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Recent Advances in Solubility, Miscibility and Material Compatibility Studies for R134a and R404A non-flammable low GWP alternative

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ABSTRACT

In commercial refrigeration, high GWP refrigerants such as R134a and R404A are huge contributors of release of HFC's to the environment. Because of increasing concerns about climate change, various environmental regulations have been proposed to phase out these refrigerants. The transition to new age environmentally friendly low GWP refrigerants R471A, requires an understanding of refrigerant/lubricant properties and material compatibility with refrigerants. In this paper, the solubility and miscibility of R471A with polyol ester-based oil (POE32) is experimentally investigated at over a temperature range of -30°C to 110°C. Further this paper also presents the compatibility results of new refrigerants and common materials used in refrigeration and air-conditioning systems.

Keywords: R471A, POE32, Solubility, Miscibility, Compatibility

1. INTRODUCTION

Refrigerants are the vital working fluid in a vapor compression refrigeration system being used in refrigeration, air-conditioning and heat pump equipment. At present, hydrofluorocarbons (HFC's), are used as refrigerants due to their safety and excellent energy performance. However, HFC's are also potent greenhouse gases owing to their high Global Warming Potential (GWP) and are under phase down process worldwide due to the rising concerns of climate change. To achieve the HFC's phase down targets, use of refrigerants with high GWP are being regulated leading towards a search for lower GWP refrigerants (Nair, 2021). Hydrofluoroolefins (HFO's), hydrocarbons (HC's) and natural refrigerants like CO₂ emerged as potential substitutes due to lower GWPs and acceptable thermodynamic properties (Heredia-Aricapa et. al., 2020; Gao et. al., 2021). Adoption and use of HC's is restricted due to their higher flammability. High operating pressures and lower energy efficiencies, specially at elevated ambient conditions (Purohit et. al., 2020) limits the use of CO₂.

R471A is a non-flammable environmentally preferable refrigerant with GWP less than 150. R471A is a low-pressure refrigerant suitable for a sustainable replacement of both R134a and R404A in medium temperature applications. Being low pressure, non-flammable and energy efficient, R471A is a business-as-usual refrigerant which eliminates specialized skills and capabilities for handling, transportation and services. It drives lower leak rates which leads to lower direct emissions and consumes lower energy than R404A which helps to reduce indirect emissions.

The transition to new age environmentally friendly refrigerants requires understanding of lubricant and material compatibility with refrigerants. For proper functioning of compressor, the oil requires to lubricate the compressor bearings and other moving parts along with minimizing gas leakage during compression of refrigerant. The dissolution of refrigerant in oil known as solubility affects the oil properties. A high degree of solubility of refrigerant in the oil decreases the overall oil viscosity affecting the lubrication of moving parts and on the other hand low solubility or partial immiscibility (liquid-liquid separation) can result in oil accumulation in the system. An optimum solubility of oil and refrigerant is preferred for proper compressor lubrication, maximum heat transfer performance in the evaporator and lubricant return to the compressor.

This article presents first in kind experimental study to evaluate interactions between R471A, lubricant (POE ISO 32 3MAF) and common materials used in HVAC&R systems. The first section of the paper discusses properties of materials tested followed by detailed description of the test setup, test procedure and test results. Results to characterize the solubility (VLE) and miscibility of R471A with POE ISO 32 3MAF lubricant is presented followed by compatibility of R471A and POE ISO 32 3MAF lubricant with different materials used throughout the system. The solubility, miscibility and material compatibility data presented for R471A is also compared to that of R134a and R404A.

2. MATERIALS

2.1 Materials

The solubility and miscibility of R471A with POE32 is performed and compared with R134a and R404A with POE32 whereas the material compatibility studies of R471A is compared with R134a. Table 1 shows the blend compositions and refrigerant properties of all three refrigerants. R471A is a non-flammable and non-toxic (ASHRAE class A1) HFO blend of R1234ze(E), R227ea and R1336mzz(E). It has a Global Warming Potential (GWP, AR5) of 148 which is 90% lower than R134a. The solubility, miscibility and material compatibility studies for R471A, R134a and R404A were performed with polyol ester oil of 32cst viscosity grade. Table 2 shows typical physical and chemical properties of POE32 lubricant. The material compatibility studies focus on the effect of refrigerant and refrigerant/oil on elastomers and plastics. The list of elastomers and plastics used in material compatibility studies are listed in Table 3.

