

Effect of Mass transfer and Reaction Kinetics in Transesterification of *Jatropha curcas* oil

Jahangir Chowdhury, Xianbing Cao, Fahad Al Basir, Priti Kumar Roy

Abstract— This paper presents the formulation and analysis of a mathematical model for biodiesel production through transesterification of vegetable oil such as *Jatropha* oil using alkaline catalysts in industrial scale. Previous works indicates that the transesterification reaction is mass transfer limited and it can be resolved with small changes in the kinetic properties experimentally. With this view, our main focus is to develop the mathematical model for discussing the effect of parameters such as catalyst loading, stirring, temperature on mass transfer and reaction kinetics in different phases of transesterification process. Optimal control theoretic approach is applied on catalyst loading to administer the said dynamics for the maximum production of biodiesel. Validity of the mathematical model is established by experimental results.

Keywords— Biodiesel, Reaction Kinetics, Mass transfer, Transesterification, *Jatropha curcas* oil, Mathematical modeling, Optimal control.

I. INTRODUCTION

Biodiesel has shown great promise to serve as an alternative fuel to petro-diesel fuel. It is produced from a variety of oils such as *Jatropha*, rapeseed, soybean, palm oil etc. [1]. Vegetable oil from *Jatropha curcas* plant is the most viable source for the production of biodiesel. It is significant to point out that the biodiesel from *Jatropha curcas* oil has the requisite potential for providing a promising and commercially good alternative to diesel oil since it has desirable physicochemical characteristics comparable to diesel [2,3]. In addition, *Jatropha curcas* plant can be cultivated in any wasteland which requires minimum irrigation [4].

Biodiesel can be produced through transesterification reaction in presence of catalysts from *Jatropha* oil. Industrial process which is widely used for biodiesel production is transesterification of triglycerides with alcohols (methanol and ethanol) in presence of catalyst [5, 6, 7]. The most notable catalyst used in producing biodiesel is the homogeneous alkaline catalyst (NaOH or KOH) [8,9]. Transesterification of

oil consists of mass transfer-controlled region at initial phase and a kinetically controlled region in the later phase [10, 11]. The immiscibility of methanol and vegetable oil causes initial mass transfer resistance in the transesterification of vegetable oil [12,13].

Initial mass transfer limitation is avoided by applying stirring on the system [14, 15]. Roy et al. [16], studied the effect of stirring on mass transfer in transesterification of *Jatropha* oil. They have shown that after a certain level of stirring (600 rpm) mass transfer resistance is negligible. The effect of the stirring intensity of the transesterification of cotton seed oil has also been analyzed using several catalysts and molar ratio [17, 18].

Many researchers have shown that transesterification reaction is highly dependent on temperature. It is observed that with an increase in reaction temperature, conversion of oil increased significantly. At constant stirring and catalyst amount, mass transfer rate is directly proportional to its temperature [19]. It is reported that in the production of biodiesel from *Jatropha curcas* oil by transesterification can be affected by catalyst loading. Noriega et al. [20] studied the transesterification of cottonseed oil for various alcohol and 1 wt.% KOH catalyst. They have proposed a three consecutive reversible reaction with a third order kinetic model for the ethanol and a first order kinetic model for the glyceride, biodiesel and glycerol. Da et al. [21] studied biodiesel production from castor oil and ethanol using 1 wt.% and 2.5 wt.% NaOH as the catalyst. Both the initial mass transfer and reaction rates increase with increasing catalyst amount [22].

The major drawback of using base catalytic method is the saponification of glycerides. Catalyst used in the reaction reacts with reactants and complicates the process by forming soap [23]. This causes lower production, washing difficulties and gives biodiesel with lower purity [24, 25, 26]. This can be avoided by selecting proper rate of catalyst loading for transesterification *Jatropha* oil. Therefore, optimization of catalyst loading is essential for maximum biodiesel production.

In this research article, we assume the same reaction mechanism as described in our previous works [16]. A mathematical model has been developed for transesterification of *Jatropha* oil with some additional assumptions. Here, the aim is to elucidate the effect of mass transfer and reaction kinetics in terms of reaction parameters. Also, an optimal profile of catalyst is determined with the help of Pontryagin

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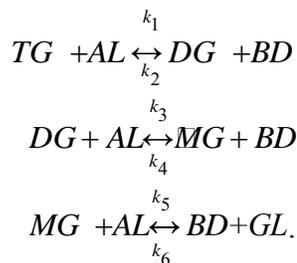
minimum principle for maximum production of biodiesel. Numerical analysis has also been done to find out the suitable reaction conditions for which biodiesel production process can be optimized.

