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To cite this article: Ibrahim Kayali, Khawla Qamhieh, Monzer Fanun, Salam Wadaah, Jyothi Chembolli Kunhatta & Khalid Kanan (2017) Transport properties of alternative fuel microemulsions based on sugar surfactant, Journal of Dispersion Science and Technology, 38:7, 917-922, DOI: [10.1080/01932691.2016.1215924](https://doi.org/10.1080/01932691.2016.1215924)

To link to this article: <https://doi.org/10.1080/01932691.2016.1215924>



Accepted author version posted online: 29 Jul 2016.  
Published online: 29 Jul 2017.



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## Transport properties of alternative fuel microemulsions based on sugar surfactant

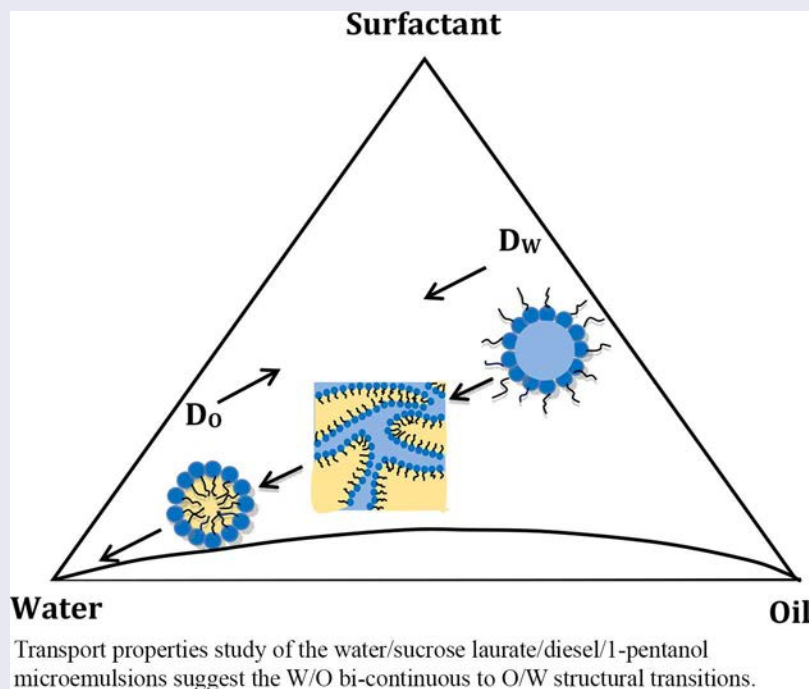
Ibrahim Kayali<sup>a</sup>, Khawla Qamhieh<sup>a</sup>, Monzer Fanun<sup>a</sup>, Salam Wadaah<sup>a</sup>, Jyothi Chembolli Kunhatta<sup>b</sup>, and Khalid Kanan<sup>a</sup>

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### ABSTRACT

Electrical conductivity of fuel microemulsion composed of diesel, pentanol, water, and sucrose laurate as surfactant was investigated over a wide range of water contents varying from 0 to 90 wt% and temperature varying from 10°C to 50°C. Conductivity measurements were performed on samples, the composition of which lie along the one-phase channel using a conductivity meter. Activation energy of conduction flow was evaluated. The hydrodynamic radius as a function of temperature in the aqueous phase-rich region (90 wt%) was measured using the dynamic light scattering (DLS) method. The microstructure of the microemulsion was further investigated by NMR diffusometry by which the self-diffusion coefficients for water were determined at 25°C. Electrical conductivity increases with water content up to 40 wt% and the percolation threshold was observed, and then stabilizes between 40 and 80 wt% then decreases. Percolation threshold temperature at constant composition was monitored as 36°C for water contents below 80 wt% and as 34°C for water contents above that. As predicted by the conductivity measurements, the determined self-diffusion coefficients of water confirmed the structural transition from discrete W/O droplets to bi-continuous phase and finally to O/W droplet microemulsion.

