NIPES
Journal of Science and Technology Research
Journal homepage: www.nipesjournals.org.ng

Studies on Phase Overlapping Analysis of Jatropha Oil Biodiesel Ternary Components at Different Temperatures

Kenneth Kennedy Adama*[1] and Lucy Uju Modebe[2]
[1] *Department of Chemical Engineering, Edo State University Uzairue, Edo State, Nigeria*
[2] *Department of Chemical Engineering, University of Delta, Agbor, Delta State, Nigeria*
* adama.kenneth@edouniversity.edu.ng +2348028536012

Article Info

***Keywords*:**
Jatropha oil biodiesel, Ternary component analysis, Separation, Temperature, Phase overlapping.

Received 15 January 2023
Revised 28 January 2023
Accepted 28 January 2023
Available online 13 March 2023

https://doi.org/10.5281/zenodo.7728321

Abstract

In this study, components phase partitioning of jatropha oil biodiesel ternary system is investigated. Modified turbidimetric technique was used to investigate the constituent compositions at nine different temperatures (20 – 60) °C. Analysis of the homogeneous mixture was conducted using gas chromatographic approach. The influence of temperature on the immiscibility region and the separating capacity of the solvent (methanol) was determined from distribution coefficient (K) and solvent selectivity (S) analysis. The results revealed how the individual components of the ternary system distributed in the homogeneous solution at the different temperatures thereby displaying large two-phase envelope. However, high level of immiscibility between the extract and raffinate phases at all investigated temperatures was also observed. The results further revealed overlapping tie lines which was linked to the presence of excess methanol in the feed composition and further emphasized the fact that the tie lines overlap because other components were simultaneously distributed between the extract and raffinate phases. This study provides important property statistics for bio-based biodiesel components separation which are of interest in process design, optimization and simulation.

1.0 Introduction

Transesterification reaction remains the widely used method for the synthesis of biodiesel from edible and non-edible renewable biomass making use of excess alcohol. The product of the process is usually the formation of a homogeneous mixture consisting of specific biodiesel, alcohol, and glycerol [1-3]. Large scale production of biodiesel and the industrialization of the process are hampered due to the challenge of separating the components in the homogeneous mixture [4, 5]. Other challenges include the understanding of the phenomena in the equilibrium behaviour of the homogeneous mixture in terms of transition of the alcohol from the raffinate glycerol phase to extract biodiesel phase for the regeneration of the alcohol [6]. Generally, immiscibility of biodiesel and glycerol is due to differences in density and therefore, partitioning of the two components would require enormous time, resources, energy and capital investment in operation of a continuous biodiesel production and processing plant [7, 8]. Thus, separation and purification of biodiesel from glycerol is the limiting step in the biodiesel production chain requiring extensive research and development of viable options [9, 10]. These options can further assist in the design and optimization of novel biodiesel production units [4, 11, 12].

Few studies [5, 9-10] exist on related phenomena associated with the separation and purification of jatropha curcas oil biodiesel systems after production. Silva et al, 2013 [13] reported component equilibrium composition for binary, ternary and quaternary homogeneous systems formed by fatty acid methyl esters and fatty acid ethyl ester of jatropha curcas oil, water, glycerol, methanol and ethanol at different temperatures (303.15, 318.15, and 333.15) K with a temperature interval of 15 K each. The influence of a wide range of temperature variation on the system component circulation and balance was not investigated. Zhou et al, 2006 [14] reported the inter-solubility of jatropha curcas L oil, methanol, fatty acid methyl ester and glycerol at four different temperatures (298.15, 308.15, 318.15 and 328.15) K with a temperature interval of 10 K each. The result obtained showed that the inter-solubility of the components in the equilibrium was temperature-insensitive within the range of temperature investigated resulting in component circulation changing as the reaction proceeded. Campo et al, 2017 [15] reported experimental equilibrium composition for jatropha biodiesel homogeneous mixture system at a temperature of 45 °C in addition to fuel properties evaluation. The separation and purification characteristics of the biodiesel as well as the other properties were also evaluated at 45 °C and one atmospheric pressure. The components equilibrium compositions available in these studies are still limited within the context of researches conducted so far in the utilization of jatropha curcas oil for biodiesel production [5, 16-17]; hence the research interest in temperature-dependent components partitioning of jatropha oil biodiesel ternary system in addition to fuel property characterizations of the jatropha biodiesel system.

