

Physiochemical and Phase behaviour study of *Jatropha curcus* oil - ethanol microemulsion fuels using sorbitane fatty esters

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Abstract: Vegetable oil reverse micelle microemulsions have been an alternative method of biodiesel production to eliminate and avoid transesterification as well as unpurified glycerol. Sorbitane fatty ester surfactants due to their high solubilization capacity forms microemulsions with oils and thus span based reverse micelle microemulsion systems have been studied. *Jatropha* oil-ethanol microemulsions have been prepared using span 80 and 85 surfactants and optimized as biofuel, their phase behavior with physicochemical parameters: density, viscosity and surface tension were analyzed for formulation. The surface tension has been an important physicochemical parameter in addition to kinematic viscosity elucidating *Jatropha* oil-ethanol microemulsion with span 80 than with span 85, as a better biofuel. Comparatively, a lower amount of span 80 than span 85 was utilized for microemulsion formulations and resulted viscosities were in close agreement with ASTM biodiesel standards. The microemulsification approach has been found a sustainable method for producing biofuels without chemical reactions and their fuel properties have been adjusted through variable formulations.

Keywords: Microemulsion, Phase Behavior, Biofuel, Kinematic Viscosity, Surface Tension

1. Introduction

Presently, with the increasing impact of environmental pollution due to increase in exhaust emissions and depletion of petroleum reserves, there is a burning need to develop alternative energy resources, such as biofuel which is greener and environment friendly. On account of several advantages such as renewable and non-toxic nature, vegetable oils are being considered as an alternative fuel source. Therefore, vegetable oils have emerged as an alternative renewable source of green fuels globally [1-3]. Vegetable oils such as soybean, peanut, sunflower, rape, coconut, karanj, neem, cotton, mustard, *jatropha*, linseed and castor have shown a performance quite comparable to diesel fuels [4-7].

Vegetable oil as engine fuel is not a new concept because the inventor of diesel engine, Rudolph Diesel ran his diesel engine on peanut oil as the fuel [8]. Vegetable oils contain triglycerides as a primary component producing high

viscosity. Due to the high viscosity, long term use of neat vegetable oils affects the engine durability resulting in injector coking and ring sticking [9, 10]. Additionally, the vegetable oils have higher cloud and pour points considered as unfavorable parameters in cold weather conditions [11]. So several methodologies have been applied to overcome these difficulties for preparing biofuel such as dilution method where vegetable oils in small portions are blended with diesel; microemulsion (ME) method where oils are mixed with alcohols using surfactants; pyrolysis by simple cracking of vegetable oils either by heating directly or with catalysts, a less energy efficient method and lastly transesterification comprising a reversible chemical reaction between oil and short chain alcohols producing biodiesel and glycerol with catalysts [12-15]. The biofuel obtained through these methods are biodegradable and environmentally friendly however their high cost, poor cold flow properties and increasing nitrogen oxides (NO_x) in exhaust emissions have been few of the disadvantages [16-18].

Transesterification is widely applied for reducing viscosity of oils and obtained biodiesel has kinematic viscosity, specific gravity, cetane number and gross heat of combustion as compared to No. 2 diesel. In transesterification reaction, the triglycerides react with alcohols in the presence of catalysts producing glycerol as a byproduct with biodiesel. Purification or conversion of glycerol into a value added product is an expensive process and its disposal causes environmental pollution [19, 20]. Therefore, biodiesel preparation by microemulsification of oils could be applied to prevent formation of glycerol. The biofuels obtained from this method are transparent and thermodynamically stable, reported as Winsor Type II microemulsions where the polar ethanol phase is dispersed in non-polar phase due to reverse micelle formation [21-24]. The fact that *Jatropha curcus* oil (JCO) cannot be used for dietary purposes without detoxification that makes it a very attractive feedstock for biodiesel. Earlier studies have shown that JCO based biodiesel has similar properties to that of petroleum-based diesel [25]. In view of all these facts, JCO and ethanol were chosen as renewable energy sources for ME preparation with spans where, JCO is an extract of *jatropha curcus* plant and ethanol is the fermentation product of sugarcane and corn while spans 80 (sorbitane monooleate) and span 85 (sorbitane trioleate) are bio-compatible surfactants. Mixing oil with alcohols is one of the approaches to achieve standard biodiesel viscosity alongside other physicochemical properties (PCP): density and surface tension are essential indicators for proper analysis of biodiesel. For instance, the cetane number and

