Foaming Characteristics of Refrigerant/Lubricant Mixtures

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Abstract

The air-conditioning and refrigeration industry has moved to HFC refrigerants which have zero ozone depletion and low global warming potential due to regulations on CFC and HCFC refrigerants and concerns for the environment. The change in refrigerants has prompted the switch from mineral oil and alkylbenzene lubricants to polyolester-based lubricants. This change has also brought about a desire for lubricant, refrigerant and compressor manufacturers to understand the foaming properties of alternative refrigerant/lubricant mixtures, as well as the mechanisms which affect these properties.

The objectives of this investigation are to experimentally determine the foaming absorption and desorption rates of HFC and blended refrigerants in polyolester lubricant and to define the characteristics of the foam formed when the refrigerant leaves the refrigerant/lubricant mixture after being exposed to a pressure drop.

The refrigerants being examined include baseline refrigerants: CFC-12 (R-12) and HCFC-22 (R-22); alternative refrigerants: HFC-32 (R-32), R-125, R-134a, and R-143a; and blended refrigerants: R-404A, R-407C, and R-410A. The baseline refrigerants are tested with ISO 32 (Witco 3GS) and ISO 68 (4GS) mineral oils while the alternative and blended refrigerants are tested with two ISO 68 polyolesters (Witco SL68 and ICI RL68H).

Initially, properties of the pure lubricants were measured at room temperature to establish basis for possible comparison amongst the refrigerant/lubricant pairs. The properties that were measured include density, viscosity and static surface tension. Viscosities of the four lubricants were measured using a cone-and-plate viscometer equipped with a temperature bath. Equilibrium surface tension measurements were taken using the Wilhelmy plate method. Theoretically, the lower the equilibrium surface tension, the less the work that is needed to expand that surface (i.e. from aeration) and thus, form foam bubbles from that liquid.

Experimentally, it was found that the mineral oils have a density of approximately 0.8 g/ml while the two polyolesters (POEs) had densities closer to 0.9 g/ml. The three ISO 68 lubricants (Witco 4GS, Witco SL68 and ICI RL68H) all exhibited viscosities around 100 cp, but the most important result from these trials was that the Witco POE appeared to be 20 cp more viscous than the ICI POE at room temperature. In terms of static surface tension, both mineral oils had similar values (31 dynes/cm) that were greater than those of the POEs (29 dynes/cm).

Dynamic surface tension measurements of the pure lubricants and of the refrigerant/lubricant pairs were experimentally determined using the maximum bubble pressure technique. This method involves injecting a gas phase (air for dynamic surface tension, refrigerant for dynamic interfacial tension) into a liquid (lubricant) while monitoring the pressure inside the bubbles formed. In
essence, curves for dynamic surface tension versus bubble lifetime (surface age) are established and compared. Theoretically, the greater the reduction in dynamic surface (or interfacial) tension, the greater the amount of foam produced.

The dynamic surface tension tests with the pure lubricants (with air injection) revealed that the mineral oils behave quite differently compared to each other. Each deviates more with increasing bubble frequency. The polyolesters, however, exhibit similar dynamic behavior at all bubble rates (0-40 bubbles/sec).

Dynamic surface tension measurements of the baseline refrigerant/lubricant mixtures (10% refrigerant) using air injection revealed that both R-12 and R-22 reduce the dynamic surface tension curves the same amount for 3GS or 4GS. However, the reduction was considerably less pronounced for the 3GS mineral oil than the 4GS mineral oil.

The baseline dynamic interfacial tension experiments (oils aerated with refrigerant gas) revealed dynamic differences between the two mineral oils. 3GS exhibited lower surface tension values than 4GS at all bubble rates using R-12 as the injected gas phase. In addition, it was found that the dynamic interfacial tension of R-12/mineral oil mixtures was already lowered by 2 dyne/cm at very low bubble frequencies, differing from dynamic surface tension data using air as the injected gas phase.

The single-component HFCs revealed some unique behavior in that R-125 and R-143a curves were higher than that of the dynamic surface tension curve for POE injected with air (i.e. the control). This condition does not favor foaming as the influence of refrigerant appears to increase the amount of work required to expand the surface of the POE in these cases. R-134a and R-32, on the contrary, lowered the dynamic interfacial tension curve and thus, appear to favor foam production more than R-125 and R-143a with POE lubricants.

The blended HFCs exhibited less deviant behavior from the control curve as both R-404A and R-407C curves were within 1 dyne/cm over a bubble frequency range from 0-30 bubbles/sec. R-410A exhibited a higher dynamic interfacial tension by approximately 5 dyne/cm over the aforementioned bubble frequency range. In short, none of the blended HFC refrigerants appear to favor the foaming process from an interfacial standpoint.