The oil tree of Jatropha belongs to the family Euphorbiaceae. Four different varieties are currently grown in tropical Nigeria which are jatropha gossypiifolia, jatropha podagrica, jatropha multifida and jatropha curcas. Jatropha is an oilseed-bearing shrub that has huge potentials for oil and biodiesel production [5, 13-15, 18]. The chemical constituents and medicinal or toxic effects of jatropha had attracted a lot of interest in the past but there has been gradual shift in its use for oil and biodiesel production [5, 14, 18].

In the reaction to produce biodiesel and the subsequent separation and purification of the products, it is important to know how the individual components distribute in the homogeneous solution at the different temperatures. This work aims to expand upon and improve on the existing available equilibrium component composition involved in the production, separation and purification of methanolic biodiesel from jatropha curcas oil.

2. Materials and Methods

2.1 Materials

Analytical grade chemicals used in the research include glycerol, methanol and potassium hydroxide pellets purchased from Acros Organics, Merck and Sigma Aldrich respectively. Other materials include jatropha oil; mechanically extracted from jatropha oil seed and jatropha oil biodiesel produced via transesterification of the oil with excess methanol in the laboratory. The major equipment used were gas chromatography mass spectroscopy coupled to a flame ionization detector (GCMS FID), beakers of different sizes, digital stopwatches, different sizes of conical flasks, in-built temperature-control thermostatic water bath, diagnostic weighing balances, power-driven agitator, separating funnels, pipettes and burettes.

2.2 Methods

2.2.1 Phase components composition of jatropha oil biodiesel system for determining binodal solubility composition and tie lines

The binodal solubility composition and tie lines for jatropha oil biodiesel ternary system at the investigated temperatures were obtained using cloud-point titration methods employed by several researchers [14, 19-21] with some modifications [2].

2.2.1. Extract phase composition

The phase components composition experiment was performed using the following procedures. 25 ml of glycerol was obtained using a micro-burette and the mass was determined using an analytical balance (XP 3000 Denver instrument). Separate volumes of jatropha oil biodiesel (10 ml) and methanol (15 ml) were weighed into different beakers thoroughly mixed together in a 250 ml beaker. Using a micro-burette, glycerol was titrated into mixture of biodiesel and methanol and continuously stirred with a Fisatom 752A magnetic stirrer. An indication of colour change of the mixture during the titration from transparent to turbid marked the end-point of the reaction. This was the saturation point of glycerol in the mixture of biodiesel and methanol [2, 13, 15, 22].

2.2.2. Raffinate phase composition

In a like manner, the initial volume of jatropha biodiesel (25 ml) was obtained using a micro-burette and the mass similarly determined using a XP 3000 Denver instrument analytical balance. Glycerol (10 ml) and methanol (15 ml) were weighed into different beakers and thoroughly mixed together in a 250 ml beaker. Titration of the jatropha biodiesel was initiated using the micro-burette into the mixture of glycerol and methanol with constant stirring via Fisatom 752A magnetic stirrer. Titration was terminated where there was color change in mixture from transparent to turbid. This was the saturation condition of biodiesel in glycerol-methanol solution. The final volume and mass of biodiesel left in the burette at the saturation point of biodiesel in the glycerol-methanol solution was then determined [2, 13, 15, 22].

2.2.3. Tie line compositions of jatropha oil biodiesel system

Jatropha oil biodiesel, glycerol and methanol samples were prepared in a 250 ml conical flask with ratio of jatropha oil biodiesel to glycerol held constant in a of 1:1 while volume of methanol was increased gradually. The homogeneous mixture was vigorously agitated using a Fisatom 752A magnetic stirrer and a vortex-touch mixer for 4 hours to ensure homogeneity and maintained in a thermostatic water bath at the different investigated temperatures. Equilibrium was achieved after 24 hours in a separating vessel where two separate clear liquid phases was observed. Thereafter, the mixture was separated into two liquid phases namely; extract and raffinate. After the separation of the phases, 5 ml of each sample from both phases was carefully collected with the aid of syringes at various intervals while still vigorously agitating the mixture at the investigated temperatures using the thermostatic water bath. Thereafter, samples were withdrawn for analysis via GC/MS FID to obtain the components and the tie-line compositions [2, 13, 15, 22].

