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Trans-esterification of Palm Oil in Series of Continuous Stirred Tank Reactors

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Abstract. Design of a continuous reactor for producing saleable biodiesel based on making its mixing performance as high as possible is inefficient. One way to solve the inefficiency is by simulation. In this study a simulation was performed to optimize a mixing performance of a continuous reactor for producing saleable biodiesel from palm oil and to predict the required residence times at the selected purities for the transesterification of palm oil in the optimized reactor. In the simulation, continuous reactors were modeled as *n* ideal continuous stirred tank reactors (CSTRs) in series. The equations systems for the transesterification of palm oil in CSTR and plug flow reactor (PFR) were constructed and solved at various residence times. The intrinsic rate was based on the experimental results for the transesterification of palm oil with methanol in the presence of NaOH as a catalyst at the reported optimum reaction condition [1]. The optimum mixing performance of a continuous reactor was 6 ideal CSTRs in series. The predicted residence times of the optimized reactor to produce palm methyl esters at purities 96.5, 97.0, 97.5, 98.0, and 98.5 %wt were 2.85, 3.15, 3.61, 4.32, and 6.24 min, respectively. In addition, the efficiencies of this reactor at purities 96.5, 97.0, 97.5, 98.0, and 98.5 %wt were 66%, 64%, 62%, 59% and 53%, respectively.

Keywords: Biodiesel, Palm Oil, Transesterification, Triglycerides, Methyl Esters, Continuous Reactor.

Introduction

Presently, biodiesel has become more attractive in many countries, including Thailand. It is a renewable and environmental friendly energy resource; it can be produced from vegetable oils and tallow; and it also gives environmental benefits, especially carbon dioxide saving [2,3]. In Thailand, several vegetable oils can be used as a feedstock; however, the highest potential source is oil from palm fruits: in the year 2001, 535 kilotons of palm oil were produced [4]. Consequently, success of using biodiesel from palm oil commercially is an important factor for Thailand's sustainable development; the positive economic effects and an increase of energy security would be achieved. However, first of all, the production technology must be developed.

The most recent method for biodiesel production is batch transesterification processes; however, generally, continuous processes give lower production cost and more uniform product quality than batch processes. A number of researchers have tried to develop continuous processes and continuous reactors for biodiesel production. Darnoko and Cheryan [5] evaluated a simple continuous stirred tank reactor (CSTR) for transesterification of palm oil. They reported that its mixing performance followed an ideal CSTR pattern; it could produce the highest purity at 97.3 %wt within the optimum residence time of 60 min at temperature 60 ^{0}C, molar ratio of methanol to oil 6:1, and KOH concentration 1.0 %wt of oil. Noureddini *et. al.* [6] evaluated a continuous process for transesterification of soybean oil in a pilot plant. This process consists of 2 motionless mixers, 1 high-shear mixer, and 1 residence tube. This process could produce a purity of more than 98 %wt within a residence time of 7 min at temperature 80 ^{0}C, molar ratio of methanol to oil 6:1, and NaOH 0.4 %wt of oil. However, its mixing performance was not reported. Harvey and Mackley [7] evaluated the production performance of oscillatory flow reactor (OFR) for rapeseed methyl esters production. Its mixing performance could be equivalent to as many as 80 ideal CSTRs in series. It could produce saleable biodiesel within 15 to 30 min for the following reaction conditions: temperature 60 ^{0}C and 70 ^{0}C, 50% excess methanol, and NaOH 0.3 to 0.5 %wt of oil.

Although an ideal CSTR could produce saleable biodiesel, it still needed long residence time. In fact, a higher mixing performance reactor can produce saleable biodiesel in a shorter time; and a higher ratio of production capacity per unit volume of reactor can be achieved; as a result, both capital cost and operation cost can be lowered. However, excess mixing performance is also a disadvantage. Generally, a high mixing performance reactor is often a complex form; it needs more equipment, complex operation, and complex control system; both higher costs can come up again. Significantly, design of a continuous reactor for producing saleable biodiesel based on making its mixing performance as high as possible is inefficient.