### GRAPHICAL ABSTRACT



### ARTICLE HISTORY

Received 29 June 2016  
Accepted 19 July 2016



### KEYWORDS

Fuel microemulsion;  
microstructure; percolation  
temperature; sugar  
surfactant

## 1. Introduction

With the increasing awareness of the environmental hazardous nature of emissions produced by the combustion of fossil fuel and their effect on human health, the potential application of alternative fuel has been growing steadily in the last two decades. Furthermore, concerns over the dwindling fossil fuel

sources have raised interest in developing viable alternative fuel from renewable resources. Vegetable oils have been regarded as major candidates for alternative fuel; however, due to their high viscosity they cannot be used directly without engine modifications. Trans-esterification process performed by reacting the oils with alcohol will convert them into biodiesel, which can

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substitute fossil fuel. Another way that can be used to reduce the high viscosity of vegetable oils and turn them to suitable biofuel is through the microemulsification process.<sup>[1–3]</sup>

Microemulsions are transparent, low-viscous, thermodynamically stable solutions formed spontaneously and typically contain both water and oil stabilized by surfactant and co-surfactant.<sup>[4]</sup> Microemulsions as diesel fuel have been tested in the past and proved to lower the levels of exhaust particulate matter and nitrogen oxide compounds.<sup>[5]</sup> Studies showed that incorporating water in the range of 5–15 wt% in microemulsion fuel reduced drastically the particulate matter emission from a direct-injection diesel engine.<sup>[6]</sup> The surfactants to be used in formulating microemulsion fuel should contain only carbon, hydrogen, and oxygen atoms. This requirement put the nonionic surfactants, including sugar surfactants, as prime candidates. In a previous work, water in diesel microemulsion was tested in a four-cylinder diesel engine. The obtained results showed a significant reduction in all types of emissions.<sup>[7]</sup>

Various studies showed that the addition of alcohol, such as methanol, ethanol, propanol, butanol, and pentanol, to fossil fuel can reduce particulate matter emissions. In addition, alcohol is considered to be an important member of the biofuel family and has been considered as a renewable bio-based resource.<sup>[8,9]</sup>

The measurement of electrical conductivity is a useful and convenient method to probe the structural transition in the microstructure from W/O to bi-continuous to O/W microemulsion. Microemulsion electrical conductivity increases sharply while titrating with water to a certain threshold volume fraction at constant temperature or after a threshold temperature at a constant composition. This sharp increase in conductivity is attributed to the percolative behavior of the existing nanostructures in the system. Two probable mechanisms are suggested for inducing percolation: one in which droplets in microemulsion come in close contact, allowing ions to hop from droplet to droplet, and the other when droplets coalesce, forming clusters that can permit the exchange of ions. The static percolation model has been proposed to describe the mechanism of percolation due to the appearance of bi-continuous microemulsion, while the dynamic percolation model is related to the rapid processes of fusion–fission among droplets.<sup>[10–14]</sup> NMR self-diffusion measurements can provide a direct and noninvasive technique for the investigation of the microemulsion structure. Confinement and obstruction of molecules have a major effect on molecular self-diffusion. Therefore, from the water and oil self-diffusion coefficients, one can decide whether a given microemulsion is of the bi-continuous, the W/O, or the O/W type. In the bi-continuous type, the water and oil self-diffusion coefficients will have values close to those of the neat liquids.<sup>[15]</sup>

Recently, we have investigated the role that sucrose laurate surfactant and different alcohols play in the formulation of microemulsions that contain water and diesel. Pseudoternary-phase diagrams were determined.<sup>[16]</sup> In the present contribution, the effect that the types of microstructure and different temperatures have on conductivity is studied for microemulsions formulated with sucrose laurate surfactant, diesel, water, and 1-pentanol. NMR self-diffusion measurements confirming the microstructure transition from W/O to bi-continuous to O/W microemulsion will be presented.

## 2. Experimental

### 2.1. Materials

Sucrose laurate (L1695) was obtained from Mitsubishi-Kasei Food Corp. (Mie, Japan). The purity of combined lauric acid equals 95%, the ester compositions are 80% monoester and 20% di-, tri-, and poly-ester, HLB equals 16. 1-Pentanol (purity 99%) was purchased from Sigma-Aldrich, Sweden. European ultralow sulfur diesel was obtained from a local gasoline station in Lund, Sweden. All chemicals were used as supplied without any further purification. Millipore water was used in all formulations.

