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Vapor-liquid equilibria of binary and ternary mixtures of acetahdehyde with Versatic 9 and Veova 9

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Abstract

In continuation of our earlier publication on the phase behaviour of binary and ternary mixtures involving acetaldehyde, Versatic 10, and Veova 10, in this work we present bubble point pressures of the binary and ternary systems of acetaldehyde, Versatic 9, and Veova 9. The measurements were carried out in the Cailletet equipment, which operates according to the synthetic method. The solubility data are presented within a temperature range of (298-353) K for the two binaries of acetaldehyde + Versatic 9 and acetaldehyde + Veova 9. The pressures required for complete solubility into one homogeneous phase ranged from (0.0832-0.414) MPa in the temperature and concentration range considered. While both Versatic and Veova contain a C=O group, Versatic also contains an alcoholic OH, which makes its solubility in acetaldehyde more difficult. The solubility curve of Versatic 9 not only shifts to higher pressures compared to that of Veova 9, but also has an increased curvature, indicating its greater deviations from ideality. As the alkyl chain is increased by one carbon number to Veova 10 and Versatic 10, the absence and presence of the OH group in Veova and Versatic also result in greater and smaller curvatures of the solubility curves, respectively. The Peng-Robinson (PR) and Soave-Redlick-Kwong (SRK) equations of state were used to model phase equilibria of the binary systems of acetaldehyde + Versatic 9 and acetaldehyde + Veova 9. Results showed that both models have good agreement with the experimental values in this study. Two ternary mixtures of acetaldehyde + Versatic 9 + Veova 9 were also investigated experimentally for their bubble point pressures.

While both components have one C=O group, Versatic 9 also contains an alcoholic OH, which Veova 9 lacks. Therefore, nonideal interactions are increased between the molecules of

acetaldehyde and Versatic 9 due to the presence of this hydrogen-bonding group, as compared to the system of acetaldehyde + Veova 9.

Keywords: vinyl neononanoate; vinyl ester of Versatic acid 9; vinyl ester of neononanoic acid; vinyl ester of tertiary carboxylic acid; monomer; [equation of state](#)

1. Introduction

“Versatic” is a registered Shell trademark for a series of tertiary aliphatic carboxylic acids [1]. To distinguish the particular Versatic acid from the others, the total number of carbon atoms in the molecule is mentioned as well, therefore, Versatic 9 is the tertiary carboxylic acid having 9 carbon atoms in its molecule. It is highly branched and fully saturated [1].

Versatic is an intermediate substance for a number of Versatic-based compounds, with well-established track records of cost effective performance. Such products are used for a wide range of applications, such as adhesives, decorative paints, agrochemicals, pharmaceuticals, peroxides, catalysts, preservatives, adhesives, lubricants, and cosmetics, as well as for protecting coatings and improving the strength and durability of concrete [2]. For example, Versatic and its monoglycidyl and monovinyl esters are used in the paint and resin industries [1].

Veova 9 and 10, monomers with many similarities, are among the important Versatic derivatives. They are branched tertiary structures with bulky hydrocarbon groups. This causes the monomers to have very low surface tension and to be hydrophobic, resulting in a strong resistance to saponification [3]. Through the reaction of their vinyl ester functional group, Veova monomers can polymerise with various other co-monomers. Therefore, the specific properties of

