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Christopher J. Seeton

*University of Illinois at Urbana-Champaign*

Predrag Hrnjak

*University of Illinois at Urbana-Champaign*

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# Thermophysical Properties of CO<sub>2</sub>-Lubricant Mixtures and their affect on 2-Phase flow in small channels (less than 1mm)

Christopher J. SEETON<sup>1</sup>, Pega HRNJAK<sup>2\*</sup>

<sup>1</sup> University of Illinois at Urbana-Champaign  
1206 W. Green Street, Urbana, IL 61801 USA  
Tel: 217-390-5677  
E-mail: [seeton@uiuc.edu](mailto:seeton@uiuc.edu)

<sup>2</sup> University of Illinois at Urbana-Champaign  
1206 W. Green Street, Urbana, IL 61801 USA  
Tel: (217) 333-3115, Fax: (217) 333-1942  
E-mail: [pega@uiuc.edu](mailto:pega@uiuc.edu)  
\*Corresponding Author

## ABSTRACT

This paper will develop an equation based approach for the VLE and VLLE properties of CO<sub>2</sub>-lubricant mixtures. Since carbon dioxide is only partially soluble in many of the working lubricants now available, knowing the separation temperature (miscibility) and phase inversion temperature is of importance in the design of heat exchangers, compressors and auxiliary equipment. Also, of special interest to compressor design engineers is the knowledge of the CO<sub>2</sub>-lubricant liquid viscosity; and for microchannel heat exchanger designers, the interfacial surface tension can be vitally important.

The second part of the part will investigate how these thermophysical properties of CO<sub>2</sub>-lubricant mixtures affect the two-phase flow patterns in tubes less than 1 mm. This will focus on how the mixture vapor to liquid density and viscosity ratios ( $\rho_v/\rho_l$ ,  $\mu_v/\mu_l$ ) and the CO<sub>2</sub>-lubricant mixture surface tension ( $\sigma_m$ ) interact in the formation of flow patterns in tubes less than 1mm in diameter.

## 1. INTRODUCTION

Microchannel heat exchangers are gaining popularity and market share due to increased heat transfer, ease of handling high pressures, reduced refrigerant charge and size of the heat exchangers. Previous research indicates that reduced diameters provide better performance of the heat exchangers due to both increased refrigerant side heat transfer and compactness, which allow for greater space available for the fins on the airside. Current working models ignore the thermophysical properties of the actual working stream, a mixture of refrigerant with lubricant. These mixture thermophysical properties limit progress toward even smaller channels due to an increased pressure drop resulting from increased liquid viscosity and surface tension associated with the lubricant in circulation.

Past authors involved with flow mapping and pressure drop have often postulated that surface tension and contact angle (wettability) play a large role in channels sizes less than 1.0 mm ID (Damianides and Westwater (1988); Graska & Westwater (1986); Coleman and Garimella (1999); Nino (2002); and Tu (2004)). Unfortunately, these authors either have left the phenomena as a hypothesis or have performed experiments with only a single fluid – in which the properties essentially remain constant. While capillary forces are well known in the medical, space and thermosiphon specialties, this phenomenon is not currently incorporated in HVAC heat exchanger correlations.

## 2. THERMOPHYSICAL PROPERTIES OF REFRIGERANT/LUBRICANT MIXTURES

### 2.1 Thermophysical Properties of Refrigerant & Lubricants

Properties of refrigerant / lubricant mixtures found in the literature are solubility, liquid density (specific volume), liquid viscosity, miscibility, and surface tension. Solubility is also referred to as vapor-liquid equilibrium, VLE, or

vapor-liquid-liquid equilibrium, VLLE, if the mixture exhibits immiscibility (critical solution temperature loci) and is the measure of dilution of the refrigerant (or lubricant). Liquid viscosity data collected over a range of refrigerant compositions and combined with solubility data can be found in the literature by its characteristic chart, a Daniel Chart. Liquid density data, while usually only a side measurement in determining the solubility of refrigerant in the lubricant, can usually be found in papers containing viscosity data since it is used to convert from absolute viscosity to kinematic viscosity. Scarce literature references are found for the measurement of surface tension, and its counterpart, contact angle for refrigerant-lubricant mixtures.