Table 1: Properties of refrigerants

Refrigerant properties	R471A	R134a	R404A
Chemical composition	R1234ze/R227ea/ R1336mzz(E) (78.7%/4.3%/17%)	R134a (100%)	R125/R143a/ R134a (44%/52%/4%)
Global Warming Potential (GWP, AR5)	148	1300	3940
Ozone Depletion Potential (ODP)	0	0	0
ASHRAE Safety Classification	A1	A1	A1
Boiling point (°C)	-16.9	-26.1	-46.2
Critical Temperature (°C)	112.24	101.06	72.14
Critical Pressure (bar)	35.3	40.59	37.35
Evaporator Glide (ΔT_{glide}) (°C)	2.4	None	0.8
Liquid density at -8°C (kg/m ³)	1290.4	1320.8	1179.7
Vapor density at -8°C (kg/m ³)	7.5	10.8	23.4

*These are just some of a mosaic of properties that must be considered in identifying a refrigerant

Table 2: Properties of POE32 lubricant

Properties	Values	Method	Units
Name	POE ISO 32 3MAF	-	-
Physical State	Liquid	-	-
Color	Straw (light)	-	-
Odor	Mild	-	-
Density at 20°C	978	ISO 12185 / ASTM D4052	kg/m ³
Base Oil Viscosity @ 40 °C	32	ISO 3104 / ASTM D445	mm ² /s
Base Oil Viscosity @ 100 °C	5.4	ISO 3104 / ASTM D445	mm ² /s
Pour Point	46	ISO 3016 / ASTM D97	°C

Table 3: Materials tested for compatibility studies

Refrigerant	Lubricant	Material Samples	
R471A, R134a, R404A	POE32	Plastics: 1. Nylon 2. Polyamide-imide (PAI) 3. Polyethylene Terephthalate (PET) 4. Polyimide 5. Polyacetal 6. ABS 7. Polyetherimide (PEI) 8. Polypropylene 9. PTFE 10. Polyphenylene sulfide (PPS) 11. Phenolic 12. PEEK 13. Polyurethane 14. Polyvinylidene Fluoride (PVDF)	Elastomers: 1. Neoprene (Chloroprene) 2. HNBR 3. NBR (Buna- N) 4. EPDM 5. Silicone Butyl Rubber

3. EXPERIMENTAL APPRATUS AND PROCEDURE

3.1 Solubility Studies

The solubility studies are performed in a PVT (Pressure/Viscosity/Temperature) test rig designed to measure liquid and vapor temperature, pressure, density, viscosity and mass flow for mixtures of refrigerant and oil. Figure 1 shows a schematic of the test facility. The main components of the test facility are variable speed pump, oscillating piston liquid viscometers, flow meter, bulk fluid reservoir, pressure transducer, balance, over pressure relief containment vessel and environmental chamber control and power. The oil refrigerant mixture is circulated in a continuous loop by a high-pressure gear pump to maintain thermodynamic equilibrium at a constant temperature. The test loop is placed inside a thermal chamber to maintain a desired temperature. The chamber has a thermal ramping feature that sweeps a range of temperature at a constant bulk composition of oil refrigerant mixture. The experimental uncertainties are summarized in Table 4 (Seeton, 2009).