Even though there are a number of publications concerning the development of continuous reactors for biodiesel production, there is no publication dealing with optimization of a mixing performance of a continuous reactor to produce saleable biodiesel. In principle, if the intrinsic rates of the chemical reactions are known, any type of reactor may be designed by introducing the rates of the appropriate physical processes associated with that type of equipment [8]. Moreover, the mixing characteristic of any continuous reactor can be modeled as n ideal CSTRs in series [9]. As a result, simulation study based on intrinsic rate of chemical reaction and design equation of an ideal CSTR can be used to optimize a mixing performance of a continuous reactor for a particular chemical reaction.

The objectives of this work were to optimize a mixing performance of a continuous reactor for producing saleable biodiesel from palm oil and to predict the required residence times at the selected purities for the transesterification of palm oil in the optimized reactor. The obtained result can be used efficiently to design a suitable continuous reactor for producing saleable biodiesel from palm oil.

Methods

System model

To optimize a mixing performance of a continuous reactor, a system model of a continuous reactor was constructed consisting of *n* tanks in series of an equal residence time (r_i) ideal CSTR as shown in Fig 1.

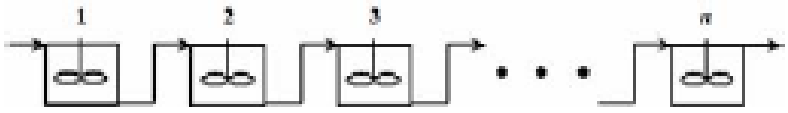

Fig. 1 System model of a continuous reactor as *n* ideal CSTRs in series.

Thus, the total residence time of a continuous reactor or *n* ideal CSTRs in series is

$$\tau_t = \sum_{i=1}^{n} \tau_i \qquad (1)$$

where τ_t is total residence time of *n* ideal CSTRs in series (s); τ_i is a residence time of tank *i* (s); and *n* is number of tanks.

Transesterification of palm oil in an ideal CSTR

In order to predict the amount of methyl esters produced either in an ideal CSTR or in *n* ideal CSTRs in series at any residence time, the system of calculation equations for transesterification of palm oil in an ideal CSTR must be constructed and solved.

The system of calculation equations for transesterification of palm oil in an ideal CSTR was constructed based on: the design equation of an ideal CSTR operated at a steady state [9] and the second order rate equations without the shunt reaction characterizing the stepwise reactions for transesterification of vegetable oils [10,11]. In addition, generally, the volume of liquid-phase reaction changes insignificantly during the reaction. Which is unlike gas-phase reaction? The constant volume is often applied for liquid-phase reaction [9]. Moreover, observations in batch transesterification of palm oil did not find a significant change of the volume of reaction mixture during the reaction. As a result, the system of calculation equations was also based on constant volume of reaction mixture. The obtained system of calculation equations is shown as following.

$$
\begin{aligned}
&C_{TG,Out} + (k_1 C_{TG} C_A - k_2 C_{DG} C_{ME})\tau && = C_{TG,In} \\
&C_{DG,Out} + (-k_1 C_{TG} C_A + k_2 C_{DG} C_{ME} + k_3 C_{DG} C_A \\
&\quad - k_4 C_{MG} C_{ME})\tau && = C_{DG,In} \\
&C_{MG,Out} + (-k_3 C_{DG} C_A + k_4 C_{MG} C_{ME} + k_5 C_{MG} C_A \\
&\quad - k_6 C_{GL} C_{ME})\tau && = C_{MG,In} \\
&C_{GL,Out} + (-k_5 C_{MG} C_A + k_6 C_{GL} C_{ME})\tau && = C_{GL,In} \\
&C_{ME,Out} + (- k_1 C_{TG} C_A + k_2 C_{DG} C_{ME} - k_3 C_{DG} C_A \\
&\quad + k_4 C_{MG} C_{ME} - k_5 C_{MG} C_A + k_6 C_{GL} C_{ME})\tau && = C_{ME,In} \\
&C_{A,Out} + (k_1 C_{TG} C_A - k_2 C_{DG} C_{ME} + k_3 C_{DG} C_A \\
&\quad - k_4 C_{MG} C_{ME} + k_5 C_{MG} C_A - k_6 C_{GL} C_{ME})\tau && = C_{A,In}
\end{aligned}
\qquad (2)
$$