gross heating values are directly related to density and affect fuel efficiency [26-28]. Similarly, viscosity is an important parameter for development of fuels that governs the engine operation [29-30]. Surface tension is one of the vital PCP that directly affects fuel atomization due to cohesive forces and hence the biodiesel with higher surface tension values hamper the correct fuel atomization on an engine combustion chamber due to difficulty in small fuel droplet formation [31]. Despite such an important role in fuel combustion, there is only a limited literature reported on surface tension. Therefore, our work reports experimental density, viscosity and surface tension of span based JCO microemulsions (MEs) in a wide temperature range of $T = (298.15, 303.15, 308.15 \text{ and } 313.15) \text{ K}$ and ambient pressure to bring them to a usable form.

2. Materials and Methods

2.1. Materials

Non-edible grade JCO was acquired from Agarwal oils (Udaipur, Rajasthan, India) and its chemical composition was analyzed by gas chromatography (GC) with flame ionization detector, given in Table 1. JCO has shown iodine and acid values as 102g/100g and 2.8 mg KOH/g, respectively. Ethanol ($\geq 99.8\%$), span 80 (HLB number 4.3) and span 85 (HLB number 1.8) were used as received from Sigma-Aldrich, span molecular structures are depicted in Fig. 1.

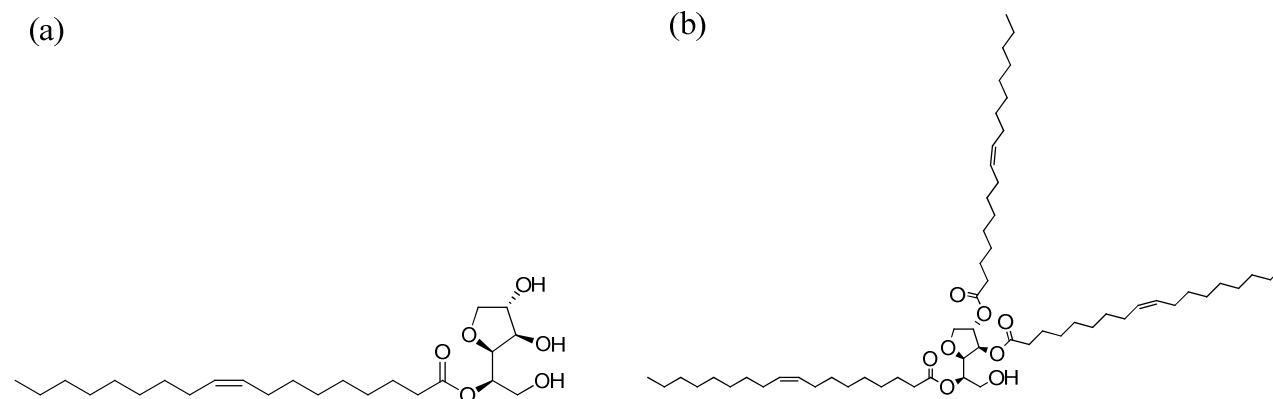


Figure 1. Structure of non-ionic surfactants: (a) span 80, (b) span 85.

Table 1. Fatty acid profile of JCO

Fatty acid	% Composition ^a
Palmitic (C16:0)	13.48
Stearic (C18:0)	7.25
Oleic (C18:1)	42.25
Linoleic (C18:2)	36.56
Arachidic (C20:0)	0.18
Behenic (C22:0)	0.13

^a analyzed by gas chromatography (GC) with flame ionization detector (FID)

2.2. Methods

2.2.1. Microemulsion Preparation

Binary mixtures of ethanol and JCO from 0 - 100 % (w/w) were prepared with an accuracy of $\pm 0.0001\text{g}$ using Mettler Toledo New Classic MS with continuous stirring at 500 RPM. Solutions up to 20 % were transparent while above which were cloudy. For 20 – 90 % JCO-ethanol binary mixtures, the variable amounts of span were added to formulate stable and transparent MEs. Solutions above 90% were clear without span addition. JCO-ethanol and JCO-ethanol-span mixtures were continuously stirred at 500

RPM and their thermodynamic stability was checked from 298.15 to 313.15 K using a thermal water bath.