### 2.2. Methods

#### 2.2.1. Pseudoternary, phase behavior

The phase diagram for the system of water, L1695, and diesel: 1-pentanol (1:1) was determined and described earlier.<sup>[16]</sup>

#### 2.2.2. Electrical conductivity

Conductivity measurements were performed at temperatures of  $\pm 0.3^\circ\text{C}$  on samples, the compositions of which lie along the one-phase channel, using a conductivity meter; the conductivity cell used is Tetra Con@325, the electrode material is graphite, and the cell constant is  $0.475\text{ cm}^{-1} \pm 1.5\%$ . The temperature range is from  $14^\circ\text{C}$  to  $50^\circ\text{C}$ . In the case of nonionic microemulsions, a small amount of an aqueous electrolyte must be added for electrical conduction.<sup>[17]</sup> The electrode was dipped in the microemulsion sample until equilibrium was reached and the reading becomes stable at 1 min. Reproducibility was checked for certain samples and no significant differences were observed. The constant of the conductivity cell was calibrated using standard KCl solutions at the first of every month during the characterization or when the conductivity meter needs that.

#### 2.2.3. Dynamic light scattering

Particle size measurements were performed using Zetasizer Nano S (ZEN 1600) for the measurements of size and molecular weight of dispersed particles and molecules in solution, by Malvern Instruments Ltd. (Worcestershire, United Kingdom). The equipment includes a 4 mW, 633 nm He-Ne laser. Size measurement ranges between 0.6 nm and 6  $\mu\text{m}$ , size measurement angle equals  $173^\circ$ , concentration range for size measurement was between 0.1 ppm (0.0001 vol%) and 40 wt%, molecular weight range was between  $10^3$  and  $10^7$  Da, and temperature measurement range between  $2^\circ\text{C}$  and  $90^\circ\text{C}$ . A 1.5 ml microemulsion sample was introduced in disposable polystyrene cuvettes and measured at temperatures ranging between  $20^\circ\text{C}$  and  $50^\circ\text{C}$  by steps of  $5^\circ\text{C}$ . The particle hydrodynamic radius is calculated from the translational diffusion coefficient ( $D$ ) using the Stokes–Einstein relationship:

$$R_h = \frac{k_B T}{6\pi\eta D} \quad [1]$$

where  $R_h$  is the hydrodynamic radius,  $k_B$  is Boltzmann's constant,  $T$  is the absolute temperature, and  $\eta$  is the solvent viscosity. The results are averages of three experiments.

### 2.2.4. NMR experiments

Self-diffusion coefficients were measured by the stimulated echo pulsed field gradient (PGSE-NMR) method using a Bruker AVANCE II 200 spectrometer with Bruker field gradient probe operating at a proton resonance frequency of 200 MHz. The temperature was kept at 25°C with an accuracy of  $\pm 0.1^\circ\text{C}$ . The spin-echo amplitude,  $I$ , of a given NMR resonance is described as follows:

$$I = I_0 \exp(-kD) \quad [2]$$

where  $I_0$  is the echo amplitude in the absence of field gradient pulses,  $D$  is the self-diffusion coefficient of the molecule, and  $k = \gamma^2 g^2 \delta^2 (\Delta - \delta/3)$ , where  $\gamma$  is the gyromagnetic ratio,  $g$  is the gradient strength,  $\delta$  is the gradient length, and  $\Delta$  is the time between the gradient pulses. Experiments were carried out at constant  $\Delta$ , 50, and 100 ms and at appropriate  $g$ . The self-diffusion coefficients of water were obtained from the spin-echo signal at around 4.8 ppm.

## 3. Results and discussion

### 3.1. Phase behavior

Figure 1 presents the phase behavior of the water/sucrose laurate (L1695)/diesel: pentanol 1:1 pseudo-ternary-phase diagram. The phase diagram indicates the presence of an isotropic, low-viscous, continuous microemulsion region denoted  $L_2$ , in addition to the presence of an anisotropic liquid crystalline region denoted LC. The remainder of the phase diagram is considered as a multiple-phase region. Detailed discussion of the phase behavior was presented in our recently published paper.<sup>[16]</sup>