Thome (1995), Conde (1996), Mermond et al. (1999), and Medvedev et al. (2004) give appraisals of estimation methods for the mixture thermophysical properties based on pure refrigerant and lubricant information. However, this reviewer advises care with the Mermond et al. paper as some of the reference data was taken out of context in writing their appraisal.

## 2.2 Refrigerant / Lubricant Solubility and Miscibility

Measurements of refrigerant / lubricant properties have been underway since the early 1950s. Little (1952), Bambach (1955), and Albright (1956) were three of the earliest publishers of refrigerant / lubricant solubility. Spauschus (1961) described an automated method of measuring the vapor pressure of refrigerant / lubricant mixtures in an apparatus capable of withstanding 3.5 MPa. In 1963, Spauschus wrote a series of two papers in the ASHRAE Journal on the thermodynamic properties of CFC12-oil and HCFC22-oil solutions. These works were the pioneering steps in describing the thermodynamic properties of refrigerant-lubricant mixtures. Since that time, researchers have developed new apparatuses to measure the solubility of refrigerant-lubricant mixtures, however most still rely on the same principle that the lubricant does not have a significant activity in the vapor phase and thus can be considered to exist only in the liquid due to its extremely low vapor pressure (activity).

While most lubricants are chosen to be completely miscible over the operation range, some trends use an immiscible lubricant mixture in the refrigeration system. These mixtures are described as VLLE mixtures and have two liquid phases where each contains an amount of refrigerant and lubricant. Seeton et al. (2000) report that carbon dioxide exhibits an immiscibility with mineral oil (MO), alkylbenzene (AB), poly- $\alpha$ -olefin (PAO), polyol ester (POE) and polyalkylene glycol (PAG) lubricants at some composition.

More recent authors have chosen to attempt to predict the properties based on activity coefficient models or equations of state (cubic, multi-parameter or corresponding states) models with each based on different mixture models such as van der Waals, Heil, Wilson, or UNIQUAC. Notable authors that have researched the application of equation of state models or activity coefficient models to HFC, CFC and HCFC refrigerants include Hesse and Kruse (1988), Grebner and Crawford (1992), Martz et al. (1996), and Burton and Jacobi (1997). Tsuji et al. (2004) studied the bubble point pressure for CO<sub>2</sub> with decane and CO<sub>2</sub> with PAG lubrication oil from Japan Sun Oil (Sunoco) and correlated their data with the Peng-Robinson equation of state.

## 2.3 Refrigerant / Lubricant Liquid Viscosity

While not primarily concerned with measurements of viscosity and truly ahead of his time, Parmelee (1964) gives an excellent description of the lubricant mixture viscosity as it warms in the suction line in return to the compressor at nearly constant. Daniel et al. (1982) introduce a new style chart that is a combination of the ASTM viscosity chart with lines of refrigerant dilution (composition) combined with solubility data to allow reading of viscosity as a function of temperature and pressure on a single chart. This new chart style has become known as a "Daniel Chart" (although Parmelee's similar charts predate Daniel's by nearly 20 years).

While Daniel et al. used the ASTM viscosity chart to linearize the viscosity of refrigerant-lubricant mixtures at lubricant rich compositions, the ASTM chart has a serious deficiency in linearizing the viscosity of fluids lower than 0.21 cSt (refrigerant rich compositions). The current ASTM Standard charts (D 341-93 (1998)) are based entirely on the work of Wright (1969). Unfortunately, the trouble with Wright's equation is that once viscosity drops below 0.21 cSt, the equation is undefined. Manning (1974) curvefit the Wright formulation and allows for extrapolation beyond the 0.21 cSt limit; however, as the viscosity is reduced below 0.18 cSt, the viscosity is not linear due to the basis on Wright's work. Since most refrigerant viscosities exist in the range below 0.21 cSt, the use of the ASTM charts has not been used in the charting of refrigerant/lubricant mixtures in the range of high refrigerant composition.

## 2.4 Refrigerant / Lubricant Liquid Density

Refrigerant / lubricant mixture density is reported in the literature as part of either solubility or liquid viscosity studies. In either case, the liquid density is normally presented as a by product of the solution for the vapor space correction. Mixture density data is presented in the form of charts, correlations, raw data, or as deviations from ideal mixing.

