

HFC-404a와 Polyol ester 오일 혼합물의 증기압 측정

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Measurement of Vapor Pressure of HFC-404a and Polyol ester Mixture System*

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요 약

기존의 CFC 계열 및 HCFC 계열 냉매들이 오존층 파괴와 지구온난화의 원인들 중의 한가지로 밝혀지면서 이것을 대체할 수 있는 냉매의 개발은 주요한 연구과제였다. 이제 몇 가지 대체 냉매들이 개발되면서 이것들이 냉동시스템에 사용될 때 발생하는 상용성 (Compatibility)이 연구될 필요가 있다. 본 연구에서는 대체냉매의 상용성 연구의 일환으로 1,1,1,2-Tetrafluoroethane(HFC-134a) +1,1,1-Trifluoroethane(HFC-143a) + Pentafluoroethane (HFC-125)의 혼합대체냉매 HFC-404a 와 냉매 윤활유의 하나인 POE 오일 혼합물의 증기압을 측정하였다. 측정온도범위는 냉매 시스템의 운전온도를 고려하여, 263.15K 에서 323.15 K로 하였으며 오일농도범위는 0 mass% 에서 90 mass% 까지로 하였다. 측정결과 273.15K 이하의 온도에서 오일 농도 30 mass%까지 증기압에 대한 오일의 영향은 미미하였으며 50 mass% 이상에서는 증기압이 급격히 떨어지는 것이 밝혀졌다. 측정자료를 이용하여 HFC-404a와 POE 오일 혼합물의 증기압을 예측할 수 있는 모형을 개발하기 위하여 Rault 모델과 Flory-Huggins 모델을 사용하여 측정치와 비교하였다. 그리고 보다 정확하고 실용적으로 증기압을 예측할 수 있는 경험식을 도출하였다.

주요어 : 증기압, 끓는 점, 대체냉매 HFC404a , POE오일, 혼합물, 기-액상평형

Abstract — Vapor pressure of HFC-404a and polyol ester system were measured at 56 points from 263.15 to 323.15 K and from 0 to 90 mass % polyol ester. It was found that below 273.15 K, the effect of the polyol ester on the vapor pressure was negligible up to 30 mass % polyol ester. The vapor pressure of the system significantly decreased as the mass fraction of polyol ester increased over 50 percent. Raoult's model and Flory-Huggins model were tested for data reduction. Empirical vapor pressure equations were obtained in terms of temperature and mass fraction of polyol ester.

Key words : vapor pressure, bubble point, HFC404a, Polyol ester, mixture, vapor-liquid equilibria.

1. Introduction

In view of ozone layer depletion and global warming, developing new alternative refrigerants has been a

key issue in refrigeration industry. The refrigerant HFC-404a{4-52-44 blend of 1,1,1,2-Tetrafluoroethane (HFC-134a) + 1,1,1-Trifluoroethane (HFC-143a) + Penta-fluoroethane (HFC-125)} is recognized as an environmentally safe refrigerant in place of chlorodifluoro ethane (HCFC-22) and chlorodifluoroethane