2.3. Calculations

The selection and choice of solvent for the separation and purification process is very important when economies and cost factors of the process are taken into account [23]. This is due to the fact that the process involves completely non-ideal interactions in which a feed containing a solute/solvent is added to an immiscible solvent in which the solute/solvent is soluble thereby making it to distribute between two phases. Agitation of the two phases is continued until equilibrium is achieved. The liquids are then allowed to settle before phase separation or disengagement [1, 24, 25]. Taking into consideration of the above process, a distribution coefficient, K, is defined as ratio of solute/solvent in each liquid phase. Distribution coefficient, K, is one of the main parameters in determining the minimum feed composition to solvent relationship that can be used in any separation process. Large values of K are desirable for the solute since it would imply that less solvent will be required for the separation [26]. Distribution coefficients between any two components in a solution or mixture is important and useful in separation processes. This ratio is referred to as the relative separation or selectivity, S. For feasible, efficient and effective separation, the selectivity must be greater than one. The higher the value from unity (one), the better the separation [26]. Chemical equilibrium between two phases is strongly influenced by distribution coefficient and selectivity. At equilibrium, the activity of a component in each phase is equal [26].

2.4. Distribution coefficient, K, and solvent selectivity, S

Distribution coefficient K, and solvent selectivity, S, were determined using constituents compositions of extract and raffinate phases of the mixture as reported by different authors [2, 13-15, 22]:

$$K = \frac{\text{methanol composition in extract}}{\text{methanol composition in raffinate}} \tag{1}$$

$$= \frac{\text{glycerol composition in extract}}{\text{glycerol composition in raffinate}} \tag{2}$$

$$S = \frac{K_{\text{methanol}}}{K_{\text{glycerol}}} \tag{3}$$

3.0 Results and Discussion

3.1 Phase components analyses

The components compositions for jatropha oil biodiesel mixture at 30 °C and 60 °C are as contained in Tables 1 and 2. Tables 1 and 2 also shows the distribution coefficients of methanol, glycerol and the solvent selectivity values at 30 °C and 60 °C. The feed composition, extract phase composition, raffinate phase compositions as well as the binodal solubility composition at the specific temperatures are also presented in Tables 1 and 2.

The distribution coefficient of methanol (K_{methanol}) in the extract phase was higher causing the jatropha oil biodiesel component to attract the highest amount of methanol in the extract phase giving substantial purification with minimal fraction of raffinate phase in the biodiesel. This was in agreement with studies conducted on different biodiesel systems [20, 21]. Hence, the drying process of the extract phase was greatly favored. At other temperatures (Tables 1 and 2), the distribution coefficient of methanol (K_{methanol}) exhibited consistent values of relatively less than one. The selectivity of jatropha oil biodiesel in the extract phase showed its preference to solubilize methanol. At all system temperatures and atmospheric pressure, the solubility values (S), were positive whole number values. Also, the mass composition of methanol in the raffinate phase showed an increase in the selectivity of jatropha biodiesel for methanol. This was due to the presence of methanol in the raffinate phase that solubilized an amount of glycerol in addition to methanol, thus affecting the selectivity (S) values. This observation was also in agreement with studies conducted by some other researchers [13, 15, 20, 27]. Basically, distribution coefficient and selectivity are important parameters in understanding the ability of the solvent to effectively separate the phases. Essentially, low values of distribution coefficient imply high concentrations of glycerol in raffinate phase and low volume of glycerol solubilized in extract phase [26]. The extraction capacity of any solvent has direct relationship with selectivity. All values of selectivity in this study were greater than one. However, biodiesel product solubility and selectivity is dependent on alcohol concentration [19].

Table 1: Tie-line for jatropha oil biodiesel(1)/methanol(2)/glycerol(3), binodal solubility composition for jatropha oil biodiesel(1)/glycerol(2)/methanol(3), distribution coefficient (K) and selectivity (S) at T = 30 °C