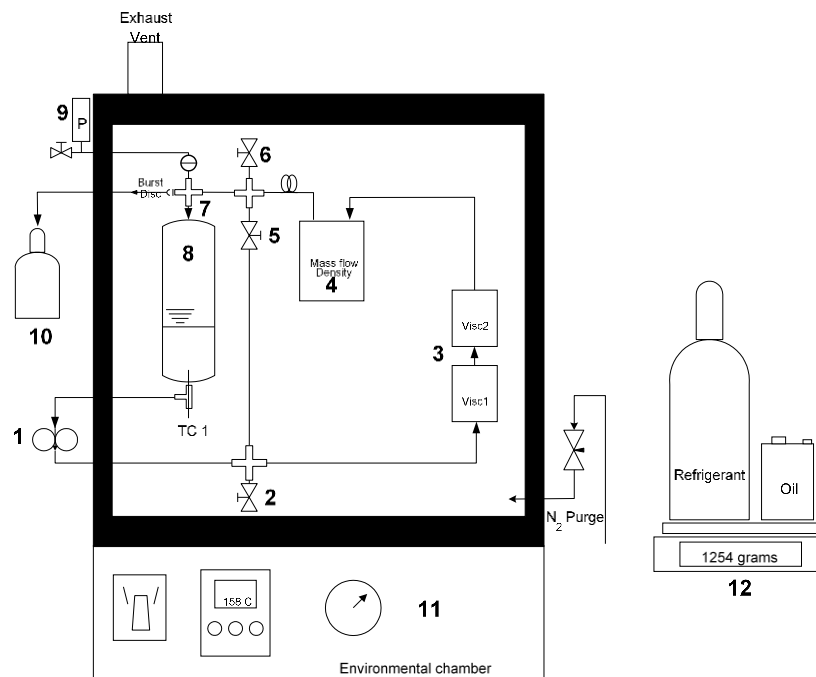


Figure 1: Thermophysical Property Test System Schematic

1. Variable speed gear pump; 2. Liquid filling valve; 3. Oscillating piston liquid viscometers; 4. Vibrating tube densitometer/ mass flowmeter; 5. Circulation valve; 6. Gas filling valve; 7. Burst disc; 8. Bulk fluid reservoir; 9. Pressure transducer; 10. Burst disc/over pressure relief containment vessel; 11. Environmental chamber controls and power; 12. Balance

Table 4: Uncertainty of experimental measurements

Instrument/Measurement	Measurement Uncertainty
Balance/Mass	± 0.02 grams
Densitometer/Liquid Density	± 0.002 g/cm ³
RTD/Temperature – Bulk Fluid Reservoir	± 0.15 K
RTD/Temperature – Viscometer	± 0.15 K
RTD/Temperature – Densitometer	± 0.15 K
Viscometer/Liquid Viscosity: 0.5 – 10 cP	± 0.1 cP
Viscometer/Liquid Viscosity: 5 – 100 cP	± 1 cP

Experimental Procedure: The solubility rig is thoroughly evacuated using the vapor charging valve and known mass of lubricant is injected into the evacuated system. The weights of the beaker containing the lubricant, charging tube and wipes to absorb any excess lubricant are measured before and after charging. The mass of lubricant charged in the solubility rig is the difference of before charging and after charging weights of flask, charging tube and absorbent wipes. The oil is charged inside the bulk fluid reservoir via the liquid charging valve. The first run conducted in the solubility rig is of neat oil and subsequently, refrigerant is charged in the system for measurements of oil/refrigerant mixtures at five compositions 5%, 10%, 20%, 30% and 40%, weight% of

refrigerant. For charging refrigerant in the experimental set-up, a small sample cylinder of desired amount of refrigerant is used to charge refrigerant in the system via vapor charging valve (6). The refrigerant is forced into the system due to pressure differential between the chamber (-30 °C) and sample cylinder (room temperature). The mass of the refrigerant sample cylinder (± 0.02 grams) is recorded before and after charging. The mass of the lubricant and refrigerant charged into the experimental system create a constant bulk composition in the system (mass of refrigerant / total mass of refrigerant and lubricant).

After the thermal chamber is stabilized, the gear pump (1) is started to circulate the oil refrigerant mixture through the circulating loop. The gear pump increases the liquid pressure of the refrigerant-oil mixture to ensure slightly subcooled liquid in the system such that no vapor bubbles are generated in the system to ensure accurate measurements. The density and viscosity of the refrigerant-lubricant mixture are recorded over the wide range of temperature in the densitometer (4) and viscometer (3) respectively. The bypass circulation valve (5) is opened to allow proper mixing and maintaining equilibrium without overloading the pump. The temperature ramp feature of the chamber is selected where the temperature of the system gradually drops from 125 °C to -30 °C over the period of 18 hours maintaining the steady state of the mixture at any given instance. At an interval of 10 seconds the dynamic measurements of pressure, density, viscosity, mass flow and temperature are recorded for a specific bulk composition of oil refrigerant mixture over a temperature range of 125 °C to -30 °C. After the completion of 18 hours, the data of the run is collected from the data logger and more refrigerant is added into the experimental system to increase the composition of oil refrigerant mixture for the next run. The measurement of thermodynamic and transport properties is collected for neat oil, 5%, 10%, 15%, 20%, 30% and 40% w/w refrigerant to oil ratio. Further details on the experimental test facility can be found in Seeton (2009).