2.2.2. Ternary Phase Diagram

For the study of phase behavior and stability of MEs, ternary phase diagram was used where the three vertices of the equilateral triangle represent the three components of ME [17]. The two vertices at the bottom depict weight percent of JCO at left side and ethanol at right, while the upper vertex represents span. The composition at each point in diagram demonstrates weight percent of three components A, B, C as under [30].

$$x\%A + y\%B + z\%C = 100\% \quad (1)$$

Miscibility curve is plotted between two liquid phases and single phase MEs of Oil-Ethanol-Span. The region above the curve is a single phase transparent and thermodynamically stable MEs with minimum surfactant. The two immiscible phases appear below the curve, and found to be similar as Winsor Type II [32, 33].

2.2.3. Density, Viscosity and Surface Tension Measurements

Density (ρ) of pure components and mixtures were measured using Anton Paar DSA 5000M densimeter whose temperature was controlled to ± 0.001 K built-in Peltier device. Sensitivity of the instrument corresponds to $\pm 5 \cdot 10^{-3}$ kg·m⁻³ precision. The densimeter was calibrated with Millipore water and reported densities are an average of three consecutive measurements. Viscosity and surface tension measurements were done with Borosil Mansingh Survismeter (Cal.no. 06070582/1.01/ C-0395, NPL, India) by flow time and pendant drop methods controlled by Lauda Alpha KA 8 thermostat with ± 0.01 K control [34-37]. The survismeter was calibrated with ethanol and water, and compared with literature values [38-40]. Viscosity and surface tension are an average of three consecutive measurements with ± 0.0001 mm²·s⁻¹ and ± 0.03 mN·m⁻¹ uncertainties respectively.

3. Results and Discussion

3.1. Phase Behavior

Figs. 2 and 3 depict the miscibility curves of prepared MEs, and show a critical role of linear and branched structures of span 80 and 85 respectively with JCO-Ethanol at 298.15 K. The results distinguished the surfactants attributing maximum solubilisation of ethanol in the oil phase. For example, 51, 45, 27, 14 and 2 % less amount of span 85 was required than span 80 for 25, 30, 35, 40 and 45 % ethanol in JCO, respectively whereas for 50, 55, 60, 65, 70, 75, 80, 85 and 90% ethanol in JCO; 7, 19, 19, 28, 31, 34, 39, 52 and 69% less span 80 was required than span 85 (Figs. 2 and 3). The span 80 contains three OH groups with a straight oleate chain and span 85 contains one OH group with branched trioleate chain (Fig.1), have increased

the oil and ethanol solubility respectively. Thus, it has proven that the branching and number of OH groups affected the hydrophobicity and hydrophilicity of MEs system respectively. It is also evident that the oils containing triglycerides are highly hydrophobic with longer and bulkier alkyl chains of triglycerides [41, 42], and thus, the span 80 is identified as the potential surfactant to achieve a single phase ME with less amount.

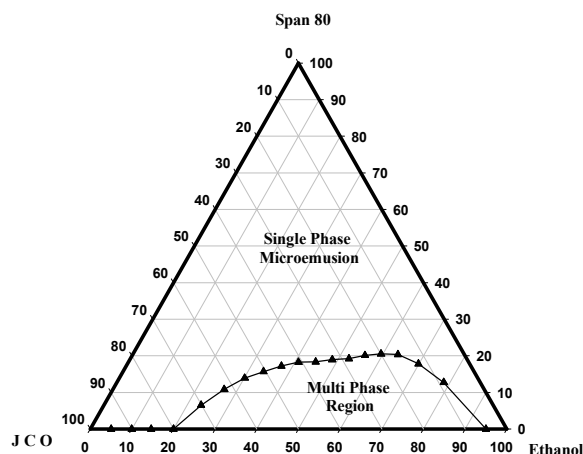


Figure 2. Ternary phase diagram of JCO-ethanol-span 80 system at room temperature

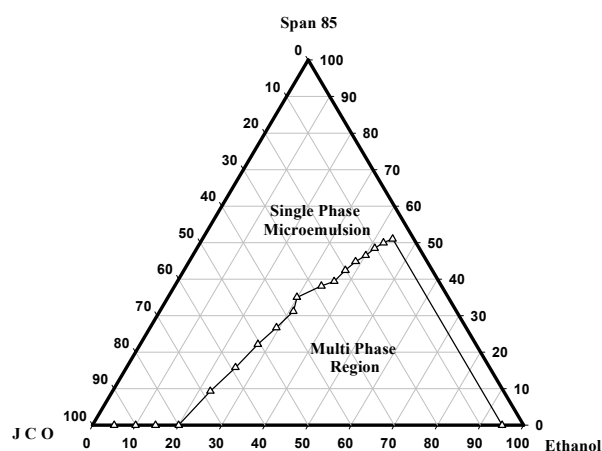


Figure 3. Ternary phase diagram of JCO-ethanol-span 85 system at room temperature.