## 2.5 Refrigerant / Lubricant Surface Tension and Contact Angle

Goswami et al. (1998) measured the surface tension of POEs and MOs with various CFC, HCFC and HFC refrigerants. The “static” surface tension was measured using a Wilhelmy plate method, and the “dynamic” surface tension was measured using a single tube maximum bubble pressure method. While the authors claim to have 10% refrigerant in solution for their tests, all tests were performed at room temperature and atmospheric pressure where refrigerant dilution is minimal.

Feenstra et al. (2001) detail a single tube maximum bubble pressure device to measure the surface tension of volatile fluids including refrigerants. While this device suffers from the same design limitations of other single tube bubble pressure devices, measurements of the surface tension of HFC134a were performed with a stated error of  $\pm 2\%$ . No measurements of refrigerant-lubricant surface tension were reported with this device.

# 3. MEASUREMENT OF THERMOPHYSICAL PROPERTIES

The focus of this work evolves around selecting the proper CO<sub>2</sub> / lubricant mixture for use in micro-channel heat exchangers (250  $\mu\text{m}$  < ID < 3.0 mm). Carbon dioxide benefits from advanced design microchannel heat exchangers due to its thermodynamic properties and operating conditions. Experiments were performed with an commercial ISO 32 weight POE lubricant (2002) and carbon dioxide. The range of the experiments were compositions from neat lubricant to 85% weight percent CO<sub>2</sub> over a temperature range of -30°C to 125°C and pressures up to 140 bar.

## 3.1 Solubility, Miscibility, Density and Viscosity Test System

An experimental test facility has been constructed, tested and calibrated for the measurement of refrigerant-lubricant mixture properties. The design of the loop allows for the continuous circulation of the liquid mixture as well as providing agitation to achieve vapor-liquid equilibrium. The lubricant is first gravimetrically charged ( $\pm 0.02$  grams) into the system and cooled to approximately 10 K below room temperature (to speed refrigerant charging) at which time the refrigerant is gravimetrically charged ( $\pm 0.02$  grams) from a small stainless steel cell to achieve the desired bulk composition in the system (total grams of refrigerant / total grams of refrigerant and lubricant). The bulk composition is not the same as the measured liquid composition as, for safety, there exists a small vapor space to allow for the expansion and contraction of the liquid volume; therefore a vapor space correction must be applied. Once the system has been charged, a small gear pump is activated to circulate the liquid mixture through each of the measurement stages. The bulk mixture pressure is measured in the bulk fluid reservoir with a calibrated pressure transducer (0.10% FS,  $\pm 0.2$  bar). The liquid density is measured at the first measurement stage with a Micromotion massflow meter. The massflow meter measures the liquid density ( $\pm 0.002$  g/cc), circulating liquid temperature ( $\pm 0.5$  K), as well as, the circulating liquid massflow rate ( $\pm 0.10\%$  of rate). The liquid viscosity is measured at the second measurement stage with a high-pressure viscometer (viscosity  $\pm 1.0\%$  FS; temperature  $\pm 0.20$  K). Incorporated into the viscometer housing are two high-pressure sight glasses to allow a viewing stage for liquid miscibility where if bulk mixture pressure is within 1% of the saturated refrigerant pressure, the mixture is observed for possible phase separation. Once passing through the sight glass the liquid mixture flows through the main stage of the temperature control loop where the temperature is set for cooling and heating modes. A simplified schematic is shown in Figure 1. The entire test loop is installed inside a temperature chamber as to keep the temperature variations in the different components below 3 K. Also key to the system operation is that the gear pump applies a pressure rise throughout the massflow meter and viscometer housing to correspond to 10 K subcooling ensures that no vapor bubbles are generated in these sections and that the properties are the liquid properties very near saturation.