II. FORMULATION OF THE MATHEMATICAL MODEL

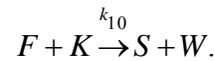
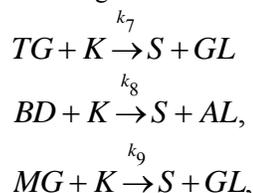
Here, we have chosen molar ratio, temperature, stirring speed and catalyst have been chosen as the variable parameters to study the mass transfer and kinetics of transesterification reaction. The following assumptions are made to developed the mathematical model.

An important consideration in the feedstock selection for biodiesel production is the content of free fatty acid (FFA) in the oil. In this article, *Jatropha curcas* oil is considered as the feedstock for biodiesel production. To use this oil as a feedstock, it should contain a low percentage of FFA so that the oil can directly be used in transesterification reaction [27].

Base catalyzed transesterification reaction for biodiesel production from *Jatropha Curcas* oil with methanol (AL) involves three reversible steps [28]. Here, in the first reversible step, triglycerides (TG) reacts with alcohol (AL) to produce diglycerides (DG) which further reacts with AL to produce monoglycerides (MG) in the second reversible step. Finally, MG reacts with AL to give glycerol (GL) as by product. At each reaction step, one molecule of biodiesel (BD) is produced for each molecule of AL consumed. The reaction system is as follows:



It is shown above that the alcoholysis kinetic reaction scheme consists of three reversible reactions. As catalyst(K), present in the transesterification process, it will react with triglyceride (oil), monoglycerides and biodiesel to produced soap. Catalyst also reacts with DG. But it has no significant effect on the system dynamics as the reaction rates are very slow [13]. So, we do not consider this mater while formulation the model. Although after esterification oil contain a few amount of FFA compare to it concentration still we consider soap formation through FFA due to higher reaction rate with catalyst. It is shown below that the saponification kinetic reaction scheme consists mainly of followings three reactions [8, 13].



Here, $k_1 - k_8$ are reaction rate constants. We denote the concentrations of triglycerides, diglycerides, monoglycerides, biodiesel (methyl ester), methanol (alcohol) and glycerol by C_T, C_D, C_M, C_B, C_A and C_G respectively. Whereas concentration of catalyst, soap, free fatty acid (FFA) and water are denoted by C_K, C_S, C_F, C_W respectively. The catalyst effect on rate constants are shown respectively using the following equations [28],

$$k_i = a'_i e^{\frac{-b_i}{T}}$$

and a'_i are given by

$$a'_i = a_i \left(\frac{C_K}{k_{icat} + C_K} \right). \quad (2)$$

Here, a_i is the standard reaction frequency factor, k_{icat} is a value that quantifies the catalyst effect on the reaction and C_K the catalyst concentration.

$$b_i = \frac{Ea_i}{R},$$

T is the reaction temperature, Ea_i is the activation energy for each component and R is the universal gas constant. The values of a_i 's and b_i 's are given in Table 2. To reduce initial mass transfer resistance, stirring is employed in the system. We assume k_s as the mass transfer rate due to stirring which is defined as below [19]:

$$k_s = \frac{a}{(1 + \exp(-b(N - c)))},$$

where N is the speed of stirrer and its unit is rpm. a , b and c are different parameters of this expression. Using the above assumption, we get the following differential equations to characterize the transesterification process:

$$\frac{dC_B}{dt} = k_1 C_T C_A - k_2 C_D C_B + k_3 C_D C_A$$

$$- k_4 C_M C_B + k_5 C_M C_A$$

$$- k_6 C_G C_B - k_8 C_B C_K +$$

$$k_s x_B \left(1 - \frac{x_B}{B_{max}} \right)$$

$$\frac{dC_T}{dt} = -k_1 C_T C_A + k_2 C_D C_B - k_7 C_T C_K,$$

$\frac{dC_D}{dt} = k_1 C_T C_A - k_2 C_D C_B - k_3 C_D C_A + k_4 C_M C_B$, Here, $u(t)$ satisfy $0 \leq u(t) \leq 1$ [19].