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(HCFC-22) + chloropentafluoroethane (CFC-115) system(R-502) which have been implicated in depletion of the ozone layer. After a new refrigerant is developed, a proper lubricant should be chosen so that the lubricant should be compatible with the refrigerant. Compatibility of a lubricant is evaluated in terms of miscibility and thermophysical properties of the mixture system such as vapor pressure, viscosity, and thermal conductivity. Particularly, the vapor pressure of the mixture system makes effects on the performance of refrigerating systems. Therefore, in this study the vapor pressure of the refrigerant HFC-404a[4-52-44 blend of 1,1,1,2-Tetrafluoroethane(HFC-134a) + 1,1,1-Trifluoroethane (HFC-143a) + Pentafluoroethane (HFC-125)] and polyol ester mixture system was investigated. Experimental vapor pressure data for the refrigerant+oil systems are found in the open literatures. Thomas and Pham [1] measured the vapor pressures of the 1,1,1,2-tetrafluoroethane + polyalkylene glycol system over the temperature range from 283.15 to 343.15 K and the composition range from 10 to 90 mass % polyalkylene glycol. Martz, W.L et al [2,3] presented a local composition modeling of the thermodynamic properties of refrigerant and oil mixtures. Huber, M.L. et al [4] used a Helmholtz energy mixing model to describe the bubble point pressures of mixtures of hydrofluorocarbon refrigerants and polyol ester (POE) based lubricants. It is insisted that the model incorporates the best available equations of state for the refrigerants, is valid over the entire range of composition, is not limited to states below the refrigerant critical temperature, and can represent volumetric properties in addition to calculating reliable phase equilibria. Thome, J.R. [5] presented a comprehensive thermodynamic approach for modeling mixtures of refrigerants and lubricating oils. The approach included generalized methods for predicting the thermodynamic properties of refrigerant-oil mixtures such as bubble point temperatures, local oil concentrations, liquid specific heats and enthalpy changes during evaporation. Yokozeki, A. [6] presented solubility data of refrigerant lubricant mixtures that were modeled with cubic equations of state (EOS): van der Waals (vdW), Soave-modified Redlich-Kwong (SRK), or Peng-Robinson (PR) types. In the model, the temperature dependence on the "a" parameter in the EOS is modeled with a common empirical function for pure

refrigerants, including oils. Park et al[7] measured vapor pressures of the 1,1,1,2-tetrafluoroethane(R-134a) + polyalkylene glycol system over the temperature range from 253.15 to 333.15 K at 10 K intervals and the composition range from 0 to 90 mass % polyalkylene glycol. Recently, Shen, B. et al[8] give us critical review on the lubricant influence on refrigerant heat transfer and pressure drop. While not few researches have been on the mixture of refrigerant and lubricant mixtures, available data are not enough to meet engineering analysis and design of system involving HFC-404a+POE. Some relevant and important aspects of HFC404a+POE have not yet been satisfactorily answered. In this study, the vapor pressures of the HFC-404a + polyol ester system were measured over the temperature range from 253.15 K to 333.15 K at 10 K intervals and the composition range from 0 to 90 mass % polyol ester. In order to analyze the equilibrium behavior of the mixture system, Raoult's model and Flory-Huggins model were tested as data reduction models. Also, based on the experimental data, an empirical working equation for the vapor pressure of the mixture system was developed in terms of temperature and mass fraction of polyol ester. Also, the effect of the polyol ester on the vapor pressure of the pure refrigerant was discussed.

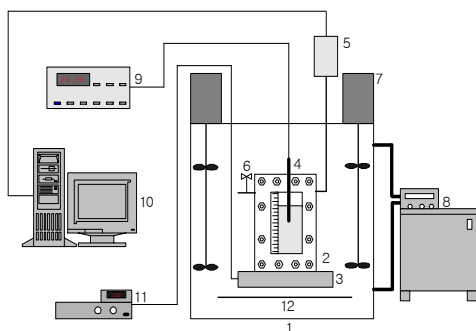
2. Experimental Section

The samples of the refrigerant HFC-404a [4-52-44 blend of 1,1,1,2-Tetrafluoroethane(HFC-134a) + 1,1,1-Trifluoroethane(HFC-143a) + Pentafluoroethane (HFC-125)] were obtained from Honeywell International Inc., and polyol ester was obtained from ICI Eng. The chemicals were used without further purification. Typical properties of the polyol ester and HFC-404a are listed in Table 1.

An experimental apparatus was developed to measure the vapor pressure of the refrigerant HFC-404a{4-52-44 blend of 1,1,1,2-Tetrafluoroethane (HFC-134a) + 1,1,1-Trifluoroethane(HFC-143a) + Pentafluoroethane (HFC-125)} and polyol ester mixture system. The apparatus is shown schematically in Figure 1. The apparatus consists of an equilibrium unit, a temperature control unit, and a data measurement and acquisition unit. The experimental apparatus is de-

Table 1. Properties of Polyol ester and 1,1,1,2-tetrafluoroethane(HFC-134a) + 1,1,1-trifluoroethane(R-143a) + pentafluoroethane(R-125) (HFC-125)(4-52-44)(HFC-404a)¹⁰

