

# A comparative study on predictions of vapor liquid equilibrium of biodiesel systems

K J Suthar, M H Joshipura

**Abstract--**In process simulation, reliable and accurate property estimation methods play an important role in the solution of various simulation problems where convergence is often traced to failures in the reliable predictions of physical and thermodynamic properties. Convergence and reliability of the simulation is heavily dependent on the fluid package selected. Predictions of phase equilibrium using various thermodynamic models provide an idea about which model will be able to represent the entire process in better way. No such systematic studies are done in the literature for biodiesel. Paper presents a review on various simulation studies done for biodiesel production to highlight the fact that the thermodynamic models were used with little knowledge or by intuitions. A comparative study is presented for the proper selection of fluid package by determining vapor liquid equilibrium data for various (fatty acid-alcohol, fatty acid-glycerol, alcohol-glycerol, fatty acid ester- alcohol etc) biodiesel systems. The comparison was done with the help of making a model of experimental VLE data and simulated VLE data (by various thermodynamic models).

**Key word--** Aspen HYSYS, Biodiesel Simulation, VLE

## I. INTRODUCTION

High cost of biodiesel is mainly because of the feed stock. To face the biodiesel process simulation and the prediction of the normalized final properties using a process simulator, it is necessary to take into account several issues which include the quantity and quality of the constituents of the departing oils, the topology of the process, the detailed kinetic models and the thermodynamic packages. Also triolein, tripalmitin, trilinolein and tristearin etc. should be considered precisely as they are the major triglycerides in most commonly used oils for biodiesel production. The fatty acids alkyl esters resulting from its alcoholysis account for more than 90 wt.% of palm, soybean, rapeseed and sunflower oils[1]. In process simulation, reliable and accurate property estimation methods play an important role in the solution of various simulation problems where convergence is often traced to failures in the reliable predictions of physical and thermodynamic properties.

We can partly describe oils by the original source, like peanut oil, canola oil, soy oil, beef tallow, and on and on. There are drying oils and non drying oils. Waste oils need more information, like FFA content, and water content. "Organic oils are three long strings of fatty acids attached to a glycerin molecule. The Fatty Acids can vary in length and in how they are bonded or put together. The way we describe Fatty Acids is by the number of carbon atoms in it and the number of double bonds. We typically see between 12 and 26 carbon molecules in our oils and oils with zero to three double bonds. The purpose of this study is to compare different

thermodynamic models available for the simulation of process using HYSYS and select the best model for the simulation of biodiesel process production.

### 1) Biodiesel System

Biodiesel (mono-alkyl esters of long chain fatty acids) is a promising alternative or extender to conventional petroleum based diesel fuel. Biodiesel has a number of advantages [2] – it is derived from a renewable domestic resource (vegetable oil), reduces carbon dioxide emissions by 78% when compared to diesel fuel on a life cycle basis, and is non-toxic and biodegradable, making it a more environmentally benign fuel. Biodiesel system includes fatty acid, alcohol, fatty acid ester and glycerol. But fatty acid is not directly represented as oil, it consists of different fatty acid chains, which vary in carbon chain length and in the number of unsaturated bonds (double bonds).

To simplify the analysis of kinetics and thermodynamics, oil can be regarded as pure compound like triolein, trilinolein, tripalmitin, tristearin etc [3]. Different oils and their composition used as biodiesel system have been reviewed in Table-1. For simulation of biodiesel production process, detailed information about oil and its composition is needed. Also thermodynamic models have to be studied with great care.

### 2) Binary VLE phase diagram

The knowledge and the capability to describe the phase equilibria of binary systems formed by a fatty acid, alcohol, FEMA and glycerol are of great interest in biodiesel production. The knowledge about the vapor-liquid equilibrium (VLE) conditions of these systems is also essential for a correct sizing of the operation units of biodiesel production. The miscibility of triglycerides and methanol are rather poor due to their dissimilarity in size and polarity, and they form two liquid phases upon their initial introduction into reactors. One factor of particular importance in the alcoholysis process is the degree of mixing between the alcohol and triglyceride phases [4]. In other words, the phase behavior of the reaction mixture is crucial for the reaction process. The appropriate selection of thermodynamic models has a strong influence on calculations. The appropriate selection of thermodynamic models has a strong influence on VLE and LLE calculations. This reason makes this comparison of model using VLE, especially important.

