

Physical and Chemical Analysis of Ultrasonic Transesterification through Numerical Simulation

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Abstract

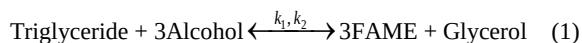
Ultrasound assisted transesterification is increasingly standing out as a highly efficient, reliable and faster way to produce biodiesel from vegetable oil. Applying the same to other feedstock such as used cooking oil, beef tallow or fish oil etc. provides great advantage in terms of yield quality and production time. However, large-scale biodiesel production through ultrasound assisted transesterification is limited by the lack of a continuous sono-chemical reactor, which effectively processes a flow of reactants by simultaneously sonicating them. Design of such a reactor is a complex process since the mechanism is governed by multiple physics such as the ultrasound wave propagation, acoustic cavitation, reactive flow, chemical kinetics etc. In this work a previously designed sono-chemical reactor by the same author is worked upon using numerical simulation to analyze the effectiveness of sonication on the transesterification reaction. The ultrasound mechanism is simulated using the linear wave equation. The acoustic cavitation phenomena which also causes an attenuation of the wave has been accounted for using the complex wave number and impedance. A logical reaction rate coupling model is used to estimate the collective effect of sonication and flow agitation in the reactor. This model system is then applied to study the effect of sonication on the kinetics of reaction and a sensitivity study is carried out. Results show positive effect of alcohol molar ratio in flow agitation case whereas increased molar ratio decreased the sonication rate constant. Biodiesel formation had direct proportionality with applied power and fluid temperature, whereas for frequency sensitivity the results depended on wave number and impedance.

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Keywords: Biodiesel; Ultrasonic; Sono-chemical; Transesterification; Reactor Design.

1. Introduction

Transesterification is the reaction between triglycerides found in vegetable oil, or used cooking oil and an alcohol such as methanol, which is catalyzed by homogeneous, heterogeneous or enzyme catalysts to produce Fatty Acid Alkyl Esters (FAAE) i.e. biodiesel and a byproduct glycerol. The overall reversible reaction is shown in Eq. 1. It is a slow reaction which needs mechanical agitation to proliferate [1]. Conventionally this can be accomplished by means of a stirrer but such a method is inefficient and needs a relatively long time [2].



Sonication has proved to be a much more efficient option to assist transesterification. The ultrasound wave causes intense cycles of compression and rarefaction at micro levels in the fluid volume which creates cavitation voids or bubbles that contain highly activated vapors of the reactants. The temperature and pressure in these micro bubbles can reach as high as 5,000 K and 1,000 atm [3]. Millions of such bubbles are formed as soon as the sonication is applied. When these bubbles implode they cause tremendous mass transfer in localized zones which intensify the reaction with a localized rate several orders higher than the conventional or stirring flow cases.

There are several works in literature that demonstrate the advantage of sonication over conventional methods of transesterification. Stavarache et al. [4] reported higher yields in shorter time using ultrasonic transesterification under homogeneous catalysts of NaOH and KOH and for the same molar ratio and catalyst amount compared to conventional stirring method. Manickman et al. [5] reported that mechanical agitation takes triple the time to give 78% yield as weighed to ultrasonic transesterification which gives about 93% yield with 1% KOH and 3:1 methanol to oil molar ratio. Apart from the physical effects it is also important to gauge the chemical effects, like free radical formation so as to be reasonable in carrying out kinetic study on sonicated transesterification. Radical chemical specie formation may make such a study highly complex and inaccurate. Extensive work has been carried out in understanding the physical mechanism of ultrasound by some researchers. The results from these works show favorable advantage of physical effects such as formation of fine emulsion, micro mixing etc. over chemical effects. For instance Abhishek et al. [6] studied the prominence of the physical and chemical effects of sonication for the transesterification reaction. Using soybean oil and methanol, they experimented with four molar ratios of 6, 12, 16 and 24 of methanol to 1 mole of oil. They used a 20 KHz frequency ultrasound equipment at moderate input power. Their approach was to couple experimental results with simulation of cavitation bubbles using the Keller-

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miksis equation. They found out that the most beneficial aspect of sonication for transesterification reaction is the physical effect like cavitation. Priyanka et al. [7] have also worked on mechanistic investigation of transesterification for soybean oil with methanol and H₂SO₄ using coupled cavitation bubble simulation and determined that physical effects outweigh the chemical effects of ultrasound. Hanif et al. [8] studied the effect of sonication on Jatropha curcas oil at various temperature and molar ratio combinations. They found that the most important physical effect that causes increase in reaction rates pertaining to sonication is the micro level mixing. Design of sonochemical reactors has been carried out by researchers like Sutkar et al. [9]. They simulated the ultrasound wave with modification in the wave number and impedance to predict the cavitation activity in a chemical reactor. They found that the cavitation is high in the zone close to the transducer and then attenuates over the distance away from the transducer due to bubbles.