Items	Lubricant	Items	Refrigerant
Company	ICI Eng.	Model	HFC-404a
Model	POE 22H	Molecular mass, g/mol	97.6
Molecular mass, g/mol	608	Critical temperature (C)	72.14
Viscosity, cSt @ 40C	18.9	Critical pressure (kPa)	3735
Viscosity, cSt @ 100C	4.2	Critical density, kg/m ³	488.5
Density, kg/m ³ @ 20C	0.995	Mass fraction (%) (HFC -125/ HFC -143A/ HFC -134A)	44 : 52 : 4
Pour Point (C)	-52		
Flash Point (C)	240	Mole fraction (%) (HFC -125: HFC -143A: HFC -134A)	35.78:60.39:3.83

**Fig. 1.** Schematic diagram of the experimental apparatus: 1, Constant temperature bath; 2, Equilibrium cell; 3, Magnetic stirrer; 4, Resistance temperature detector; 5, Digital pressure transducer; 6, Charging valve; 7, DC Motor and stirrer; 8, Constant temperature bath(cold reservoir source); 9, Digital thermometer; 10, Personal computer; 11, Magnetic stirrer controller; 12, Heater.

scribed in details by Park et al [7].

The experiment is carried out in two parts consecutively. The first part is to measure the mass fraction of polyol ester and the second part is to measure the vapor pressure of the mixture system. For the first part of the experiment, all parts of the equilibrium cell were cleaned. Then, the assembled equilibrium cell was evacuated to 10^{-3} torr. First, the mass of the evacuated cell was gravimetrically determined. Second, the polyol ester was injected into the cell and the polyol ester-charged cell was again evacuated to 10^{-3} torr.

Then, the mass of the polyol ester-charged cell was gravimetrically determined. Third, HFC-404a was injected into the polyol ester-charged cell and the mass of the HFC-404a + polyol ester-charged cell was determined again gravimetrically. Then, the mass percentage of the polyol ester was calculated. The precision of the balance (Sartorius F3200) was 0.1 %. For the second part of the experiment, the charged equilibrium cell was immersed in the thermostatic bath that was filled with silicon oil. The temperature of the isothermal bath and equilibrium cell was maintained at a set point by a cold reservoir, a heater, and a circulator. It took two or three hours for the temperature of the bath and cell to stabilize at a set point. After confirming that the bath and cell temperature were maintained at a set point within ± 0.1 K, the magnetic stirrer in the equilibrium cell was started. It stirred the HFC-404a + polyol ester to ensure proper mixing, thus enabling equilibrium to be quickly reached. It took about 30 minutes for the HFC-404a + polyol ester system to reach equilibrium to where the cell temperature was stable and the vapor-liquid boundary did not change. After equilibrium was reached, the bath and cell temperatures were measured with a 100Ω platinum resistance sensor immersed in the cell and a precision thermometer (Yokogawa, 7563) with a precision of 0.01 K. The sensor and thermometer were calibrated on the ITS-90. The uncertainty in the temperature measurements was estimated to be ± 0.1 K. The pressure was measured

with a pressure transducer (Druck, PDCR 922) with a precision of 0.1 kPa. The pressure transducer was calibrated on a dead weight pressure gauge (Ruska, Budenberg) after each series of experiments. The pressure was measured three times when the cell pressure was constant within 5 kPa. The uncertainty of the pressure measurement was estimated to be 0.2%. After a set of measurements was completed, the above procedure was repeated for another mass fraction of polyol ester.