$$\frac{dC_M}{dt} = k_3 C_D C_A - k_4 C_M C_B - k_5 C_M C_A + k_6 C_G C_B - k_9 C_M C_K,$$

$$\frac{dC_A}{dt} = -k_1 C_T C_A + k_2 C_D C_B - k_3 C_D C_A + k_4 C_M C_B - k_5 C_M C_A + k_6 C_G C_B + k_8 C_B C_K,$$

$$\frac{dC_G}{dt} = k_5 C_M C_A - k_6 C_G C_B + k_7 C_T C_K + k_9 C_M C_K,$$

$$\frac{dC_K}{dt} = -(k_7 C_T C_K + k_8 C_B C_K + k_9 C_M C_K + k_{10} C_F C_K) = -\frac{dC_S}{dt},$$

$$\frac{dC_F}{dt} = -k_{10} C_F C_K = -\frac{dC_W}{dt} \quad (3)$$

with initial conditions: $C_B(0) = 0$, $C_T(0) = C_{T_0}$, $C_D(0) = 0$, $C_M(0) = 0$, $C_A(0) = C_{A_0}$, $C_G(0) = 0$, $C_S(0) = 0$, $C_K(0) = C_{K_0}$, $C_F(0) = C_{F_0}$, $C_W(0) = 0$.

This model includes the temperature effect on the rate constants through the Arrhenius form of reaction rates [35] and stirring effect by the term k_s defined above [19].

Equation (2) was proposed because the experimental results in the Jatropha oil methanolysis showed that, when C_K is low, a slight increase in concentration promotes a significant increase in the rate constants. However, when C_K is higher than 1.5 wt%, an increase in concentration does not generate a proportional increase in the rate constants [24, 34].

III. THE OPTIMAL CONTROL PROBLEM (OCP)

Dynamics of the system in terms of process parameters is analysed in order to have a better understanding of the biodiesel production in an industrial scale. In model system (3), reaction rates are defined as function of catalyst concentration and thus can be controlled by amount of catalyst. For this reason, the control parameter $u(t)$ is introduced into the system and can be treat as the effect of catalyst. The control input $u(t)$ is applied to control the reaction rates by amount of catalyst to get maximum biodiesel

yield through transesterification by minimizing side reactions.

Table 1: Values of parameters used in numerical simulations [23, 30].

Parameters	Value (%wt. cat/wt. oil)
k_{1cat}	1.80
k_{2cat}	0.32
k_{3cat}	1.49
k_{4cat}	0.22
k_{5cat}	24.36
k_{6cat}	1.7×10^{-3}
k_{7cat}	1.40
k_{8cat}	0.20
k_{9cat}	0.21
k_{10cat}	0.195

(6)

Also $u(t) = 1$ represents the maximal use of catalyst and $u(t) = 0$, which signifies no effect of catalyst. Taking into consideration of the parameter $u(t)$, we have the following control induced system corresponding to the system (3):

$$\begin{aligned} \frac{dC_B}{dt} = & uk_1 C_T C_A - uk_2 C_D C_B + uk_3 C_D C_A \\ & - uk_4 C_M C_B + uk_5 C_M C_A - uk_6 C_G C_B \\ & - k_8 u C_B C_K + k_s C_B \left(1 - \frac{C_B}{B_{max}}\right), \end{aligned}$$

$$\frac{dC_T}{dt} = -uk_1 C_T C_A + uk_2 C_D C_B - k_7 u C_T C_K,$$

$$\begin{aligned} \frac{dC_D}{dt} = & uk_1 C_T C_A - uk_2 C_D C_B - uk_3 C_D C_A \\ & + uk_4 C_M C_B, \end{aligned}$$

$$\begin{aligned} \frac{dC_M}{dt} = & uk_3 C_D C_A - uk_4 C_M C_B - uk_5 C_M C_A \\ & + uk_6 C_G C_B, \end{aligned}$$