3.2. Physicochemical Study

3.2.1. Density, Kinematic Viscosity and Surface Tension

The effect of increasing ethanol concentration on density of the miscible systems from $T = (298.15 \text{ to } 313.15) \text{ K}$, illustrated in Figs. 4 and 5. The density has decreased with increase in ethanol concentration where with span 80, between 25-90 % ethanol, the rate of decrease in density is 0.52% while with span 85 is 0.11%. From 90% onwards without span, the density decreased sharply. It seems that on addition of ethanol up to 20%, the strongest hydrophobic-hydrophobic interactions (H_bH_bI) within oil molecules have weakened due to the development of

hydrophilic-hydrophilic interactions (HHI) between oil and ethanol and caused a decrease in density. The higher rate of decrease in density with span 80 might be due to a dominance of weaker HHI between ethanol-span over stronger H_bH_bI between oil-oil and weaker H_bH_bI between span-oil. These interactions have developed due to the presence of one oleate chain in span 80 and 3:1 ratio of OH in span 80 and ethanol. With span 85, the lower rate of decrease in density might be due to increase in H_bH_bI which increased the hydrophobicity induced by trioleate chain of span 85, as well as decrease in HHI because of 1:1 ratio of OH in span 85 and ethanol. From 80 % onwards, hydrophobicity decreased due to decrease in oil and span concentrations which developed stronger HHI due to increase in ethanol concentration that caused a sharp decrease in density. MEs with span 85 have higher densities than span 80 because span 85 with three oleate branching developed stronger H_bH_bI between oil and span 85.

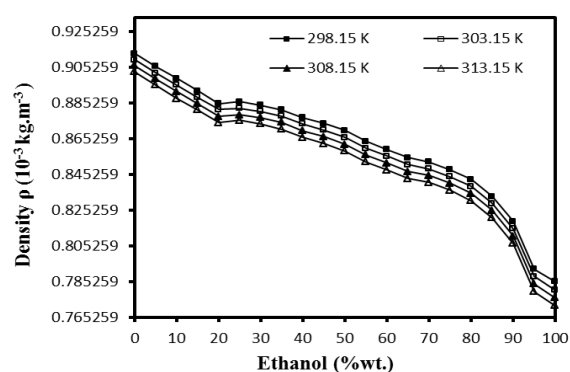


Figure 4. Densities of JCO-ethanol microemulsions with span 80

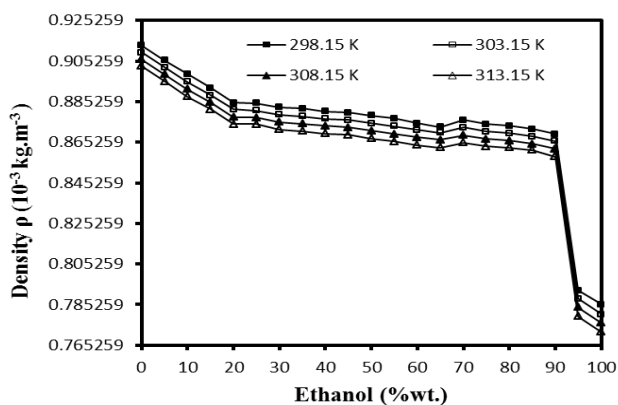


Figure 5. Densities of JCO-ethanol microemulsions with span 85

Kinematic viscosities are illustrated in Figs. 6 and 7, which decreased with increase in ethanol concentration. Initially, up to 20% ethanol addition, the kinematic viscosity has decreased sharply within $5\text{--}12\text{ mm}^2\cdot\text{s}^{-1}$ and after 20 % onwards with span 80, the same decreased steadily within $0.7\text{--}2.3\text{ mm}^2\cdot\text{s}^{-1}$. Initially at zero ethanol concentration, only strong H_bH_bI prevailed within the oil system. When the concentration of ethanol was increased, the stronger H_bH_bI were weakened with development of weaker H_bH_bI between