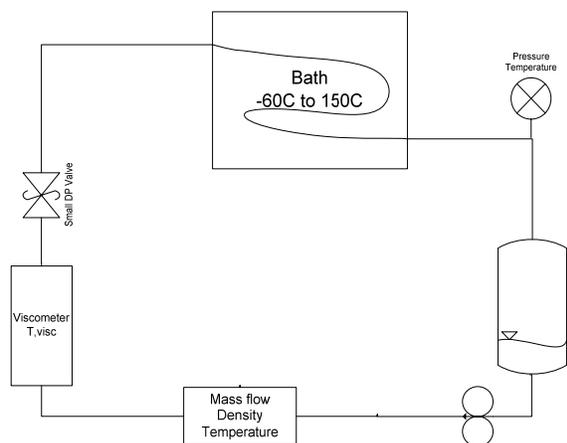


Figure 1. Thermophysical Property Test System Schematic

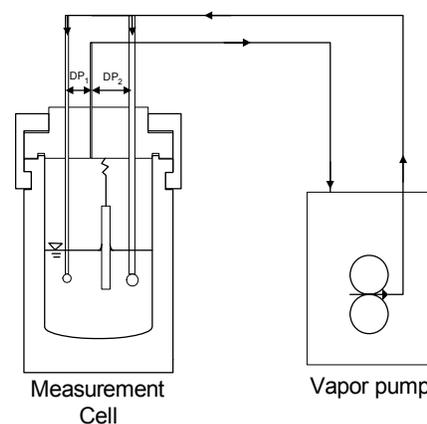


Figure 2. Surface Tension System Schematic

### 3.2 Refrigerant / Lubricant Surface Tension

The surface tension apparatus has been built to utilize a two tube maximum bubble pressure method. The two tube method has advantages over the single tube, in that, knowledge of the fluid density and a precise measurement of the insertion depth are not required if the tubes are at the same depth. The maximum bubble pressure method involves the formation of a vapor bubble using a syringe of known diameter in a liquid. The pressure of the bubble is measured as it grows and the difference in the pressure and  $\theta = 0^\circ$  (semispherical bubble) and  $\theta = 90^\circ$  (bubble initiation) is related to the surface tension through the Laplace equation ( $\sigma = \Delta P * R/2$ ). Since two tubes of different radii are utilized at the same time and under the same conditions, the bubble initiation pressure ( $\theta = 90^\circ$ ) can be calculated and only the maximum pressure in each tube needs to be accurately measured, greatly simplifying the procedure. Figure 2 shows a schematic of the surface tension setup, and Figure 5 shows the results of the surface tension measurements in POE 32 with CO<sub>2</sub>. The composition listed in the legend is an average composition as there are slight variations due to the change in the vapor space.

### 3.3 Refrigerant / Lubricant Solubility, Miscibility, and Density

To correlate the solubility and liquid density of the refrigerant / lubricant mixtures, both empirical equations are used. The empirical equations allow for quick correlation and are convenient for computer calculations. These equations have the following forms:

$$\text{Log}_{10}(P) = a_1 + \frac{a_2}{T} + \frac{a_3}{T^2} + \text{Log}_{10}(w) \left( a_4 + \frac{a_5}{T} + \frac{a_6}{T^2} \right) + \text{Log}_{10}^2(w) \left( a_7 + \frac{a_8}{T} + \frac{a_9}{T^2} \right) \quad (1)$$

$$\rho = b_1 + b_2 T + b_3 T^2 + w(b_4 + b_5 T + b_6 T^2) + w^2(b_7 + b_8 T + b_9 T^2) \quad (2)$$

where the constants  $a_1$  through  $a_9$  and  $b_1$  through  $b_9$  are empirical correlation constants for each equation, Table 1, the temperature,  $T$ , is in Kelvin; the composition,  $w$ , is liquid phase mass fraction of refrigerant; the pressure,  $P$ , is in bar, the density,  $\rho$ , is in g/cm<sup>3</sup>. Figure 3 and Figure 4 chart the mixture density and solubility.

### 3.4 Refrigerant / Lubricant Viscosity

An empirical correlation has also been developed to fit the refrigerant / lubricant viscosity measurements.

$$\begin{aligned} \text{Log}_e \left( \text{Log}_e(v + 0.7 + f(v)) \right) = & c_1 + c_2 \text{Log}_e(T) + c_3 \text{Log}_e^2(T) \\ & + w(c_4 + c_5 \text{Log}_e(T) + c_6 \text{Log}_e^2(T)) \\ & + w^2(c_7 + c_8 \text{Log}_e(T) + c_9 \text{Log}_e^2(T)) \end{aligned} \quad (3)$$

where  $c_1$  through  $c_9$  are empirical correlation constants, the temperature, Table 1,  $T$ , is in Kelvin, the composition,  $w$ , is liquid phase mass fraction of refrigerant, the kinematic viscosity,  $v$ , is in centistokes and  $\text{Log}_e$  is the natural logarithm. Initially,  $f(v)$  was defined as the ASTM standard above in Section 2.3; however, due to the inability to cover the entire composition range from pure lubricant to pure refrigerant, a new function and corresponding chart