$$\frac{dC_A}{dt} = -uk_1 C_T C_A + uk_2 C_D C_B - uk_3 C_D C_A$$

$$+uk_4C_M C_B - uk_5C_M C_A + uk_6C_G C_B \\ + uk_8C_B C_K,$$

$$\frac{dC_G}{dt} = uk_5C_M C_A - uk_6C_G C_B + uk_7C_T C_K \\ + uk_9C_M C_K,$$

$$\frac{dC_K}{dt} = -(uk_7C_T C_K + uk_8C_B C_K + uk_9C_M C_K \\ + uk_{10}C_F C_K)$$

$$= -\frac{dC_S}{dt},$$

$$\frac{dC_F}{dt} = -uk_{10}C_F C_K = -\frac{dC_W}{dt} \quad (4)$$

with initial conditions:

$$C_B(0) = 0, C_T(0) = C_{T_0}, C_D(0) = 0, C_M(0) = 0, \\ C_A(0) = C_{A_0}, \text{ and } C_G(0) = 0.$$

The system (4) can be written in compact form as:

$$\frac{dC}{dt} = f(u(t), C(t)),$$

where,

$$C = [C_B, C_T, C_D, C_M, C_A, C_G, C_K, C_S, C_F, C_W]^T \text{ and} \\ f = [f_1, f_2, f_3, f_4, f_5, f_6, f_7, f_8, f_9, f_{10}]^T, \text{ and}$$

$f_i (i=1, \dots, 10)$ are the right sides of system (4). Here, in order to maximize bio-diesel (C_B) production and minimize soap (C_S), the objective cost function for the minimization problem as,

$$J(u) = \int_{t_i}^{t_f} [Pu^2(t) + QC_S^2 - RC_B^2(t)] dt. \quad (5)$$

The cost function (i.e. the objective functional) is taken in quadratic to ensure the existence of optimal catalyst profile. The objective functional is taken in such a way that we can account the costs of production, expressed by the first term, the soap formation, whose presence needs to be minimized by second term, and for getting maximum biodiesel, expressed by the last term. Here, the objective is to minimize the cost by finding a suitable $u(t)$.

The parameters P, Q and R are the positive weight constants on the benefit of the cost of production. The benefit is based on the minimization of cost together with maximization of biodiesel concentration. The aim is to find out the optimal control $u^*(t)$ such that

$$J(u^*) = \min (J(u) : u \in U),$$

where,

$$U = \{u(t) : u \text{ is measurable and } 0 \leq u \leq 1, \\ t \in [t_i, t_f]\}.$$

We use "Pontryagin Minimum Principle" [29] to find the optimal control $u^*(t)$.

A. Optimality of the System

For optimal control of the system, the Hamiltonian is defined as follows:

$$H = [Pu^2(t) + QC_S^2(t) - RC_B^2(t)] + \sum \xi_i f_i, \quad (6)$$

where $i=1, 2, \dots, 10$ and $\xi_1, \xi_2, \dots, \xi_{10}$ are adjoint variables and the corresponding adjoint equations [36] are given by,

$$\frac{d\xi_i}{dt} = -\frac{\partial H}{\partial C_i}, i = 1, 2, \dots, 10. \quad (7)$$

Where $C_1 = C_B, C_2 = C_T, \dots, C_{10} = C_W, \text{ etc.}$

This gives,

$$\frac{d\xi_1}{dt} = 2RC_B + \xi_1(uk_2C_D + uk_6C_G \\ + uk_4C_M + k_8uC_K - k_s(1 - \frac{2C_B}{B_{max}})) \\ - \xi_2uk_2C_D + \xi_3(uk_2C_D + uk_4C_M) \\ - \xi_4(uk_4C_M + uk_6C_G) - \xi_5(uk_2C_D \\ + uk_6C_G + uk_4C_M) \\ + \xi_6(-uk_6C_G) + \xi_7k_8uC_K \\ - \xi_8k_8uC_K - \xi_5k_5uC_K,$$

$$\frac{d\xi_2}{dt} = uk_1C_A(-\xi_1 + \xi_2 - \xi_3 + \xi_5) \\ + uk_7C_K(-\xi_6 + \xi_7 - \xi_8 + \xi_2)$$

$$\frac{d\xi_3}{dt} = \xi_1u(k_2C_B - k_3C_A) - \xi_2uk_2C_B \\ + \xi_3u(k_2C_B + k_3C_A) - \xi_4uk_3C_A \\ + \xi_5(-uk_2C_B + uk_3C_A),$$