where C is mole concentration (mol/L); τ is residence time (s); k_1 to k_6 are kinetic rate constants (L/mol.s); subscript *TG, DG, MG, GL, ME,* and *A* are denoted for triglycerides (TG), diglycerides (DG), monoglycerides (MG), glycerolr (GL), methyl esters (ME) and alcohol; subscript *In* is denoted for entering condition; subscript *Out* is denoted for exit condition.

In addition, an ideal CSTR is referred to a tank reactor that is completely mixed; thus, the concentration is identical everywhere within the tank and similar to the concentration at the exit point [9].

The reaction rate constants, k_1 to k_6, for transesterification of palm oil with methanol in the presence of NaOH as catalyst at atmospheric pressure were obtained experimentally from our previous work. The experiment was based on the reported optimum condition for transesterification of palm oil as follows: 6:1 molar ratio of alcohol to oil, temperature 60 ^{0}C, and catalyst concentration 1% wt of oil [1]. The obtained reaction rate constants are shown in Table 1. These reaction rateconstants provided a good fit with experimental results. The overall goodness of fit to predict a weight percentage of ME in the product during the reaction was quantified through two standards: the correlation coefficient (*R2*) and the mean relative deviation (MRD). The R2 was 0.9936 and the MRD was 1.835%.

Table 1 The reaction rate constants (L/mol.s)

k_1	1.057×10^{-2}
k_2	0.000
k_3	1.184×10^{-1}
k_4	8.187×10^{-2}
k_5	1.310×10^{-1}
k_6	2.011×10^{-3}

The system of equations, Eq. (2), can be rewritten in matrix form of system of nonlinear equations as following.

$$\begin{bmatrix} \frac{\partial g_1}{\partial C_1} & \frac{\partial g_1}{\partial C_2} & \frac{\partial g_1}{\partial C_3} & \frac{\partial g_1}{\partial C_4} & \frac{\partial g_1}{\partial C_5} & \frac{\partial g_1}{\partial C_6} \\ \frac{\partial g_2}{\partial C_1} & \frac{\partial g_2}{\partial C_2} & \frac{\partial g_2}{\partial C_3} & \frac{\partial g_2}{\partial C_4} & \frac{\partial g_2}{\partial C_5} & \frac{\partial g_2}{\partial C_6} \\ & & \vdots & & & \\ \frac{\partial g_6}{\partial C_1} & \frac{\partial g_6}{\partial C_2} & \frac{\partial g_6}{\partial C_3} & \frac{\partial g_6}{\partial C_4} & \frac{\partial g_6}{\partial C_5} & \frac{\partial g_6}{\partial C_6} \end{bmatrix} \begin{Bmatrix} C_1 \\ C_2 \\ C_3 \\ C_4 \\ C_5 \\ C_6 \end{Bmatrix} = \begin{Bmatrix} C_{1,In} \\ C_{2,In} \\ C_{3,In} \\ C_{4,In} \\ C_{5,In} \\ C_{6,In} \end{Bmatrix} \qquad (3)$$

where g_1 to $g6$ are the functions that represent the terms on the left hand side of equations system, Eq. (2), respectively; C_1 to $C6$ are the exit mole concentrations of TG, DG, MG, GL, ME, and alcohol; C_1,In to C_6,In are the entering mole concentrations of TG, DG, MG, GL, ME, and alcohol. Eq. (3) is 6 unknowns and 6 equations system. The unknowns are the exit mole concentrations, C_1 to C_6. Generally, the approximate solutions of the above system of nonlinear equations can be solved by various numerical techniques [12]. A couple of least-squares regression technique and the ability of solver tool in Microsoft Excel 2000 program also can be used to solve the above system of nonlinear equations, which was used to find the best approximate solutions that gave the minimize value of sum of error squares (*E2*) as shown in the following equation.

