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ORIGINAL ARTICLE

# Characterization of Water/Sucrose Laurate/*n*-Propanol/Allylbenzene Microemulsions

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**Abstract** Water/*n*-propanol/sucrose laurate/allylbenzene micellar systems were formulated and applied in the isomerization of allylbenzene in the presence of heterogenized derivatives of some platinum group catalysts. The ratio (w/w) of *n*-propanol/surfactant studied herewith was 2/1. Temperature insensitive microemulsions were found. The microemulsions were characterized by the volumetric parameters, density, excess volume, ultrasonic velocity, and isentropic compressibility. The densities increase with increases in the water volume fraction. Excess volumes of the microemulsions decrease for water volume fractions below 0.2, level off for water volume fractions between 0.2 and 0.6 then increase for water volume fractions above 0.6. Excess volumes of the studied micellar systems increase with temperature. Isentropic compressibilities increase with temperature for water volume fractions below 0.8 and decrease for water volume fractions above 0.8. Structural transitions from water-in-oil to bicontinuous to oil-in-water occur along the microemulsion phase. The particle hydrodynamic diameter of the oil-in-water microemulsions at the 0.95 water volume fraction was found to decrease with temperature.

**Keywords** Phase behavior · Ultrasonic velocity · Density · Isentropic compressibility · Hydrodynamic diameter

## Introduction

Microemulsions are homogeneous, transparent, thermodynamically stable dispersions of water and oil stabilized by a relatively large amount of surfactant [1–3]. In order to allow additional steric flexibility for surfactant rearrangements, a cosurfactant (typically a short-chain alcohol) is combined with the surfactant. A microemulsion system can exist as water-in-oil, bicontinuous, or oil-in-water [1–3] types. Microemulsions are considered to be ideal microreactors for various chemical and biochemical reactions because of their ease of preparation and thermodynamic stability [1–3]. However, to be used as microreactors, detailed characterization of their properties is required. This includes studies of the phase behavior (composition of the mixture at which the microemulsion is formed), size of the microemulsion droplets. Various spectroscopic and scattering techniques have been employed for this characterization [1–3]. Traditional spectroscopy (light scattering techniques, NMR, etc.) employs the electromagnetic wave. Another kind of wave that propagate through materials is ultrasound [high frequency (above 100 kHz) acoustical waves]. These waves explore the elastic rather than the electric and magnetic properties of materials. As it transverses a sample, compressions and decompressions in the ultrasonic wave change the distance between molecules within the sample, which, in turn, respond by intermolecular repulsions and attractions. This ability of ultrasonic waves to explore intermolecular forces allows access to molecular levels of organization. Measurements of the

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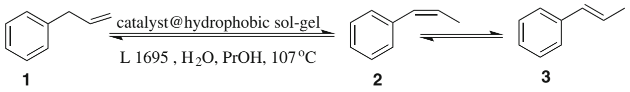
scattering effects of ultrasonic waves allow distinction between different structural organization within the microemulsion phase [4–8].

In the course of our attempts to replace conventional, but harmful solvents used in organic synthesis by benign media, a three phase emulsion (or microemulsion)/solid state transport system (EST) has been developed [9, 10]. This system which consists of a mixture of the organic substrates and water, together with surfactants that solubilize the reagents, has already proven useful in several hydrogen transfer reactions [9–11] as well as in C–C coupling processes [12–15]. Successful application of the EST system requires careful studies on the various parameters of the emulsions and microemulsions to be employed. In the present paper we report on the properties of aqueous microemulsions suitable for double bond migration in allylic compounds by a heterogenized organometallic catalyst. Alkene isomerizations are involved in the fabrication of fatty acids, terpenes, alkaloids and steroids, and are also key steps in the preparation of Shell's higher olefin process and in the fabrication of a variety of fragrances. Allylbenzene which is water-insoluble and can be converted into the isomeric 1-phenyl-1-propenes by the sol–gel supported ion pair $[(C_8H_{17})_3NCH_3][RhCl_4]$ [16] and by other heterogenized catalysts employed in the framework of this study. Among the various surfactants studied in connection with our isomerization in microemulsions, we found sucrose laurate (L1695), together with the co-surfactant *n*-propanol, of particular utility. Room temperature measurements of the phase behavior, the volumetric properties and droplet size should be a good indication of the properties of the media applied at 107 °C at which the catalytic double bond migration in allylbenzene has been recorded (Scheme 1).