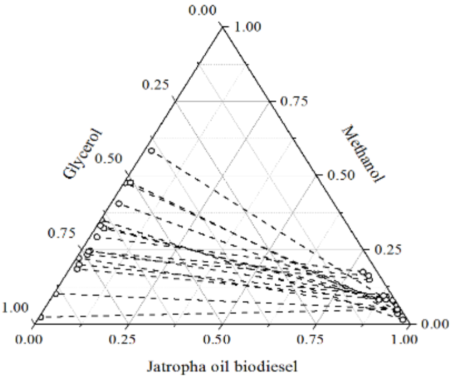
Feed composition			Extract phase			Raffinate phase			Binodal solubility composition			K_{methanol}	K_{glycerol}	S
w_1	w_2	w_3	w_1	w_2	w_3	w_1	w_2	w_3	w_1	w_2	w_3			
0.495	0.040	0.465	0.964	0.023	0.013	0.010	0.553	0.437	0.015	0.877	0.108	0.042	0.029	1.448
0.489	0.028	0.483	0.968	0.018	0.014	0.008	0.559	0.433	0.024	0.780	0.196	0.032	0.032	1.000
0.478	0.047	0.475	0.961	0.022	0.017	0.005	0.509	0.486	0.025	0.668	0.307	0.043	0.035	1.229
0.474	0.072	0.454	0.889	0.096	0.015	0.002	0.473	0.525	0.049	0.560	0.391	0.203	0.029	7.000
0.463	0.093	0.444	0.837	0.163	0.000	0.005	0.359	0.636	0.200	0.300	0.500	0.454	0.002	227.000
0.456	0.101	0.443	0.891	0.105	0.004	0.003	0.240	0.757	0.300	0.200	0.500	0.438	0.005	87.600
0.442	0.113	0.445	0.913	0.078	0.009	0.011	0.184	0.805	0.420	0.130	0.450	0.424	0.011	38.545
0.438	0.137	0.425	0.897	0.101	0.002	0.003	0.210	0.787	0.540	0.075	0.385	0.481	0.003	160.333
0.434	0.171	0.395	0.789	0.181	0.030	0.004	0.077	0.919	0.680	0.050	0.270	2.351	0.033	71.242
0.419	0.200	0.381	0.874	0.103	0.023	0.005	0.134	0.861	0.800	0.030	0.170	0.769	0.023	33.435

0.386	0.233	0.381	0.913	0.081	0.006	0.005	0.112	0.883	0.889	0.016	0.095	0.723	0.007	103.286
0.377	0.175	0.448	0.944	0.047	0.009	0.004	0.125	0.871	0.889	0.016	0.095	0.376	0.010	37.600
0.356	0.247	0.397	0.948	0.036	0.016	0.003	0.078	0.919	0.889	0.016	0.095	0.462	0.017	27.176
0.312	0.234	0.454	0.951	0.039	0.010	0.002	0.086	0.912	0.953	0.040	0.007	0.453	0.011	41.182
0.396	0.237	0.367	0.947	0.031	0.022	0.002	0.083	0.915	0.970	0.020	0.010	0.373	0.024	15.542
0.387	0.168	0.445	0.946	0.031	0.023	0.002	0.081	0.917	0.994	0.006	0.000	0.383	0.025	15.320

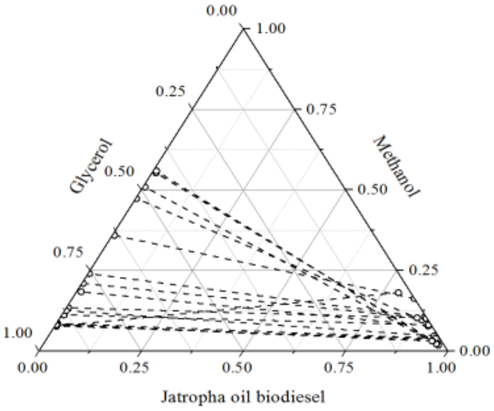
Table 2: Tie-line for jatropha oil biodiesel(1)/methanol(2)/glycerol(3), binodal solubility composition for jatropha oil biodiesel(1)/glycerol(2)/methanol(3), distribution coefficient (K) and selectivity (S) at T = 60 °C

Feed composition			Extract phase			Raffinate phase			Binodal solubility composition			$K_{methanol}$	$K_{glycerol}$	S
w_1	w_2	w_3	w_1	w_2	w_3	w_1	w_2	w_3	w_1	w_2	w_3			
0.539	0.028	0.433	0.956	0.041	0.003	0.002	0.256	0.742	0.015	0.815	0.170	0.160	0.004	40.000
0.538	0.029	0.433	0.953	0.039	0.008	0.004	0.378	0.618	0.017	0.706	0.277	0.103	0.013	7.923
0.533	0.026	0.441	0.947	0.035	0.018	0.002	0.316	0.682	0.014	0.697	0.289	0.111	0.026	4.269
0.488	0.054	0.434	0.913	0.081	0.006	0.001	0.305	0.694	0.078	0.579	0.343	0.266	0.009	29.556
0.443	0.166	0.391	0.893	0.083	0.024	0.012	0.256	0.732	0.120	0.550	0.330	0.324	0.033	9.818
0.423	0.202	0.375	0.887	0.096	0.017	0.010	0.307	0.683	0.160	0.500	0.340	0.313	0.025	12.520
0.400	0.176	0.424	0.853	0.082	0.065	0.008	0.318	0.674	0.210	0.441	0.350	0.258	0.096	2.688
0.384	0.271	0.345	0.848	0.107	0.045	0.011	0.401	0.588	0.260	0.392	0.350	0.267	0.077	3.468
0.354	0.154	0.492	0.928	0.057	0.015	0.013	0.476	0.511	0.360	0.321	0.320	0.120	0.029	4.138
0.351	0.233	0.416	0.906	0.081	0.013	0.018	0.348	0.634	0.679	0.120	0.201	0.233	0.021	11.095
0.332	0.298	0.370	0.881	0.102	0.031	0.019	0.087	0.894	0.796	0.084	0.120	1.172	0.035	33.486
0.316	0.367	0.317	0.887	0.082	0.031	0.015	0.077	0.908	0.869	0.064	0.067	1.065	0.034	31.323
0.302	0.353	0.345	0.904	0.079	0.017	0.017	0.067	0.916	0.910	0.060	0.067	1.179	0.019	62.053
0.296	0.237	0.467	0.936	0.063	0.001	0.014	0.059	0.927	0.911	0.056	0.033	1.068	0.001	1.068
0.380	0.208	0.412	0.889	0.094	0.017	0.002	0.060	0.938	0.984	0.014	0.002	1.567	0.018	87.056
0.463	0.214	0.323	0.914	0.082	0.004	0.002	0.057	0.941	0.985	0.015	0.000	1.439	0.004	359.750