TABLE-1  
LITERATURE SURVEY: OIL, SIMULATOR AND THERMODYNAMIC MODEL USED FOR BIODIESEL PRODUCTION PROCESS

Oil Used	Oil composition	Simulator used	Thermodynamic model used	Reference
Vegetable Oil: rapeseed and Rapeseed Oil	Triolein and Oleic acid represent Oil. Methyl Oleate represent biodiesel	ASPEN Plus®	NRTL and UNIQUAC	[7]
High quality Rapeseed Oil	Oleic acid represent as the overall fatty acid and biodiesel as methyl oleate	Aspen Plus 2006.5	A UNIFAC-DMD (Dortmund)	[8]
Castor Oil	Triolein represents Oil and Methyl Oleate represent fatty acid ester	ASPEN Engineering Suite™ 11.1	Wilson Equation	[9]
Vegetable oil (sunflower Oil)	Triricinolein represents castor oil and ethyl ricinoleate to represent biodiesel.	HYSYS (Hyprotec system)	NRTL and UNIFAC	[10]
Waster vegetable oils	Triolein, Trilinolein, Tripalmitin, Tristearin, Oleic, Linoleic, Palmitic and Stearic acid represent oil and methyloleate, M-linoleate, M-palmitate and M-stearate represent biodiesel	Aspen HYSYS V7.0 software	NRTL and UNIQUAC for polar compound and UNIFAC	[5]
Renewable lipids feedstock	94 wt% Triolein and 6 wt% oleic acid (C <sub>18</sub> H <sub>34</sub> O <sub>2</sub> ). Consequently, methyl oleate (C <sub>19</sub> H <sub>36</sub> O <sub>2</sub> ) is used to represent biodiesel	Aspen Hysys	UNIFAC LLE model, NRTL for polar compound	[11]
Vegetable oil	Oleic acid is used as the key component representing triglycerides in this study (triolein) and methyl oleate (C <sub>19</sub> H <sub>36</sub> O <sub>2</sub> ) represents the final product (FAME or biodiesel)	ASPEN Plus® V2006	RK Aspen EOS, UNIQUAC and UNIFAC-LL, electrolyte NRTL	[12]
Conola Oil	Triolein represents oil and methyl oleate as biodiesel	HYSYS and plant NetVer 3.2	NRTL	[13]
Jatropha Oil	Triolein represents oil and methyl oleate represents the resulting biodiesel	HYSYS plant NetVers 2.1.3	NRTL and UNIQUAC	[14]
Rapeseed oil	oleic acid 44.7%, linoleic acid 32.8%, palmitic acid 14.2%, steric acid 7.0%, and others 1.3% represent oil and methyl palmitate, methyl oleate and methyl linoleate are biodiesel.	ASPEN Plus®	NRTL and UNIQUAC	[15]
Virgin vegetable oil and waste oil	Triolein is used to represent and methyl oleate represents biodiesel	ASPEN Plus	NRTL and UNIFAC -LLE	[16]
Palm oil	Triolein represents the virgin vegetable oils and triolein and oleic acid in the case of waste cooking oils. Methyl or ethyl oleate represent biodiesel	Aspen plus 2006	SRK and Electrolyte NRTL	[17]
	Triolein represents oil and ethyl oleate represent biodiesel	Aspen Plus v. 11.1	NRTL and UNIFAC	[18]

### 3) Thermodynamic Properties Packages

Various thermodynamic property packages used for the simulation of biodiesel production process along with the simulator is reviewed in Table-1 which showed most of the process have used NRTL or UNIQUAC. The presence of polar compounds such as methanol and glycerol makes necessary the use of the non-random two liquids (NRTL) model or Universal Quasi Chemical (UNIQUAC) model [5]

On other hand, fatty acid like triolein are highly non polar organic phase[6]. Biodiesel systems includes both oil and alcohol and it ultimately the extent to which both react forming the product biodiesel. Hence, estimating thermodynamic model suitable for biodiesel system is essentially a primary work for the simulation of biodiesel production process.

### II. COMPARING FLUID PACKAGES BY DETERMINING VLE DATA OF VARIOUS BIODIESEL SYSTEMS

Various fluid packages were compared on the basis of available VLE data from various literatures [19-24]. Vapor-Liquid equilibrium data of various system including fatty acid-alcohol, fatty acid-glycerol, alcohol-glycerol, fatty acid ester- alcohol etc. were plotted using HYSYS. Such plots were formed for every different fluid package and every different system. Data tables were generated from these plots and from the generated data, a plot of liquid composition (x) vs. temperature (°C) was plotted to obtain base equation from trend line using MS excel® for the particular system. Similarly, base equation was generated for all the fluid packages for further calculations. A base equation was used to calculate temperature/pressure at common composition points for different fluid packages for a particular system. An example of methyl oleate-methanol system is demonstrated further.

