Assisted Sonication vs Conventional Transesterification Numerical Simulation and Sensitivity Study

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Abstract—Tranesterification is known as slow reaction that can take over several hours to complete as the two immiscible liquid reactants combine to form biodiesel and the less favorable glycerol. The quest of finding the perfect catalyst, optimal operational conditions, and reactor configuration to accelerate the reaction in mere few minutes that ensures high quality biodiesel, in economically viable way is coming along with sonication. This drastic reduction is a key enabler for the development of a continuous processing that otherwise is fairly costly and low throughput using conventional method. The reaction kinetics of sonication assisted as inferred by several authors is several time faster and this work implements these rates in a high fidelity numerical simulation model. This flow model is based on Navier-Stokes equations coupled with energy equation for non-isothermal flow and the transport equations of the multiple reactive species. The model is initially validated against experimental data from previous work of the authors using an annular reactor configuration. Following the validation, comparison of the reaction rate is shown to gain more insight to the distribution of the reaction and its attained rates. The two models (conventional and sonication) then compared on the basis of their sensitivity to the methane to oil molar ratio as the most pronounced process parameter. Both the exit reactor yield and the distribution of the species are evaluated with favorable yield under sonication process. These results pave the way to build a more robust process intensified reactor having an integrated selective heterogeneous catalyst to steer the reaction. This can avoid the downstream cleaning processes, cutting reaction time, and render economic benefit to the process.

1. Introduction:

Whether conventional or sonication assisted the transesterification process undergoes the following main and reversible reactions:

\[
\begin{align*}
\text{Triglyceride} + \text{Alcohol} & \quad \frac{K_1}{K_2} \quad \leftrightarrow \quad \text{FAME} + \text{Diglyceride} \\
\text{Diglyceride} + \text{Alcohol} & \quad \frac{K_3}{K_4} \quad \leftrightarrow \quad \text{FAME} + \text{Monoglyceride} \\
\text{Monoglyceride} + \text{Alcohol} & \quad \frac{K_5}{K_6} \quad \leftrightarrow \quad \text{FAME} + \text{Glycerol} \\
\text{Triglyceride} + 3\text{Alcohol} & \quad \frac{K_7}{K_8} \quad \leftrightarrow \quad 3\text{FAME} + \text{Glycerol}
\end{align*}
\]

Summing the first three reactions provide the overall fourth reaction in which one mole of oil reacts with three moles of alcohol leading to the production of three moles of Fatty Acid Methyl Ester (FAME) and the less desired one mole of glycerol. Remains of Triglycerides (TG), Diglyceride (DG), or Monoglyceride (MG) in the reaction are considered impurities and thus one needs find the conditions to speed up the overall forward reaction to reach to the favorable reaction equilibrium state. Contrary to hydrocarbon fuel in which their thermodynamic properties is well documented and are known as priori, the Triglyceride, Diglyceride, and Monoglyceride is not a fixed formula and their properties including their standard enthalpy, specific heat, and even their physical...
properties including density, viscosity, etc. are not surprisingly inconsistent in literature. This adds some uncertainty in modeling of their reactivity as far as establishing robust energy minimization approach such as Gibbs free energy based modeling. Alternatively, intrinsic modeling that based on experimental evaluated chemical kinetic data comes very handy. In this modeling quest the most recurring and cited properties is been used in this work. Conventional transesterification method is slow to be considered as a viable continuous method at high throughput. Ultrasound assisted method, however, reduces the immiscibility of the two reacting fluids as the sonication energy creates cavitation bubbles which continue to grow rapidly, collapsing violently, generating energy and mechanical effect leading to drastic mass transfer rate enhancement at the boundary. The pressure within the bubbles can be as high as 5000 atm which can cause a very high local temperature rise (about 7200°C) [2]. As this takes place at the phase boundary of the two immiscible species the reactions between the two species intensify [3].