## Experimental

### Materials

Sucrose laurate (L1695) was obtained from Mitsubishi-Kasei Food Corp. (Mie, Japan). Allylbenzene (ALB), *n*-propanol, methyltrioctyl ammonium chloride (Aliquat 336), and rhodium trichloride trihydrate were obtained from the Sigma-Aldrich Chemical Company. Dichlorotris(triphenylphosphine) ruthenium, di-$\mu$-chlorobis[(1,2,5,6-$\eta$)-1,5-cyclooctadiene] dirhodium, *E*-Chlorocarbonylbis (diphenyl-phosphine) iridium, palladium acetate and chlorocarbonyl bis-triphenyl phosphine were purchased from Strem. Hexachloroplatinic acid hexahydrate was obtained from Alfa Aesar. Tetramethoxy silane (TMOS) and *n*-octyltriethoxysilane (OTEOS) were purchased from ABCR-Glest, Inc. The reference compounds (*E*)- and (*Z*)-1-phenyl-1-propene were prepared as reported earlier from allylbenzene and polystyrene-bound dichloro-bis (triphenylphosphine) ruthenium (II) [17]. All compounds were used as supplied without further purification. Triply distilled water was used for all experiments.



**Scheme 1** Catalytic double bond migration in allylbenzene

## Methods

### Sample Preparation for a Pseudoternary Phase Diagram at Constant Temperature

The phase behavior of water, propanol, sucrose laurate, and allylbenzene mixture is described in pseudoternary phase diagrams in which the weight ratio of propanol/sucrose laurate is fixed at two. The determination of the phase diagram was performed in a thermostated bath (at 298, 310 and 318 ± 0.1 K). Ten weighed samples composed of mixtures of propanol; sucrose laurate and allylbenzene were prepared in culture tubes sealed with viton-lined screw caps at predetermined weight ratios of allylbenzene/propanol/sucrose laurate. The initial composition of the samples in grams before water titration is presented in Table 1. The mixtures were titrated with water along dilution lines by steps of 3 wt% each time drawn to the aqueous phase apex from the opposite side of the triangle. In all of the samples tested, evaporative losses were negligible. All samples were equilibrated during a time interval of up to 24 h. The different phases were determined using visual and optical (crossed polarizers) methods. The appearance of turbidity was considered as an indication for

**Table 1** Composition of the samples before water titration in grams

| # | Sucrose laurate (L1695) | *n*-Propanol (*n*-PrOH) | Allylbenzene (ALB) |
|---|---|---|---|
| 1. | 0.30 | 0.60 | 0.10 |
| 2. | 0.27 | 0.53 | 0.20 |
| 3. | 0.23 | 0.47 | 0.30 |
| 4. | 0.20 | 0.40 | 0.40 |
| 5. | 0.17 | 0.33 | 0.50 |
| 6. | 0.13 | 0.27 | 0.60 |
| 7. | 0.10 | 0.20 | 0.70 |
| 8. | 0.07 | 0.13 | 0.80 |
| 9. | 0.03 | 0.07 | 0.90 |
| 10. | 0.33 | 0.67 | 0.00 |

phase separation. The phase behavior of such samples was affirmed only after sharp interfaces had become visible. The conclusion of this procedure was hastened by centrifuging the samples. Every sample that stayed clear and uniform after energetic vortexing was considered as belonging to a one-phase area in the phase diagram [18].

### Ultrasonic Velocity and Density

The volumetric properties (ultrasonic velocity and density) of the different microemulsion samples were measured by means of a density and sound velocity analyzer (DSA 500M-Anton Paar, Austria) with a sound velocity resolution of 0.5 $ms^{-1}$ and density resolution of $5 \times 10^{-6}$ g $cm^{-3}$. A 3-ml degassed sample was introduced using Hamilton glass syringe into a U-shaped borosilicate glass tube that was being excited electronically to vibrate at its characteristic frequency. The characteristic frequency changed depending on the density of the sample. Through exact determination of the characteristic frequency and a mathematical conversion, the density of the sample can be calculated. The measuring cell was closed by an ultrasonic transmitter on the one side and by a receiver on the other side. The transmitter sent sound waves of a known frequency through the sample. The velocity of sound was calculated by determining of the period of received sound waves and by considering the distance between transmitter and receiver. Due to the high dependency of the density and velocity of sound values on the temperature, the measuring cells had to be thermostated precisely with two integrated Pt 100 platinum thermometers together with Peltier elements that provided a precise thermostating of the sample to within ±0.01 °C. Viscosity-related errors were automatically corrected over the full viscosity range by measuring the damping effect of the viscous sample followed by a mathematical correction of the density value. The instrument automatically detected gas bubbles in the density measuring cell by an advanced analysis of its oscillation pattern and generated a warning message. Measurements were made at 298, 310, 318 K.