1) VLE Plot using HYSYS

Two types of vapor-liquid equilibrium diagrams are widely used to represent data for two-component (binary) systems in the study. The first is a ‘temperature versus x and y’ diagram (Txy). The x term represents the liquid composition, usually expressed in terms of mole fraction. The y term represents the vapor composition. These types of diagrams are generated at a constant pressure. And the other type is ‘pressure versus x and y’ which are generated at a constant temperature [25]. The diagram is easily generated using Aspen HYSYS by going to flowsheet-add operation-extension-equilibrium plots. But an equilibrium plot unit operation extension should be registered. Various VLE for the biodiesel systems were plotted as shown in an example of methyl-oleate and ethanol at 101.32KPa pressure in figure-1.

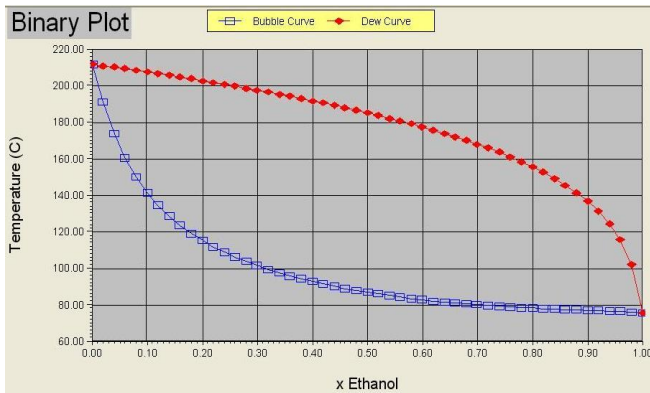


Fig 1. VLE plot for Methyl oleate-Ethanol system at 1atm

2) Computing the base equation

VLE data collected from various literatures have temperature/pressure range at varying composition points from that of the data available from HYSYS. Hence to obtain the data at common point for their comparison, a base equation is generated using the trend line as shown in figure-2. Similarly, Composition(x) vs. Temperature (°C) is plotted for various fluid packages as shown in figure-3.

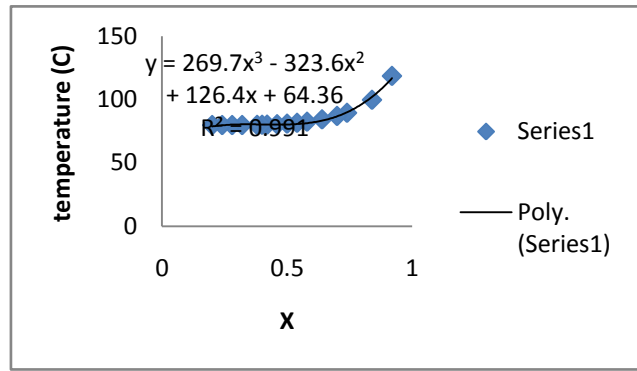


Fig 2 Composition(x) vs. Temperature (°C) plot for base equation for methyl oleate-ethanol system at 101.31KPa

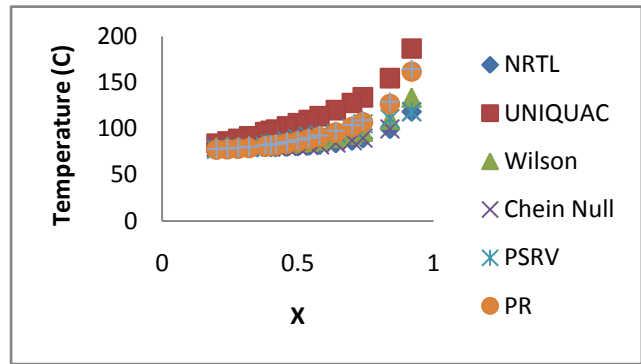


Fig 3 Composition(x) vs. Temperature (°C) plot for methyl oleate-ethanol at 101.3KPa for various FP

3) Calculating %AAD for Methyl oleate-Ethanol at 101.3KPa using various fluid packages

Once base equation is generated, temperature/pressure at common composition can be obtained. Similarly, obtaining it for various fluid packages and tabulating it. The values obtained from the literature are considered as the basis for the comparative study with the one obtained from HYSYS. Table-2 shows %AAD for methyl oleate-Ethanol at 101.32