Apart from the authors work on numerical simulation of the transesterification there no other reported literature treating this subject. There are, however, several works that focused on kinetics of these reactions. Noureddini et al. [4] calculated the activation energies and rate constants for forward and backward overall reaction and subsidiary reactions for soybean oil transesterification at different conditions of a well-stirred batch reactor. Jose et al. [3] compared kinetics of sonicated and conventional transesterification and of soybean oil and reported that the rate constant for the sonication reactions were three times to half order of magnitude higher than the conventional process. This suggests that for similar activation energies the reaction constant must be compensated so that the reaction rates need to be much higher in the case of sonication. Vishwanath et al. [5] demonstrated the dependency of rate constants on molar ratio, catalyst percentage and temperature for forward and backward reaction of palm oil fatty distillate ultrasound assisted transesterification with isopropanol. The highest rate constants for forward reaction were reported at five to one of alcohol to oil molar ratio. At catalyst concentration 7%, the highest forward rate constants was achieved. The forward rate constant also depicted a direct proportionality with temperature. The highest attempted experimental temperature was 600°C which resulted in the highest forward reaction constant. Other parameters showed direct proportionality with the reverse reaction constant, however molar ratio increase resulted in a lower backward reaction constant. Investigation of the kinetics of transesterification reaction of waste cooking oil with a heterogeneous catalyst (K3PO4) and methanol carried out by Dipak et al. [6]. A 375 W at 22 kHz ultrasonic sonication device is used. The reaction was carried out at six to one molar ratio. Similar to the Vishwanath and coworkers they reported the increase in the forward rate constant with the increase in temperature. The evaluated activation energy was 64,241 KJ/Mol and the rate constant was in the range 0.02 to 0.18 when the temperature swept from 300°C to 600°C. Earlier conducted works of the authors demonstrate the feasibility of CFD in evaluating species concentrations as far as their distributions and output product. Less emphasis was given to the influence of key reaction parameters including molar ratio and flow speed. Another work by the same author shows the evaluation of the reaction kinetics of the waste cooking oil transesterification followed by implementing these values in a reactive flow model [7]. In this work sensitivity study is carried out considering molar ratio, flow condition, and different geometrical configuration. The details of transesterification process inside the three dimensional cylindrical single tubular reactor is considered. The influence of the molar ratio for both conventional and sonicated transesterification reaction will be carried out. The results aim to gain deeper insight of the difference in the two reaction methods as far as the yield and their distributions.

2. Model Development

The none-isothermal, viscous, turbulent flow is governed by the Navier-Stokes equations associated with temporal, advective, viscous, and source terms and in general form is written as:

$$\frac{\partial}{\partial t} (\rho u_i) + \frac{\partial}{\partial x_j} (\rho u_i u_j) = -\frac{\partial}{\partial x_j} \left( \Gamma \frac{\partial \phi}{\partial x_j} \right) + S_{\phi} \tag{5}$$

Where $u$ is the velocity and $S_{\phi}$ is the source term due to the interaction, destruction or creation of other species. $\Phi$ is the dependent variable which is density ($\rho$), the velocity density multiple ($\mu u$), or the temperature ($T$) to constitute the continuity, momentum, and the energy, respectively. Equation (5) also can represent the turbulence scalars, the kinetic energy and the turbulence dissipation rate, and these are written as:

$$\rho \frac{\partial \epsilon}{\partial t} = \nabla \cdot \left( \mu \nabla \epsilon \right) + \frac{\epsilon}{k} \left( \frac{\partial u_i}{\partial x_i} + \frac{\partial u_j}{\partial x_j} \right) \frac{\partial u_i}{\partial x_j} - C_{\epsilon} \rho \epsilon \frac{\partial \rho \epsilon}{\partial t} \tag{6}$$

$$\rho \frac{\partial k}{\partial t} = \nabla \cdot \left( \frac{\mu \nabla k}{\rho} \right) + \frac{k}{C_U} \left( \frac{\partial u_i}{\partial x_i} + \frac{\partial u_j}{\partial x_j} \right) \frac{\partial u_i}{\partial x_j} - C_U \frac{\rho \epsilon}{k} \tag{7}$$