For a batch process sonication is easily applicable, however, for a continuous process the integration of flow and sonication is crucial and an effective design is tough to achieve. Previously we have worked on studying the chemical effects of sonication [10][11]. In this work, further high fidelity analysis is carried out on a previously designed sono-chemical reactor [12][13]. The goal is to gain a fundamental insight into the localized conversion and species distribution by using numerical simulation and also to analyze the effectiveness of sonication on the transesterification reaction. The ultrasound mechanism is simulated using the linear wave equation. The acoustic cavitation phenomena which also causes an attenuation of the wave has been accounted for using the complex wave number and impedance as per Sutkar et al. [9]. A logical reaction rate coupling model as previously applied in [12][13] and by Jordens et al. [14] is used to estimate the collective effect of sonication and flow agitation in the reactor. This model system is then applied to study the effect of sonication on the kinetics of reaction.

2. Methodology

In this work we have designed a sono-chemical reactor for the transesterification reaction. This reactor has unique sectioning which allows it to utilize all areas of its geometry, and also helps in increasing the throughput of the continuous sono-chemical conversion process. The top section of the reactor is designed for sonication. It uses the sonic energy from the sonotrode and concentrates it in a small section of the reactor such that the maximum volume of the fluid is sonicated. It acts as a pseudo flow cell. The following section is a static mixer section. Having a static mixer helps in further conversion of the unreacted chemicals coming from the sonication section. The design allows the use of different types of static mixers depending on the viscosity of the reacting fluids. The last section is the collection section which helps in initiating the separation of biodiesel and glycerol. The reactor height is 25 cm and diameter is 6 cm. A 2D model is made in COMSOL Multiphysics package for simulating the acoustic, reacting flow and chemical kinetic physics. The reactor geometry is given in Fig. 1.

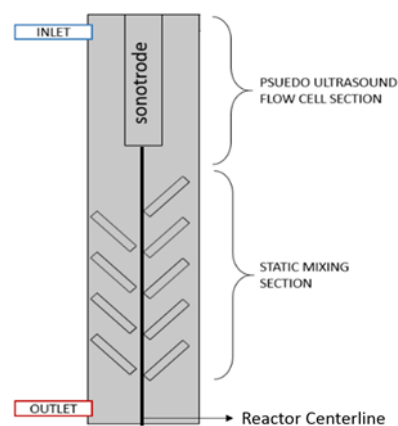


Figure 1: Reactor Geometry

The methodology followed to carry out the intended analysis is illustrated in Fig. 2.

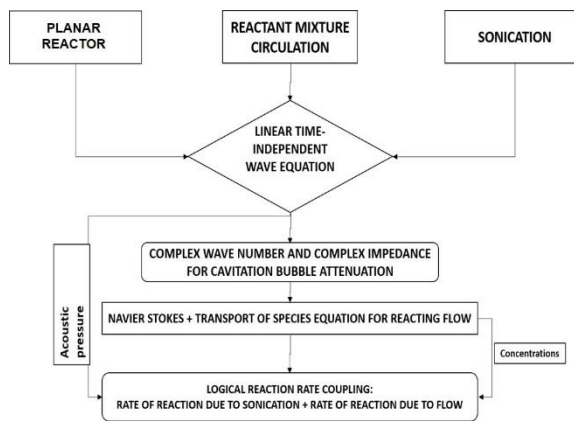


Figure 2: Methodology for analysis

2.1. Linear time-independent wave equation

For simulating the acoustic phenomena, the linear time independent wave equation was used. In this form of the equation the pressure is considered as time harmonic function, i.e. $P(x, t) = P(x)e^{i\omega t}$. Therefore, the linear wave equation is as Eq. 2.

$$\frac{1}{\rho} \nabla^2 \mathbf{P} - \frac{k_c^2}{\rho} \mathbf{P} = 0 \quad (2)$$

Where $\mathbf{P}$ is the acoustic pressure and k_c is the attenuated wave. When cavitation bubbles are formed they allow the reaction to proceed faster, however they decrease the wave energy by causing attenuation. This restricts the wave from reaching far ends of the reactor. This has to be accounted for when designing a sono-chemical reactor. In linearized models that appear in literature this is done by using a complex wave number. As per Sutkar et al. [7] the attenuated wave is simulated using the complex wave number and acoustic impedance as shown in Eq. 3 and 4.

$$k_c = \frac{\omega}{c \sqrt{1 + \left(\frac{i\omega\mu}{\rho c^2}\right)}}, \quad z_c = \frac{\rho c}{c \sqrt{1 + \left(\frac{i\omega\mu}{\rho c^2}\right)}} \quad (3)$$

$$\rho_c = \frac{Z_c k_c}{\omega}, c_c = \frac{\omega}{k_c} \quad (4)$$

Where ρ is the density, c is speed of sound in the medium, Z_c is the complex impedance, ρ_c is the complex density, ω is the angular frequency, μ is the viscosity and C_c is the complex speed of sound.