Foam stability and foamability measurements were performed with a variety of techniques. The first method, namely aeration, was used to perform tests on the baseline CFC and HCFC refrigerants with the corresponding mineral oils. The HFC/polyolester pairs required a different approach to producing foam, in that a pressure drop was required to form the foams that could be measured and compared. These methods were performed to study foaming ability and foam stability of the various refrigerant/lubricant pairs.

The foamability and foam stability tests for the baseline pairs were performed using 30 ml of lubricant aerated with air at standard room conditions, using flow rates between 250 and 1000 ml/min. While the aeration column (with air injection) revealed that the 4GS/R-22 pair produced the largest amount of foam and the most persistent foam column, it also exposed a significant fact concerning the R-22 trials with both 3GS and 4GS lubricants. In essence, both mineral oils were tested using a variety of air flow rates, however both lubricants exhibited maximum foaming ability and foam stability for one of the middle flow rates tested. This suggests that there is an optimum amount of refrigerant needed to produce the most or longest lasting foam. It should also be noted
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that R-22 exhibited more foaming than R-12. In terms of bubble size, the bubbles produced were small (on the order of hundreds of microns), corresponding to slow drainage rates and higher foam stability.

The HFC pressure-release foaming apparatus was used to test the HFC/POE mixtures. The experimental variables considered and varied during these experiments are pressure drop (20, 50 and 70 psi), time of pressure drop (10, 30, 60 and 180 sec) and refrigerant/lubricant mixture ratio (1:1, 3:1 and 6:1). The trials revealed that, in terms of foam stability, that none of the HFC/POE pairs were stable under any of the conditions tested (foam lifetimes did not last longer than 10 seconds for any given trial). In addition, it was found that the 60 and 180 second pressure drops yielded little foam height (less than 1 cm). Thus, foam stability and foamability due to “slow” pressure drops were not considered to be valid comparative parameters in this study. However, the trials did reveal that the R-134a/POE system was the most foamy system for “fast” (10 and 30 sec) pressure drops and low (20 psi) pressure drops, and this definitely corresponds to the dynamic interfacial tension data, as R-134a showed the greatest reduction in dynamic surface tension. In addition, bubble sizes were generally larger (on the order of 1 mm) than those observed for the baseline aeration study.

The ASTM standard test method for foaming was also performed on the HFC/POE systems. This method involves the injection of air through a gas diffuser. However, since this method does not utilize a pressure drop to induce foaming, the HFC/POE pairs did exhibit any foaming with this apparatus.

Desorption rates of the HFC/POE pairs were measured using a weight-loss method. In essence, before and after the pressure-release foaming trials were conducted, the mass of the HFC/POE mixture was measured and compared. Desorption rate was taken as the amount of refrigerant lost per time of the pressure drop. It was found that the greater the refrigerant/lubricant ratio and the slower the pressure drop, the greater the desorption rate.

Absorption of refrigerant in the lubricant was studied by a weight gain method. An open container of lubricant in a pressure vessel is continuously weighed by a force transducer when the vessel is filled with a known refrigerant under predetermined pressure and temperature conditions. The change in the weight is plotted as a function of time and absorption rate is calculated. R-134a was shown to have the greatest absorption rate in POE lubricant at both room temperature and 34°C for pressure drops of 20, 50 and 70 psi.

In conclusion, the surface properties, foaming characteristics, absorption and desorption rates measured support each other in that slow absorption rates seem to correspond to a smaller reduction in surface tension, and thus, lower foamability. Greater desorption rates are also characteristic of these systems as the refrigerant leaves the mixture before it can effectively enhance the foaming potential of the mixture. The R-143a/POE system was indicative of these properties. On the contrary, R-134a/POE exhibited a fast absorption rate and a slow desorption rate, thus being able to reduce dynamic interfacial tension of the system the greatest, allowing for the greatest foamability.