3. Theory and Calculation

Before measuring the vapor pressure of HFC-404A {4-52-44 blend of 1,1,1,2-Tetrafluoroethane(HFC-134a) +1,1,1-Trifluoroethane(HFC-143a) + Pentafluoro- ethane (HFC-125)} polyol ester system, the vapor pressure of HFC-404a was measured in order to determine the system accuracy of the experimental apparatus. The measured vapor pressures of the refrigerant HFC-404a were compared with those of ASHRAE[9]. The deviations are shown in Figure 2. The root-mean-square deviation between the measured vapor pressures and those of ASHRAE[11] is about $\pm 0.5\%$. Based on the measured vapor pressures, a vapor pressure(bubble point) equation for the HFC-404a was developed as follows:

$$\ln(P/P_c) = \frac{1}{1-\tau} \sum_{i=1}^5 a_i \tau^i \quad 263.15 \leq T \leq 323.15 \text{ [K]} \quad (1)$$

where $\tau = T/T_c$, P , kPa is the vapor pressure, T_c , K and P_c are the critical temperature and the critical pressure, respectively, and the a_i coefficients from the least squares fit are given in Table 2.

The vapor pressures of the HFC-404a + polyol ester system were measured in a temperature range from 263.15 to 323.15 K at 10 K intervals from about 5 to 90 mass % polyol ester. The measured vapor pressures are presented in Table 3. Thomas and Pham[1] reported their measured data only plotted on figures. So, quantitative comparison with reference [1] was not possible in the present study. The variation of the vapor pressure of the system with the mass fraction polyol ester is presented in Figure 3. The vapor pressure decreases as the polyol ester increases. The effect of polyol ester on the vapor pressure is more and more significant as the temperature and the oil mass % increases. As shown in table 3, below 273.15 K, the effect of polyol ester is negligible within 2% up to 30 mass % polyol ester. Based on the measured data, vapor pressure-temperature- mass percent of polyol ester (P - T - ω) correlation was obtained. The empirical correlation is as follows:

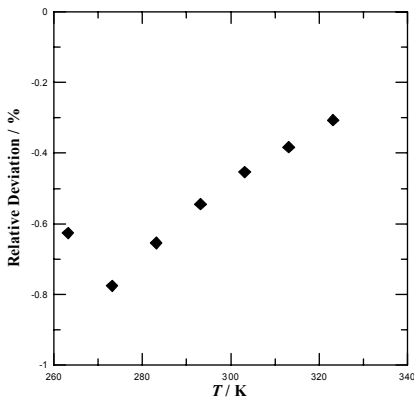


Fig. 2. Comparison of the measured vapor pressure of 1,1,1,2-Tetrafluoroethane(HFC-134a) + 1,1,1-Trifluoroethane (R-143A) + Pentafluoroethane(R-125) ; (HFC-125)(4-52-44)(HFC-404a) with data from ASHRAE Handbook¹¹.

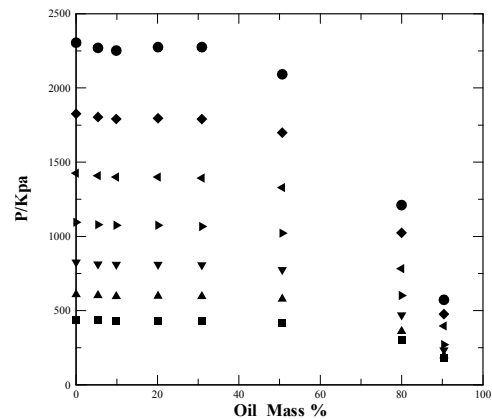


Fig. 3. Vapor pressure of HFC-404a + Polyol ester as a function of temperature and polyol ester oil mass percent : ■ 263.15K; ▲ 273.15K; ▼ 283.15 K; ► 293.15K; ◄ 303.15K; ◆ 313.15K; ● 323.15K.

Table 2. Coefficients of Vapor Pressure Equation(1), (2) and (3)

Coefficients	HFC-404a + POE System		
	Equation (1)	Equation (2)	Equation (3)
a_1	-223.4064	0.07054702876766	6.0680
a_2	579.1638	0.00002069785029	-0.0212
a_3	-397.0609	-1.92173575443379	3.06E-05
a_4	47.9743	-0.01227809410747	-1.5636
a_5	-6.6788	-0.05999087203979	-9.71E-06
a_6		-0.00001777997596	1.62E-08
a_7		-1.56104037964929	
a_8		0.01053950756561	
a_9		0.00280012868410	
a_{10}		0.00000084594835	
a_{11}		-0.07183139570713	
a_{12}		-0.00049343871595	
k_1	1		
k_2	1.5		
k_3	2		
k_4	4		
k_5	6.5		