TABLE- 2  
CALCULATION OF DEVIATION FOR METHYL OLEATE-ETHANOL SYSTEM AT 101.3KPa USING NRTL

N	X	T °C	X exp	Base case value, T(°C)		NRTL, T(°C)	Deviation
				y = 269.7x <sup>3</sup> - 323.6x <sup>2</sup> + 126.4x + 64.36			
1	0.5814	88.34	0.58	86.04		81.43	0.054
2	0.5305	86.76	0.54	85.09		80.72	0.051
3	0.4964	85.63	0.5	84.77		80.37	0.052
4	0.4653	85.22	0.46	84.82		80.28	0.054
5	0.4279	84.09	0.42	85.02		80.35	0.055
6	0.4025	83.55	0.4	85.10		80.40	0.055
7	0.3791	83.13	0.38	85.12		80.46	0.055
8	0.3184	83.57	0.32	84.53		80.51	0.048
9	0.2793	81.74	0.28	83.30		80.30	0.036
10	0.2369	81.31	0.24	81.10		79.78	0.016
11	0.2125	81.04	0.2	77.70		78.85	0.015
%AAD for NRTL is 4.38				<b>83.65</b>		<b>80.22</b>	<b>0.0438</b>

Where N is number of points

### III. RESULTS AND DISCUSSION

Two types of systems i.e. isothermal and isobaric were considered for the comparative study on vapor liquid equilibrium prediction. For the example considered, methyl oleate-ethanol at 101.32KPa Wilson activity model showed minimum deviation of 3.005%. Similarly, various biodiesel systems were compared using different property packages. FEMA were not recommended for PR and SRK equation of states as there use would invalidate the simulation result. Also compounds like Glycol and ethanol were not recommended for PR and SRK. Results for the isobaric systems are tabulated in Table-3.

From all isobaric systems considered, Wilson model gave minimum percentage absolute average deviation of 12.99. Considering isothermal systems, deviation have increased for Wilson activity model. The results for the isothermal systems are tabulated in Table-4. The absolute average deviation for PR and SRK is 13.94% and 11.93% respectively but most of their values were found for the missing binary coefficient. Similarly, PRSV and Chein Null had 17.12% and 13.54% of absolute average deviation respectively.

TABLE-3  
% DEVIATION FOR THE ISOBARIC SYSTEM

System	Isobaric	N	% Deviation						
			PR	SRK	NRTL	UNIQUAC	Wilson	PRSV	Chein Null
Methyl Oleate-Methanol	101.32KPa	15	3.2*	3.88*	3.0363	17.508	1.967	3.9507*	2.934*
Methyl Oleate-Ethanol	101.32KPa	16	3.451*	4.2*	7.334	20.318	3.005	19.28*	7.275*
Ethanol-Glycol	101.32KPa	15	15.5*	14.9*	26.404	26.3	9.885	27.42*	9.06*
Methyl Laurate-Methanol	101.32KPa	15	9.85*	9.85*	1.5001	1.166	1.52	9.81*	1.59*
Ethanol-Ethyl Stearate <sup>a</sup>	92KPa	11	1.6*	2.4*	6.11	37.13	10.9	2.25*	6.38*
Methyl Myristate-Methanol	101.32KPa	9	6.91*	5.48*	19.36	27.69	15.48	7.44*	19.33*
Methyl Laurate-Ethanol	101.32KPa	9	12*	13.07*	3.67	3.32	3.07	11.08*	3.77*
Methyl Myristate-Ethanol	101.32KPa	8	10.3*	11.43*	4.801	25.59	1.89	10.01*	2.95*
Ethyl palmitate-ethyl stearate <sup>b</sup>	5.3329KPa	9	53.65	53.1	56	55.98	55.99	53.65	55.96
Glycol-Water	101.32KPa	9	25.88	25.67*	26.17	26.66	26.26	26.35*	26.16*
%AAD			14.24	14.39	15.43	24.16	12.99	17.12	13.54

Where, N is number of points and \*Binary interaction parameters are missing, <sup>a</sup>Systems for which one component is hypothetically created and <sup>b</sup>System for which both components are hypothetically created