[2] 2015 012016 doi:10.1088/1757-899X/92/1/012016
The three terms on the right hand are the diffusion, the generation, and destruction/dissipation of the turbulent quantities, respectively. They are coupled through the eddy viscosity ($\mu_t$) coefficient which is expressed as:

$$\mu_t = f_{\mu}C_pr^2/\varepsilon \quad (8)$$

Where $f$ and $C_p$ are pre-defined constants additional to the empirical constants $C1\varepsilon$, $C2\varepsilon$, and that are tuned for a specific flow categories, e.g. channel, swirl, separating, and jet flows.

For system closer, additional transportation specie equations of equal numbers to the numbers of reacting and product species are used. Each equation incorporates a reaction source term and is written in the following general form [10]:

$$\frac{\partial}{\partial t}(\rho m_i) + \frac{\partial}{\partial x_j}(\rho u_j m_i) - \frac{\partial}{\partial x_j}(\rho D_{ij,m} + \mu /Sc_i)\frac{\partial m_i}{\partial x_j} + R_i + S_i \quad (9)$$

Where $D_{i,m}$ is the diffusion coefficient of $m_i$ species, $Sc$ is the turbulent Schmidt number and is defined as the ratio of the eddy viscosity $\mu_t$ to the eddy diffusivity $D_{i,m}$. The reaction source term $R_i$ accounts for formation or destruction of species according to the stoichiometric chemical reaction which is written as:

$$\sum_{j=1}^{N} \nu_{r,j}S_i \Leftrightarrow \sum_{j=1}^{N} \nu_{p,j}S_i \quad (10)$$

Where $\nu$ are the stoichiometric coefficients of reactant (prime) and product (double prime) $k_f$ and $k_b$ are the forward and backward reaction constants respectively and follows the Arrhenius for as:

$$k_f or b = A T^{\alpha} e^{-E/RT} \quad (11)$$

Where $A$ is the reaction pre-constant, $T$ and $\alpha$ are the temperature and its exponential dependency constant. $E$ is the activation energy, and $R$ is the universal gas constant.

The reaction constants ($k_f$ and $k_b$) are coupled with the $R_i$ source term in the transport equation according to the species concentrations and following the stoichiometry of equation (10) and is written as:

$$R_{i,r} = M_{i,r}(\nu_{r}^{*} - \nu_{p}^{*}) \left( k_f \prod_{j=1}^{N} C_{i,r}^{\nu_{r,j}} - k_b \prod_{j=1}^{N} C_{i,r}^{\nu_{p,j}} \right) \quad (12)$$

Where $C_{j}$ is the molar concentration of $j$th specie raised to stoichiometric coefficients $\nu$ and reaction order $\eta$, and $M_i$ is the molecular weight of species $i$. The analyses and reactions are achieved within the framework of Ansys Fluent [8]. Janajreh and Alshrah [9] have carried out both non-reactive and reactive flow for tubular reactor to model the transesterification of the two reactants and their products. The properties of the species are listed in Table 1 and the reaction kinetics of each reaction are given elsewhere and are summarized in Table 2 [4].

| Species                  | Chem. formula | Mol. weight | Viscosity (kg/m.s) | Cp (J/kg.0C) | Density Kg/m3 |
|-------------------------|---------------|-------------|--------------------|--------------|---------------|
| Methanol                | CH4O          | 32          | 3.96e-4            | 1.470e3      | 791.8         |
| Waste oil or Triglyceride| C54H104O6     | 848         | 1.61e-2            | 2.2e3        | 883.3         |
| Diglyceride             | C37H72O5      | 596         | -                  | -            | 880           |
| Monoglyceride           | C20H40O4      | 344         | -                  | -            | 875           |
| Biodiesel               | C18H36O6      | 284         | 1.12e-3            | 1.187e3      | 870           |
| Glycerol                | C3H9O3        | 93          | 1.412e0            | 0238.6       | 1261          |
Table 2. Chemical Kinetic Constants of Reactions [4]