The results of the solubility and liquid viscosity measurements are plotted on the viscosity-temperature axis and the resulting plot is known as Daniel Chart (Figure 4). The Daniel Chart allows interpretation of the interactions between solubility, pressure, and temperature on its effect on liquid viscosity on a single chart and provides crucial information for compressor bearing wear protection.

3.2 Miscibility Studies

The miscibility test set up comprises of a thermal chamber, stainless steel pressure cells for different compositions of refrigerant and oil samples, control cell with POE32 and temperature sensor. A temperature sensor is mounted on the control miscibility cell to measure the accurate temperature of oil-refrigerant mixture inside the cell. The accuracy of the temperature sensor is ± 0.1 °C in the temperature test range of -30 °C to 75 °C. The cells are kept in the thermal chamber to maintain a desired equilibrium temperature inside the cell. The back wall of the thermal chamber has LED for ease of visualization of cells. The refrigerant and oil mass are measured with an A&D FP-6000 laboratory balance, ± 0.02 grams.

Experimental Procedure: Miscibility is typically studied by mixing known concentrations of refrigerant and oil in between temperature range of -35 °C to 75 °C at an interval of 5 °C or until a phase separation is observed. The typical oil/ refrigerant compositions charged in equilibrium cells for miscibility testing are 60%, 70%, 80%, 85%, 90%, and 95% of refrigerant concentration. After thorough cleaning and pressure testing, the equilibrium cells are evacuated to a vacuum pressure of 20 microns. The tare weight of the evacuated equilibrium cells is recorded. The oil is charged in the equilibrium cell from the oil charging port with a syringe. The oil filled equilibrium cell is evacuated to remove any dissolved moisture or air. After thorough evacuation, the weight of each cell is recorded again to determine the accurate amount of oil filled in each cell. Based on the oil/refrigerant composition, pre-calculated refrigerant amount is charged into each equilibrium cell. The equilibrium cell with all its constituents (oil and refrigerant inside) is weighed again to determine the amount of refrigerant charged in the cell. The final oil and mass fraction is calculated based on weights of oil and refrigerant charged in the equilibrium cell. Next, the six miscibility cells are kept inside a temperature controlled thermal chamber. The miscibility tests are performed over the desired temperature range (-35 °C to 75 °C) and if needed, fine tuning is done in between two temperatures to determine the precise temperature point of immiscibility. A thermodynamic equilibrium is considered when the temperature within the control cell reaches the set temperature within an error of 0.1 °C and difference between the control cell and the chamber temperature is within 4 °C (difference is higher at very low temperatures). At a specific temperature the refrigerant oil miscibility is observed through a sight glass.

3.3 Material Compatibility Studies

Three samples of each material were cut in strips of approximately $\frac{3}{4}$ " (0.02m) by $\frac{1}{2}$ " (0.013m) from the 20 different sheet materials and O-rings (See Table 3). The stainless-steel pressure vessels were used to expose the materials in oil and refrigerants at 90 °C for 21 days. Sealed tubes were prepared according to the general procedure described in ASHRAE Standard 97 (Standard, A.S.H.R.A.E., 2007). Investigations were conducted using refrigerants R471A and R134a with polyol ester (POE32) lubricant.