### 3.2. Electrical conductivity

The variation in electrical conductivity of the microemulsion upon titrating with aqueous phase was determined along the water dilution line N50 as shown in Figure 1. The electrical conductivity  $\sigma$  ( $\mu\text{S}/\text{cm}$ ) as a function of water content at different temperatures is presented in Figure 2. Two distinct levels of electrical conductivity values at two different temperature ranges could be recognized. The first ranges from 26°C to 38°C and the other from 42°C to 50°C. Both ranges start with very low conductivity up to a certain volume fraction of water

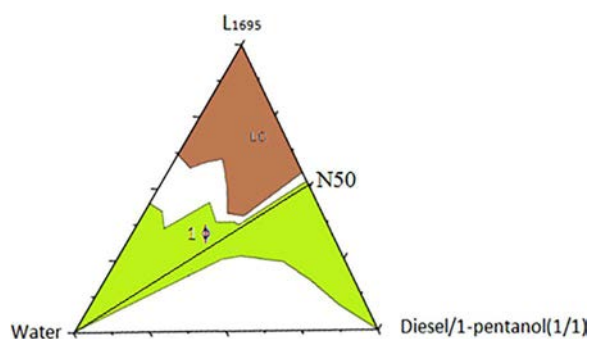


Figure 1. Pseudo-ternary-phase diagram of water/sucrose laurate/diesel oil/1-pentanol (1/1) at 25°C. LC indicates a liquid crystal region and  $1 \Phi$  indicates a microemulsion region (Ref. 16).

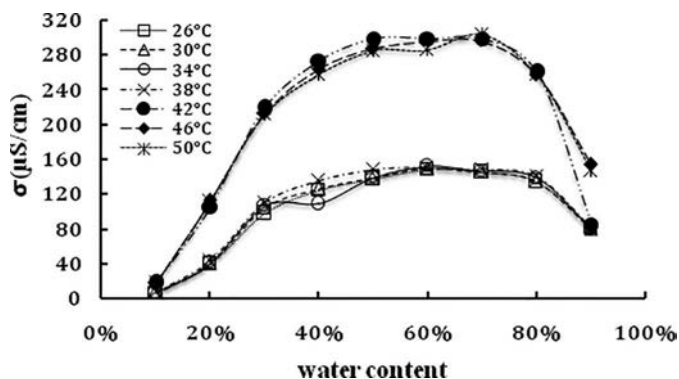


Figure 2. Electrical conductivity,  $\sigma$  ( $\mu\text{S}/\text{cm}$ ), as a function of water content at different temperatures.

( $\theta_c$ ). Below this volume fraction threshold, W/O droplets are isolated from each other and embedded in a nonconducting continuum oil phase, thus contributing very little to the conductance. With increasing water content, partial fusion of droplets and formation of a percolated network may occur and form clusters. The number of these nano clusters increases very rapidly above the percolation threshold, giving rise to the observed large increase of the conductivity values. With increasing water content, structural transition, from the W/O droplet-like structure to a bi-continuous or connected clusters, will occur, causing further increase and stabilization in conductivity. This bi-continuous region is quite large and exists within 0.30–0.75 aqueous phase volume fractions. For the bi-continuous microemulsions both the water and oil domains are considered to be continuous.<sup>[18]</sup> As a consequence of this structure, both oil and water would be free to diffuse over macroscopic distance with high self-diffusion coefficients  $\sim D_o$ , where  $D_o$  is the diffusion coefficient in the pure liquids (water and oil, respectively).<sup>[13]</sup> Finally a decrease in conductivity values is observed with increasing water content. This occurs in the O/W structure that is formed at high water content. It should be noted here that the nonionic surfactant used in this study is not 100% pure but contains some impurities including ions that would contribute to the electrical conductance. At high water content, the concentration of these ions decreases and thus the electrical conductivity.