| Reaction | \( K \) at 50°C and Reynold’s Number=6200 | Activation Energy (J/mol) | Pre-exponential factor (A) -calculated |
|----------|------------------------------------------|---------------------------|---------------------------------------|
| 1. Triglyceride → Diglyceride             | 0.05                                     | 56,902.4                  | 77,983,755.8                          |
| 2. Diglyceride → Triglyceride             | 0.11                                     | 40,919.52                 | 449,020.085                           |
| 3. Diglyceride → Monoglyceride            | 0.215                                    | 78,659.2                  | 1.0976E+12                            |
| 4. Monoglyceride → Diglyceride            | 1.228                                    | 46,860.8                  | 4,570,170.3                           |
| 5. Monoglyceride → Glycerol               | 0.242                                    | 21,673.12                 | 767.86                                |
| 6. Glycerol → Monoglyceride               | 0.007                                    | 41,296.08                 | 32,870.58                             |

The overall reactions for the conventional and sonication transesterification process are reported in the literature and are summarized in Table 3.

Table 3. Overall chemical kinetic constants as reported [3] [4]

| Reaction                  | K      | Activation Energy (J/mol) | Pre-exponential factor (A) -calculated |
|---------------------------|--------|---------------------------|---------------------------------------|
| Forwards: Conventional.   | 7.8E-05| 164958.38                 | 3.4868E+22                            |
| Forwards: with sonication.| 2.34E-04| 164958.38               | 1.046E+23                             |

The main assumption in these analysis is the state of well mixing between the two immiscible reactants represented by the methanol and triglyceride. Therefore, the two fluid enters the reactor as premixed fluid implying significant agitation in the fluid prior its introduction to the reactor. This assumption simplified the physical aspect of the problem by pursuing the Euler multiple species numerical approach. This modeling departs from the multiple phase approach where bubbly, sludge, and intermittent flow can take place in the case of none premixed and slowly moving flow. Nevertheless, it is well known that transesterification requires intense agitations to avoid any mass transfer limitation of the two reactants and thus additional to premixing flow needs to be configured to preserve the well mixing state of the reactants. Experimentally, the increase in molar ratio of methanol to triglyceride would push the reaction forward [11], numerically this considered indirectly as the increase in the molar ratio will deplete the triglyceride faster forming the FAME and the Glycerol. Because the FAME and Glycerol now at lower concentration stage there revers reaction proceeds at slower rate.

Simulation Setup and model validation
The model setup is in the line of the author’s previous work [9]. It consists of a single cylindrical reactor (30cm length) flowing axially as depicted in Fig. 1.

Fig. 1: The topography of the reactor, the considered wedge and its discretized hybrid hexagonal and prismatic mesh
A 3D cylindrical wedge of 5deg is considered where two periodic faces coincided at the centerline. It is subjected to prescribed dirichlet inlet (velocity, species concentration, and temperatures), and Neumann of zero axial gradient at the outlet, and no-slip and no-penetration at the outer cylindrical reactor wall. A hybrid
hexagonal and prismatic mesh is considered at three refined levels to assess mesh independency comprises of a course, baseline, and refined. Table 4 shows the independency of the mesh as far as pressure drop near the turbulence Reynolds values. It demonstrates the suitability of the baseline mesh as a compromise between the accuracy and computational time.

Table 4. Summary of the mesh sensitivity results

| Mesh level  | Number of cells | Pressure Drop (Pas) and Rel. Error |
|-------------|----------------|-----------------------------------|
| Coarse      | 6,300          | 2.503; 28.5%                      |
| Baseline    | 50,400         | 2.007; 3.2%                       |
| Fine Mesh   | 403,200        | 1.948;---                          |

The validation of the model is carried out against the experimental measurement obtained elsewhere [4] under conventional transesterification. These measurements of the triglyceride (TG), FAME, Glycerol, and Alcohol (AL) are conducted using Thermoscientific GC/MS that equipped with FID detector. FAME column is calibrated against several standard biodiesel samples for qualitative and quantitative species measurements. Results of the model and experimental data are plotted in Fig. 2 in function of the residence time. The model residence time corresponds to the length of the reactor for the specific downstream location divided by the inlet flow velocity. These measurements are obtained at 6:1 AL to TG ratio, in 3,600rpm stirred batch reactor operated at atmospheric pressure and 50oC temperature. The trend of species depletion and formation is well-produced despite the small deviation shown in the initial reaction stage (20 minutes) in the biodiesel, glycerol and alcohol. A fairly decent match between the model and experimental measurements prevails to over the rest of the time for all species.