Comparing the baseline pairs with the HFC/POE pairs, R-134a was the only HFC that exhibited any kind of similarity in terms of dynamic interfacial tension reduction. Foamability and foam stability were considerably greater for the baseline pairs, albeit for a completely different method for foam production.
Objectives

- To define the characteristics of the foam formed when refrigerant leaves solution with a lubricant after being exposed to a pressure drop

- To determine the rate at which an HFC refrigerant leaves solution with a polyolester (POE) when exposed to a pressure drop

- To determine the rate at which POE lubricants absorb HFC refrigerant
Measurement of Properties

• Lubricant properties
  a. Density
  b. Viscosity
  c. Surface tension

• Refrigerant/lubricant mixture properties
  a. Static surface tension
  b. Dynamic surface tension

• Foaming characteristics
  a. Aeration (foam column, ASTM)
  b. Pressure drop induced foaming

• Desorption rates

• Absorption rates
## Refrigerant-Lubricant Pairs

<table>
<thead>
<tr>
<th></th>
<th>REFRIGERANT</th>
<th>LUBRICANT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Baseline Pairs</strong></td>
<td>(CFC) R-12</td>
<td>ISO 32, 68 mineral oil</td>
</tr>
<tr>
<td></td>
<td>(HCFC) R-22</td>
<td>ISO 32, 68 mineral oil</td>
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<tr>
<td><strong>Single-component HFCs</strong></td>
<td>R-32</td>
<td>ISO 68 polyolester</td>
</tr>
<tr>
<td></td>
<td>R-125</td>
<td>ISO 68 polyolester</td>
</tr>
<tr>
<td></td>
<td>R-134a</td>
<td>ISO 68 polyolester</td>
</tr>
<tr>
<td></td>
<td>R-143a</td>
<td>ISO 68 polyolester</td>
</tr>
<tr>
<td><strong>Blended HFCs</strong></td>
<td>R-404A</td>
<td>ISO 68 polyolester</td>
</tr>
<tr>
<td></td>
<td>R-407C</td>
<td>ISO 68 polyolester</td>
</tr>
<tr>
<td></td>
<td>R-410A</td>
<td>ISO 68 polyolester</td>
</tr>
</tbody>
</table>
Experimental Conditions

- **Foaming characteristics & desorption rates**
  a. Baseline test with
     - CFC-12 and ISO 32, 68 mineral oil
     - HCFC-22 and ISO 32, 68 mineral oil
  b. HFC refrigerant with ISO 68 POE
  c. Refrigerant/lubricant mixture subjected to pressure drop
     - Three pressure drops (20, 50, 70 psi)
     - Two “rapid” pressure drops (10 and 30 seconds)
     - Two “slow” pressure drops (1 and 3 min)
     - Three refrigerant : lubricant ratios (1:1, 3:1, 6:1)

- **Absorption rates** (for pressure drops of 20 and 50 psi)
  a. Refrigerant/lubricant at room temp (24°C)
  b. Lubricant 10°C warmer than refrigerant
  c. Lubricant 20°C warmer than refrigerant
  d. Refrigerant/lubricant at 40°C
  e. Refrigerant/lubricant at -10°C
Lubricant Viscosity Measurements: Cone & Plate Viscometer

Diagram:
- Digital Display
- Temperature Bath (25°C)
- Spindle (rotating)
- Cone
- Cup (25°C)
- Plate
- Lubricant Sample (2 ml)
- Lubricant
- Cone
- Angle < 4°
Lubricant Surface Tension Measurements: Wilhelmy Plate Method

![Diagram of the Wilhelmy Plate Method]

- **Tensiometer**
- **Beam (adjustable)**
- **Platinum Wire**
- **Platinum Plate**
- **Liquid Surface**
- **Lab Jack**

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**Contact angle**
$\theta = 0^\circ$

- **Surface Tensiometer**
- **Platinum wire**
- **Platinum plate**
- **Liquid surface**

---

- **Weight**
# Lubricant Property Data Summary

<table>
<thead>
<tr>
<th>Lubricant</th>
<th>Density (g/ml)</th>
<th>Viscosity (cp)</th>
<th>St. Dev.</th>
<th>Surface Tension (dynes/cm)</th>
<th>St. Dev.</th>
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</thead>
<tbody>
<tr>
<td>Baseline Mineral Oils</td>
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<td></td>
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<tr>
<td>Synthetic Polyolesters</td>
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<tr>
<td>ISO 68A</td>
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<td>111.2</td>
<td>2.2</td>
<td>28.9</td>
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<tr>
<td>ISO 68B</td>
<td>0.88</td>
<td>90.3</td>
<td>2.3</td>
<td>28.6</td>
<td>0.06</td>
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</tbody>
</table>

- all tests performed at 25°C, 1 atm
- POEs are more dense than mineral oils
- ISO 68A POE has a significantly higher viscosity than the ISO 68B lubricant
- POEs have lower static surface tension than mineral oils
Measurement of Dynamic Surface Tension: Maximum Bubble Pressure Method