As mentioned in the introduction, electrical conductivity undergoes a significant jump either when the volume fraction of the dispersed aqueous phase is increased to the percolation threshold at constant temperature or when the threshold temperature is reached at constant composition. Electrical conductivity data presented in Figure 2 clearly shows a sharp increase at temperatures above 38°C over the whole range of water content. Studies on percolation induced by increasing temperature have been reported before.<sup>[19]</sup> A dramatic increase in electrical conductivity with increasing temperature for microemulsions at constant composition (indicative of a percolation phenomenon) was found.<sup>[12]</sup> It was explained that an increase in temperature would increase the effective diameter of the droplets, the rate of coalescence, and the exchange of material, leading to cluster formation and percolation. In the conductivity versus temperature curve, the percolation temperature is defined as the point at which a sharp increase

in the slope occurs. Percolation threshold temperatures for mixed reverse micellar systems using sodium bis(2-ethylhexyl) sulfosuccinate (AOT) and nonionic surfactants were reported.<sup>[20]</sup> The values varied between 17°C and 39°C depending on the head, chain length of the tail, and the type of oil used. The excess volumes of sucrose monolaurate-based microemulsions have been found to decrease with the increase in water contents below 20 wt% and then stabilize for water contents between 20 and 60 wt%. For water contents above 60 wt% the excess volumes increase. This behavior indicates structural transitions from W/O to bi-continuous to O/W.<sup>[21,22]</sup> It was also observed that the values of excess volume for all water contents change at temperatures around 37°C. This means that if for a given water content, for example 30 wt%, the value of excess volume was lower than that of 40 wt% water content, at temperatures around 37°C the order inverts and the values of excess volume for 30 wt% became higher than those of 40 wt%. This behavior could be explained by expansion or networking of the systems at temperatures around 37°C (i.e., percolation). This behavior is related to certain dissociations or to the breakage of hydrogen bonds. The same behavior was also observed for isentropic compressibility.<sup>[22]</sup> These behaviors are similar to the behavior observed in this study.

The variations of  $\ln \sigma$  as a function of the reciprocal absolute temperatures at different water contents (10, 20, 30), (40, 50, 60), and (70, 80, 90) wt% are shown in Figure 3. For all water contents below 80 wt%, a break in the slope around the percolation temperature of 36°C occurs, while for the 90 wt% the break in the slope is around the percolation temperature of 34°C.

The activation energies of conductive flow ( $E_{cond}$ ) have been estimated using an Arrhenius form of the relation:

$$\sigma = A \exp(-E_{cond}/RT) \quad [3]$$

Or

$$\ln \sigma = \ln A - E_{cond}/RT \quad [4]$$

where  $\sigma$  is the conductance,  $A$  is a pre-exponential constant,  $R$  is the universal gas constant, and  $T$  is the absolute temperature. The values for  $E_{cond}$  can be obtained from the slope of  $\ln \sigma$  versus  $1/T$  plots.

The obtained conductivity data at different water contents (10–90 wt%) were treated separately at two temperature ranges. Figure 4a shows the plot between  $\ln \sigma$  and  $1/T$  for the temperature range (26–38°C), while Figure 4b shows the same plot but for the range of (42–50°C). From the slopes of the produced linear fit,  $E_{cond}$  values were calculated and are presented in Figures 5 and 6, respectively. Figure 5 shows the variation of  $E_{cond}$  for temperatures below and above the percolation temperature and Figure 6 presents the variation of electrical conductivity around the percolation temperatures for the whole range of water contents examined. In Figure 5, for temperatures below the percolation temperature ( $T_p$ ),  $E_{cond}$  value at 10 wt% water was equal to 25.8 kJ/mol, indicating a non-percolating state. At 20 wt% water, the value of  $E_{cond}$  decreases dramatically to 6.7 kJ/mol. This decrease is attributed to the increasing interaction between the W/O droplets. It must be emphasized here that the sugar surfactant used in this study