$$E^2 = \sum_{i=1}^{6} \left[C_{1,In} - \left(\frac{\partial g_1}{\partial C_1} C_1 + \frac{\partial g_1}{\partial C_2} C_2 + \frac{\partial g_1}{\partial C_3} C_3 + \frac{\partial g_1}{\partial C_4} C_4 + \frac{\partial g_1}{\partial C_5} C_5 + \frac{\partial g_1}{\partial C_6} C_6 \right) \right]^2 \qquad (4)$$

However, to ensure that this method provides correct solutions, the error square of each equation must be checked to evaluate the correctness of the obtained solutions. This value should as near zero as possible. The observed error squares in this simulation ranged from 2.27 x 10^{-17} to 6.07 x 10^{-4}. The biggest error square occurred when the biggest residence time was used in calculation (60 min). These results confirmed that this method could be used to solve the above system of nonlinear equations while the correct solutions were obtained. On the other hand, if the other numerical methods were used, the largest acceptability of the error squares also had to be given in a computer code.

Furthermore, it is a nature of a numerical technique that initial guesses affect the obtained results. Some initial guesses give divergent results; some initial guesses give convergent results. For the latter case, different initial guesses can also give different results. Hence, different initial guesses were used. The ranges of results for reaction rate constants were obtained. However, the ranges were very narrow.

A purity of biodiesel is represented by the weight percentage of ME in the product as shown by the following equation

$$\text{Purity (\%wt)} = \frac{W_{ME}}{W_{ME} + W_{TG} + W_{DG} + W_{MG}} \times 100\%$$

where W_{ME}, W_{TG}, W_{DG}, W_{MG} are weight percentages of ME, TG, DG, and MG on a glycerol-free basis (%wt).

The weight percentages of TG, DG, MG, and ME on a glycerol-free basis were calculated from the known exit mole concentrations of ME, TG, DG, and MG coupled with their molecular weights. These molecular weights were calculated from the known fatty acids of palm oil [13]; they were 849.5, 597.0, 344.5, and 284.5 kg/kmol, respectively.

Transesterification of palm oil in n ideal CSTRs in series

To predict the amount of methyl esters produced in *n* ideal CSTRs in series at any specified residence time, the system of calculation equation for transesterification of palm oil in an ideal CSTR must be solved sequentially. For example, the exit mole concentrations of the 1st tank were determined by solving the system of calculation equations. Then, the exit mole concentrations of the 1st tank were used as the entering mole concentrations for the 2nd tank. Again, the exit mole concentrations of the 2nd tank were determined by solving the system of calculation equations. Similarly, the exit mole concentrations of the 2nd tank were used as the entering mole concentrations for the 3rd tank and so on. As a result, by sequential solving of the system of calculation equations, the exit mole concentrations of n^{th} tank can be determined.

Reactor efficiency

To know the efficiency of a continuous reactor that its mixing performance equivalent to *n* ideal CSTRs in series, the conversion yields at various residence times in a plug flow reactor (PFR) must be determined. Consequently, the system of calculation equations for transesterification of palm oil in PFR was constructed based on the same conditions as constructing the system of calculation equations for transesterification of palm oil in an ideal CSTR, but it was based on the design equation of PFR [9]. The obtained system of calculation equations is shown as following.