$$\frac{d\xi_4}{dt} = \xi_1uk_4C_B - \xi_1uk_5C_A - \xi_3uk_4C_B \\ - \xi_4(-uk_5C_A - uk_4C_B) \\ - \xi_5(-uk_5C_A + uk_4C_B) - \xi_6uk_5C_A,$$

$$\frac{d\xi_5}{dt} = -\xi_1(uk_1C_T + uk_3C_D)$$

$$\begin{aligned}
& +uk_5C_M) + \xi_2uk_1C_T + \xi_3uk_3C_A - \\
& \xi_4(uk_3C_D - uk_5C_M) - \xi_5(-uk_1C_T \\
& - uk_3C_A - uk_5C_M) - \xi_6uk_5C_M \\
& - \xi_3uk_1C_T, \\
\frac{d\xi_6}{dt} &= \xi_1uk_6C_B - \xi_4uk_6C_B - \xi_5uk_6C_B \\
& - \xi_6uk_6C_B \\
\frac{d\xi_7}{dt} &= \xi_1uk_8C_B + \xi_2uk_7C_T - \xi_6uk_7C_T \\
& + \xi_7(uk_7C_T \\
& + uk_8C_B + uk_{10}C_F) - \xi_8(uk_8C_B \\
& + uk_7C_T) - \xi_5uk_8C_B + \xi_8uk_{10}C_F, \\
\frac{d\xi_8}{dt} &= -2QC_S, \\
\frac{d\xi_9}{dt} &= uk_{10}C_K(\xi_7 - \xi_8 + \xi_9 - \xi_{10}), \\
\frac{d\xi_{10}}{dt} &= 0,
\end{aligned} \tag{8}$$

along with the transversality condition $\xi_i(t_f) = 0$, for $i = 1, 2, \dots, 10$.

According to Pontryagin Minimum Principle [36], the unconstrained optimal control variables u^* satisfies,

$$\frac{\partial H}{\partial u^*} = 0. \tag{9}$$

Thus from (6) and (9), we have

$$u^*(t) = \frac{-\sum_{i=1}^{10} \xi_i f_i(x, T)}{2P}. \tag{10}$$

Finally, we have the optimal system for maximum biodiesel production of biodiesel as a system given by equation (4), (8) and (10).

IV. NUMERICAL RESULTS AND DISCUSSION

In this section, the model system and the optimal system have been solved through Matlab to understand the behaviour of the transesterification reaction. Different reaction parameters such as temperature, catalyst loading, stirring are employed for better understanding of their effects on reaction kinetics.

Figure 1 reveals that at low mixing speed (300 rpm) the rate of production of biodiesel is very low. This is due to mass transfer resistance in this region. At 600 rpm stirring, concentration of biodiesel increases significantly. That means stirring is the most significant parameter at this stage the

reaction. But stirring higher than 600 rpm, there is no significant change in concentration of biodiesel.

Table 2: Values of parameters used in numerical simulations [23, 30].

Parameters	Value
a_1	6614.83
a_2	4997.98
a_3	9993.96
a_4	7366.64
a_5	3231.18
a_6	4824.87
a_7	6614.83
a_8	4997.98
a_9	5440.01
a_{10}	4335.09

Parameters	Value
b_1	6614.83
b_2	4997.98
b_3	9993.96
b_4	7366.64
b_5	3231.18
b_6	4824.87
b_7	3231.18
b_8	4824.87
b_9	5123.17
b_{10}	4326.80

Figure 1 also shows that transesterification is dependent on temperature also. Rate of production is increased with increasing Temperature. Taking 600 rpm stirrer speed and $50^\circ C$ temperature mass transfer resistance is almost eliminated.

It is observed in Figure 2 that with an increase in reaction temperature, conversion of oil increased significantly which is an indication of increasing mass transfer. Figure 3 shows that at increasing temperature, yield of biodiesel is increasing for fixed amount of catalyst (0.5%w/v) and stirring (600 rpm). Little amount of catalyst increases the yield of biodiesel at low

temperature by reducing the activation energy. From the figure 4 we can observe linear positive relation among FFA and soap content. It is evident as the FFA content increase the soap formation also increases which is in synchronization with the chemical reaction fate.