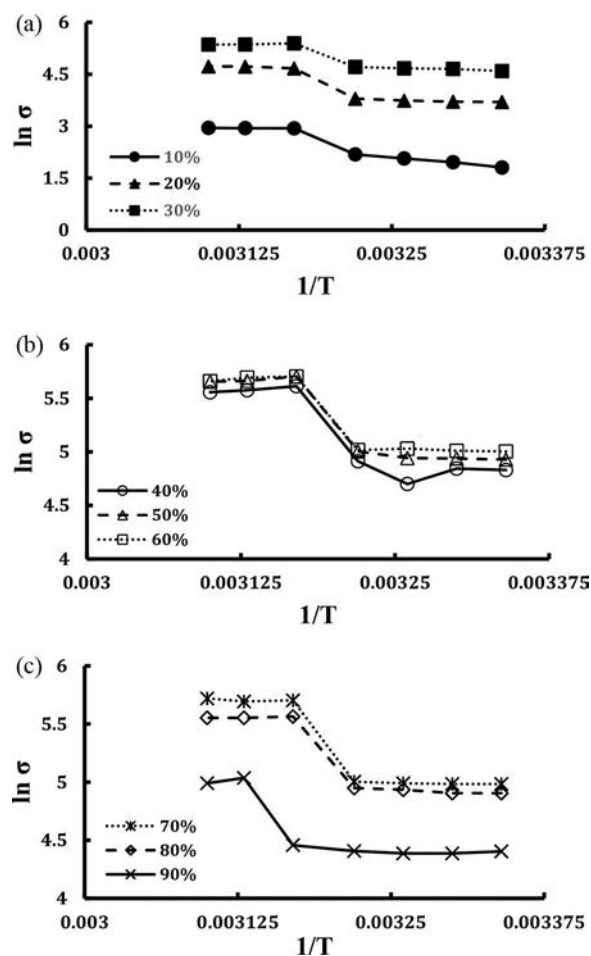


Figure 3.  $\ln \sigma$  versus  $1/T$  at water contents of 10, 20, 30 (a); 40, 50, 60 (b); and 70, 80, 90 (c).

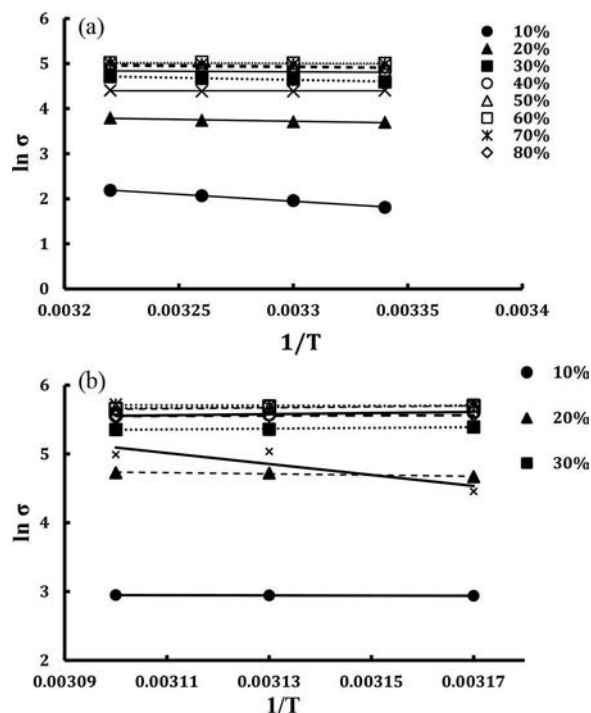


Figure 4.  $\ln \sigma$  versus  $1/T$  at different water contents for temperatures 26°C, 30°C, 34°C, 38°C (a) and 42°C, 46°C, 50°C (b).

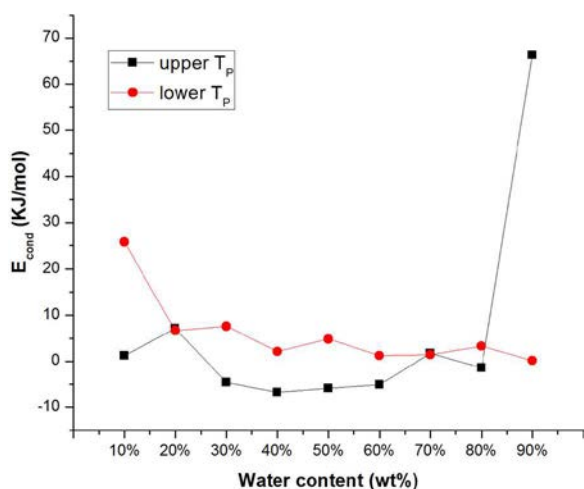


Figure 5.  $E$  (kJ/mol) versus water content at temperature ranges 26–38°C (circles) and 42–50°C (squares).

