using receiver operating characteristic (ROC) curve that is a graphical plot that illustrates the excellent performance of a classifier system for esterification condition with AUC . In application for quality monitoring, the influence of various parameters such as temperature set in the reactor has relation to the quality of product. By using this system, we obtained the optimum process conditions is 200^{o}C and time needed for the process was 200 minutes.

Keyword: *Quality monitoring, glycerol esterification, infrared sensor, KNN clustering, experimental design*

1. PENDAHULUAN

In chemical industrial process scale, the productionto react fatty acid to make esters of glycerol is carried out by two different ways: direct esterification of fatty acid with the glycerol and catalyzed by a homogenous acid (Isabel et al., 2003). Synthesis by direct esterification widely used in the esterification of glycerol because these process is simple and feasible in batch production system (Pardi, 2005). In general, industrial engineering is concerned with the quality of production and service systems. The major factors affecting the quality of esterification process are molar ratio of oil, amount of catalyst, reaction temperature, catalyst type and stirring speed according the reaction duration (Mostafa & Maher, 2013). But in these research we focus on quality monitoring with related by the temperature and process time needed. Determination of the quality for esterification is highly desirable for Glycerol ester product in order to increase the efficiency energy and cost of production. A current method for determination of esterification is sampling and needs highly cost and time. The qualities of some products of chemical reaction are generally measured using chromatographic methods (Patz et al., 2004; Fairbrother et al., 1991; Yu et al., 2006), which are highly accurate; however, these methods also have some disadvantages such as the long time required and high operating costs, which limit their application in a rapid process control system (Sinelli et al., 2004). today a main objective is to adapt quickly in order to start a new production or to react in a failure occurrence. Intelligent manufacturing systems (IMS) offer not only both flexibility and reconfigurability, but also this concept brings more than a few ideas of software intelligence meanings, which contemplated characteristics such as autonomy, decentralization, flexibility, reliability, efficiency, learning, and self-regeneration (Revilla

& Cadena, 2008). Thus, a spectroscopic methodone of the most powerful nondestructive techniques, has been applied in process control to manage fermentation substrates and products. In industrial engineering aplication ,methods of classification for robust system needed in monitoring especially has dynamic condition (Turney, 1993) In this paper to determine the estimation quality esterification process in real time, online monitoring directly in the product and close to critical components is highly desirable. Optical measurement techniques are promising candi dates spectral region i.e., multi-channel NDIR (non-diffractive infrared) absorption or IR spectroscopy. The later method is also used in laboratories, thus allowing better correlation online data and laboratorium results with data acquisition interfaced by database to the computer. The correlation used for calibration parameter in online measurement sensors especially to identification esterification condition. Sinelli et al. demonstrated a set up with detector array combined with the gradient filter to avoid the need for movable parts (Wiesent et al., 2011). Wiesent et al. presented a system with an infrared source, a fluid cell consisting of two sapphire windows and aquadruple infrared detector equipped with different filter windows for analysis of phosphate ester . The objective of these paper is to develop the system for identification of esterification condition um variable related to temperature in the reactor and process time of the esterification processusing LED mid IR sensors and data acquisition system and find the optimum variable related to temperature in the reactor and process time. In this paper for Section 2 we briefly explain materials and methods. In Section 3 we briefly review the concept of classyfying esterification condition with KNN and the conclusion are discussed in Section 4.

2. MATERIALS AND METHODS

2.1 Materials

In this research we are using pure glycerol with purity 85-90% and synthesize it, with Oleic acids pro analysis grade specification in 1:1 ratio, mixed in reactor with heater and stirring blade. For the catalyst we used Metyl Ester Sulfonic Acid (MESA) 0.5%.

2.2 Equipment

Esterification reactions were carried out in a laboratory-built apparatus. An apparatus consists of laboratory conical flask 1000 ml with 450 ml working volume. Esterification reaction was under atmospheric pressure (opened system), temperature of the reactor was controlled using hot plate (controlled with internal thermostat) as in Fig 1. All the reactants (oleic acids, pure glycerol and catalyst) were weighted and charged into the reactor. Then the temperature was increased through adjusting the thermostat. The magnetic stirrer was allowed to operate after 2 - 3 min (to heat up the mixture). After passing the desired reaction time, the reactor was removed from the hot plate. Samples were withdrawn from the reaction mixture for analysis. The reaction mixture was cooled to the ambient temperature by immersing it into a water bath.Also the esterification process in closed system was investigated, where all there actors were isolated.