2.2. Flow simulation

The desired sonicators capacity is between 10 L/hr to 50 L/hr. Therefore, with this in consideration an inlet velocity of 0.0044 m/s is prescribed at the inlet, to have an approximate residence time of 1 min. At such a low velocity the fluid is in the laminar region. Hence, the Navier–Stokes equations for 2D, incompressible, viscous, laminar flow are used to simulate the reactant flow in the reactor as given in Eq. 5.

$$\rho(\mathbf{u}\nabla\mathbf{u}) = -\nabla P_{flow} + \mu\nabla^2\mathbf{u} + \rho\mathbf{g} \quad (5)$$

Where, $\mathbf{u}$ is the velocity field, $\mathbf{g}$ is the gravitational acceleration, P_{flow} is the pressure.

2.3. Reactant transport

For simulating the reaction within the flow and the conversion of species the transport of dilute species equation is used (see Eq. 6).

$$\nabla(-D\nabla c_i) + \mathbf{u}\nabla c_i = R_{rate} \quad (6)$$

D is the diffusion coefficient, c_i is molar concentration of the i^{th} specie, R_{rate} is the rate of reaction and $\mathbf{u}$ is the velocity profile from the flow simulation. The Arrhenius model for the reaction kinetics is given in Eq. 7 as:

$$K_{flow} = A \cdot e^{\frac{-E}{R_u T}} \quad (7)$$

Where K is the rate constant, A is the pre-exponential factor, E is the activation energy, R_u is the universal gas constant and T is the temperature. The activation energy and pre-constants are obtained from the work of Nouredini et al [15].

2.4. Reaction rate coupling

The rate of reaction R_{rate} was taken as pseudo first order model as per the work of Freedman et al. [16]. To couple the rate of reaction due to sonication and flow agitation the method by Jorden's et al. [14] was adopted. In this procedure the Arrhenius rate constant of sonication was calculated, where the temperature is the one that inside the cavitation bubble. The temperature is calculated using the adiabatic ideas gas transition equation. The rate constant and the cavitation bubble temperature are as per Eq. 8 and 9.

$$k_{son} = A \cdot e^{\frac{-E}{R_u T_{bubble}}} \quad (8)$$

$$T_{bubble} = \frac{T_L P (\gamma - 1)}{P_{vapor}} \quad (9)$$

Where k_{son} is the sonication rate constant, T_{bubble} is the

cavitation bubble temperature, γ is the specific heat ratio, P is the acoustic pressure and P_{vapor} is the vapor pressure. T_L is the liquid temperature however in this study we have taken a fixed value of T_L at 333 K, to have at-least the vapor phase of methanol, since methanol evaporates at 333 K. P_{vapor} was taken as the molar average of the vapor pressures of vegetable oil and methanol.

Using Eq. 8 and 9 in a logical coupling model the coupled reaction rate was calculated. This is setup such that the sonication reaction rate is applied only when the acoustic pressure is above the blake pressure. The coupling model is given in Eq. 10.

$$-R_{rate} = [(P > P_{blake})\beta k_{son} * [Oil * Methanol] + (1 - \beta)k_{flow} * [Oil * Methanol]] \quad (10)$$

And β is the cavitation bubble volume which is calculated from Eq. 11 [12] [14] and [17].

$$\beta = 2 \times 10^{-9} P \quad \text{for } P_{blake} < P < 1 \times 10^8 \text{ Pa.} \quad (11)$$

2.5. Boundary conditions

The initial amplitude at the transducer was calculated from Eq. 12.

$$P_w = \sqrt{\frac{2\rho C P_d}{A}} \quad (12)$$

P_w is the initial amplitude in Pa, P_d is the rated power in Watt and A is the area of the transducer. At the walls of the reactor the dirichlet boundary condition $P=0$ was applied implying pressure release, which is valid when the material is highly absorbing such as Teflon [18].

For the laminar flow velocity of 0.0044 m/s was specified at the inlet and at the outlet $P_{flow} = 0$ was applied. For the transport equation the inlet was specified in terms of concentrations.