- MBP is measured for various bubble lifetimes

- Capillary submerged at a constant depth for all trials

- MBP inside a bubble occurs when bubble is perfectly hemispherical

- Differential pressure signal reads voltage, which is then related to surface tension
Lubricant Dynamic Surface Tension at 25°C

- Mineral oils behave differently (each vary with bubble frequency)
- Polyolesters have similar curves (at all bubble frequencies)
- 3GS sample, due to its lower viscosity, can resolve bubbles at higher bubble rates
Dynamic Surface Tension of Baseline Refrigerant/Lubricant Mixtures

- Both refrigerants behave similarly for each mineral oil
- Lowering of dynamic surface tension is less pronounced for 3GS than for 4GS (hence, 4GS should form foam easier than 3GS)
Dynamic Interfacial Tension: Baseline Oils Aerated with R-12

- Dynamic interfacial tension of refrigerant gas with oil is already lowered by 2 dynes/cm at very low bubble frequencies (which differs from dynamic surface tension with air bubbles)

- 3GS exhibits lower values than 4GS (agreeing with previous data on baseline mixtures)
Dynamic Interfacial Tension: POE Lubricant with HFCs

- R-134a and R-32 favor foaming (lowering of dynamic IFT)

- R-125 and R-143a appear to disfavor foaming (increase dynamic IFT)

- R-134a should produce foam easier than other single-component HFCs tested
Dynamic Interfacial Tension: POE Lubricant with HFC Blends

- R-404A and R-407C appear to have little influence on the dynamic IFT of POE lubricant

- R-410A exhibits higher dynamic IFT than air or other HFC blends

- None of the HFC blends appear to favor foaming based on the MBP experiments
Foam Column Apparatus

- Refrigerant aerated through fritted disk filter
- 30 ml lubricant, 200-1000 ml/min refrigerant
- Flowrate maintained until steady-state foam height reached then shut off
Baseline Foaming using Aeration Column

30 ml 4GS & R-22

30 ml 3GS & R-22

- Each case (3GS and 4GS) exhibits maximum foaming ability and foam stability for the middle flow rate tested, suggesting that there is an optimum amount of refrigerant needed to produced the most/longest lasting foam
Further Conclusions from Baseline Aeration Experiments

• 4GS/R-22 produced the largest amount/most persistent foam column (supported by dynamic surface tension data)

• R-22 exhibited more foaming than R-12

• Average bubble size is small for stable foam columns. This corresponds to slower drainage rate and higher foam stability.
HFC Pressure-release Foaming Apparatus

Diagram:
- 1/4" Copper Tubing
- Isolation Valve (D)
- Pressure Gauge
- Toggle Valve (B)
- Coupling Adapter
- Transient Valve (C)
- Safety Valve (A)
- Safety Tube (with graduations)
- HFC/POE Mixture
- Stirring Bar
- Magnetic Stirrer
- Vacuum Pump
- Liquid Nitrogen
- HFC Recovery Tube
Foamability for 1:1 HFC/POE Mixtures Subjected to 20 psi Pressure Drops

- R-134a is clearly the most foam producing HFC with POE at a 1:1 ratio (which agrees with the lowest value of dynamic IFT of R-134a)

- 10 sec drop yields greatest foamability (for all trials, including those not pictured here)

- R-32 could not be tested at 20 psi due the constraints of the apparatus and R-32’s physical properties (it immediately built pressures of 30+ psi without agitation)
Further Conclusions from HFC Pressure-Release Foaming Trials

- In general, foam lifetimes were less than 10 seconds (i.e. *not* a valid comparative parameter)

- R-32, due to its high vapor pressure, could only be tested at pressure drops of 50 and 70 psi

- “Slow” pressure drops (60 and 180 sec) yielded little foam height (less than 1 cm)

- In general, the average bubble sizes of the HFC/POE foams were larger than those for the baseline aeration tests

- All trials conducted using a 6:1 HFC:POE ratio (for R-134a and R-143a) yielded maximum foam heights that equaled the height of the pressure tube (28+ cm)
ASTM Standard Test for Foaming

- Standard conditions:
  - 1000 ml graduated cylinder
  - water bath immerses cylinder 90%
  - constant flowrate
  - 200 ml lubricant
  - suitable gas diffuser

- 1) Sample tested at 75°F
- 2) Sample tested at 200°F, then cooled to 75°F

- NO FOAMING OBSERVED FOR HFC/POE pairs
Initial Desorption Rates for R-134a, 20 psi Pressure Drop, Mixture Volume 10-15 ml