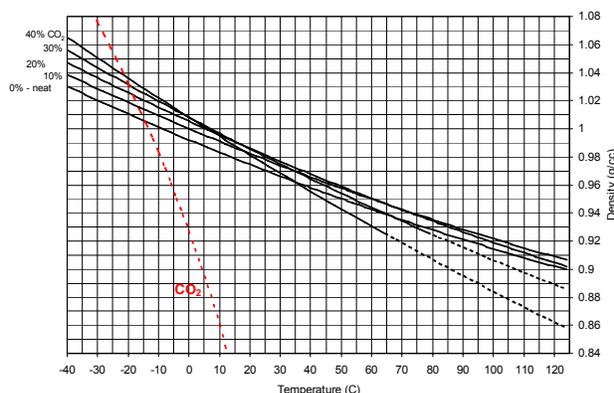
was developed. A new chart and equation have been developed and extends the temperature and viscosity range for hydrocarbons and, for the first time, has the ability to extend to the low viscosity regime of halocarbons and low temperature fluids. The function of viscosity,  $f(\nu)$  becomes:

$$f(\nu) = e^{-\nu} K_0(\nu + \psi) \tag{4}$$

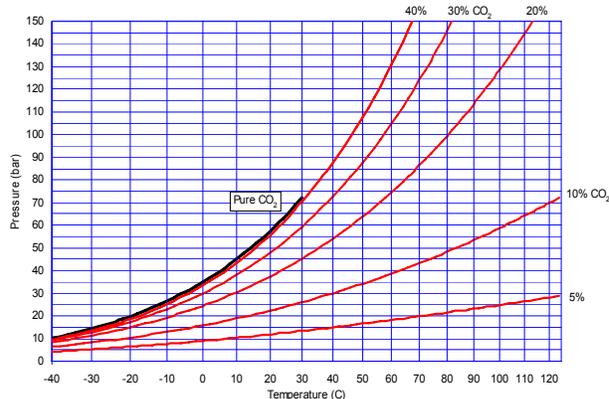
The function  $K_0$  is the zero order modified Bessel function of the second kind. The historical additive constant 0.7, defines  $\psi$  as 1.244068.  $K_0$  can be calculated on most advance engineering calculators and computer software programs (as an example, in Microsoft Excel  $\rightarrow$  BesselK( $\nu + 1.244068, 0$ )). Figure 6 displays the Daniel chart.

**Table 1. Property Fit Coefficients for POE32 with CO<sub>2</sub>**

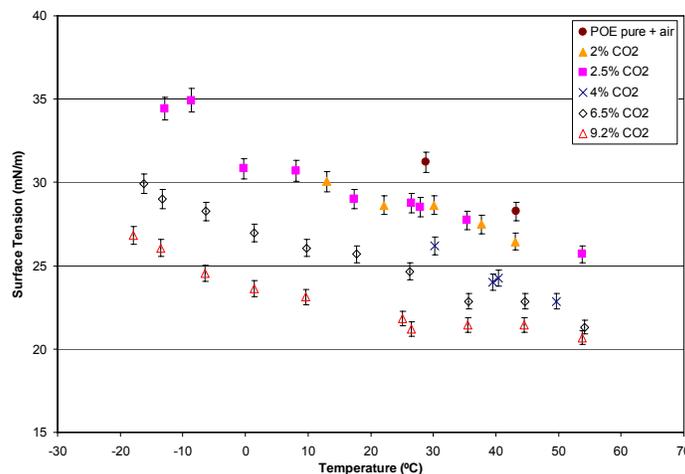
Coefficient	Vapor Pressure Equation (1)	Density Equation (2)	Kinematic Viscosity Equation (3)
1	6.59664	1.33306	3.15665E+01
2	-1.61705E+03	-1.59464E-03	-7.08386
3	7.00550E+04	1.26792E-06	3.14380E-01
4	3.88634	1.67745E-02	-3.40071E+02
5	-1.46846E+03	2.77192E-04	1.28631E+02
6	1.19438E+05	0	-1.22477E+01
7	3.40692E-01	8.57576E-01	1.30003E+03
8	-2.54276E+02	-3.61583E-03	-4.69484E+02
9	2.11410E+04	0	4.22881E+01
$\sigma$	0.999960	0.9977	0.9960