3. Results And Discussion

In previous studies related to sono-chemical reactors the simulations were carried out with only single liquid in perspective, but in our study we have a mixture of liquids. Hence the effective properties need to be evaluated. The speed of sound was calculated using Eq. 13.

$$C = \sqrt{\frac{K_E}{\rho_E}} \quad (13)$$

Where K_E and ρ_E are equivalent bulk modulus and equivalent density calculated from volume fractions of the reactants.

For calculating equivalent viscosity the Refutas equation was used. In this method the viscosity blend for the reactants is calculated (see Eq. 14). Using mass fractions the blend index of the mixture is evaluated (see Eq. 15) and finally the effective viscosity is calculated from Eq. 16.

$$VBN_i = 14.534 \times \ln(\ln(v_i + 0.8)) + 10.975 \quad (14)$$

$$VBN_{mixture} = \sum_{i=0}^N x_i \times VBN_i \quad (15)$$

$$\begin{aligned} v_{mixture} &= \left(\exp\left(\exp\left(\frac{VBN_{mixture} - 10.975}{14.534}\right)\right) \right) \\ &- 0.8 \end{aligned} \quad (16)$$

Where VBN is the viscosity blend index, v_i is the kinematic viscosity of the element in cSt, and x_i is the mass fraction.

3.1. Acoustic Pressure Simulation

The acoustic simulation was initially carried out at a frequency of 24,000 Hz and rated power of 100 W. The wave attenuation was clearly evident from the results as can be seen in Fig. 3. The peak pressures were not very different in the two cases but the acoustic pressure waveform was flattened much closer to the sonotrode tip in the case of attenuated pressure. It is observed that in both the cases the acoustic pressure over the static mixer is lower as compared to the acoustic pressure in the flow cell section. This is due to the mixer blades obstructing the

sound wave. Due to this obstruction the sonic energy is concentrated in the region close to the sonotrode. The energy concentration per unit area is higher between the sonotrode and the start of the static blades as compared to any other area in the reactor. This helps in better conversion of reactants and without using additional energy. Using such a design reduces the need for having multiple sonotrodes, which add up to the power consumption and cost. Hence, the design seems superior in terms of utilizing the acoustic energy effectively.

3.1.1 Acoustic sensitivity

To evaluate the design a parametric study was carried out. This study is aimed at identifying the best values of the acoustic parameters to achieve better conversion. The parameters that best govern the acoustic performance of the reactor are the rated power and frequency of the equipment. A parametric study with 5 cases of rated powers and 3 cases of wave frequency was carried out. The rated power was varied from 100 to 300 W in steps of 50 W and three frequencies i.e. 24, 36 and 70 kHz were studied. The results of the attenuated acoustic pressure at different powers is shown in Fig. 4 and the results from the frequency study are shown in Fig. 5.

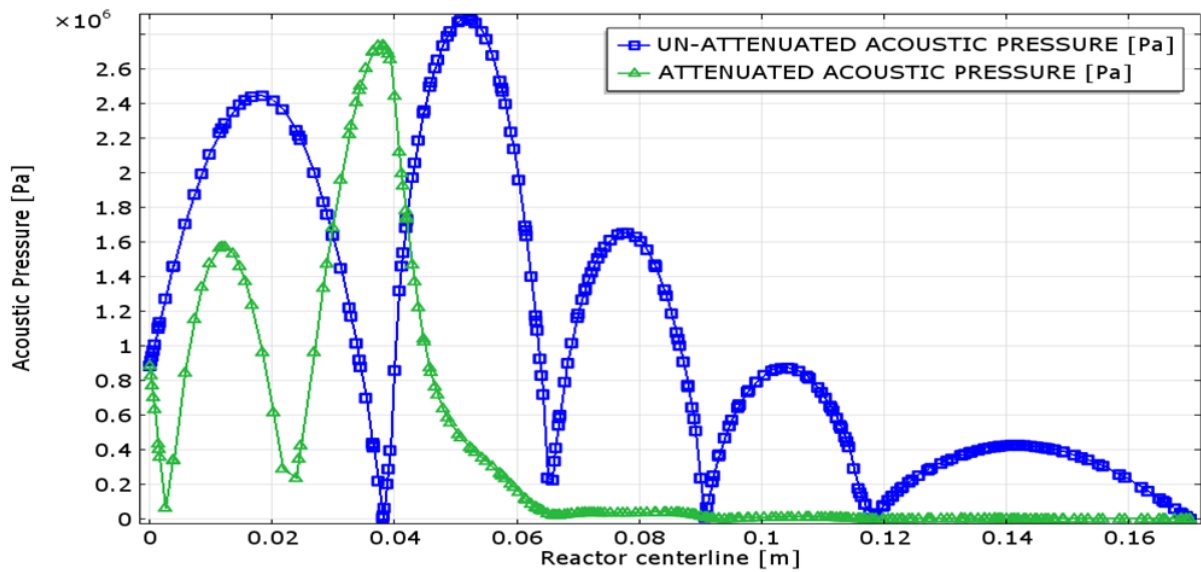


Figure 3: Acoustic Pressure simulation at 100 W and 24000 Hz.