Table 3. Measured Vapor Pressures of 1,1,1,2-Tetrafluoroethane(HFC-134a) + 1,1,1-Trifluoroethane(R-143a) + Pentafluoroethane(R-125) (HFC-125)(4-52-44)(HFC-404a)

T (K)	HFC-404a + POE System							
	Vapor Pressure, kPa							
	0 mass %	5.36 mass %	9.87 mass %	20.12 mass %	30.89 mass %	50.65 mass %	79.97 mass %	90.38 mass %
263.15	437.	435.	433.	433.	429.	420.	303.	182.
273.15	609.	603.	596.	599.	596.	579.	361.	197.
283.15	826.	813.	811.	811.	807.	775.	470.	234.
293.15	1095.	1079.	1075.	1075.	1067.	1022.	602.	271.
303.15	1426.	1409.	1400.	1400.	1393.	1329.	783.	397.
313.15	1826.	1804.	1791.	1796.	1790.	1699.	1024.	477.
323.15	2305.	2270.	2252.	2275.	2275.	2092.	1211.	573.

$$\ln P(\omega, T) = (a_1 + a_2 T + a_3 / T + a_4 \ln T) + (a_5 + a_6 T + a_7 / T + a_8 \ln T) / (1 - \omega) + (a_9 + a_{10} T + a_{11} / T + a_{12} \ln T) / (1 - \omega^2) \quad (2)$$

where P , is the vapor pressure in kPa, T is the temperature in K, ω is the mass fraction polyol ester in percent, and a_1 to a_{12} are coefficients determined by least square fit.

The empirical equation (2) was compared with another type of empirical equation by Cavestri [10] that was a function of mass fraction of polyol ester, temperature, and saturation pressure of the refrigerant

HFC-404a of the system at the same temperature.

$$P(\omega, T) = \omega P_{sat}(T) + \omega(1 - \omega) \times (a_1 + a_2 T + a_3 T^2 + a_4 \omega + a_5 \omega T + a_6 \omega T^2) P_{sat}(T) \quad (3)$$

P is the vapor pressure in kPa, P_{sat} is the saturation pressure in kPa, which is calculated from equation (1), T is the temperature in K, and ω is the mass fraction of lubricant in percent. Coefficients of equation (1), (2) and (3) are presented in Table 2. In order to investigate the accuracy of these equations, the root-mean-square

Table 4. Root-Mean-Square Deviations between Measured Vapor Pressures and Calculated Vapor pressures from Empirical Equation for 1,1,1,2-Tetrafluoroethane(HFC-134a) + 1,1,1-Trifluoroethane(R-143a) + Pentafluoroethane(R-125)(HFC-125)(4-52-44)(HFC-404a) + Polyol ester

HFC-404a + POE System		
POE 22H mass %	Equation(2)	Equation(3)
0	1.761	0
5.36	2.163	0.937
9.87	2.056	1.527
20.12	0.156	1.387
30.89	2.350	1.684
50.65	5.934	1.489
79.97	2.545	4.978
90.38	2.629	13.519
Average	2.449	3.190

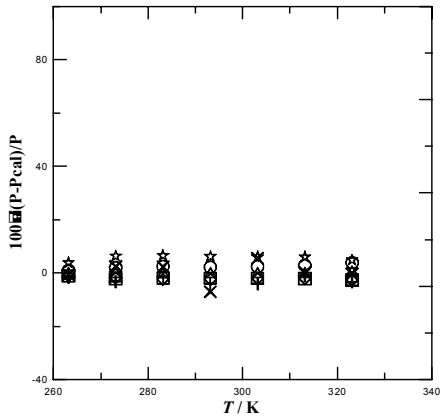


Fig. 4. Percent deviation of the vapor pressure of the HFC-404a + POE system from the empirical correlation Equation (2) : \diamond , 5.36 mass%; \square , 9.87 mass%; \triangle , 20.12 mass%; \circ , 30.89 mass%; \star , 50.65 mass%; $+$, 79.97 mass%; \times , 90.38 mass%.