the Veova monomers can be imparted to the corresponding copolymers [3]. The difference in the degree of branching and the length of the hydrocarbon groups, lead to differences in the glass transition temperatures of their homopolymers, and therefore, to their different behavior as modifying co-monomers. While, Veova 10 monomer is a flexibilising co-monomer, Veova 9 provides rigidity to the copolymer [3]. Veova 10 was previously investigated in our earlier publication [4]. The present study focuses on the Veova 9 monomer, also known as vinyl neononanoate, which is the vinyl ester of Versatic acid 9, a synthetic saturated monocarboxylic acid with a highly branched structure containing nine carbon atoms, as shown in Table 1. In this table, R1 and R2 are alkyl groups containing a total of 6 carbon atoms [5]. Veova 9 is very attractive for the manufacture of polymers through reactions of the vinyl group. It is hydrophobic, has very good chemical and UV resistance, and high glass transition temperature (high polymer hardness) [5]. This has resulted in Veova 9 being used as a modifying co-monomer in producing high-quality vinyl acetate-based polymer latices, which are used for the manufacture of specialty emulsion paints, or as a co-monomer with acrylates in the manufacture of emulsion, solution and bulk polymers [5]. Some examples of Veova 9 vinyl ester-based polymer applications include acid or anhydride-cured acrylic automotive coatings, acrylic powder coatings, and specialty emulsion paints, such as glossy emulsion paints and varnishes, water repellent coatings, anti-corrosion paints, and coatings for polyolefins [5].

In order to develop new processes involving Versatic 9 and/or Veova 9, or to modify and optimize current processes, it is necessary to have accurate information on the phase behavior of the mixtures under questions. Since acetaldehyde is a well-known chain stopper in polymerization reactions, which is used to control the molecular weight of the polymer product, the phase behavior of mixtures of acetaldehyde with either Versatic 9 or Veova 9 are of

importance. In this study, vapor-liquid equilibrium data on binary mixtures of acetaldehyde + Versatic 9 and acetaldehyde + Veova 9, as well as ternary mixtures of acetaldehyde + Versatic 9 + Veova 9, are determined experimentally. Such solubility data is not yet available in open literature. Also, in order to have a thermodynamic modelling investigation on the systems considered, two cubic equations of state were used in both predictive and correlative modes for the binary systems of this study.

2. Experimental

The equipment used for the experimental determination of solubilities is known as the Cailletet apparatus, which is based on the synthetic method of measuring phase equilibria. Since synthetic methods generally operate by knowledge of only the overall composition of the contents in an equilibrium cell without taking samples of the individual phases in equilibrium, they have the specific advantage that errors resulting from sampling are not inherent in the data. Therefore, data obtained by accurate synthetic equipment are generally considered to be among the more accurate phase equilibrium data. The disadvantage, however, is that information on the concentrations of the phases in equilibrium is restricted to the limiting situations of two-phase equilibria in which one of the equilibrium phases is at the verge of disappearing, such as bubble points and dew points. In such cases, the concentration of the remaining phase can be considered equal to the known concentration of the constituents within the equilibrium cell.

The equilibrium cell is in the form a 3 mm diameter tube of Pyrex, with a closed end where the sample is constrained within, and an open end which is placed inside an autoclave. Mercury is injected into this tube following the injection of the experimental sample in order to

seal the sample from the hydraulic oil in the autoclave that is transferring the pressure. Pressure is generated by a screw hand pump acting on the hydraulic oil. A dead-weight pressure gauge measures the pressure with an accuracy of 0.03% of the reading. Temperature is also controlled and fixed, with a constancy better than 0.01 K, by a thermostat fluid which continuously circulates around the Cailletet tube. The temperature is measured with a platinum resistance thermometer with an accuracy better than 0.02 K.

Bubble points are measured for each sample by allowing the sample to reach the temperature of the thermostat liquid, reducing the pressure until two phases (vapor and liquid) are generated, then gradually increasing the pressure until the point where the last bubble of vapor disappears while the mixture is being continuously stirred with a steel ball within the sample, activated by reciprocating magnets situated outside of the Cailletet tube. A microscope is used for a more accurate observation of the disappearing bubble. When the pressure at which the vapor completely dissolves in the liquid is recorded, the temperature is increased to the next desired temperature, and the procedure above is repeated. In this way, a curve of bubble point pressures versus temperature is measured for a mixture of constant overall composition (isopleth). The schematic diagram of the Cailletet equipment, as well as more detailed explanations on the device and the procedure, are available in earlier publications [6,7]. The specifications and suppliers of the substances used are presented in Table 1. The substances were used without further purification.