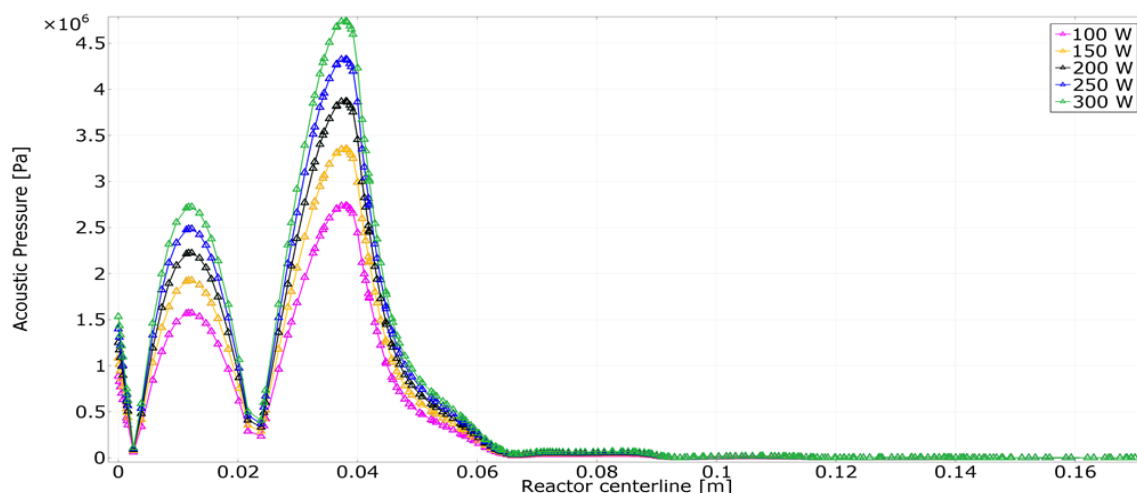


Figure 4: Attenuated acoustic pressure at different power levels at 24000 Hz.

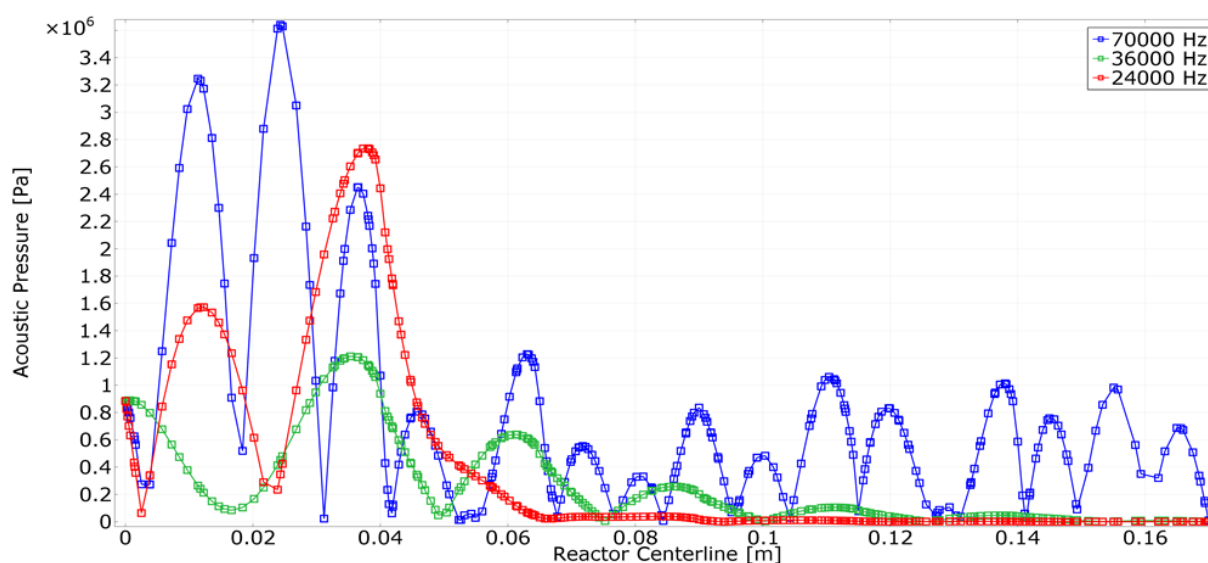


Figure 5: Attenuated acoustic pressure at different frequencies and 100 W power

Power sensitivity: The simulated peak acoustic pressure showed a direct proportionality to the rated power. As the power increased the peak pressure also increased but the pressure distribution profile remained similar. The least acoustic peak pressure of 2.6 MPa was observed under 100 W power at a distance of 0.04 m from the sonotrode. Under a power of 150 W the acoustic pressure was raised to 3.4 MPa whereas the maximum peak pressure of 4.6 MPa was observed at 300 W power at the same position. After a distance of 0.06 m from the sonotrode the pressure started to flatten out.