### Dynamic Light Scattering

A Zetasizer Nano S (ZEN 1600) from Malvern Instruments Ltd. (Worcestershire, UK) was used for the measurements of the size of the dispersed particles. The equipment consisted of a 4 mW, 633 nm He–Ne laser. The size measurement range was between 0.6 nm and 6 μm, the size measurement angle equaled 173°, the concentration range for size measurement was between 0.1 ppm (0.00001 vol%)—40 wt% and the temperature measurement range was between 275 and 363 K. A 1.5-ml microemulsion sample was introduced into a disposable polystyrene cuvette and measured over a temperature range between 273 and 323 K in steps of 5 K. The particle hydrodynamic diameter was calculated from the translational diffusion coefficient ($D$) using the Stokes–Einstein relationship:

$$d_H = k_B T / 6\pi\eta D \quad (1)$$

where $d_H$ is the hydrodynamic diameter, $k_B$ is Boltzmann's constant, $T$ is the absolute temperature and $\eta$ is the solvent viscosity. The results are averages of 3 experiments.

### Catalytic Isomerization of Allylbenzene

A glass-lined mini-autoclave was charged with an oil-in-water microemulsion composed of allylbenzene (0.18 ml, 0.81 wt%) sucrose laurate (L1695, 0.65 g, 3.30 wt%), *n*-propanol (1.63 ml, 6.64 wt%), and triply distilled water (TDW, 17.6 ml, 89.25 wt%) together with the sol–gel encaged precatalyst (0.037 mmol), prepared by the method described for the model catalyst [16]), TMOS (1.8 ml, 17 mmol), OTEOS (1.05 ml, 6.35 mmol)]. The mixture was stirred magnetically (300 rpm) at 107 °C for the desired length of time. The immobilized catalyst was filtered off, washed and sonicated first with TDW (30 ml) followed by $CH_2Cl_2$ ($3 \times 10$ ml). The filtrate was treated with NaCl (2 g) which caused a break down of the microemulsion. The organic phase was separated and the aqueous layer extracted with $CH_2Cl_2$ (20 ml) and analyzed by gas chromatography as previously described [16].

## Results and Discussion

### Phase Behavior

Figure 1 presents the phase behavior of water/sucrose laurate/*n*-propanol/allylbenzene systems at 298 K. As

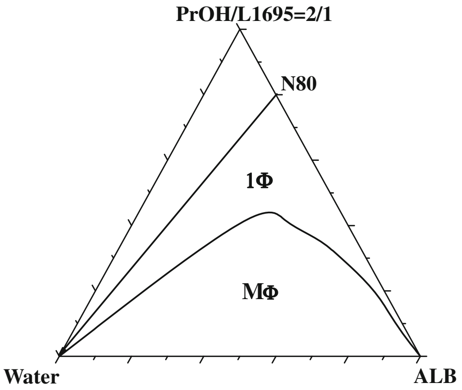


**Fig. 1** Pseudoternary phase diagram of the water/*n*-propanol/sucrose laurate allylbenzene system at 298 K. The mixing ratio (w/w) of *n*-propanol/surfactant is 2/1. The one phase region is designated by 1Φ, and the multiple phase regions are designated by (*M*Φ). N80 is the dilution line where the weight ratio of (surfactant + propanol)/allylbenzene is 4/1

shown in the figure, the transparent water-in-oil microemulsion region appeared immediately on the first addition of water to the mixture of surfactant and allylbenzene. Similar findings on the behavior of sucrose laurate in the presence of other aromatic oils have been reported elsewhere [19, 20]. In these systems, temperature insensitive microemulsions were observed, because no significant change in the hydration of the surfactant head groups occurred within the studied temperature range between 298 and 318 K. Similar behavior of nonionic sugar-based surfactants has been reported elsewhere [19, 21–24].