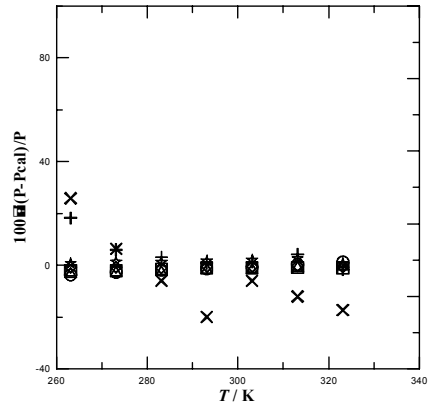


Fig. 5. Percent deviation of the vapor pressure of the HFC-404a + POE system from the empirical correlation Equation (3) : \diamond , 5.36 mass%; \square , 9.87 mass%; \triangle , 20.12 mass%; \circ , 30.89 mass%; \star , 50.65 mass%; $+$, 79.97 mass%; \times , 90.38 mass%.

deviations between measured and calculated vapor pressures from the two empirical equations are presented in Table 4. In Figure 4 and 5, the measured vapor pressures were compared with the calculated pressures from equation (2) and (3). Average root-mean-square deviation of equation (2) and (3) is 2.45 % and 3.19% , respectively, as shown in Table 4.

4. Discussion

4.1. Raoult's Model

As the partial pressure of polyol ester in the vapor

phase is negligible compared to that of HFC-404a, the partial pressure of the HFC-404a in the vapor phase of the HFC-404a + polyol ester system is assumed to be equal to the vapor pressure of the refrigerant HFC-404a. Then, Raoult's model is applied to this system as follows [11]:

$$P_{r,vap} = P_{vap} = x_{ref} P_{sat,ref}(T) \quad (4)$$

$P_{r,vap}$ is the partial pressure of the refrigerant HFC-404a in the vapor phase, P_{vap} is the pressure of the refrigerant in the vapor phase, x_{ref} is the mole

fraction of the HFC-404a in the liquid phase, and $P_{sat,ref}$ is the saturation vapor pressure of the pure HFC-404a.

In Figure 6, percent deviation of the vapor pressure of the HFC-404a + POE system from the Raoult's model is presented. In Table 5, root-mean-square deviations between the measured and predicted values are presented. Below 20 mass % polyol ester, Raoult's law predicted predicted vapor pressure within 10%. However, as the mass percent of polyol ester increased, Raoult's model predicted significantly lower than the measured data. It seems to be that in the binary solution where the difference between the molecular weights of two components is significant, the equilibrium behavior deviates from Raoult's model.

4.2. Flory-Huggins' Model

The molecular weight of the lubricant is an order of magnitude larger than that of the refrigerant. Thus, the equilibrium behavior of refrigerant and lubricant system is not accurately predicted by Raoult's model. Thomas and Pham [1] suggested that the Flory-Huggins' model should be used to predict the equilibrium properties of the refrigerant and lubricant system.

In the Flory-Huggins' model [12, 13], the ratio of the pressure of the system to that of the pure refrigerant is presented in terms of the Flory-Huggins' interaction parameter x as follows [14]:

$$\ln(P/P_{sat,1}) = \ln \phi_1 + \left(1 - \frac{1}{m}\right) \phi_2 + x \phi_2^2 \tag{5}$$

where

$$\phi_1 = \frac{f_1 / \rho_1}{f_1 / \rho_1 + f_2 / \rho_2} \tag{6}$$

$$\phi_2 = \frac{f_2 / \rho_2}{f_1 / \rho_1 + f_2 / \rho_2} \tag{7}$$

$$m = \frac{v_2^L}{v_1^L} \tag{8}$$

P is the vapor pressure of the refrigerant in kPa, $P_{sat,1}$ is the saturation pressure of the refrigerant in kPa, f_i and ρ_i are the mass fraction and density of component i in $kg\ m^{-3}$, respectively, and v_i is the