Frequency Sensitivity: In the frequency study, no defined relation between the acoustic pressure and applied frequency was observed. The peak pressure was highest in case of the highest frequency of 70 kHz, which was about 3.7 MPa. The lowest peak acoustic pressure was observed at 36 kHz which was about 1.2 MPa. This undulation in the peak pressure at different frequencies can be attributed to the relative variation between the transducer initial amplitude, complex wave number and complex impedance. It is observed that a higher acoustic pressures are favored by either a higher imaginary part of the impedance or a higher real part of the wave number. At the frequency of 24 kHz the imaginary part of the impedance is higher whereas at a frequency of 70 kHz the real part of

wave number is higher. At 36 kHz their combination does not support increase in acoustic pressures. At a higher frequency acoustic pressures were observed to be stronger in the whole length of the reactor including the static mixer section. At 36 kHz, though the acoustic pressure is low the wave flattens at a distance of 0.12 m which is higher than what is observed at 24 kHz.

Based on this study it can be said that higher frequencies are better for this reactor and this fluid medium. For different fluids this depends on the density and speed of sound.

3.2. Reactive Flow Simulation

The transesterification reaction highly depends on two factors, the molar ratio and temperature. But when carried out under sonication, the rated power and frequency also affect the product proliferation. As per stoichiometry, one mole of oil needs three moles of methanol to produce three moles of FAME. But this is the ideal case. Increase in molar ratio usually needed and that provides better conversion. This is simply since there is more methanol for every mole of oil which increases the interaction between the reactants on in accordance to Le Chatelier's principle. However, there is a limit to increasing molar ratio. Excess

methanol causes post-process separation difficulties. Excess methanol in the produced fuel renders the fuel unsuitable as per the ASTM standards.

As for temperature, since the Arrhenius kinetic model is adopted the rate constant increases with increase in temperature, thereby increasing the reaction rate. For the reactant volume the max temperature that the reactants can be raised to is 333 K, which is the boiling point of methanol. The power and frequency only affect the cavitation bubble temperature and the sonication rate constant. The calculated bubble temperatures were much higher than the fluid domain temperatures. The sonication rate constants were several orders higher than the conventional rate constants.

Results clearly show that sonication is indisputably better than agitation methods. Visualizations of biodiesel concentrations showed higher concentrations forming between the static mixer blades. A sensitivity study has been carried out to study the effect of molar ratio, temperature, power and frequency on the sonication rate constant.

3.2.1 Reaction sensitivity

Molar ratio sensitivity: For the flow agitation, with increasing molar ratio the biodiesel concentration in the

reactor also increased which is the obvious effect of the increased interaction of reactants. Benefit of having static mixer is proved since higher concentrations of biodiesel were observed between the blades. On the sonication part, with increasing molar ratio it was observed that the temperature of the bubble decreased. This decreased the sonication rate constant. This decrease is caused by the rise in vapor pressure of the cavitation bubble with increase in methanol. This result implies that higher molar ratios are not actually beneficial at the localized level, or more specifically at the scale of a cavitation bubble. This also provides the benefit of adopting lower molar ratios for sonicated transesterification. Fig. 6 shows the concentration profile in the reactor for different molar ratios. The max bubble temperature was observed at molar ratio of 1: 3 at all power levels.

Temperature Sensitivity: As a result of the Arrhenius model, higher concentrations were observed for higher fluid temperatures. There was no effect of temperature on the cavitation bubble temperature or the sonication reaction constant. Fig. 7 gives the concentration profile of biodiesel in the reactor at different temperatures.