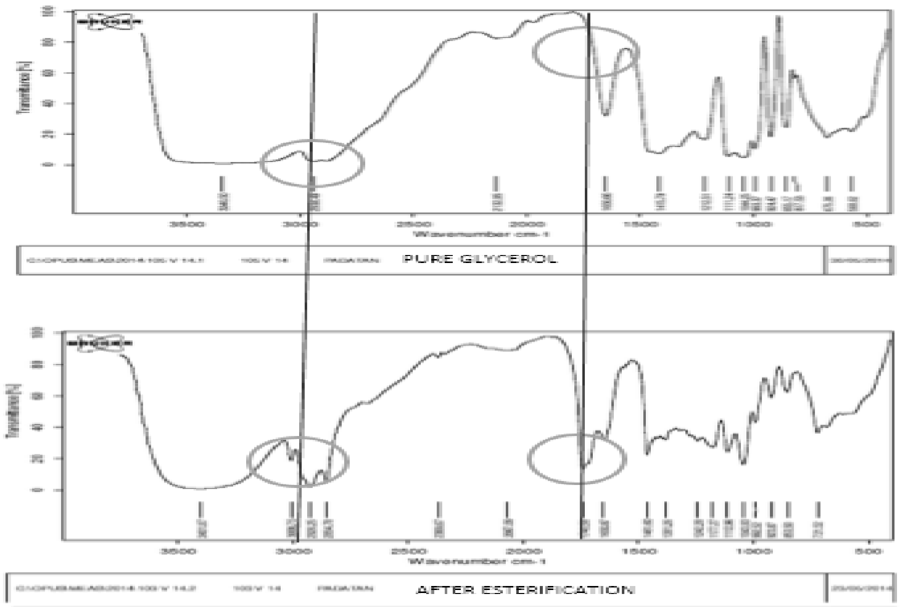

Figure 1. Mid IRSpectrum Before and After Esterification Proces

2.3 Methods

Variation Condition

Several reaction condition were tried to get variation condition in temperature and reaction time. For the temperature being varied from150 ^{o}C to 230 ^{o}C. For the process time between 180-260 minutes

Sensor System

A commercial infrared LED source as original growth of narrow gap semiconductor alloys onto n+-InAs substrate, optical coupling through the use of chalcogenide glasses and Si lenses with antireflection coating(Boston Electronic,2014) 3,4 micron LED-34SR full thread body and 5,5 micron LED-55SR full thread body and also the thermopile detectors (Heimann 2014) HTIA DxTx as in Figure 2. to reach steady state and to record signal amplitude with a good signal to-noise ratio.

System Data Acquisition

To ensure the data parameters get collected from the sensors , the system was interfaced with database like MySQL. In this research we build the data acquisition system using Arduino Leonardo microcontoler with Universal Serial Bus (USB) connector to connect data streaming in real time with computer. And processing the output with KNN method (Hall et al., 2008).

Figure 2. LED IR Source

Table 1. Specification of LED34Sr

Peak wavelength	µm	3,4±0.05
Pulse Power	mW	0.25÷0.35CW
Voltage	V	Drive Current0.2A 0.26÷0.29

Table 2. Specification of LED55Sr

Peak wavelength	µm	5,4÷5.5
Pulse Power	mW	5÷7 CW
Voltage	V	Drive Current 0.2A 1.5÷2.5