## Volumetric Properties

Figure 2 presents the variation in the density as a function of the water volume fraction for the water/*n*-propanol/sucrose laurate/allylbenzene microemulsions along the N80 dilution line presented in Fig. 1. The density increases with the water volume fraction. For better understanding of the variation of density as a function of the water volume fraction, the excess volume of the microemulsions formation should be evaluated. The relation (2) can evaluate the excess volume of microemulsions formation keeping in view the additivity of volumes of the aqueous micellar and oil phase,

$$V^{\mathrm{E}} = V_{\mathrm{mic}} - \sum_{i} \phi_i V_i \tag{2}$$

where $V_{\mathrm{E}}$ is the excess volume, $V_{\mathrm{mic}}$ is the measured specific volume of the solution, $\phi_i$ is the volume fraction of component $i$ in the micellar system and $V_i$ is the specific volume of component $i$. Figure 3 presents the variation in the excess volume as function of the water volume fraction at 298, 310 and 318 K. The excess volumes are negative and decrease for water volume fraction below 0.2. For water volume fractions between 0.2 and 0.6, the excess volumes are constant. For water volume fractions above 0.6 the excess volume increases. Negative values of excess volumes indicate that the system contracts upon mixing. This behavior of excess volume indicates that structural transition in the microemulsion region occurs at 0.2 and at 0.6 water volume fractions. The values of the excess volume increase with temperature. This behavior could be related to breakage of hydrogen bonds or to the dissociation of ionic head groups. Figure 4 presents the variation of the ultrasonic velocity as function of the water volume fraction for the water/*n*-propanol/sucrose laurate/allylbenzene system. The ultrasonic velocity increases with the water volume fraction up to 0.8, and thereafter decreases. The variation in the values of ultrasonic velocity as a function of the water volume fraction provides information on the state of the water. At a low

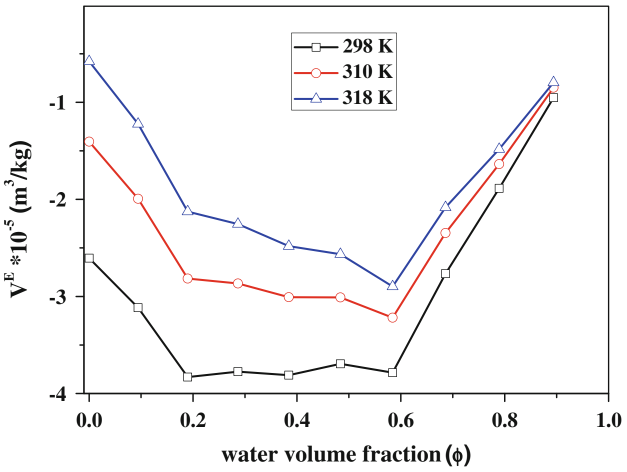


**Fig. 3** Variation of the excess volume as a function of the water volume fraction for water/*n*-propanol/sucrose laurate/allylbenzene micellar systems along the N80 dilution line at 298, 310 and 318 K

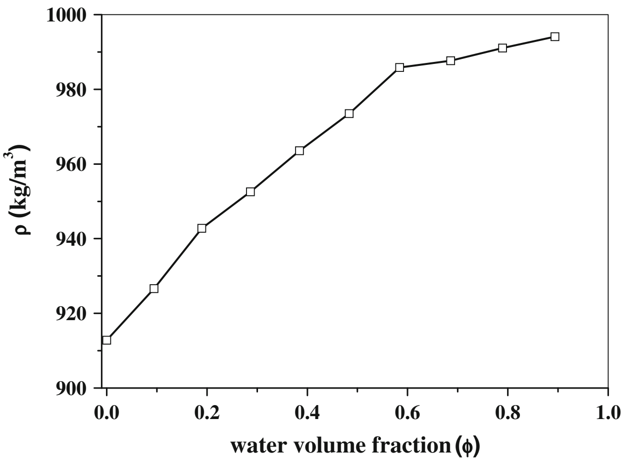


**Fig. 2** Variation of the density as a function of the water volume fraction for water/*n*-propanol/sucrose laurate/allylbenzene micellar systems along the N80 dilution line at 298 K