$$
\begin{aligned}
\frac{dC_{TG}}{d\tau} &= -k_1 C_{TG} C_A + k_2 C_{DG} C_{ME} \\
\frac{dC_{DG}}{d\tau} &= k_1 C_{TG} C_A - k_2 C_{DG} C_{ME} - k_3 C_{DG} C_A + k_4 C_{MG} C_{ME} \\
\frac{dC_{MG}}{d\tau} &= k_3 C_{DG} C_A - k_4 C_{MG} C_{ME} - k_5 C_{MG} C_A + k_6 C_{GL} C_{ME} \\
\frac{dC_{GL}}{d\tau} &= k_5 C_{MG} C_A - k_6 C_{GL} C_{ME} \\
\frac{dC_{ME}}{d\tau} &= k_1 C_{TG} C_A - k_2 C_{DG} C_{ME} + k_3 C_{DG} C_A \\
&\quad - k_4 C_{MG} C_{ME} + k_5 C_{MG} C_A - k_6 C_{GL} C_{ME} \\
\frac{dC_A}{d\tau} &= -k_1 C_{TG} C_A + k_2 C_{DG} C_{ME} - k_3 C_{DG} C_A \\
&\quad + k_4 C_{MG} C_{ME} - k_5 C_{MG} C_A + k_6 C_{GL} C_{ME}
\end{aligned}
\qquad (6)
$$

where *C* is mole concentration (mol/L); τ is residence time (s); *k1* to *k6* are kinetic rate constants (L/mol.s); and subscript *TG, DG*, *MG*, *GL*, *ME*, and *A* are denoted for TG, DG, MG, GL, A computer code for finite different method was employed on program MATLAB 6.1 to solve the system of calculation equations for transeterification of palm oil in a PFR. The system of design equations was solved for residence time up to 60 min. In fact, generally for numerical technique,

the size of step time (Δt) used in simulation affects the obtained results. The effect of size of step time was tested. However, step time less than 1 sec gave insignificant different results compared to step time 1 sec. As a result, step time 1 sec was used for simulation.

Results and Discussion

Transesterification of refined palm oil in continuous reactor modeled as *n* ideal CSTRs in series and PFR were simulated. The intrinsic rate was based on our previous result for transesterification of palm oil at temperature 60 ^{0}C and NaOH concentration 1 %wt of oil. The entering molar ratio of methanol to oil was specified at 6:1.

For transesterification of refined palm oil in *n* ideal CSTRs in series, the system of calculation equations was solved for various values of *n*; however, the results of *n* in the range of 1 to 7 were presented here. These equations were solved in the range of residence time (τ_i) from 5 sec to 60 min. For transesterification of refined palm oil in PFR, the system of calculation equations was solved for residence time up to 60 min, while step time (Δt) equal to 1 sec was used.

The purities at various total residence times up to 20 min of *n* ideal CSTRs in series and PFR are shown in Fig. 2. An ideal CSTR could produce the highest purity at 97.8 %wt. Increasing of residence time more than 60 min, the purity was increased slowly. Darnoko and Cheryan [5] also found that the highest purity in their experiment was 97.3 %wt. For *n* >1 and PFR, they could produce the same highest purity at 98.7 %wt. However, the required residence times were different. The required residence times to yield the selected purities at 96.0, 96.5, 97.0, 97.5, 98.0, and 98.5 %wt of *n* ideal CSTRs in series were extracted. These data are shown in Fig. 3.