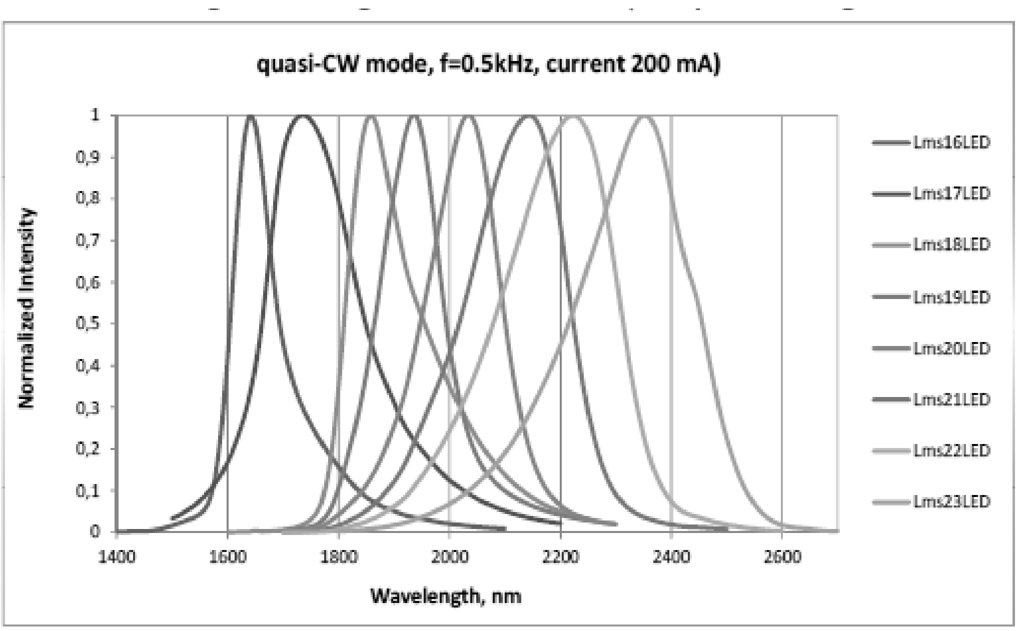

Figure 3. LED IR Source Spectral Range

Table 3. Example of data acquisition

Sample	Parameter Value(Volt)			Quality
	Sensor1	Sensor2	Sensor3	
1	1.300	1.400	1.100	2
2	1.400	1.900	1.200	1
3	1.600	1.700	1.100	1
4	1.200	1.700	1.900	3
5	1.400	1.400	1.800	2
6	1.200	1.400	1.200	2
7	1.000	1.500	1.000	3
8	2.000	1.800	1.800	1
9	1.000	2.000	1.900	2
10	1.600	1.200	1.200	2

3. RESULTS AND DISCUSSION

3.1 Quality Esterification with KNN

In this research for data acquisition we are using online data measurement interfaced with SQL database and identification parameter inputs by optical sensors MID IR. The input database from sensor collected in database and using KNN method to clustering the quality of esterification product to analyze the performance of system parameter like temperature and process time because esterification process was closely related to that parameter. With computer software application Orange ver 2.6.1 the KNN method was trained and tested by processing data file 250 examples, 2 attributes and Classification Discrete Class with 3 values.

From Hall (2008), We can represent our data set as a matrix D=NxP , containing P scenarios (data acquisition) $s^1,...,s^p$, where each scenario s^i contains N featuressi=$\{s_1^i,...s_N^i\}$ from sensor 1 and sensor 2 . A vector o with length P of output values o=$\{o^1,...,o^p\}$ accompanies this matrix, listing the output value o^i for each scenario s^i .

It should be noted that the vector can also be seen as a column matrix; if multiple output values are desired, the width of the matrix may be expanded.

KNN can be run in these steps:

1. Store the output values of the M nearest neighbors to query scenario q in vector r=$\{r^1,...,r^m\}$ by repeating the following loop M times:

a. Go to the next scenario in the data set, where is the current iteration within the domain {1,...,P}
b. If is not set or $q<d(q,s^i):q\leftarrow d(q,s^i)$
c. Loop until we reach the end of the data set (i.e.i=P)
d. Store q into vector c and into vector r

2. Calculate the arithmetic mean output across as follows:

$$\bar{r} = \frac{1}{M}\sum_{i=1}^{M} r_i$$

3. Return r as the output value for the query scenario q

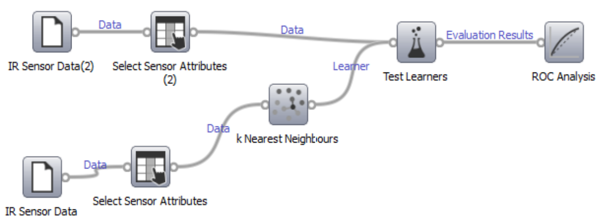

Figure 4. Schema in Orange data mining software model

Table 4. KNN Parameter

Metrics	Euclidean
Continuous Attributes	Normalized
Number of Neighbours	3
Weighting	By distance

As a result from our research, we have using KNN method to classify the input parameter that identified by the sensor as a voltage parameter and clustered into quality .After the quality was clustered the performance of KNN was tested using ROC analysis.

3.2 ROC Analysis

With ROC curve that is a graphical plot that illustrates the performance of a classifier system as its discrimination threshold is varied. The curve that is generated by Orange Software data mining shows by plotting the true positive rate against the false positive rate at various threshold settings. For the three predicted class as in Fig 5- 7. we can find the performance of classifying of KNN.