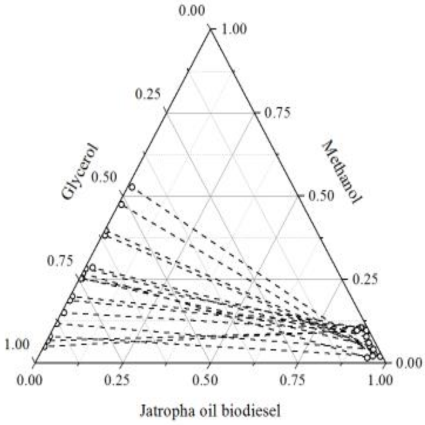
Figure 1 shows the tie line analysis of extract and raffinate phase compositions for jatropha oil biodiesel system at the investigated temperatures. The compositions between the equilibrium extract and raffinate phases are represented by the tie lines which also represent the conjugate phases in equilibrium for the homogeneous mixture. The solubility of methanol in raffinate phase is greater than in extract phase as shown by the size of phase region adjoining methanol and glycerol and gradient of tie lines. Methanol readily solubilizes in raffinate phase due to its strong affinity for glycerol; hence increase in the size of the two phase region enclosing glycerol and methanol. However, the tie lines are seen to overlap with other tie lines as displayed in Figure I. Several reasons can be adduced for this behavior. Firstly, jatropha oil biodiesel component of the homogeneous mixture is less soluble in polar glycerol/methanol phase thereby displaying large two-phase envelope. This observation is in agreement with a similar study [14]. However, due to the presence of methyl oleate, a $C_{18:1}$ fatty acid, the homogeneous mixture exhibits immiscibility between biodiesel extract and glycerol raffinate phases throughout investigated temperatures. Furthermore, from Figure I, there appears to exist a minor single-phase region between the two 2-phase regions at the different temperatures. This scenarios does exist and have indeed been studied though uncommon and only briefly discussed in literatures [28]. It is postulated that the two separate 2-phase regions are strongly dependent on pressure and temperature [26]. Thus, the overlapping tie lines shows the composition of the system appearing as emulsion in one phase and dispersed in small droplets within the other phase. These are the tie lines that returns the constituent compositions of the two-liquid phases and do not form distinct extract and raffinate phases but rather, an emulsion [29]. This can also be described to mean the presence of excess methanol in the feed composition thereby causing the loss of jatropha oil biodiesel in the raffinate phase with further high concentration of glycerol in extract phase. Thus large quantities of methanol in the mixture hampers extract phase purification by settling. Secondly, for the coexisting extract and raffinate phases of Figure 1, the orientation angle of the ties lines at certain withdrawal time intervals increases as the methanol concentration increases. Values at low methanol concentrations support existence of different 2-phase coexistence region of extract and raffinate phases and also existence of 3-phase region separating the 2-phase regions. For obvious reasons, presence of many different fatty acids in biomembrane systems such as biodiesel systems can lead to lateral homogeneity of molecules which can organize the molecules into distinct territories with different properties [30]. This lateral molecular homogeneity is usually caused by differences in effective interaction free energies between different molecules in biodiesel system. The free energies essentially involve entropic components which cannot be obtained by simple molecular mechanics energy calculations. They are obtained in principle from atomic level molecular dynamic simulation [30 - 32]. Consequently, the tie lines overlap because other components (likely the free fatty acids, or soap) were simultaneously distributed between the extract and raffinate phases which resulted in the tie line slope behavior.