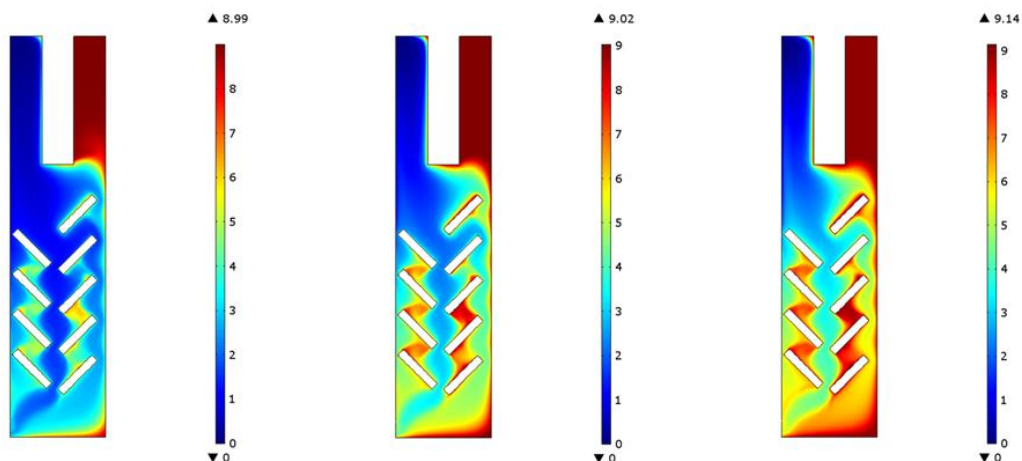


Figure 6: Biodiesel concentration profile (mol/m^3) at molar ratios of 3:1, 6:1 and 9:1 at 330 K temperature

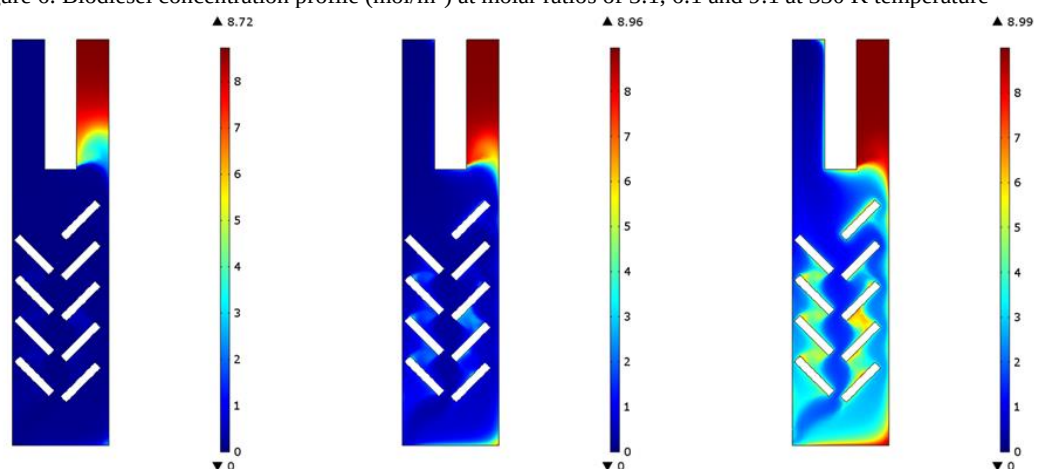


Figure 7: Biodiesel concentration profile (mol/m^3) at 310 K, 320 K and 330 K at 3:1 molar ratio.

Power Sensitivity: As mentioned in the above sections, power increase resulted in a direct increase of acoustic pressures inside the reactor. As from Eq. 9 and 10 we see that the cavitation bubble temperature and rate constant are dependent on the acoustic pressure. To study the same a sensitivity study with 5 different power levels was carried out. Results adhere to relations in the equations. Higher bubble temperature and higher sonication rate constant were observed with increase in rated power. The maximum values were calculated at a power of 300 W.

Frequency sensitivity: The variation in cavitation bubble temperature and K_{son} with frequency was closely similar to that of acoustic pressure. The highest values were obtained at a frequency of 70 kHz and the lowest values were obtained at a frequency of 36 kHz. This variation again implies that higher frequencies are better for this reactor design and fluid medium.

The results for the surface averaged cavitation bubble temperature and sonication rate constant for molar ratio sensitivity are given in Fig. 8. and those for frequency sensitivity are given in Fig. 9.

4. Conclusions

In this work a multi-physics simulation of sonicated transesterification was carried out by following procedures that were experimentally validated in literature to study the physical and chemical effects. The acoustic pressure waves were simulated by the Helmholtz equation. The wave

attenuation due to cavitation was accounted for using complex wave number and impedance. The reactive flow was simulated using a coupled Navier-Stokes and species transport equations. To simulate the cavitation bubble temperature and reaction rates, a logical coupling model based on acoustic pressure was applied. The Arrhenius kinetic model was used for calculating the reaction kinetics. A novel reactor with unique sectioning was tested. A sensitivity study was carried out to study the effect of power, frequency, molar ratio and temperature on biodiesel formation in the reactor. Results showed the rated powers is directly proportional to the bubble temperatures and the reaction rate constants. For the frequency study it was observed that the best conversion results were obtained at 70 kHz and the poorest were at 36 kHz. As for molar ratio, higher molar ratios increased biodiesel formation for the flow agitated conditions but an opposite trend was observed for the sonication case. At higher molar ratios cavitation bubble temperature was lower leading to reduction in the rate constant. This implies that at the localized level the stoichiometric molar ratio is desirable. In the temperature study it was observed that the increase in temperature had a direct relation with biodiesel formation. Apart from studying the effects of sonication, this study also aimed at broadly testing the performance of the reactor design. From the simulations it was clear that having a pseudo flow cell section and a static mixer adds to the performance of a sono-chemical reactor.