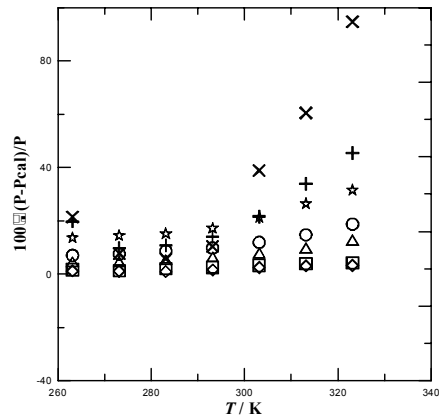


Fig. 6. Percent deviation of the vapor pressure of the HFC-404a + POE system from the Raoult's model: \diamond , 5.36 mass%; \square , 9.87 mass%; \triangle , 20.12 mass%; \circ , 30.89 mass%; \star , 50.65 mass%; $+$, 79.97 mass%; \times , 90.38 mass%.

Table 5. Root-Mean-Square Deviations for Raoult's Model and Flory-Huggins' Model

HFC-404a + POE System		
POE mass%	Raoult	Flory - Huggins
0	-	-
5.36	1.939	3.773
9.87	2.658	6.754
20.12	7.402	13.425
30.89	11.162	17.915
50.65	19.896	21.571
79.97	22.146	8.955
90.38	34.165	18.942
Average	14.195	13.049

liquid specific volume of component i . The Flory-Huggins' interaction parameter x is calculated by the least-square method that minimizes the difference between the measured data and the correlated values. In this study the parameter x is 1.4750 over the temperature range from 263.15 to 323.15 K.

In Table 5, the results from the Flory-Huggins model are compared with those from Raoult's model. Flory-Huggins' model was better than the Raoult's model for the composition range above 50 mass percent of polyol ester. In Figure 6 and 7 percent deviation of the vapor pressure between the measured data and the predicted results from the Raoult's and Flory-Huggins' models are presented along temperature and mass percent of polyol ester. The predicted values from the Flory-Huggins' model agreed within 20% with the measured data over less than 80 mass % of polyol ester.

5. Conclusions

Vapor pressures of the HFC-404a + polyol ester system were measured at 56 points over the temperature range from 253.15 to 333.15 K at 10 K intervals and the mass fraction ranged from 0 to 90 percent of polyol ester.

1. It was found that the vapor pressure of the HFC-404a + polyol ester system decreased as the

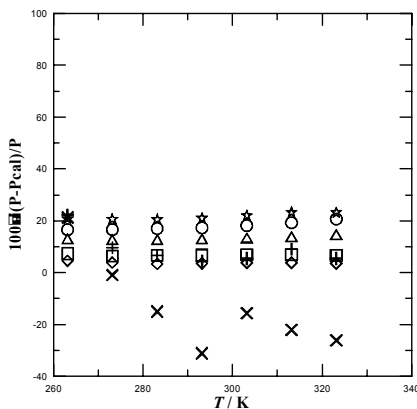


Fig. 7. Percent deviation of the vapor pressure for the HFC-404a + POE system from the Flory-Huggins' model: \diamond , 5.36 mass%; \square , 9.87 mass%; \triangle , 20.12 mass%; \circ , 30.89 mass%; \star , 50.65 mass%; $+$, 79.97 mass%; \times , 90.38 mass%.

polyol ester increased. The effect of the polyol ester on the vapor pressure was negligible within 2% up to 30 mass percent of polyol ester. The effect was significantly larger as the polyol ester increased over 50 mass %. Also it was found that the oil effect becomes significant as temperature increases.

2. Raoult's model and Flory-Huggins model were tested as a data reduction model of vapor pressures of the mixture. It was found that Raoult's law predicted vapor pressure within 10 % over less than 20 mass % of polyol ester. The Flory-Huggins model predicted the vapor pressure in a much better accuracy than the Raoult's model above 50 mass % of polyol ester.
3. Also, an empirical vapor pressure equation (2) was presented in terms of temperature and mass percent of polyol ester. The empirical equation predicts within 10% the vapor pressures of the mixture system over the range of temperature and oil mass %. The correlation is the most convenient working formula to predict the vapor pressure of the mixture system.

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