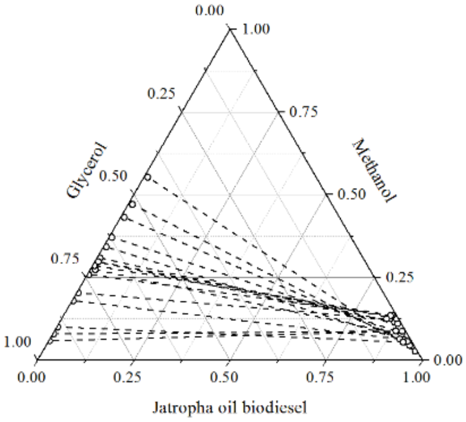
(**A**)



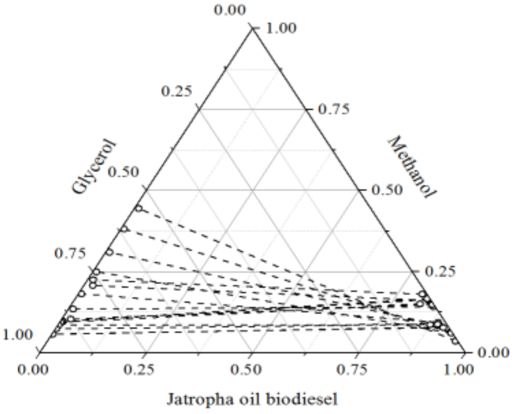
(**B**)



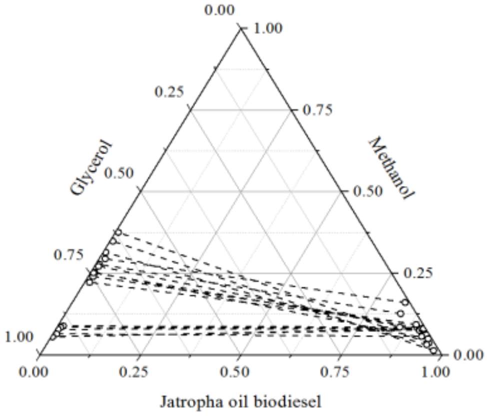
(**C**)



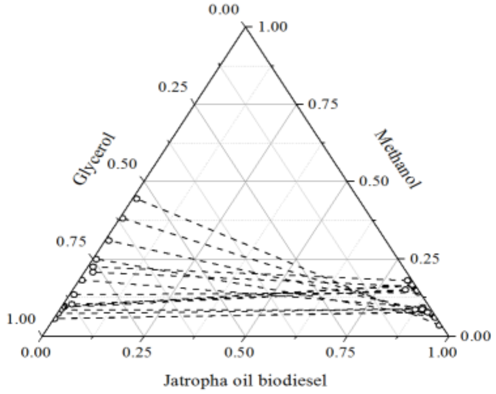
(**D**)



(**E**)



(**F**)



(**G**)

Figure 1: Tie-line extract and raffinate phase composition of jatropha oil biodiesel mixture at (**A**) 20 °C (**B**) 25 °C (**C**) 35 °C (**D**) 40 °C (**E**) 45 °C (**F**) 50 °C (**G**) 55 °C

4.0. Conclusion

In this study, extract and raffinate components of jatropha oil biodiesel ternary system was investigated at temperatures based on components composition behavior. Experimental components composition involving tie-lines compositions were measured for jatropha oil biodiesel homogeneous system at nine different temperatures (20 – 60) °C using modified cloud-point titration procedures. The results obtained showed how the individual components of the jatropha oil biodiesel system distribute in the homogeneous solution at the different temperatures. It also revealed that the jatropha oil biodiesel component of the mixture was less soluble in the highly polar glycerol/methanol phase and so displayed large two-phase region which was supported by similar studies. However, there were high levels of immiscibility between the extract and raffinate phases at the investigated temperatures. The results further revealed overlapping nature of tie lines which was connected to the presence of excess methanol in the feed composition which caused the loss of jatropha oil biodiesel in the raffinate phase and further high concentration of glycerol in the extract phase. The results emphasized the fact that the tie lines overlap because other components were simultaneously distributed between the extract and raffinate phases which resulted in the observed behavior.