a larger hydrophobic group of oil and smaller ethyl chain of ethanol. Above 20% ethanol, a dominance of HHI between ethanol-ethanol resulted in phase separation of oil and ethanol. To mutually solubilize the two phases, span was added carrying with hydrophobic domain and polar OH group. The span mutually solubilized the components, and a stable ME were obtained where a very less impact of ethanol concentration on kinematic viscosity was observed due to the counterbalance between H_bH_bI and HHI induced by span 80 (Figs. 6). When span 85 was added, the counterbalancing effect between the H_bH_bI and HHI was less than with span 80 due to presence of trioleate chain which increased the H_bH_bI , and as a result the kinematic viscosities are on the higher end as shown in Fig. 7. The kinematic viscosity of MEs with span 80 at 313.15 K with ethanol-oil ratios 55:45, 60:40, 65:35, 70:30, 75:25, 80:20, 85:15 and 90:10 have found similar to biodiesel, B100 ($1.9\text{--}6\text{ mm}^2\cdot\text{s}^{-1}$), and in closest agreement with the standard of ASTM D6751-02 [43]. Thus, with phase behavior, density and kinematic viscosity studies have proven that the span 80 based MEs have a most favorable path for making biofuel.

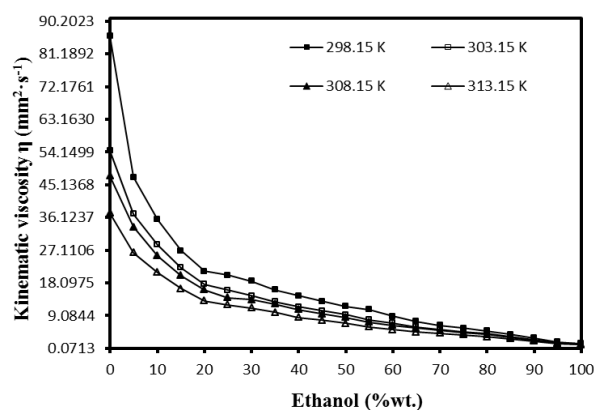


Figure 6. Viscosities of JCO-ethanol microemulsions with span 80

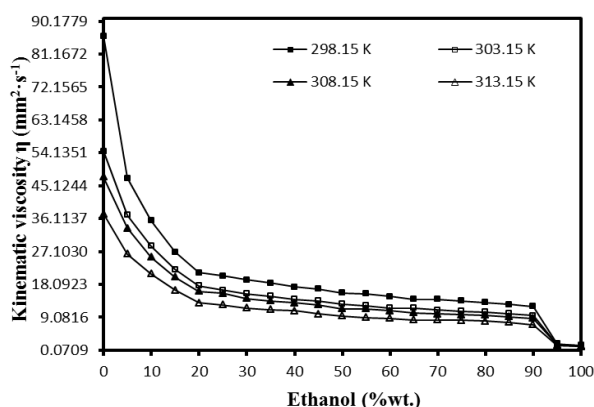


Figure 7. Viscosities of JCO-ethanol microemulsions with span 85

Surface tensions of MEs have decreased with an increase in ethanol concentration as illustrated in Figs. 8 and 9. It seems that initially up to 20% ethanol concentration, the surface tension decreased sharply, and 20 % onwards with span 80, increased at the rate of $0.04\text{ mN}\cdot\text{m}^{-1}$ with per %

increase in ethanol concentration at 293.15 K. The sharp decrease in surface tension might be due to a weakening of strong cohesive forces in the form of H_bH_bI existing in oil and development of weaker H_bH_bI between oil-ethanol. The systematic representation of H_bH_bI and HHI between oil, ethanol and surfactant is illustrated through JCO ethanol interaction model, Fig.10. After the addition of span 80 and with increasing ethanol concentration, development of stronger HHI between span and ethanol caused a constant increase in surface tension. With span 80, after 80% ethanol, the surface tension has decreased sharply due to diminishing stronger H_bH_bI of oil-oil and developing HHI of ethanol-ethanol. With span 85, after 20 % onwards, at 298.15 K, the surface tension has increased with the rate of 0.004 mN.m^{-1} while at 303.15, 308.15 and 313.15 K, decreased with the rate of 0.007, 0.003 and 0.008 respectively. This reverse trend in surface tension with temperature indicates the thermal behavior of MEs. The rate of increase or decrease in surface tension between 25-80% with span 85 is lower than span 80 because increasing HHI of span 85- ethanol and ethanol-ethanol was compensated by H_bH_bI of oil-span 85 due to the trioleate chain. The surface tension with span 80 was more than with span 85 because span 80 with three OH groups developed stronger HHI with ethanol. Thus, the three OH groups of span 80 increased the surface forces and produced higher surface tension than span 85.