with its hydroxyl and methoxy groups strongly binds water. Increasing the amount of water added to the surfactant and oil mixture will induce the swelling mechanism. At 30 wt% water a slight increase in  $E_{cond}$  value occurs (7.6 kJ/mol), which could be attributed to the reorganization of the clusters. Between 40 and 80 wt% water, there is a fairly slow decrease in the  $E_{cond}$  values, indicating that the system has been transformed into bi-continuous phase where the interfacial area remains almost the same and the rate of transport of ions decreases slowly. The sharp decrease in  $E_{cond}$  value for water content of 80–90 wt% indicates the transformation of the structure from the bi-continuous phase to the O/W microemulsion. A totally different behavior is observed when the system is treated at temperatures above the percolation threshold temperature ( $T_p$ ). At 10 wt% water, the  $E_{cond}$  value was as low as 1.2 kJ/mol, indicating the presence of a percolating state. The discrete and noninteracting droplet state that was present at temperatures below  $T_p$  no longer exists. Both charge hopping and coalescence percolation mechanisms can sufficiently

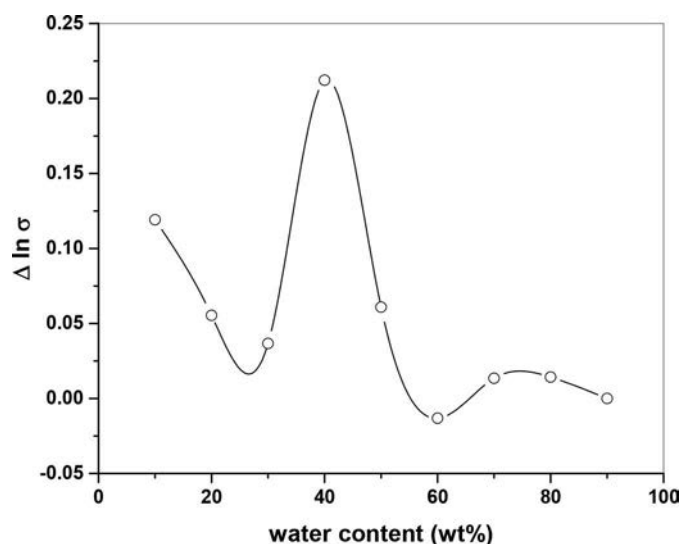


Figure 6.  $\Delta \ln \sigma$  versus water content showing the variation around the percolation temperature ( $T_p$ ).

explain the influence of temperature on conductivity.<sup>[12]</sup> At 20 wt% water, an increase in  $E_{cond}$  value to 7.1 kJ/mol is observed. Again this may indicate the reorganization of clusters. For water content ranging between 30 and 60 wt%, the  $E_{cond}$  values are actually negative. A slight increase in  $E_{cond}$  value to 1.7 kJ/mol occurs at 0.7 wt% water, then at 80 wt% water it becomes  $-1.4$  kJ/mol. The extremely low values for this region suggest a more open microstructure. It appears that the static percolation related to the appearance of bi-continuous microemulsion, where ions can move freely through the connected water channels, has been enhanced at temperatures ranging above that of the  $T_p$  of the system. A sharp increase in  $E_{cond}$  value (66.3 kJ/mol) occurs at 90 wt% water. Increasing the temperature will make the micelle grow in size. Larger droplets may require higher activation energy in order to deform a larger area of the interface.

Figure 6, which shows the variation of  $\Delta \ln \sigma$  around the percolation temperature ( $T_p$ ), suggests the following sequence of structural transitions. For 10–20 wt% water contents, W/O microemulsions are present. For water contents between 30 and 50 wt% inverted bi-continuous microstructure is proposed, while for water contents between 60 and 80 wt% regular bi-continuous microemulsions are suggested. A complete inversion to O/W microemulsions occurs at water contents above 80 wt%.