References

[1] Bazooyar, B.; F. Shaahmadi; M.A. Anbaz and A. Jomekian. (2021). Intelligent Modeling and Analysis of Biodiesel/Alcohol/Glycerol Liquid-Liquid Equilibria, Journal of Molecular Liquids, 322, 114972.

[2] Adama, K. K.; E.O Aluyor and T.O.K. Audu. (2021). Component Distribution Associated with Phase Separation and Purification of Tropical Almond Biodiesel at Different Temperatures. Renewable Energy, 165, 67 – 75.

[3] Jabeen, M.; M. Munir; M.M. Abbas; M. Ahmad; A. Waseem; M. Saeed; M.A. Kalam; M. Zafar; S. Sultana; A. Mohammed and B. Chaudhry. (2022). Sustainable Production of Biodiesel from Novel and Non-edible Ailanthus altissima (Mill.) Seed Oil from Green and Recyclable Potassium Hydroxide Activated Ailanthus Cake and Cadmium Sulfide Catalyst. Sustainability, 14, 10962.

[4] Kim, D.S.; M. Hanifzadeh and A. Kumar. (2018). Trend of Biodiesel Feedstock and its Impact on Biodiesel Emission Characteristics. Environmental Progress and Sustainable Energy, 37, 7 – 19.

[5] Kamel, D.A.; H.A. Farag; N.K. Amin; A.A. Zatont and R.M. Ali. (2018). Smart Utilization of Jatropha (Jatropha curcas Linnaeus) Seeds for Biodiesel Production: Optimization and Mechanism. Industrial Crops Production 111, 407 – 413.

[6] Guldhe, A.; B. Singh; T. Mutanda; K. Perrnaul and F. Bux. (2015). Advances in Synthesis of Biodiesel via Enzyme Catalysis: Novel and Sustainable Approaches. Renewable and Sustainable Energy Reviews 41, 1447-1464.

[7] Inam, S.; M.A. Hanif; U. Rashid; F. Nadeem; F.A. Alharthi and E.A. Kazerooni. (2022). The Novel Approach of Catalytic Interesterification, Hydrolysis and Transesterification of Pongamia pinnata Oil, Catalysts, 12 896.

[8] Uddin, W. (2022). Mobile and Area Sources of Greenhouse Gases and Abatement Strategies BT – Handbook of Climate Change Mitigation and Adaptation; Springer International Publishing: Cham, Switzerland, 743 – 807.

[9] Afolalu, S.A.; O.O. Yusuf; A.A. Abioye; M.E. Emetere; S.O. Ongbali and O.D. Samuel. (2021). Biofuels: A Sustainable Renewable Source of Energy – A Review. IOP Conference Series, Earth and Environmental Science, 665, 12040.

[10] Otieno, S.; F. Kengara; C. Kowenje and R. Mokaya. (2022). Optimization of Biodiesel Synthesis from Jatropha curcas Oil using Kaolin-derived Zeolite Na-X as a Catalyst, The Royal Society of Chemistry (RSC) Advances, 12, 22792 – 22805.

[11] Santos, T.; J.F. Gomes and J. Puna. (2018). Liquid-Liquid equilibrium for ternary systems containing biodiesel, methanol and water, Journal of Environmental Chemical Engineering, 6, 984 – 990.

[12] Adepoju, T.F. (2020). Optimization Processes of Biodiesel Production from Pig and Neem (Azadirachta indica a. Juss) Seeds Blend Oil using Alternative Catalysts from Waste Biomass. Industrial Crops Production, 149, 11.

[13] Silva, J.R.F.; M.A. Mazutti; F.A.P. Voll; L. Cardozo-Filho; M.L. Corazza; M. Lansa; W.L. Priano and J.V. Oliveira. (2013). Thermophysical Properties of Biodiesel and Related Systems: (Liquid + Liquid) Equilibrium Data for Jatropha curcas Biodiesel, Journal of Chemical Thermodynamics. 58, 467-475.

[14] Zhou, H.; H. Lu and B. Liang. (2006). Solubility of Multi-Component Systems in the Biodiesel Production by Transesterification of Jatropha Curcas L. Oil with Methanol. Journal of Chemical and Engineering Data, 51: 6, 1130 - 1135.

[15] Campo, C.E.; M. Cabarcas; A. Gonzalez-Delgado; T.E. Sanchez and D.K. Ojeda. (2017). Liquid-Liquid Equilibrium for Ternary Mixtures Methanol-Glycerol-Biodiesel from Jatropha Curcas, International Journal of ChemTech Research, 10(4), 245-254.