Fig. 2: Validation of model and experimental measurements

3. Results of Simulation and Discussion

Reaction rate
As kinetic reaction rate of the sonication transesterification process is nearly three times higher than those of conventional process as noted in Table 3, simulation results also reflect this high reaction rate trend as depicted in Fig. 3. The results clearly demonstrate the earlier higher value as well as the occurrence of the peak in sonication transesterification which is nearly three times higher than the conventional process. The reaction rates distribution is more clearly shown in the contour plot in Fig. 4 reaching nearly 54 kmol/m3.s for the sonication versus 18 kmol/m3.s for the conventional transesterification.
Species distribution
As for the resulted species, the spatial variation of the Ester and Glycerol is depicted in Fig. 5 which also depict a faster and higher concentrations are formed in the favor of the sonication transesterification.

Fig. 5: Distribution of the product species along the reactor axis for conventional (left) and sonication (right)
The results at downstream shows a substantial increase in the sonicator reaching molar ratio that goes beyond theoretical stoichiometry values (3:3:1 of Biodiesel:AL:GL). The final sonication yield in molar fraction are 0.63, 0.21, and 0.16 for Biodiesel, GL and AL, respectively. It however attained near the exact theoretical stoichiometry values (0.43 FAME, 0.14 GL, and 0.43 AL) in the case of conventional process. The results shows the anticipated trend however and demonstrates the faster and higher molar fraction formation during the sonication method. Table 5 summarizes the molar fraction at the exit of the reactor.

Table 5: Molar fraction at the exit of the reactor
TABLE 5. Molar Fraction at the exit (yield)

|        | Conventional Input & Output | Sonication Input & Output |
|--------|-----------------------------|---------------------------|
| AL     | 0.856                       | 0.159*                    |
| TG     | 0.144                       | 0.144                     |
| Biodiesel | 0                          | 0                          |
| GL     | 0                            | 0.21                       |

*lower than the anticipated value; **higher than the anticipated value

As the results reveal in Table 5, a higher molar concentration than what the stoichiometry suggests is generated in the sonication method. This may suggest the inaccuracy of the implemented sonication activation energy and rate constant values with no restriction to up-hold the reaction to the stoichiometric values. Never the less the current simulations bring more insight to the trend rather than the absolute measure for these fractions. The subsequent analysis is based on implementing these values in an attempt to verify the influence of the most pronounced transesterification parameter, i.e. molar ratio.

Molar ratio sensitivity

Fig. 7 depicts the influence of the change of the molar ratio of the alcohol to the TG for both conventional and sonication transesterification. At 3:1 AL to TG molar ratio some of the TG stayed unconverted in the conventional process while is to tally consumed during the sonication process. At higher AL to TG molar ratio of 6:1 the total TG conversion is achieved at the middle stream and albeit faster for the sonication transesterification with an asymptotic conversion starting at nearly 1/5th upstream and nearly completed at 2/5th upstream.

Further increase in the AL to TG molar ratio to 9:1 resulted in a noticeable earlier start of the asymptotic conversion of the sonication process, but would also result in near completion at the same upstream 2/5th station. As for the AL conversion, the alcohol residual for the conventional case at the 3:1 molar ratio signifies incomplete conversion contrary to the sonication process in which all the alcohol and TG are utilized. These results show clearly the considerable advantage for the conventional process in using larger molar rate to achieve completed conversion, this additional AL value, however this is not required in the case of sonication assisted transesterification.