Figure 2: Miscibility Cell

Three samples of each material were immersed at 90°C for 21 days in pressure vessels containing a 1:1 ratio of POE32 lubricant and refrigerants. Pressure vessels of volume 0.5L were used for the testing and initial weights and volumes were measured on each sample. Each pressure vessel contained 50 grams of refrigerant, 50 grams of lubricant, and the material of interest, as detailed in Table 3. Vessels were placed in an oven at 90°C for 21 days. After 21 days, following the exposure, samples were removed from the vessel and tested within 15 minutes for immediate weight and volume change. The samples were evaluated for the presence of any discoloration, cracking/crazing, blistering, and noticeable or severe swelling. The samples were removed from the hot liquid then blotted with a soft cloth and allowed to cool to room temperature. The cooled specimens were rinsed briefly in isopropyl alcohol, blotted dry with a soft cloth. The mass of each specimen was first determined by weighing it in air. The volume of each specimen was first determined by weighing it in air and then in water. Hardness of each specimen was first determined by either Shore M using a micro hardness tester for elastomer materials and Shore D using a Shore D durometer for thermoplastic materials. The average values obtained on the immersed samples were recorded. The percent change in mass, volume and hardness were calculated by difference for each specimen and the results for three specimens in each of the test liquids were averaged.

4. RESULTS AND DISCUSSION

4.1 Solubility Studies

The solubility and working viscosity of R471A with POE32 lubricant is evaluated and results are compared with R134a and R404A with POE32 lubricant. The results are reported at various concentrations of 40, 30, 20, 10, 5 and 0 w/w percent of refrigerants (R471A, R134a and R404A) over a temperature range of -30°C to 110°C. The results of the solubility and liquid viscosity measurements of R471A with POE32 are presented in the form of plot of the vapor pressure and isobaric viscosity curves as a function of temperature and composition, a Daniel Plot (See Figure 4) and are compared with R134a and R404A (Figure 5a and 5b). Figure 5a compares the viscosities of refrigerant R471A, R134a and R404A at saturated pressure corresponding to a typical evaporator temperature (-8°C) for a medium temperature refrigeration system for sump temperatures varying from 10°C to 120°C. Figure 5a illustrates that the working viscosities of all three refrigerants provided to the bearings would be very similar to each other and within the uncertainty for sump temperature range 30°C to 120°C. There is a slight drop in viscosity of R471A with POE32 due to higher solubility (See Figure 5b) as compared to R134a in temperature of 10°C to 30°C. On the other hand, viscosities of R471a with POE32 are very similar and within the uncertainty to R404A with POE32 except there is slight reduction of viscosity in the temperature range of 10~20°C.

The above results of R471A in comparison to R134a and R404A with POE32 indicate no significant changes in viscosity of POE32 (with R471A) in the sump over a wide range of temperature and hence POE32 could be used as a lubricant with R471A without any modification.