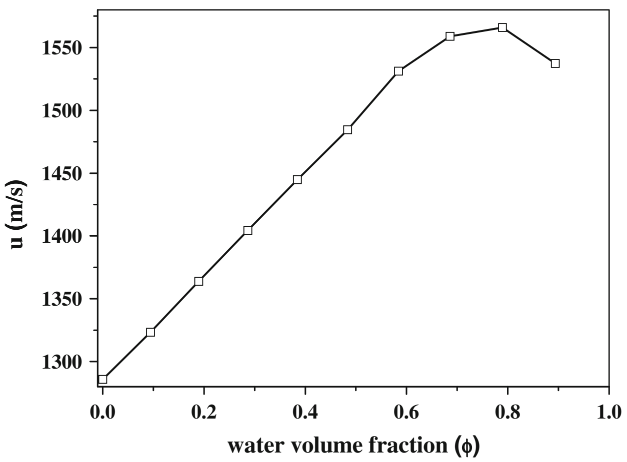


**Fig. 4** Variation of the ultrasonic velocity as a function of the water volume fraction for water/*n*-propanol/sucrose laurate/allylbenzene micellar systems along the N80 dilution line at 298 K

water volume fraction the properties of water are very different from those of the bulk (free water) indicating the entrapment of water in the micellar core suggesting the presence of water-in-oil microstructure. The increase in the values of ultrasonic velocity upon addition of water also indicates structural transitions along the water dilution line. For a water volume fraction above 0.8, the ultrasonic velocity approaches that of pure water indicating that water is the continuous phase and an oil-in-water microstructure is present. Since ultrasonic velocity is determined by the change of physical properties at the interface between the particle core and the continuous medium, the ultrasonic velocity variation will also be correlated to the variation of the size of the particle core. We analyzed the ultrasonic velocity in terms of isentropic compressibility $k_s$, which represents a relative change of volume per unit of pressure applied at constant entropy. $k_s$ is much more sensitive to structural changes than the velocity and can provide qualitative information about the physical nature of the aggregates. The isentropic compressibility $k_s$ values were evaluated with the help of the Laplace equation [25–27]:

$$k_s = \frac{1}{u^2 \rho} \tag{3}$$

Figure 5 presents the variation of the isentropic compressibility $k_s$ as a function of the water volume fraction. The isentropic compressibility decreases with the water volume fraction below 0.8 and increases thereafter. The variation in the values of isentropic compressibility as a function of the increasing water volume fraction indicates structural transitions from a water-in-oil to a bicontinuous to an oil-in-water microstructure. Figure 6 presents the values of the isentropic compressibility as a function of temperature. It increases with the temperature for water volume fractions below 0.8 while for water volume fractions above 0.8 it decreases, indicating different structures in these composition ranges. We suggest that at water volume fractions below 0.2 water-in-oil microemulsions are present while for water volume fractions between 0.2 and 0.6 it is a bicontinuous morphology, and for water volume fractions above 0.6 an oil-in-water microstructure is present. Figure 7 shows a schematic presentation of the suggested structural transitions along the studied dilution line.

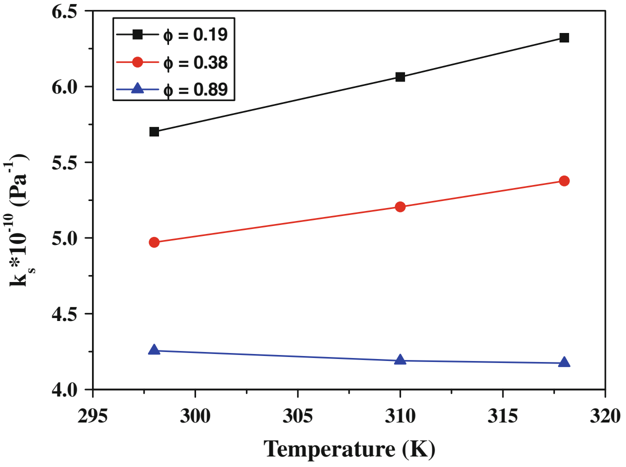


**Fig. 6** Variation of the isentropic compressibility as a function of the temperature for water/*n*-propanol/sucrose laurate/allylbenzene micellar systems along the N80 dilution line at 298 K

## Diffusion Properties

At a water volume fraction of 0.95 and on the assumption that exchange processes are negligible for the surfactant, we can estimate the hydrodynamic diameter ($d_H$) from