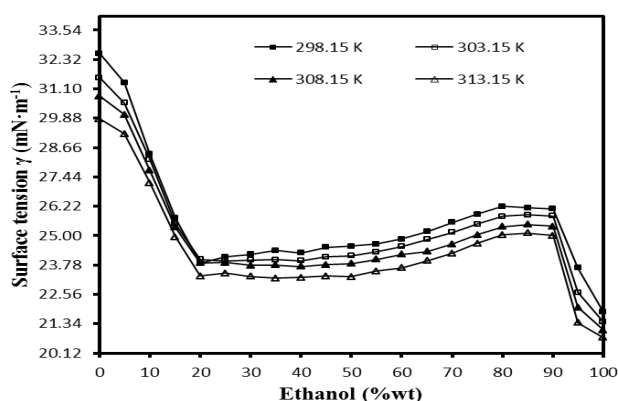


Figure 8. Surface tensions of JCO-ethanol microemulsions with span 80

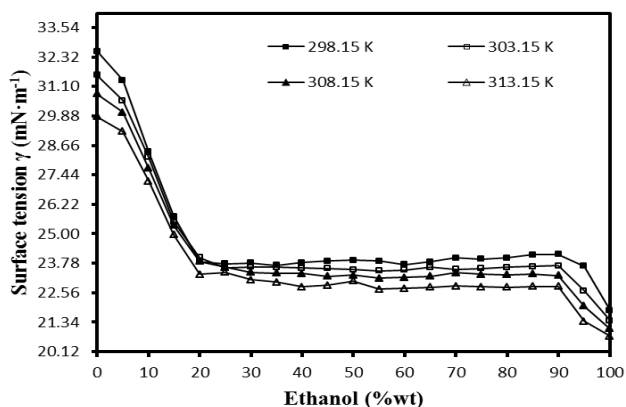


Figure 9. Surface tensions of JCO-ethanol microemulsions with span 85

4. Conclusions

This study has elucidated the physicochemical characterization of span based Jatropha oil-ethanol MEs to be used as biofuel. Unlike sulfate-based extended surfactants, ME fuel of spans are an environmentally desirable fuel; since there is no sulfur content in span preventing sulfur oxide (SO_x) emissions. Also the MEs were stable without any phase separation up to 313.15 K. From the phase behavior, density, viscosity and surface tension studies, the optimum system found is the system of span 80 based JCO-ethanol MEs. This fuel system is stable in a temperature range of 298.15-313.15 K and the optimum system for this study is span 80 based JCO-ethanol MEs. The 55:45,60:40, 65:35, 70:30, 75:25, 80:20, 85:15 and 90:10 ratios of ethanol : oil with span 80 have kinematic viscosity meeting the ASTM standard of B100 biodiesel ($1.9-6 \text{ mm}^2\text{s}^{-1}$) at 313.15 K. These newly developed JCO-ethanol-span 80 MEs could be used as renewable and eco-friendly fuels with high calorific values on designing surfactant based biofuel.

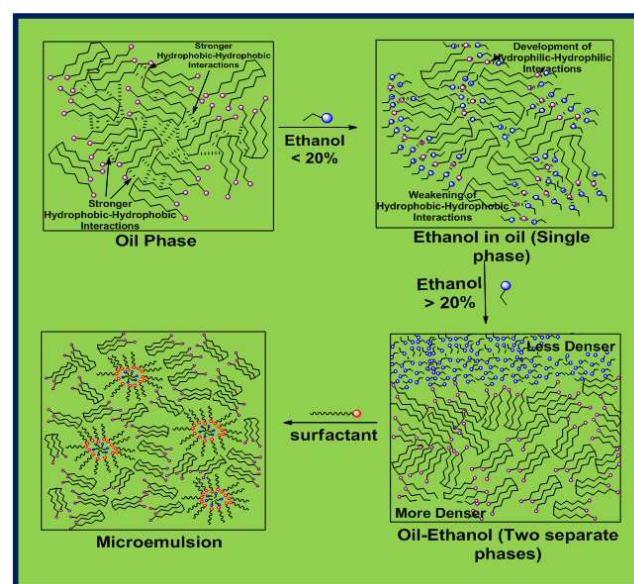


Figure 10. JCO Ethanol interaction model

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