TABLE-4  
% DEVIATION FOR THE ISOTHERMAL SYSTEM

System	Isothermal	N	% Deviation						
			PR	SRK	NRTL	UNIQUAC	Wilson	PRSV	Chein Null
Methanol-Glycol	493K	9	13.96*	10.04*	9.92	11.51	15.1	18.84*	9.57*
Triolein-Methanol <sup>a</sup>	493K	10	125.45*	109.32	301.5	95.35	291.46	128.71*	84.2*
Ethanol-Ethyl Laurate <sup>a</sup>	523K	8	4.37*	3.24*	56.34*	168.75	212.27	3.494*	21.98*
Ethanol-Ethyl Laurate <sup>a</sup>	493K	8	0.97*	0.99*	22.7*	82.51	67.95	1.03*	20.07*
Methanol-Methyl Myristate	523k	6	18.69*	19.98*	37.51	10.24	44.71	18.68*	24.17*
Methanol-Methyl Myristate	493K	6	19.70*	20.78*	37.99	13.84	26.44	18.16*	33.44*
Methanol-Methyl Laurate	523K	9	27.78*	18.98*	48.15	52.45	53.66	17.78*	25.67*
Methanol-Methyl Laurate	493K	9	17.18*	18.22*	41.88	35.89	35.65	15.55*	37.18*
Methanol-Glycol	493K	9	17.42*	15.91*	5.8321	4.4642	6.36	15.33*	4.11*
Ethanol-Glycol	493K	8	4.04*	7.43*	43.59	41.32	42.14	4.058*	40.72*
%AAD			13.94	11.93	52.63	51.63	79.57	15.2	9.38

Where, N is number of points and \*Binary interaction parameters are missing and <sup>a</sup>Systems for which one component is hypothetically created

The deviation increased because of three main systems i.e. triolein-methanol, ethanol-ethyl laurate and ethanol-ethyl laurate. All the three systems have component which are missing in HYSYS component library. It showed the high deviation in the result due to critical properties and hence they have to be studied with greater care. Table-5 shows absolute average deviation for the <sup>a,b</sup>systems for which compounds were hypothetically created and high deviation is noticed for all the systems.

TABLE-5  
% DEVIATION FOR THE SYSTEMS FOR WHICH COMPONENTS WERE HYPOTHETICALLY CREATED

Property packages	% AAD
PR	46.55
SRK	43.10
NRTL	82.7
UNIQUAC	91.71
Wilson	136.71
PRSV	48.11
Chein Null	39.23

Where, N is number of points and \*Binary interaction parameters are missing

Most recommended property packages NRTL and UNQUAC gives the deviation of 82% and 91% respectively. The accurate results could not be drawn as binary interaction parameter for most of the property packages are missing. Once those missing parameter values are calculated, they can be used for predicting vapor liquid equilibrium producing an accurate result for the selection of suitable property package. PR and SRK property packages showed comparatively 13-14% deviation but it can only be selected once the missing interaction parameters are obtained and then getting minimum deviation and if it is recommended for the biodieselsystem.

TABLE-6  
% DEVIATION FOR THE SYSTEMS FOR WHICH THE COMPONENTS ARE PRESENT IN HYSYS LIBRARY AND BINARY INTERACTION PARAMETER ARE NOT MISSING

Systems	NRTL	UNIQUAC	Wilson
Methyl Oleate-Methanol	3.03	17.508	1.967
Methyl Oleate-Ethanol	7.33	20.318	3.005
Ethanol-Glycol	26.40	26.3	9.885
Methanol-Glycol	9.92	11.51	15.1
Methyl Laurate-Methanol	1.50	1.166	1.52
Methyl Myristate-Methanol	19.36	27.69	15.48
Methyl Laurate-Ethanol	3.67	3.32	3.07
Methyl Myristate-Ethanol	4.80	25.59	1.89
Glycol-Water	26.17	26.66	26.26
%AAD	11.35	17.78	8.68

But PR and SRK property packages are not recommended for glycol, ethanol and FEMA, hence binary interaction parameters for PRSV and Chein Null are of main interest for comparative study. The results for the deviation of the systems for which components were present in HYSYS library and binary interaction parameter were not missing are tabulated in Table-6. It concludes the use of Wilson activity model over NRTL and UNIQUAC giving minimum deviation of 8.6%. Though accurate results would only be revealed once missing binary interaction parameter are calculated.

#### IV. CONCLUSION

Vapor liquid equilibrium prediction for the biodiesel systems were investigated using different property packages like Peng-Robinson, SRK, NRTL, Chein Null, UNIQUAC, PRSV and Wilson. Among those property packages, activity models have strong predictability. Though missing binary interaction parameter have to be calculated for accurately recommending best suitable property package for biodiesel system and it might have an impact on the present result. But from the present study, Wilson activity model have better accuracy for the biodiesel system. Also critical properties have to be carefully studied in the case of hypothetically added compounds for biodiesel production process.

#### V. ACKNOWLEDGEMENT

I would like to acknowledge all the faculty members of Chemical department, Nirma University for their invaluable guidance and assistance throughout the work.

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