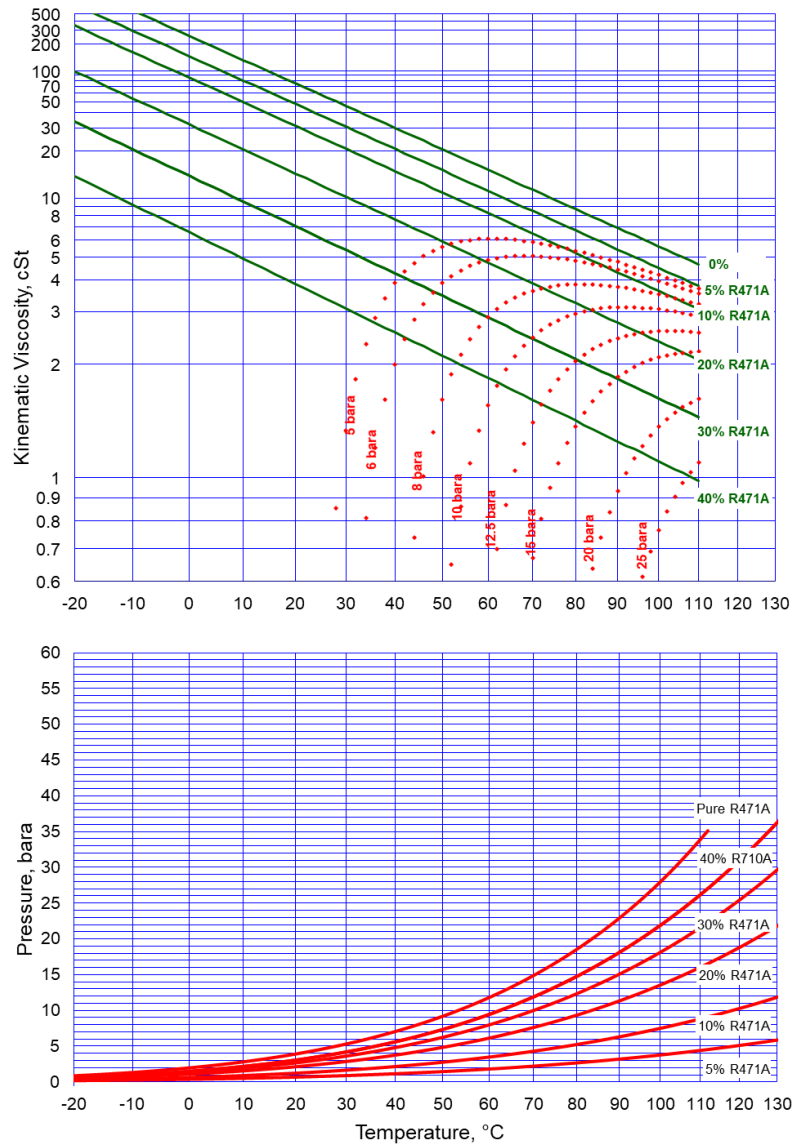


Figure 4: Pressure-Temperature and Viscosity Data for R471A with POE32

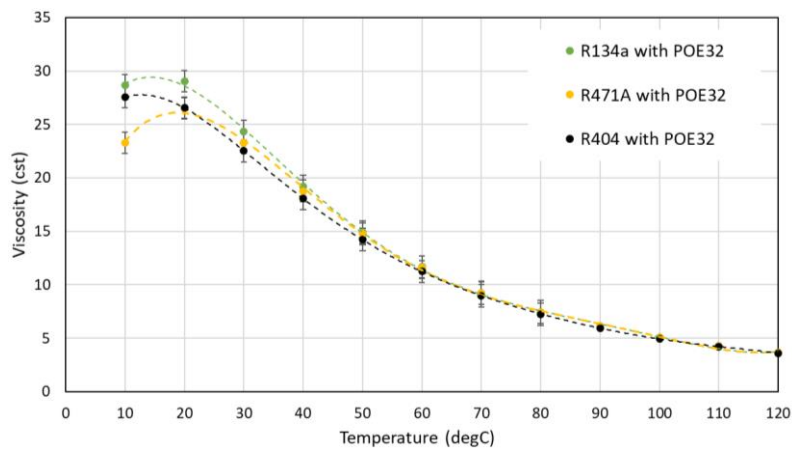


Figure 5a: Viscosity comparison at saturated pressure for medium temperature refrigeration cycle's evaporator temperature -8 °C

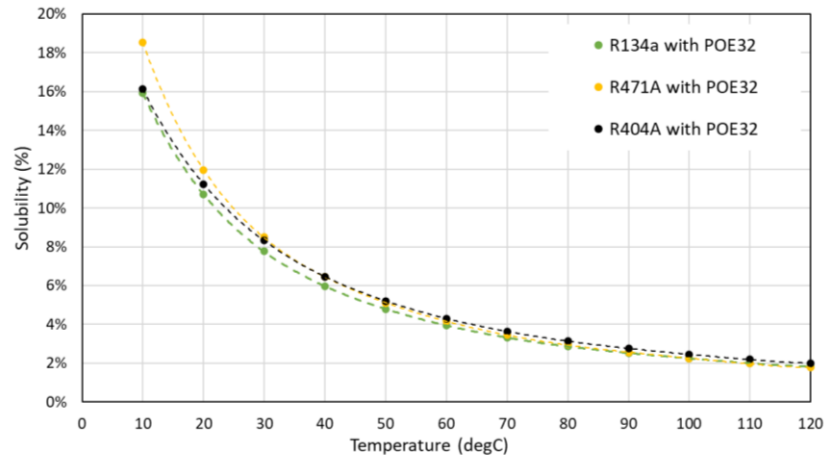


Figure 5b: Solubility (dilution) comparison at saturated pressure for medium temperature refrigeration cycle's evaporator temperature -8 °C

4.2 Miscibility Studies

The miscibility testing was conducted for refrigerants R471A, R134a and R404A with POE32. For each pair of oil and refrigerant, tests were performed at refrigerant concentrations of approximately 60%, 70%, 80%, 85%, 90%, and 95% over a temperature range of -30°C to 75°C. The criteria of immiscibility between liquid refrigerant and liquid oil are generally determined, if cloudiness, precipitate formation, or formation of a second liquid phase is noticed via the front or back sight glass of miscibility cell. However, in the following discussion, when cloudiness or precipitate formation was observed, it will be noted as such, but when the mixture separated into two liquid phases, it will be noted as immiscible. The observations for each refrigerant/lubricant pair are provided below in Figure 6.