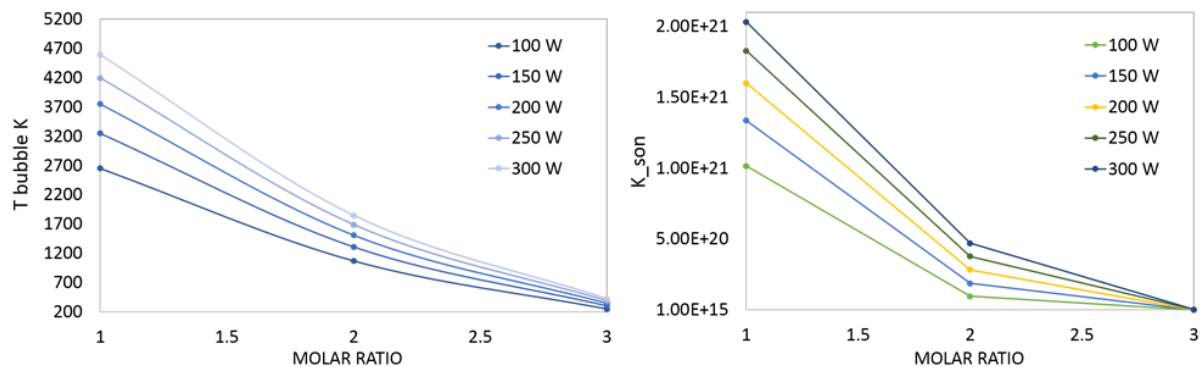


Figure 8: Cavitation bubble temperature and K_{son} at different powers and molar ratio at 24000 Hz.

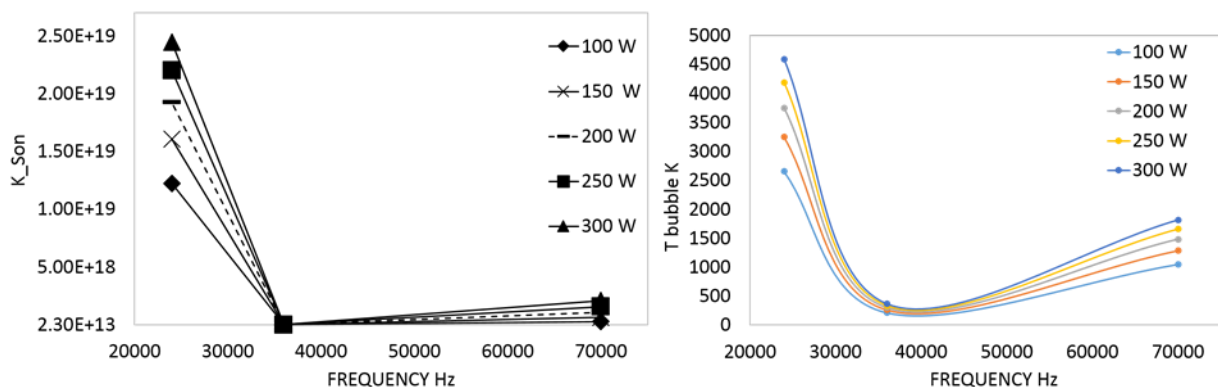


Figure 9: K_{son} and cavitation bubble temperature at different powers and frequencies at 3:1 molar ratio.

Property Table

Property	Unit	Value
Activation energy, E	J/Mol	164958.4
Adiabatic coefficient, γ	-	1.4
Ambient liquid pressure, P_{liq}	Pa	1.00E+05
Blake threshold P_{blake}	Pa	1.00E+05
Oil density [19]	Kg/m ³	883
Dynamic viscosity of oil [20]	Pa.s	1.62E-02
Density of methanol [21]	Kg/m ³	883
Dynamic viscosity of methanol [22]	mPa.s	0.545
Pre-exponential factor A	m ³ /mol/s	3.49E+22
Vapor pressure of vegetable oil [23]	Pa	543
Vapor pressure of methanol[24]	Pa	13020
Universal gas constant R_u	J/mol.K	8.314
Bulk modulus of Methanol [25]	N/m ²	0.8E9
Bulk modulus of Oil [26]	N/m ²	2.1E9

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