Also from Figure 4, it is cleared that with an increase in the reaction temperature at or above 50°C the initial reaction conversion rate increased but the ultimate biodiesel conversion appears to be lower compared to that of using a lower temperature. This is due to the fact that the saponification of oil and biodiesel by the alkali catalysts is enhanced at temperature above 50°C . In other words, higher temperature accelerates the side saponification reaction of the triglycerides. Operating the reactor at temperatures higher than 50°C is not economical under experimental conditions.

With an increase in catalyst amount, biodiesel concentration increases. The effect of catalyst loading on the reaction process is shown in Figure 5. The alkyl ester production rate increases as the catalyst concentration increases. A lower rate of transesterification is observed at a lower concentration of catalyst (0.5 wt. %). Increasing the catalyst concentration from 0.5 wt. %, 2.0 wt. % or even 2.5 wt. % it is seen that reaction rate is increased rapidly during the initial period of reaction although the maximum yield does not change. Thus it does not seem necessary to use the catalyst concentration higher than 1.5 wt. %, since adding surplus catalyst leads to the formation of excess soaps which leads to very low recovery of biodiesel during separation phase.

Reaction rate highly depends on catalyst loading (See Figure 5). With increase in catalyst amount, biodiesel yield increases but after a certain level (1.817%wt) yield is decreased significantly. This is because of the saponification of biodiesel and triglycerides. High amount of catalyst favours the saponification reaction. Thus, we can conclude that catalyst amount above 1.5%w/w is not recommended for biodiesel production.

Figure 7, illustrates the effect of control policy impact on the biodiesel production. Comparative analysis the control and without control reaction process shows an enhanced of biodiesel and less saponification problem. The saponification rate decreases with control input application.

In Figure 6 and Figure 7, comparison between the optimal concentration profiles of biodiesel and soap are carried out in two different avenues which are mentioned as without control and at the optimal control level. From the figures, it is observed that the control induced system give more production of biodiesel and reduced the soap with respect to time. Our result shows that at 60 minute of reaction time, the concentration of biodiesel at optimal level reaches its maximum value as 2.98 mol/L, while the maximum concentration is 2.795 mol/L if there is a fixed catalyst amount present in the reaction. In this way we can get 6.62% more biodiesel after 60 min of reaction. Hence, it is clearly established from numerical simulation, that the biodiesel production is optimized in transesterification reactions by the way of applying control theory.

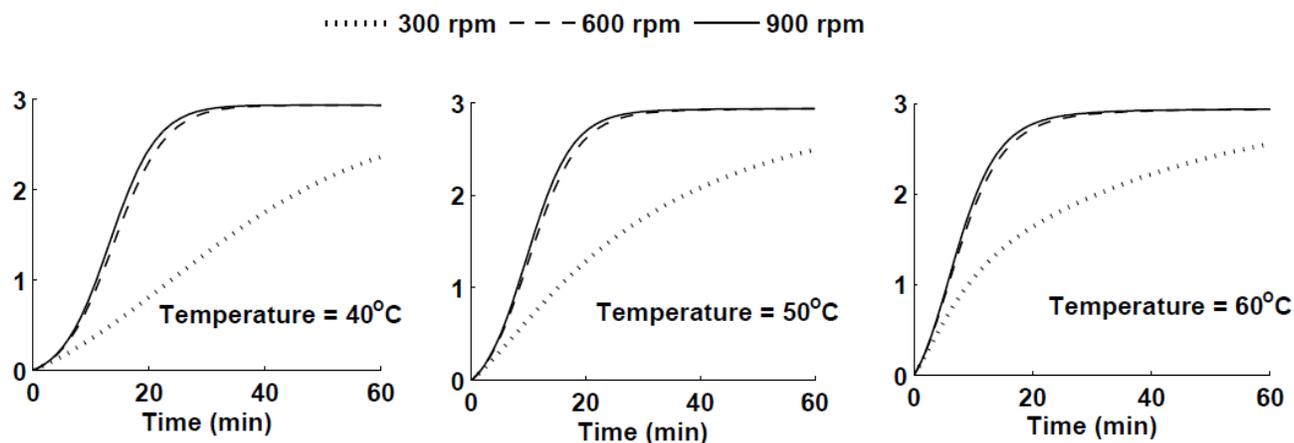


Figure 1: Effect of stirring for different temperature on BD production taking 60 min reaction time; y-label represent the concentration.