[16] Munir, M.; M. Saeed; M. Ahmad; A. Waseen; S. Sultana; M. Zafar and G.R. Srinivasan. (2021). Optimization of Novel Lepidium perfoliatum Linn. Biodiesel using Zirconium-modified montmorillonite clay catalyst. Energy Sources Part A: Recovery, Utilization and Environmental Effects 44, 6632-6647.

[17] Rozina, A.S.; M. Ahmad; M. Zafar and N. Ali. (2017). Prospects and Potentials of Fatty Acid Methyl Esters of Some Non-edible Seed Oils for use as Biodiesel. Renewable and Sustainable Energy Reviews. 74, 687-702.

[18] Adama, K. K. (2021). Physicochemical Composition and Functional Properties of Jatropha Seed Oil and Jatropha Biodiesel: An Agro-Renewable Product, Nigerian Institution of Professional Engineers and Scientists (NIPES) Journal of Science and Technology Research, 3(1), 153 – 161.

[19] Franca, B.B.; F.M. Pinto; F.L.P. Pessoa and A.M.C. Uller. (2009). Liquid-Liquid Equilibria for Castor Oil Biodiesel+Glycerol+Alcohol. Journal of Chemical and Engineering Data 54: 2359-2364.

[20] Ardila, Y.C.; A.B. Machado; G.M.F. Pinto; R.M. Filho and R.W. Maciel. (2013). Liquid-Liquid Equilibrium in Ternary Systems Present in Biodiesel Purification from Soybean Oil and Castor Oil at 298.2K and 333.2K. Journal of Chemical and Engineering Data, 58, 605-610.

[21] Rostami, M.; S. Raeissi; M. Mahmodi and M. Nowroozi. (2013). Liquid-Liquid Equilibria in Biodiesel Production. Journal of American Oil Chemical Society (AOCS), 90: 147-154, Springer.

[22] Adama, K.K.; S.K. Otoikhian; O.E. Aluyor and T.O.K Audu. (2018). Temperature Effect on the Refinement of Jatropha Oil Biodiesel Upper Rich Phase. Journal of Bioprocessing & Biotechnology, 8:5.

[23] Hajek, M.; F. Skopal; A. Vavra and J. Kocik. (2016). Transesterification of Rapeseed Oil by Butanol and Separation of Butyl Ester. Journal of Cleaner Production, 7, 254-263.

[24] Coniglio, L.; J.A.P. Coutinho; J.Y. Clavier; F. Jolibert; J. Jose; I. Mokbel; D. Pillot; M.N. Pons; M. Sergent and V. Tschamber. (2014). Review: Biodiesel via Supercritical Ethanolysis within a Global Analysis Feedstocks-Conversion-Engine for Sustainable Fuel Alternative. Progress in Energy and Combustion Science, 43, 1-35.

[25] Banga, S. and Varshney, P.K. (2016). Effects of Impurities on Performance of Biodiesel: A Review. Journal of Scientific and Industrial Research 69, 575-579.

[26] Seader, J.D. and Henley, E.J. (2006). Separation Process Principles, 2nd Edition, John Wiley and Sons Publishers, New York.

[27] Machado, A.B.; Y.C. Ardila; L. Hadlich de Oliveira; M. Aznar and R.W. Maciel. (2012). Liquid-Liquid Equilibria in Ternary and Quaternary Systems Present in Biodiesel Production from Soybean Oil at 298.2K and 333.2K, Journal of Chemical and Engineering Data, 57(5), 1417-1422.

[28] Komer, K.; J. Tichy and F. Skopal. (1995). Ternary Phase Diagram, Biodiesel Fuel-Methanol-Glycerol, Journal fur Praktische Chemie. 337, 328.

[29] Stephen, N.C. and James, T.H. (2011). Reverse-Phase Ternary Phase Diagram, Tie lines and Plait Point for Commercial Biodiesel-Glycerol-Methanol, Industrial & Engineering Chemistry Research 50, 1012-1016.

[30] Nagle, J.F. and Scott, H.L. (1978). Biomembrane Phase Transitions. Physics Today, 31, 2, 38-47.

[31] Feigenson, G.W. (2006). Phase Behaviour of Lipid Mixtures. Nature Chemical Biology 2, 11, 560-563.

[32] Frazier, M.L.; J.R. Wright; A. Pokorny and P.F.F. Almeida. (2007). Investigation of Domain Formation in Sphingomyelin/Cholesterol/Popc Mixture by Fluorescence Resonance Energy Transfer and Morte Carlos Simulations, Biophysical Journal. 92, 7, 2422-2433.