The results in Figure 6 indicate full miscibility of R471A and R134a with POE32 oil in temperature range -30°C to 75°C whereas R404A is immiscible at 30°C and above for 60%-70% refrigerant concentration and approximately 40°C and above for 65% and 90% refrigerant concentration. The full miscibility of R471A with POE32 at all operating conditions ensures oil return to the compressor under all conditions and advantages R471A over R404A.

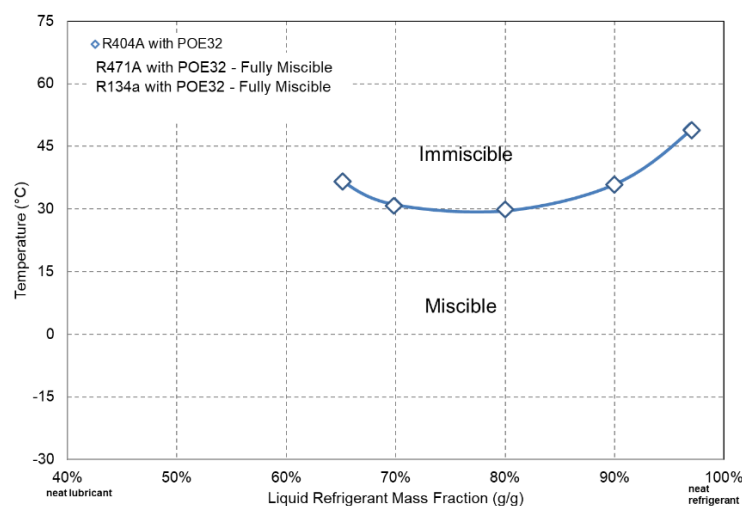


Figure 6: Miscibility results of R404A, R471A and R134a with lubricant POE32

4.3 Compatibility Studies

In this section, the experimental results of compatibility of refrigerants with thermoplastics and elastomers are presented and discussed. Material compatibility evaluations of thermoplastics and elastomers were conducted with two refrigerants R471A and R134a and lubricant POE32. All experimental results are concisely presented in Tables 6 and 7. Table 5 provides an overview of criteria of polymer- refrigerant compatibility. However, in

literature the criteria can vary depending upon the wide range of applications leading to different requirements on the materials (Eyerer, S. et. al.). The change in all measured properties (mass, volume and hardness) is depicted directly after the exposure in Table 6. Finally, Table 7 concludes and presents the acceptable compatibility (green), limited compatibility (yellow) and poor compatibility (red) of R471A and R134a with thermoplastic and elastomer materials.

As indicated in Table 6, based on the criteria defined in Table 5, most of the thermoplastics show good compatibility with R134a and POE32 with the exception of ABS and phenolic. The post exposure hardness measurements of ABS revealed considerable decrease in hardness (hardness change% -25%) indicating softness of the material after the test. However, based on literature, mass and volume change% in thermoplastics are an important criterion for compatibility and hardness change is not considered much of a problem. The second thermoplastic which could pose problem with R134a and POE32 is phenolic showing reduction in mass and volume change. On the other hand, all the thermoplastics are compatible with R471A and POE32. The hardness change % for polyurethane is -15% and reduction of hardness leads to a higher elasticity and toughness, yet to a lower tensile strength which limits its compatibility. Overall, compatibility of thermoplastics with R471A and POE32 is slightly better than with baseline R134a and POE32.