- Desorption rates increase linearly for the 30 second drop; irregularly for the 10 second drop

- The rate of initial desorption at 10 sec drop time is 7, 2.5 and 3 times greater than the rate at 30 sec drop time for 1:1, 3:1 and 6:1 refrigerant : lubricant ratios, respectively
Measurement of Absorption Rates

- Based on the weight gain method
- Apparatus
- Calibration
- Procedure and measurements
- Results and discussion
Absorption Rate Data

- Experimental results of the absorption of HFC refrigerants and their blends in POE lubricant at room temperature (24°C)

<table>
<thead>
<tr>
<th>Refrigerants</th>
<th>Pressure (psig)</th>
<th>Solubility, X (%)</th>
<th>Absorption rate, k (min⁻¹)</th>
<th>Initial absorption rate (% / min⁻¹)</th>
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<tbody>
<tr>
<td>R-32</td>
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<td>0.06296</td>
<td>0.1005</td>
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<td>50</td>
<td>2.718</td>
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Absorption Rate Data

- Experimental results of the absorption of HFC refrigerants and their blends in POE lubricant at 34°C

<table>
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<tr>
<th>Refrigerants</th>
<th>Pressure (psig)</th>
<th>Solubility, X (%)</th>
<th>Absorption rate, k (min⁻¹)</th>
<th>Initial absorption rate (% / min⁻¹)</th>
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<tr>
<td>R-32</td>
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</table>
Conclusions & Data Summary

Baseline (H)CFC/mineral oil

- Dynamic surface tension reduction
  \[3\text{GS/R-12} \sim 3\text{GS/R-22} < 4\text{GS/R-12} \sim 4\text{GS/R-22}\]

- Foamability
  \[3\text{GS/R-12} \sim 3\text{GS/R-22} < 4\text{GS/R-12} \sim 4\text{GS/R-22}\]

- Foam stability
  \[4\text{GS/R-12} < 3\text{GS/R-12} \sim 3\text{GS/R-22} < 4\text{GS/R-22}\]

Alternative HFC/POE

- Dynamic surface tension reduction
  \[R-410A < 143a < R-125 < R-404A \sim R-407C < R-32 < 134a\]

- Foamability
  \[R-32 < R-125 \sim R-143a < 134a\]

- Absorption rate
  \[R-32 < 143a < R-410A < R-404A < R-125 < R-407C < 134a\]

- Desorption rate
  \[134a < R-125 \sim R-32 < 143a\]
## Comparison of Pairs of Interest

### Dynamic Surface Tension Reduction:

<table>
<thead>
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<th>Comparison</th>
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<tbody>
<tr>
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<td></td>
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### Foamability:

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### Foam Stability:

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Relating Surface Tension, Absorption, Desorption and Foaming

- lubricant molecule
- refrigerant molecule

**System A**
- Rate of Absorption: SLOW
- Reduction in ST: SMALLER
- Foamability: LOWER
- Rate of Desorption: HIGHER

**System B**
- Rate of Absorption: FAST
- Reduction in ST: HIGHER
- Foamability: HIGHER
- Rate of Desorption: SMALLER
Summary & Conclusions

• Baseline tests
  a. Dynamic surface (interfacial) tension affected by viscosity differences (3GS vs. 4GS)
  b. Viscosity affected foamability and foam stability (4GS produced more foam and was more stable than 3GS)
  c. R-22 lowered dynamic surface tension more than R-12, also produced more foam/persisted longer in aeration study

• Alternative refrigerants
  a. From the dynamic interfacial tension tests, R-134a (especially) and R-32 favored foaming while R-125, R-143a and the blends did not
  b. No foaming was observed with ASTM standard (no pressure drop)
  c. Pressure-release foaming yielded very little stability for HFC/POE pairs
  d. R-134a/POE mixtures were the most foamable
  e. Foam only observed for “fast” pressure drops and not for “slow” pressure drops
Future Work

- Utilization of various surfactant additives to produce and stabilize (i.e. control) HFC/POE foams (foamability vs. foam stability)
  a. Surfactants are non-ionic (non-corrosive) and oil-soluble (eg. Tween 80 series)
  b. Previous work has proven that as little as 1% surfactant, by weight, added to alternative refrigerant mixtures was enough to produce HFC/POE foams with both high foamability and high foam stability
  c. Foams were stabilized for 6+ minutes at room temperature and pressure using the aforementioned aeration column (i.e. no pressure drop required)

- Use of simple POE lubricants with less components (to study interfaces on a molecular level)

- To carry out heat transfer efficiency in the presence and absence of foam