Fig. 8 shows the product yield of the process captured by the molar fraction of Ester and GL. At the stoichiometric molar ratio (3:1) the values for the sonication process are at 0.75 and 0.25 for the Easter and GL, respectively. It signifies a complete conversion which is contrary to the yield of the conventional molar fraction values for Easter and TG which are at 0.71 and 0.24, respectively. The unconverted AL and TG was at 0.04 and 0.01 respectively.
At higher molar ratio, and with complete conversion of the AL and TG, the Ester and GL reach to the theoretical stoichiometric values for the conventional transesterification, i.e. at 6 AL to one TG molar ratio, the Ester reach the 0.43 and the GL is 0.14 signifying the attainment of complete conversion. The sonication assisted, however, emerges at faster conversion and higher production of Ester and TG with values beyond what the stoichiometry would provide. It appears that AL continue to deplete also to value that is lower than the theoretical stoichiometry. The glycerol values accordingly is also increased beyond the stoichiometry suggesting that the used kinetics of the sonication assisted transesterification are loose and not that accurately measured. Therefore, this aspect needs to be taken further in the lab to capture these kinetics in a process similar to the work of Noureddini and Zhu [4]. At nine AL to one TG molar ratio similar and favorable trend is produced in the conventional transesterification (i.e. The molar fractions are 0.6 AL, 0.3 Ester, and 0.1 GL), but not so for the sonication as the AL continues to deplete to lesser value than the limited stoichiometry.

**Conclusion**

In this work, the numerical simulations of the transesterification in a three dimensional reactor is achieved. A non-isothermal Navier stokes model coupled with species transport of reactive flow of the overall reversible reactions was carried out. The results clearly demonstrate the effectiveness of the sonication assisted transesterification over the conventional process. At low molar ratio whereas conventional transesterification fall short in completing the reaction, the sonication assisted transesterification lead to complete stoichiometry conversion. These results shows clearly the considerable advantage for the conventional process in using larger molar rate to achieve completed conversion. This additional amount, however, can be avoided in the case of sonication assisted transesterification which eliminates the downstream AL separation and offset any associated cost. The results show the feasibility of reactive flow dynamics in capturing and numerically simulating the transesterification process. However, as at higher AL and TG molar ratio (6:1 and 9:1) the sonication reaction resulted in yield beyond the theoretical stoichiometric values. This is a “miss-behaved” system which is contrary to the “well behaved” conventional reaction. This suggest to seek more accurate sonication kinetic data. Hoping to continue construct a precise and insightful picture of the distribution of the reaction rates and their associated species for the development and design of more efficient reactors.

**Nomenclature**

| Symbol | Meaning |
|--------|---------|
| K1     | Forward reaction constant for first reaction |
| K2     | Backward reaction constant for first reaction |
| K3     | Forward reaction constant for second reaction |
| K4     | Backward reaction constant for second reaction |
| K5     | Forward reaction constant for third reaction |
| K6     | Backward reaction constant for third reaction |
| K7     | Forward reaction constant for overall reaction |
| K8     | Backward reaction constant for overall reaction |
| E1     | Activation energy for first reaction in forward direction |
| E2     | Activation energy for first reaction in backward direction |
| E3     | Activation energy for second reaction in forward direction |
E4 Activation energy for second reaction in backward direction
E5 Activation energy for third reaction in forward direction
E6 Activation energy for third reaction in backward direction
E7 Activation energy for overall reaction in forward direction
E8 Activation energy for overall reaction in backward direction

u Velocity component
SΦ Dependent variable - turbulent scalars.
T Temperature
ρ Density
μ blinking Eddy viscosity
Di,m Diffusion coefficient
f,C User-defined constants
σk, σε Empirical constants

mi Species
Sct Turbulent Schmidt number
kf and kb Forward and backward reaction constants
Cj Molar concentration of jth species
η Reaction order
ν Stoichiometric coefficient
Mi Molar weight
Ri Reaction source term
mf Mass fraction

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