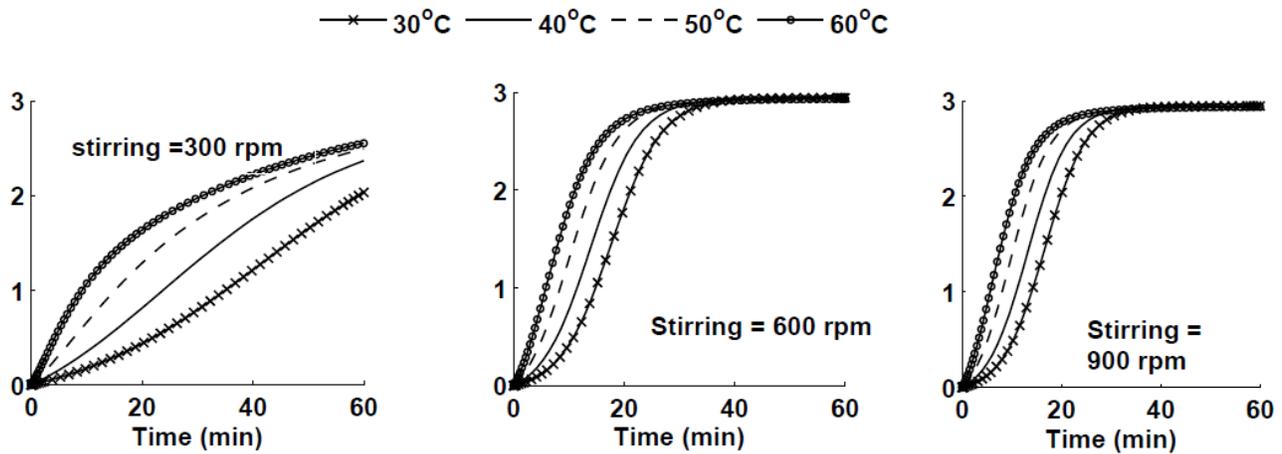


Figure 2: Effect of temperature for different stirring on BD production taking 60 min reaction time and; y-label represent the concentration.

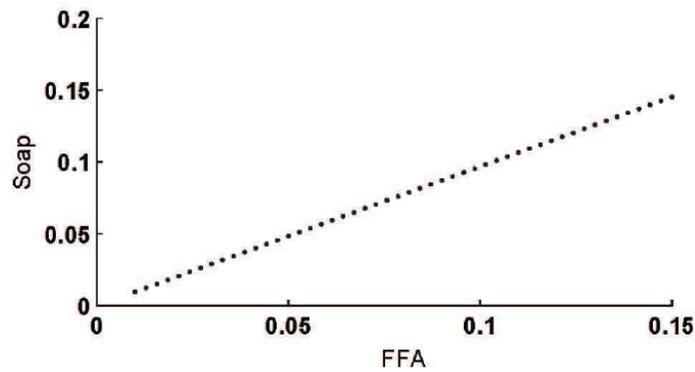


Figure 3: Effect of FFA on biodiesel production at 50°C, 6:1 molar ratio and 0.5%wt catalyst.

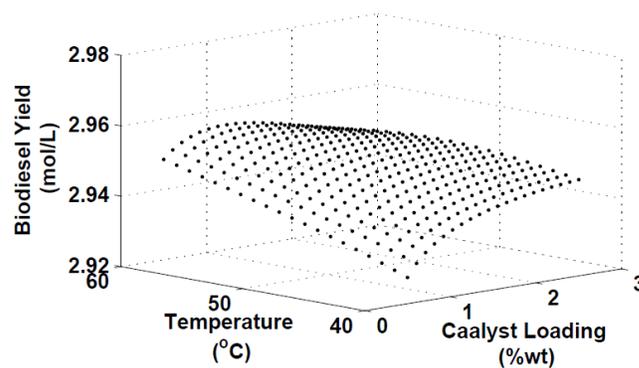


Figure 4: Combined Effect of catalyst and temperature on BD production taking 60 min reaction time.