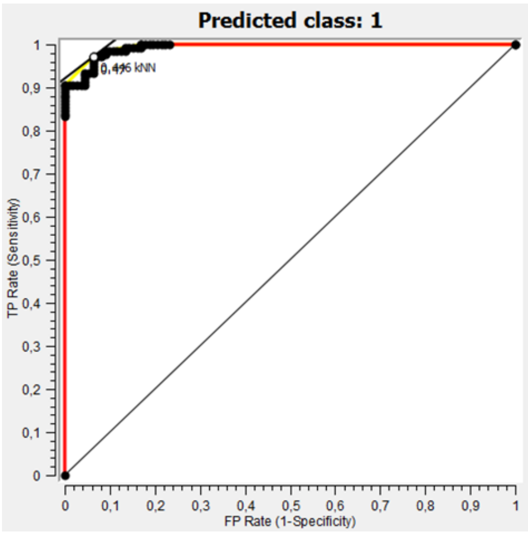

Figure 5. ROC analysis curve Predicted Class 1:Good Quality

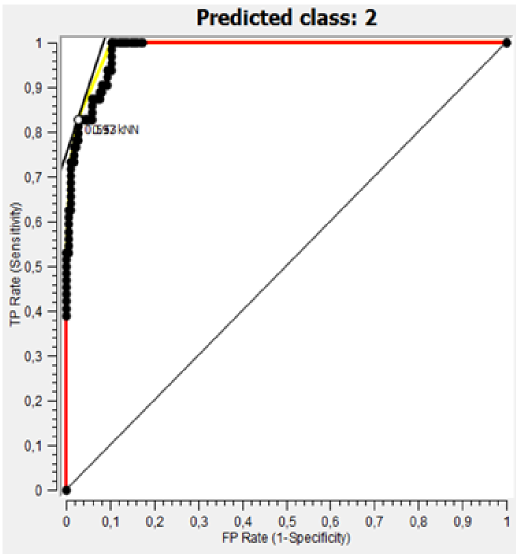

Figure 6. ROC analysis curve Predicted Class 2:Fair Quality

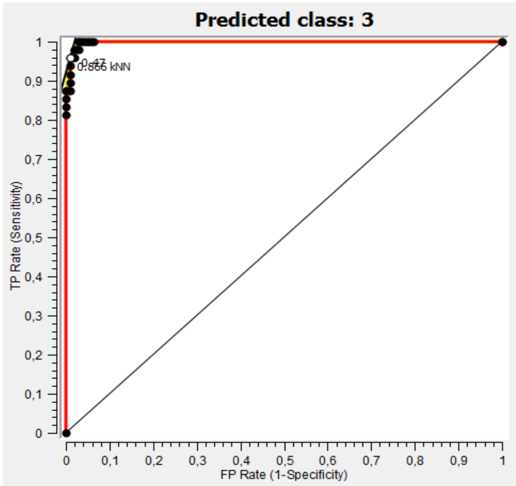

Figure 7. ROC analysis curve Predicted Class 2:Bad Quality

Evaluation Results

	Method	CA	Sens	Spec	AUC	IS	Brier
1	kNN	0.9238	0.9562	0.9375	0.9951	1.2562	0.1131

Figure 8. Evaluation Results from Orange Software

The evaluation result from data mining orange software that showed with KNN method for quality clustering in this research using data from sensor LED Mid IR . The machine learning community most often uses the ROC AUC(Area Under Curve) statistic for model comparisonin Fig 8. (Hanczar et al., 2010; Wiesent et al., 2011) and for make sure the model could be used in industrial engineering aspect. A reliable and valid AUC estimate can be interpreted as the probability that the classifier will assign a higher score to a randomly chosen positive example than to a randomly chosen negative example (Yu et al., 2006). In this research we get AUC 0.9951 it means the KNN method was valid and the quality was clustered very well with 3 cluster which is: Good, Fair and Bad.

3.3 Effect of Temperature and Time

Both temperature and time are having major effects on the conversion of the esterification process. Accordingly, they were studied and optimized together. The obtained results showed that that by increasing the reaction temperature, the reaction conversion increases rapidly. Figure 8. shows that after 240 min, the esterification reached well formed esterification condition. Therefore another esterification reaction was carried out within a temperature range of 160°C - 220°C as shown in Table 5. The results revealed that by increasing the esterification time, the esterification yield increased up to a maximum conversion. To determine the optimum temperature of esterification process, not only the maximum yield of esterification should be considered but also the time required to reach the reaction temperature was taken into account. Heating time to reaction temperature was certainly longer and energy consumption was surely greater for higher reaction temperature. Consequently, a faster reaction at a lower temperature is desirable.