3. Results and Discussion

The vapor-liquid phase equilibrium data for the binary mixtures of acetaldehyde + Versatic 9 and acetaldehyde + Veova 9, as measured for several molar concentrations for each mixture by the Cailletet equipment, are presented in Tables 2 and 3, respectively. The data cover a temperature range of (298-353) K. Bubble point experiments were also conducted on ternary mixtures of acetaldehyde + Versatic 9 + Veova 9 and the corresponding results are given Table 4. There are no phase equilibrium data available in literature on any of the investigated systems, therefore, it was not possible to compare the data of this work to others.

The pressure-temperature cross sections of the phase boundaries are shown in Figure 1 for all of the systems measured in this work. As temperature increases, it becomes more difficult to dissolve acetaldehyde in either Versatic 9, Veova 9, or their mixture, therefore increasingly higher pressures are required.

As information on P - x surfaces is often more useful in design and modeling than on P - T surfaces, The P - T - x data of the above systems have been interpolated in the form of pressure-concentration isotherms and presented in Tables 5 and 6, respectively, for acetaldehyde + Versatic 9 and acetaldehyde + Veova 9. Literature data for pure acetaldehyde [8] is also included with the above data to plot the P - x curves of both binary systems on Figures 2 and 3.

Figure 4 shows the behavior of both binary systems together on one graph for comparison. The binary mixtures with Versatic 9 have greater curvature than those with Veova 9, indicating their greater deviations from Raoult's law on the isothermal P - x curve. This is rooted in the difference in the functional groups of the two structures. While both components have one C=O group, Versatic 9 also contains an alcoholic OH, which Veova 9 lacks. Therefore, nonideal interactions

are increased between the molecules of acetaldehyde and Versatic 9 due to the presence of this hydrogen-bonding group, as compared to the system of acetaldehyde + Veova 9.

Figure 5 compares the effect of an increase in the chain length of Versatic and Veova from nine to ten [4], on their phase behavior with acetaldehyde. It is interesting to note that while the nonideality of the mixture decreases with chain length for Versatic, the opposite trend is observed for Veova. This too, can be explained by the molecular structures of the two substances. The nonideal molecular interactions of the hydrogen-bonding group of OH are decreased as the hydrocarbon chain length increases in Versatic, resulting in an increased hydrocarbon nature of Versatic 10 in comparison to Versatic 9, and hence, less deviations from Raoult's law. Veova lacks the strongly interacting OH group, so its chemical nature is closer to acetaldehyde, as they both contain the C=O group as their most prominent functional group. Therefore, increasing the chain length in Veova only results in its greater structural contrast to acetaldehyde, consequently resulting in increased curvature (nonideality) in its solubility curve with increasing carbon number.

In order to model the experimental data by thermodynamic tools, two of the simplest and most common models available for phase equilibrium calculations, namely, the Peng-Robinson (PR) and Soave-Redlich-Kwong (SRK) equations of state (EoSs) [9,10] have been chosen. The first necessity for any calculation with cubic equations of state is knowledge of the critical properties of the compounds. Acetaldehyde is a well-known compound with available thermophysical properties, however, critical properties have not been reported in open literature for Versatic 9 and Veova 9. For these compounds, the critical properties were calculated based on the Modified Lydersen-Joback-Reid group contribution method [11]. This method combines the best results and advantages of the Lydersen [12] and the Joback-Reid [13] group contribution methods, and

in doing so, it has been shown to be reliable and accurate for the estimation of the critical properties of high molecular mass substances [14,16]. The critical temperatures, critical pressures, and normal boiling points of Versatic 9 and Veova 9 were calculated by this method, while the acentric factors were estimated by the method of Valderrama and Robles [15]. The calculated critical properties and normal boiling points of the Modified Lydersen–Joback–Reid model are required as input parameters in the method of Valderrama and Robles. The values of critical properties and acentric factors for the investigated compounds are reported in Table 7.