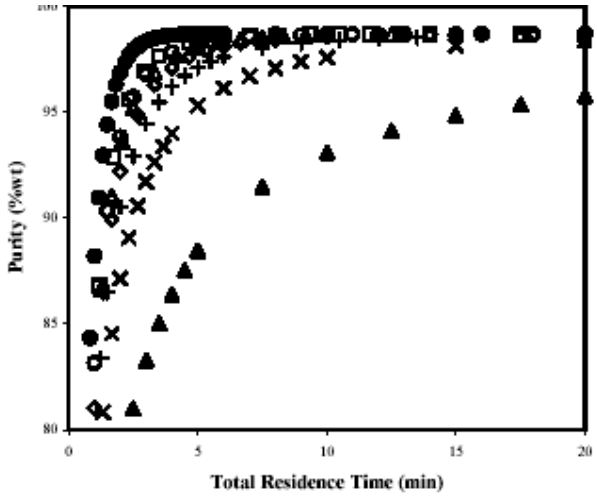

Fig. 2 Predicted purity for the transesterification of palm oil in series of CSTRs at molar ratio 6:1, temperature 60 ^{0}C, NaOH concentration 1 %wt of oil; (▲) 1-CSTR; (×) 2-CSTRs; (+) 3-CSTRs; (◊) 4-CSTRs; (Δ) 5-CSTRs; (o) 6-CSTRs; (□) 7-CSTRs; (●) PFR.

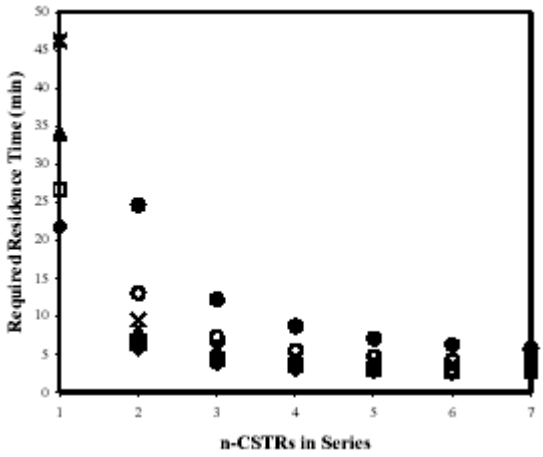

Fig. 3 Predicted required residence times to produce the selected purities (%wt) for the transesterification of palm oil in series of CSTRs at molar ratio 6:1, temperature 60 ^{0}C, NaOH concentration 1 %wt of oil; (◊) 96.0; (□) 96.5; (Δ) 97.0; (×) 97.5 (○) 98.0; (●) 98.5.

The decreased residence time (%) at any *n* of tanks of interest and selected purity level *x* was computed from

$$\text{Decreased Residence Time } (\%)_{n,x} = \left(\frac{\tau_{n-1} - \tau_n}{\tau_{n-1}} \right)_x \times 100 \qquad (7)$$

where τ is a required residence time, subscript *n* is denoted for the number of tanks of interest, subscript *n-1* is denoted for one tank less than the number of tanks of interest, and subscript *x* is denoted for a purity level *x*.

Fig. 4 shows the significant range for the decreased residence times: the range of *n* between 3 to 6 and the range of purity between 96.0 to 98.5 %wt. When $n = 2$, the decreased residence times were more than 70%; they are not shown here. On the other hand, when $n > 6$, the decreased residence times were too small; they are not also shown here. The shaded area in Fig. 4 represents the decreased residence time of less than 10 %.

The patterns of decreased residence time at all selected conversion yields were similar. The great decrease could be obtained when $n = 2$. A large decrease still could be obtained when $n \geq 5$. However, when $n = 6$; the decreased residence times at selected purities 96.0, 96.5, 97.0, 97.5, and 98.0 %wt were around 8%; the decreased residence times at selected purities 98.5 %wt was around 12%. These results revealed that for purity ≤ 98.0 %wt, increasing of *n* gave a significant decrease of required residence time up to $n = 6$. In addition, for purity ≥ 98.5 %wt, $n > 6$ still gave some significant decrease of required residence time. According to the European Union standards for alternative diesel fuels, the minimum acceptable purity of biodiesel is 96.5 %wt [14]. As a result, in order to produce saleable biodiesel, the optimum mixing performance of a continuous reactor was 6 ideal CSTRs in series. The predicted residence times of 6 ideal CSTRs in series to produce palm methyl esters at purities 96.5, 97.0, 97.5, 98.0, and 98.5 %wt were 2.85, 3.15, 3.61, 4.32, and 6.24 min, respectively.