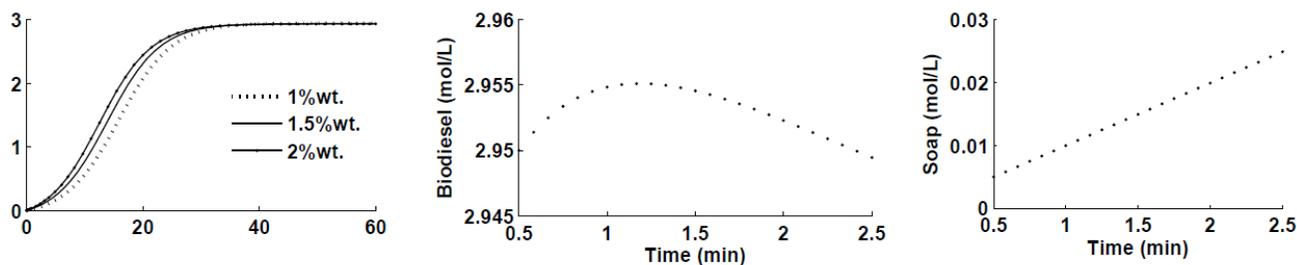


Figure 5: Effect of catalyst on biodiesel and soap (C_S) production at 50°C and 6:1 molar ratio. Concentrations are plotted after 60 min of reaction time.

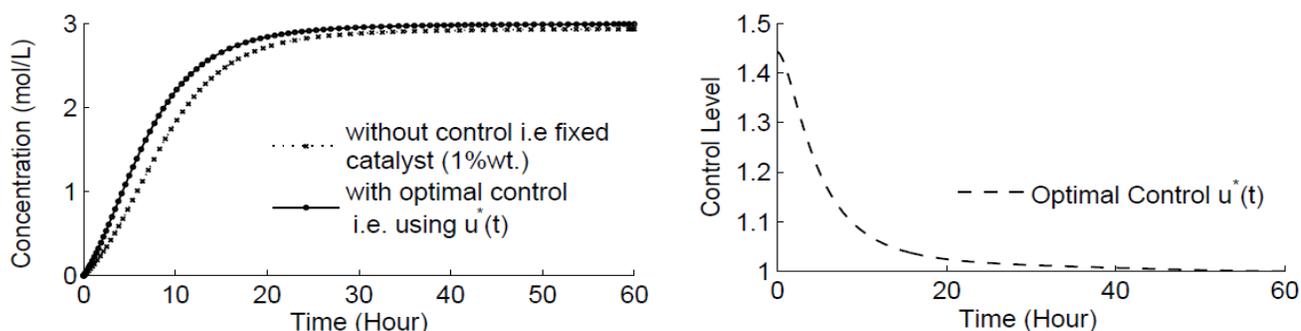


Figure 6: Comparison between two cases: (1) BD concentration with optimal control, and (2) BD concentration without control.

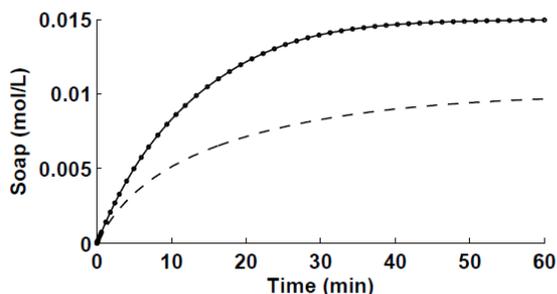


Figure 7: Comparison between two cases: (1) soap formation with optimal control, and (2) soap formation without control.

V. CONCLUSION

In this article, a mathematical model of transesterification of *Jatropha* oil is presented. Effect of reaction parameters on initial mass transfer and kinetics of reactive system is studied using the mathematical model. We have shown that mass transfer and reaction kinetics depend significantly on catalyst loading, stirring and temperature. The aim is to optimize biodiesel production by controlling catalyst loading. Control on catalyst loading is essential to increase reaction rate and reduce soap formation. It was observed through our mathematical analysis that for optimized and maximum biodiesel conversion the catalyst should be less than

1.5% to maintain a sustained reaction process. A higher amount of catalyst concentration is detrimental for the reaction as it will produce soap in the reaction process while a lower value than this threshold will also hamper production of biodiesel.

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