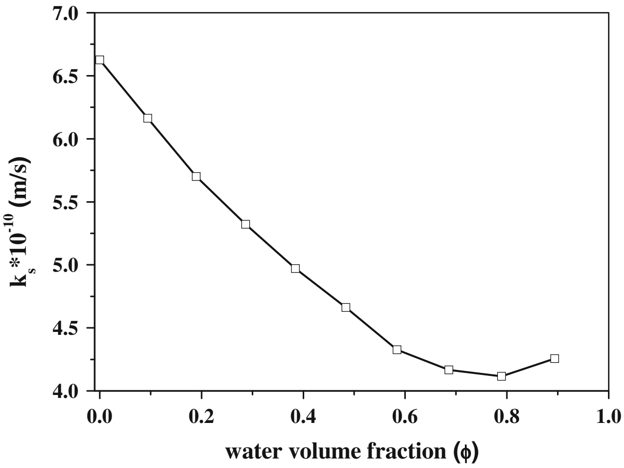


**Fig. 5** Variation of the isentropic compressibility as a function of the water volume fraction for water/*n*-propanol/sucrose laurate/allylbenzene micellar systems along the N80 dilution line at 298 K

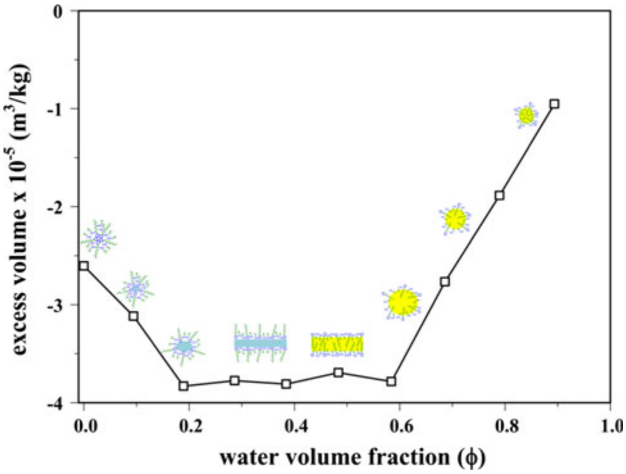


**Fig. 7** Schematic presentation of the suggested structural transitions

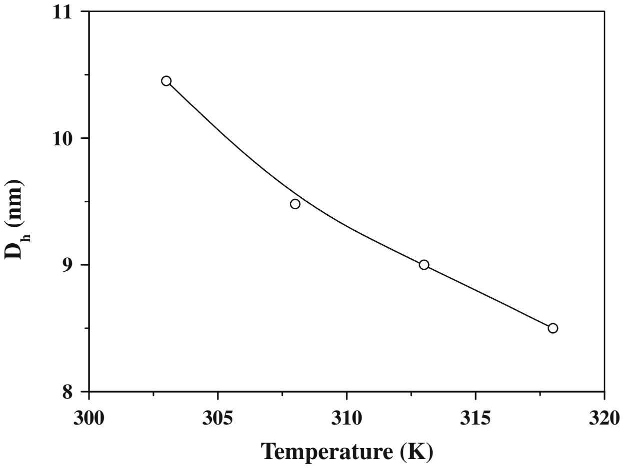


**Fig. 8** Variation of the particle hydrodynamic diameter as a function of the temperature for a water/*n*-propanol/sucrose laurate/allylbenzene oil-in-water microemulsion system at 0.95 water volume fraction along the N80 dilution line

Eq. 1. The variation in the values of the hydrodynamic diameter ($d_H$) at a water volume fraction of 0.95 along the N80 dilution line for water/*n*-propanol/sucrose laurate/allylbenzene microemulsions for different temperatures was investigated. As shown in Fig. 8 the hydrodynamic diameter decreases with temperature. It was shown that isomerization reactions of allybenzene in microemulsions depended on the droplet size of the system and that sucrose laurate microemulsions with very small aggregates were good systems for this isomerization reaction [15].