### 3.3. Hydrodynamic radius

The hydrodynamic radius of water/sucrose laurate/diesel/1-pentanol diluted microemulsions at water content equals 90 wt% along the dilution line N60 was measured using the dynamic light scattering (DLS) technique at temperatures varying from 25°C to 50°C (Figure 7). The values of the hydrodynamic radius increase with temperature from 10.9 to 12.1 nm. This behavior indicates that the micelles grow in size as the temperature increases. The microemulsion droplets deform by thermal fluctuations. The droplets undergo attractive interactions that lead to increased aggregation. Two mechanisms have been used to explain the origin of the attractive interactions: interpenetration of the tails of the

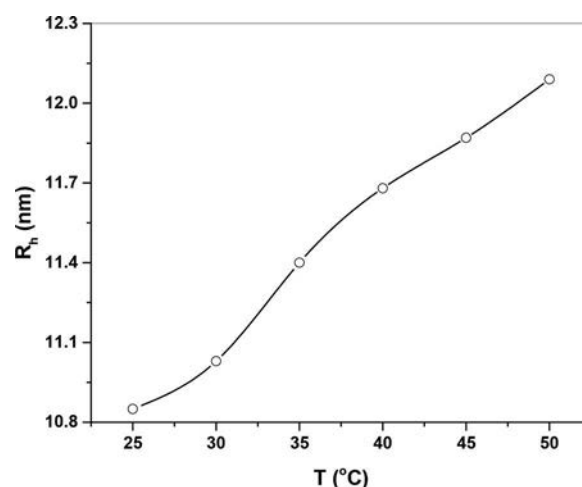


Figure 7. The hydrodynamic radius,  $d_h$ , versus temperature for the diluted microemulsion at water content 90 wt%.

**Table 1.** Diffusion coefficients of water ( $D^w$ ) for samples that lie along the one-phase channel in the microemulsion region (Figure 1).

Water (wt%)	$D^w$ ( $10^{-11}$ m <sup>2</sup> /s)
10	7.7
40	55.5
50	70.0
60	89.2
90	220.0

surfactants molecules residing on different droplets or fusion of droplets, which lowers the curvature energy.

### 3.4. Self-diffusion behavior of the system

As mentioned in the introduction, molecular self-diffusion coefficient is sensitive to obstruction and confinement into closed domains, and thus can provide a straightforward approach to the microemulsion microstructure. Table 1 shows the self-diffusion coefficients of water  $D^w$  measured along the water dilution line  $N_{50}$  in the one-phase microemulsion region (Figure 1). The very large variations in  $D^w$  indicate microemulsion structural transitions from W/O structure at low water content ( $7.7 \times 10^{-11}$  m<sup>2</sup>/s at 10 wt%) to the bi-continuous microemulsion ( $55.5$ – $89.0 \times 10^{-11}$  m<sup>2</sup>/s at 40–60 wt%) and finally to the O/W microemulsion ( $220.0 \times 10^{-11}$  m<sup>2</sup>/s at 90 wt%). The self-diffusion coefficient measured for neat water  $D_0$  was  $230 \times 10^{-11}$  m<sup>2</sup>/s. The relative self-diffusion coefficient for water,  $D^w/D_0$ , in the O/W microstructure is very close to unity (0.96), as expected.

## 4. Conclusions

Alternative fuel microemulsion using sucrose laurate surfactant was tested for its transport properties. Percolation threshold was identified through varying microemulsion composition at constant temperature, or through varying temperature at constant composition. At a temperature range lower than the percolation temperature, the  $E_{cond}$  values dropped substantially with increasing water content, indicating the transition of microemulsion structure from non-percolating, discrete, non-interacting W/O droplets to more swollen, interacting droplets, forming clusters. For water content of 30–80 wt%, the transformation to the bi-continuous phase is evident by the obtained  $E_{cond}$  values. The region for water content above 80 wt% indicates the transition from bi-continuous to O/W microstructure. The fuel microemulsions exhibited a sharp increase in electrical conductivity with increasing temperature at constant composition. For temperatures above  $T_p$ , the  $E_{cond}$  values were low even at very low water content (10 wt%), indicating the existence of percolation. The transformation to bi-continuous phase led to extremely low  $E_{cond}$  values with

enhanced effect of temperature on the ion free movement through water open channels. Finally, a sharp increase in  $E_{cond}$  value at 90 wt% water occurs due to the increase in O/W droplet size that require higher activation energy to be deformed. The considerable variations in the self-diffusion coefficients for water measured as a function of water content demonstrated clearly the structural transition from W/O to bi-continuous to O/W microemulsion.

## Funding

The stay of I. K. as a visiting professor at Lund University Sweden was generously supported by The Arab Fund Fellowship Program in Kuwait.

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