With these values of critical properties and acentric factors, the PR and SRK EoSs were used to predict the experimental phase behavior data of the binary systems in Tables 2 and 3. However, because of the polarity and different molecular interactions between these compounds, the PR and SRK did not perform well without the use of binary interaction parameters. Therefore, binary interaction coefficients (k_{ij} 's) were fit for both the PR and SRK EoSs and the optimized values are presented in Table 8. Figures 6 and 7 show the graphical pressure-temperature behavior of the PR and SRK EoSs with respect to the corresponding experimental values for different compositions of acetaldehyde + Versatic 9 and acetaldehyde + Veova 9, respectively. Both models show reliable correlations. Also, in order to have a more comprehensive comparison, the deviations of the calculated equilibrium pressures with respect to the experimental values are presented in the form of AARD%, given by the following equation,

$$AARD\% = \frac{100}{Np} \sum_1^N \left| \frac{p_i^{Exp.} - p_i^{Calc.}}{p_i^{Exp.}} \right| \quad (1)$$

Where $p_i^{Exp.}$ and $p_i^{Calc.}$ are the experimental and calculated bubble point pressures, respectively and Np is the number of data. The values of AARD% of the PR and SRK EoSs with respect to

experimental values for the different compositions of both the acetaldehyde + Versatic 9 and acetaldehyde + Veova 9 systems are reported in Table 8. PR and SRK EoS's had AARD% values of 2.6% and 2.8%, respectively, for acetaldehyde + Versatic 9 in the experimentally investigated range of compositions. For acetaldehyde + Veova 9, the AARD% values of 4.7% and 4.8% were calculated, respectively for PR and SRK.

4. Conclusions

No data is available in the literature on the solubilities of acetaldehyde in either Versatic 9 or Veova 9, which are data of concern in the polymer industries. This study presents experimentally- determined vapor-liquid equilibria of binary mixtures of acetaldehyde + Versatic 9 and acetaldehyde + Veova 9 at various temperatures and concentrations, as well as ternary mixtures of these three components. Positive deviations from ideality (Raoult's law) are observed for all of the systems investigated, but more so for Versatic 9 than Veova 9. The binary systems of acetaldehyde + Versatic 9 and acetaldehyde + Veova 9 were modeled by the well-known PR and SRK equations of state. The Modified Lydersen–Joback–Reid group contribution method and the model of Valderrama and Robles were used to calculate the unavailable critical properties and acentric factors of Versatic 9 and Veova 9, necessary for the EoS calculations. Both PR and SRK showed accurate results and reliable trends for the different compositions of both binary systems, but only when optimized binary interaction coefficients were employed. PR and SRK had AARD% values of 2.6% and 2.8%, respectively for acetaldehyde + Versatic 9, and 4.7% and 4.8%, respectively for acetaldehyde + Veova 9 systems in the experimentally investigated range of compositions.

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Table 4. Experimental bubble-point data of temperature T and pressure P , for the system acetaldehyde (1) + Versativ 9 (2) + Veova 9 (3).^a

<i>Molar composition</i>	<i>T/K</i>	<i>P/MPa</i>	<i>T/K</i>	<i>P/MPa</i>	<i>T/K</i>	<i>P/MPa</i>
$x_1=0.1997, x_2=0.4199, x_3=0.3803$	323.06	0.092	328.94	0.107	335.07	0.124
	341.08	0.141	346.86	0.161	352.78	0.182
$x_1=0.3980, x_2=0.3160, x_3=0.2860$	303.22	0.084	313.09	0.113	323.02	0.151
	332.98	0.193	342.91	0.249	352.80	0.313

^a Standard uncertainties u are $u(T)$ is 0.02 K, $u(x_1)=0.005$, and $u(P)=0.01$ MPa.

Table 5. Isothermal solubility data of liquid mole fraction x , temperature T , and pressure P for acetaldehyde (1) + Versatic 9 (2).^a

<i>T/K</i>	313.15	323.15	333.15	343.15
x_1	<i>P/MPa</i>	<i>P/MPa</i>	<i>P/MPa</i>	<i>P/MPa</i>
0.1918		0.108	0.137	0.170
0.2764	0.115	0.147	0.185	
0.3625	0.142	0.185		

^a Standard uncertainties u are $u(T)$ is 0.02 K, $u(x_1)=0.005$, and $u(P)=0.01$ MPa.