## Double Bond Migration Reactions

The catalytic isomerization of allylbenzene in solvent-free, as well as in organic solvents, had already been shown to depend strongly on the structural features of the catalyst [28]. In this study, in the aqueous media, we noticed a similar dependence of the reaction on the catalyst used. Upon application of different catalysts of the platinum group elements, we found the following order of activity: Heterogenized $RuCl_2(PPh_3)_3 > [(C_8H_{17})_3NCH_3][RhCl_4] > [Rh(cod)Cl]_2 > IrCl(CO)(PPh_3)_2 \sim Pd(OAc)_2 > H_2PtCl_2$, i.e., the catalytic potency depends both on the nature of the metal and on the type of ligand. The isomerization of allylbenzene by the aforementioned immobilized catalysts is summarized in Table 2. As in organic solvents, the ruthenium complex was shown to be the most efficient catalyst also in microemulsions. After 2 h, the reaction mixture was almost at equilibrium and the amounts of the isomeric products did not alter thereafter. Slightly less effective was the immobilized rhodium ion pair. After 3 h the pair gave 8 % of the starting allylbenzene and 92 % of the *Z*- and *E*-phenylpropenes. The heterogenized $[Rh(cod)Cl]_2$ reacted considerably slower than the ion pair. Approximately 20 h was required at 107 °C to reach a state of equilibrium. We recall that under solvent-free homogeneous conditions hardly any isomerization took place at 107 °C. Therefore in organic solvents, the experiments were carried out at 160 °C [28]. Furthermore, in these media, the addition of more than 15 % water to the reaction mixture (in the autoclave) usually stopped the catalytic reactions. The immobilized iridium and palladium catalysts were found to have quite similar catalytic potencies. They

**Table 2** Isomerization of allylbenzene by some sol–gel encaged catalysts in aqueous microemulsions under comparable conditions

| Entry | Sol–gel entrapped catalyst | Reaction time (h) | Composition of the final reaction mixture (%)[a,b] | | |
|---|---|---|---|---|---|
| | | | **1** | **2** | **3** |
| 1 | $RuCl_2(PPh_3)_2$ | 2 | 4 | 7 | 89 |
| 2 | $[(C_8H_{17})_3NCH_3][RhCl_4]$ | 3 | 8 | 8 | 84 |
| 3 | $[Rh(cod)Cl]_2$ | 5 | 59 | 4 | 37 |
| 4 | $[Rh(cod)Cl]_2$ | 20 | 5 | 7 | 88 |
| 5 | $IrCl(CO)(PPh_3)_2$ | 5 | 82 | 3 | 15 |
| 6 | $IrCl(CO)(PPh_3)_2$ | 20 | 37 | 8 | 55 |
| 7 | $Pd(OAc)_2$ | 5 | 82 | 3 | 15 |
| 8 | $Pd(OAc)_2$ | 20 | 44 | 8 | 48 |
| 9 | $H_2PtCl_4 \cdot 6H_2O$ | 5 | 66 | 4 | 30 |
| 10 | $H_2PtCl_4 \cdot 6H_2O$ | 20 | 53 | 7 | 40 |

Reaction conditions: microemulsion of allylbenzene (**1**) (0.18 ml), sucrose mono laurate (0.65 g), *n*-propanol (1.63 ml), $H_2O$ (17.6 ml), sol–gel material prepared from TMOS (1.8 ml) and trimethoxyoctylsilane (1.05 ml) and the appropriate metal complex (0.042 mmol); 107 °C

[a] **1**–**3** as shown in Scheme 1

[b] Average of two reactions that did not differ by more than ±2 %

promote ca. 50 % conversion within 20 h. Hexachloroplatinic acid is quite efficient at the beginning of the isomerization process, but soon loses its catalytic power.

## Conclusion

Temperature insensitive microemulsions were developed to be used as microreactors with hydrophobic reagents that will lead to a significant reduction in the vast amounts of organic solvents used currently in organic syntheses, and consequently increase the safety and diminish the cost of chemical processes. Structural transitions from water-in-oil to bicontinuous to oil-in-water were probed in the microemulsion region. Since the particle size of the microemulsions is an important parameter in determining the yield of isomerization reaction of allylbenzene, the results presented in this study recommend performing these reactions at water volume fractions above 0.80 at high temperatures.

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**Jochanan Blum** is professor emeritus of organic chemistry at the Hebrew University of Jerusalem. His current research interest is directed to sustainable chemistry, including studies on the replacement of organic solvents in chemical reactions by water, catalysis by organometallics, new methods for catalyst recycling and domino reactions.