**Figure 3. Density of POE 32 with CO<sub>2</sub>**



**Figure 4. Solubility of POE32 with CO<sub>2</sub>**



**Figure 5. Surface Tension of POE 32 with CO<sub>2</sub>**

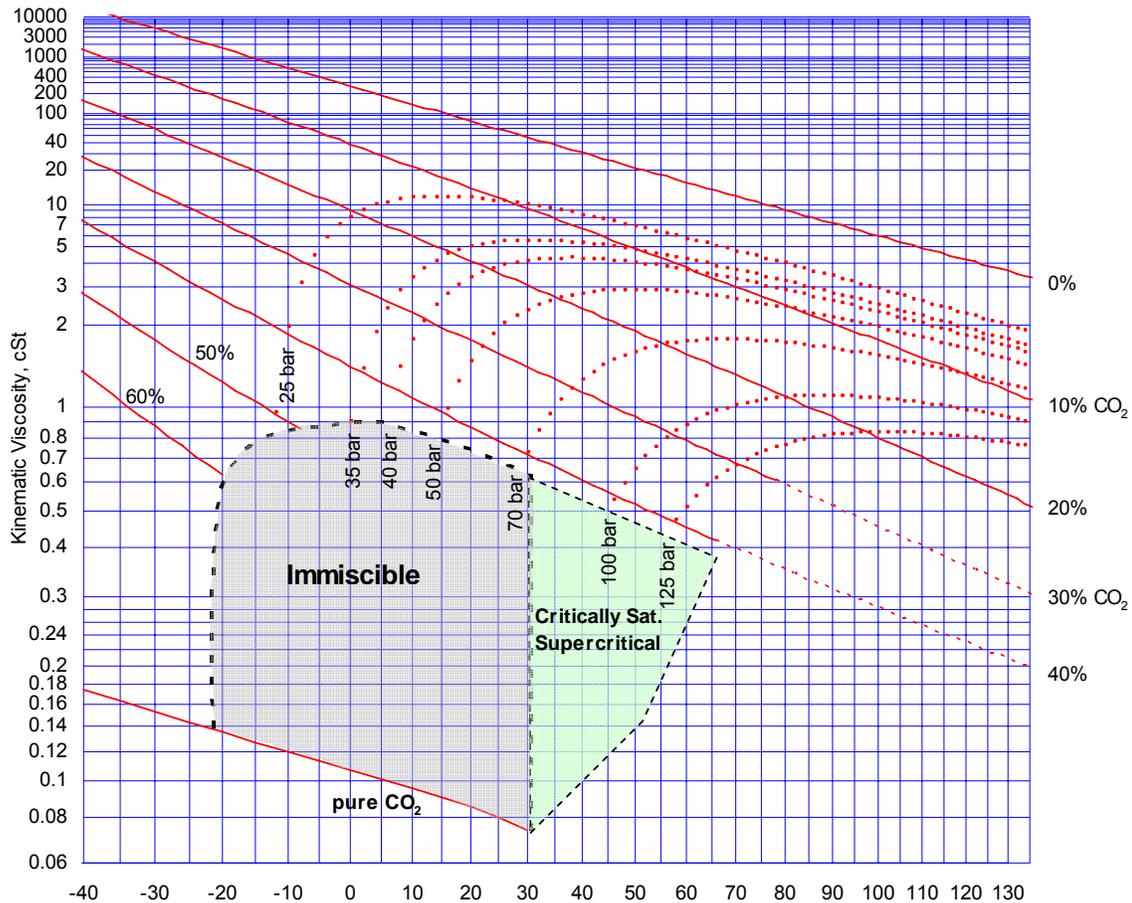


Figure 6. PvTx-v of POE32 with CO<sub>2</sub> (Daniel Chart)

#### 4. EFFECT ON TWO-PHASE FLOW

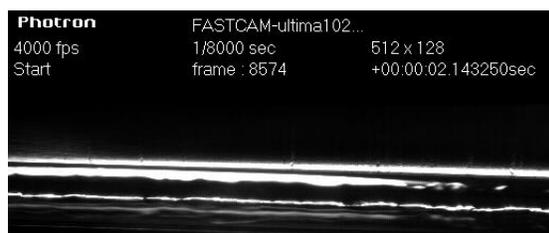
Pettersen (2001) concludes that the “unique” properties of CO<sub>2</sub> such as high density ratio, low liquid viscosity and low surface tension cause the flow patterns to be dominated by intermittent and annular flow with good wetting of the tube wall. Stratified flow was also not seen in his observations. With the exception of low temperature applications, CO<sub>2</sub> is boiled at much higher reduced pressures and temperatures than more typical HFC and HCFC refrigerants. Carbon dioxide’s vapor density ratio ( $\rho_v/\rho_l$ ), viscosity ratio ( $\mu_v/\mu_l$ ), and surface tension are also dramatically different than those of conventional refrigerants operating at the same temperatures.