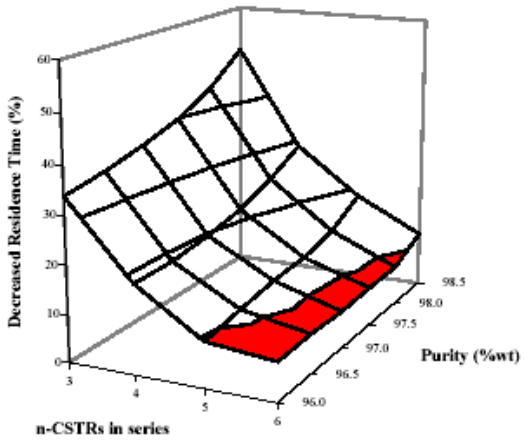

Fig. 4 Predicted decreased residence times at the selected purities for the transesterification of palm oil in series of CSTRs at molar ratio 6:1, temperature 60 ^{0}C, NaOH concentration 1 %wt of oil.

The efficiency of reactor (η) at a purity level *x* is the ratio of the required residence time to yield this level of conversion in a PFR to that of the reactor as shown in following equation [15].

$$\eta_x = \left(\frac{\tau_{pf}}{\tau}\right)_x \qquad (8)$$

Where η is efficiency of reactor, τ is required residence time, subscript *pf* is denoted for PFR, and subscript *x* is denoted for purity level *x*. The calculated efficiencies of a continuous reactor that its mixing performance equivalent to 2 until 6 ideal CSTRs in series are shown in Fig. 5. The shaded area represents the efficiencies of more than 60%. The efficiencies of 6 ideal CSTRs in series at purities 96.5, 97.0, 97.5, 98.0, and 98.5 %wt were 66%, 64%, 62%, 59% and 53%, respectively.

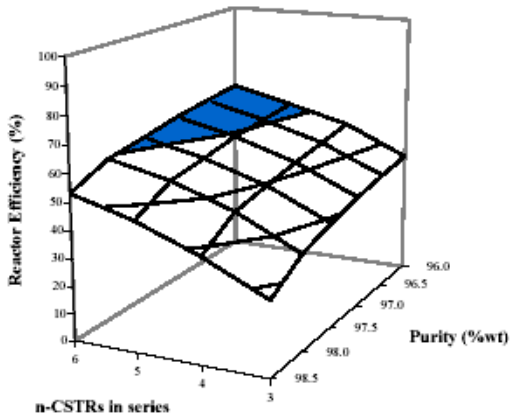

Fig. 5 Predicted reactor's efficiencies at the selected purities for the transesterification of palm oil in series of CSTRs at molar ratio 6:1, temperature 60 ^{0}C, NaOH concentration 1 %wt of oil.

Conclusions

For the transesterification of palm oil with methanol at molar ratio 6:1, temperature 60 ºC, NaOH concentration 1 %wt of oil, an ideal CSTR could produce the highest purity at 97.8 %wt. For n >1 and for PFR, they could produce the same highest purity at 98.7 %wt. However, the required residence times were different. For conversion yield ≤ 98.0 %wt, increasing of n gave a significant decrease of required residence time up to n = 6. In addition, for purity ≥ 98.5 %wt, n > 6 still gave some significant decrease of required residence time. As a result, to produce saleable biodiesel, the optimum mixing performance of a continuous reactor was 6 ideal CSTRs in series. The predicted residence times of 6 ideal CSTRs in series to produce palm methyl esters at purities 96.5, 97.0, 97.5, 98.0, and 98.5 %wt were 2.85, 3.15, 3.61, 4.32, and 6.24 min, respectively. In addition, the efficiencies of this reactor at purities 96.5, 97.0, 97.5, 98.0, and 98.5 %wt were 66%, 64%, 62%, 59% and 53%, respectively.

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