Table 5. Quality Measurement with variation condition

Average Temperature (^{o}C)	Time (minutes)	Quality Range
160	150	2-3
180	170	2-3
200	200	1-2
220	220	2-3

4. CONCLUSION

The system of real time monitoring glycerol esterification process with mid IR sensors was contribute to support identification for quality esterification and get information for the optimal condition of the process. These quality esterification has good performance by classified into 3 cluster: Good, fair and bad quality, these classification was trained and tested in Orange Software for data mining using KNN method and tested the performance of the classifier using ROC analysis. In application for esterification optimization, the influence of various parameters such as temperature set in the reactor has relation to the process time needed. By using these quality monitoring system based on the measurement and classification estrification forming using KNN from two input LED mid IR 3,4 and 5,5 µm sensors, we obtained the optimum process conditions is 200^{o}C and time needed for the process was 200 minutes (Table 4.).These parameter was different from traditionally method that set the temperature 180 ^{o}C with 170 minutes.

5. DAFTAR PUSTAKA

1. Fairbrother, P., W.O. George, J.M. Williams. (1991) Whey fermentation: on-line analysis of lactose and lactic acid by FTIR spectroscopy. *Appl Microbiol Biotechnol.* 35: 301-305
2. Hall P, Park BU, Samworth R., J. (2008). Choice of neighbor order in nearest-neighbor classification. *Annals of Statistics* 36 (5): 2135–2152. doi:10.1214/07-AOS537.
3. Hanczar, Blaise; Hua, Jianping; Sima, Chao; Weinstein, John; Bittner, Michael; and Dougherty, Edward R. (2010); Small-sample precision of ROC-related estimates, *Bioinformatics* 26 (6): 822–830
4. Hanley JA, Hajian-Tilaki KO (1997) Sampling variability of nonparametric estimates of the areas under receiver operating characteristic curves: an update. *Academic Radiology* 4:49-58.
5. Isabel, D., Federico, M., Joaquın, P. and Enrique, S. (2003). Synthesis of MCM-41 materials dialkylsilane groups and their catalytic activity in functionalised with the esterification of glycerol with fatty acids. *Applied Catalysis A*: *General*, **242**, 161-169. doi:10.1016/S0926-860X(02)00501-X
6. Lobo, J. M.; Jiménez-Valverde, Alberto; and Real, Raimundo (2008), AUC: a misleading measure of the performance of predictive distribution models, *Global Ecology and Biogeography*, 17: 145–151.
7. Mostafa NA, Maher A. (2013). Production of Mono-,Di,-, and Triglycerides from waste fatty acid through esterification with glycerol. *Advanced in Bioscience and Biotechnology*, 4: 900-907.
8. Pardi. (2005). Optimasi proses produksigliserolmonooleatdarigliserolhasilsampingpembuatan biodiesel. *Thesis Magister*. Sekolah Pascasarjana Universitas Sumatera Utara.
9. Patz, C., D., A. Blieke, R. Ristow, H. Dietrich. (2004). Application of FT-MIR spectrometry in wine analysis. *Anal ChimActa.* 513; 81-89.
10. Revilla, J., & Cadena, M. (2008). Trends in intelligent manufacturing systems. *Proceeding of the World Congress on Engineering,* London, UK.
11. Sinelli, N., de Dionigi, S., Pagani, M.A., Riva, M., Belloni, P., 2004.Application of NIR spectroscopy in on-line monitoring of doughproofing and bread staling. *Tecnica Molitoria* 55,1075–1092.
12. Turney, P. (1993). “Robust Classification with Context-Sensitive Features”, Proceedings of the Sixth International *Conference onIndustrial and Engineering Applications of Artificial Intelligence and Expert Systems (IEA/AIE-93):* 268-276. 1993.
13. Wiesent, B. R., Dorigo, D. D., and Koch, A. W. (2011). Suitability of tunable Fabry-Perot spectrometers for condition monitoring pur- poses of gear oils in offshore wind turbines, *Proceedings IRS2 2011 – 12th International Conference on Infrared Sensors and Systems,* Nuremberg, 82–87, doi:10.5162/irs11/i4.2.
14. Yu, H., Y. Ying, X. Fu, H. Lu. (2006). Quality determination of Chinese rice wine based on Fourier transform near infrared spectroscopy. *Near Infrared Spectrosc.* 14: 37-44.