Recent flow regime maps use the more natural mass flux and vapor mass quality as the coordinate system. Kattan et al. (1998) continued the work of Steiner (1997) and modified the historical Taitel and Duckler flow pattern map to this coordinate system. While a flow pattern map on this coordinate system is convenient, the Kattan et al. flow map’s basis on the Taitel and Duckler flow map limits its use to channels in which surface tension effects are not significant. Coleman and Garimella (2003) and Thome (2005) both indicate that the Kattan et al. map is not valid when extended to use with refrigerants in microchannels. Thome uses the confinement number criteria,  $Co$ , as defined by Kew and Cornwell (1997), to distinguish between microscale flow and macroscale flow for diabatic flow.

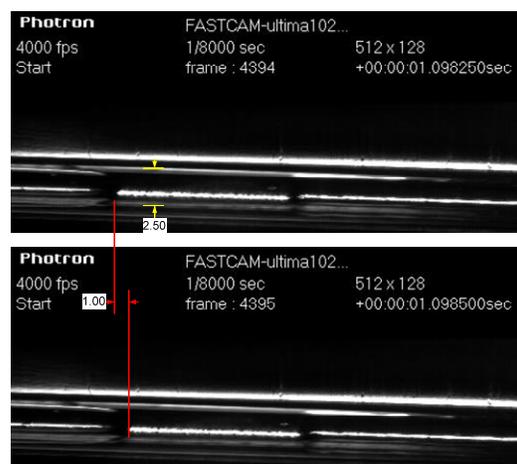
$$Co = \left( \frac{\sigma}{gD^2(\rho_l - \rho_v)} \right)^{0.5} > 0.5 \quad (5)$$

Where  $\sigma$  is the surface tension,  $g$  is the gravitational constant,  $D$  is the inner diameter of the tube, and  $\rho_l$  and  $\rho_v$  are the liquid and vapor densities, respectively. This criterion put forth by Kew and Cornwell indicates that for confinement number  $Co > 0.5$ , gravitational forces are negligible compared to surface tension forces and supports the conclusions put forth by Brauner and Moalem Maron (1992) who used the Eotvös Number,  $Eö$ .

Currently, work is progressing to produce a flow pattern map utilizing pure  $CO_2$  and  $CO_2$ /lubricant mixtures based on their mixture properties. Bubbly flow, intermittent flow (slug) and annular flow have been observed over a range of test conditions. Figure 7 shows an annular flow at a quality of approximately 0.3 and massflux of  $135 \text{ kg/m}^2\text{-sec}$ . Figure 8 shows intermittent flow of the pure  $CO_2$  flow at a quality of approximately 0.15 and massflux of  $135 \text{ kg/m}^2\text{-sec}$ .



**Figure 7. Annular flow of  $CO_2$**   
( $x \sim 0.3$ ,  $G = 135 \text{ kg/m}^2\text{-sec}$ ,  $T = 19^\circ\text{C}$ ,  $0.50 \text{ mm}$  tube)



**Figure 8. Intermittent flow of  $CO_2$**   
( $x \sim 0.15$ ,  $G = 135 \text{ kg/m}^2\text{-sec}$ ,  $T = 19^\circ\text{C}$ ,  $0.50 \text{ mm}$  tube)  
Slug velocity  $\sim 0.8 \text{ m/sec}$

## 5. CONCLUSIONS

In carbon dioxide systems utilizing microchannel heat exchangers, the selection of a proper lubricant is not just a compressor dependant selection as the selection of an inferior lubricant may have detrimental consequences in the heat exchangers. Engineers desiring to use these mixtures should have a full understanding of the  $CO_2$ /lubricant mixture properties as these properties can be dramatically different than those seen in conventional systems.

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