Table 6. Isothermal solubility data of liquid mole fraction x , temperature T , and pressure P for acetaldehyde (1) + Veova 9 (3).^a

<i>T/K</i>	313.15	323.15	333.15	343.15	353.15
x_1	<i>P/MPa</i>	<i>P/MPa</i>	<i>P/MPa</i>	<i>P/MPa</i>	<i>P/MPa</i>
0.1883		0.083	0.104	0.131	0.162
0.3850	0.111	0.147	0.189	0.238	0.300
0.5808	0.148	0.195	0.256	0.330	0.417

^a Standard uncertainties u are $u(T)$ is 0.02 K, $u(x_1)=0.005$, and $u(P)=0.01$ MPa.

Table 7. The values of critical properties and acentric factors for the investigated compounds.

Compound	T_c/K	P_c/MPa	ω	Ref.
Acetaldehyde	461.0	5.55	0.317	[17]
Versatic 9	740.9	2.16	0.578	[11,15]
Veova 9	687.3	1.62	0.370	[11,15]

Table 8. The values of optimized binary interaction coefficients and errors for the PR and SRK EoSs for binary systems of acetaldehyde +Versatic 9 and acetaldehyde + Veova 9

EoS	Acetaldehyde + Versatic 9		Acetaldehyde + Veova 9	
	k_{ij}	$AARD\%$	k_{ij}	$AARD\%$
SRK	0.089	2.8	0.026	4.7
PR	0.083	2.6	0.018	4.8

Figure captions:

Figure 1: Experimentally measured bubble point pressure vs. temperature for the binary systems of acetaldehyde (1) + Versatic 9 (2), and acetaldehyde (1) + Veova 9 (3), and ternary systems of acetaldehyde (1) + Versatic 9 (2) + Veova 9 (3) at different concentrations as indicated in the legend.

Figure 2: Solubility of acetaldehyde (1) in Versatic 9 (2) at four different temperatures. The data for pure acetaldehyde are taken from literature [8].

Figure 3: Solubility of acetaldehyde (1) in Veova 9 (3) at four different temperatures. The data for pure acetaldehyde are taken from literature [8].

Figure 4: Comparison of the bubble point curves of binary mixtures of acetaldehyde (1) + Versatic 9 (2) and acetaldehyde (1) + Veova 9 (3) at four different temperatures. The data for pure acetaldehyde are taken from literature [8].

Figure 5. Comparison of the solubilities of acetaldehyde (1) in: (a) Versatic 9 and Versatic 10; (b) Veova 9 and Veova 10 at the temperatures of 313.15 K and 333.15 K. The data for the binary mixtures of Versatic 10 and Veova 10 and for pure acetaldehyde are taken from literature [4, 8].

Figure 6. Comparison of PR ($k_{ij}=0.083$) and SRK ($k_{ij}=0.089$) EoS results with experimental values for different compositions of acetaldehyde + Versatic 9.

Figure 7. Comparison of PR ($k_{ij}=0.018$) and SRK ($k_{ij}=0.026$) EoS results with experimental values for different compositions of acetaldehyde + Veova 9.

Figure 1: Experimentally measured bubble point pressure vs. temperature for the binary systems of acetaldehyde (1) + Versatic 9 (2), and acetaldehyde (1) + Veova 9 (3), and ternary systems of acetaldehyde (1) + Versatic 9 (2) + Veova 9 (3) at different concentrations as indicated in the legend.

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Figure 4: Comparison of the bubble point curves of binary mixtures of acetaldehyde (1) + Versatic 9 (2) and acetaldehyde (1) + Veova 9 (3) at four different temperatures. The data for pure acetaldehyde are taken from literature [8].

(a)

(b)

Figure 5. Comparison of the solubilities of acetaldehyde (1) in: (a) Versatic 9 and Versatic 10; (b) Veova 9 and Veova 10 at the temperatures of 313.15 K and 333.15 K.

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