The compatibility results of elastomers with R134a and POE32 (see Table 6) indicate, neoprene, NBR (Buna-N), EPDM and butyl rubber exhibited significant shrinkage. For materials that significantly shrink, hardness measurements confirm hardening of neoprene, NBR (Buna-N), EPDM and butyl rubber. In comparison, the compatibility of neoprene and NBR (Buna-N) with R471A and POE32 is better than R134a and POE32, though EPDM and butyl rubber need to be evaluated on a case-by-case basis due to greater volume and mass change observed in the tests. Overall, compatibility results of elastomers with R471A and POE32 is better than baseline R134a and POE32

Table 5: Criteria of polymer-refrigerant compatibility

Type of polymer group	Rating/Color Scheme	Mass Change/Volume Change	Hardness Change
Elastomers	1 (best compatibility)	$\geq 0\%$ and $\leq +20\%$	$< \pm 10\%$
	2 (borderline)	$> +20\%$ and $\leq +30\%$	$< \pm 15\%$
	3 (incompatible)	$> +30\%$ or $< 0\%$	$> \pm 15\%$
Thermoplastics	1 (best compatibility)	$> -2\%$ and $\leq +10\%$	-
	2 (borderline)	$> +10\%$ and $\leq +20\%$	-
	3 (incompatible)	$> +20\%$ or $\leq -2\%$	-

Table 6: Change in mass, volume and hardness for thermoplastics and elastomers

Polymer Material	R471A with POE32			R134a with POE32		
	Mass Change %	Volume Change %	Hardness Change%	Mass Change %	Volume Change %	Hardness Change%
Thermoplastics						
Nylon	<1	<1	2	<1	<1	3
Polyamide-imide (PAI)	<1	<1	<1	<1	<1	1
Polyethylene Terephthalate (PET)	<1	<1	2	1	2	0
Polyimide	<1	<1	<1	-1	1	-2
Polyacetal	<1	<1	1	1	2	-1
ABS	2	2	5	5	7	-25
Polyetherimide	<1	<1	3	<1	<1	4
Polypropylene	3	3	2	2	2	0

PTFE	<1	<1	2	4	3	2
Polyphenylene sulfide (PPS)	<1	<1	1	<1	<1	-1
Phenolic	<1	<1	3	-2	-2	3
PEEK	<1	<1	<1	<1	<1	1
Polyurethane	2	<1	-15	1	2	2
Polyvinylidene Fluoride (PVDF)	<1	<1	<1	3	2	-3
Elastomers						
Neoprene (chloroprene)	25	25	-8	-10	-8	1
HNBR	5	5	<1	16	15	<1
NBR (Buna-N)	12	12	-15	-3	-2	1
EPDM	-12	-12	22	-25	-19	6
Silicone	<1	<1	<1	2	1	0
Butyl Rubber	-12	-12	3	-18	-14	2

Table 7: Compatibility results of R471A and R134a and POE32 with elastomers and thermoplastics

Polymer Material	R471A with POE32	R134a with POE32	Polymer Material	R471A with POE32	R134a with POE32
Thermoplastics			Thermoplastics		
Nylon			Phenolic		
Polyamide-imide (PAI)			PEEK		
Polyethylene Terephthalate (PET)			Polyurethane		
Polyimide			Polyvinylidene Fluoride (PVDF)		
Polyacetal			Elastomers		
			Neoprene (chloroprene)		
ABS			HNBR		
Polyetherimide			NBR (Buna-N)		
Polypropylene			EPDM		
PTFE			Silicone		
Polyphenylene sulfide (PPS)			Butyl Rubber		

5. CONCLUSION

This paper evaluated the solubility and miscibility studies of R471A, R134a and R404A with lubricant POE32 and compatibility studies of various thermoplastics and elastomers with R471A (and POE32) and R134a

(and POE32). The solubility measurements of R471A, R404A and R134a with POE32 showed working viscosities of all three refrigerants provided to the bearings would be very similar and within the uncertainty measurements for medium temperature refrigeration system. The results of R471A in comparison to R134a and R404A with POE32 indicate no significant changes in viscosity of POE32 (with R471A) in the sump over a wide range of temperature and hence POE32 could be used as a lubricant with R471A without any modification. Secondly, the miscibility measurements indicate complete miscibility of R471A with POE32 ensuring proper oil return to the compressor under all conditions in medium temperature refrigeration system as compared to R404A with POE32 which is immiscible at certain operating conditions. Thirdly, material compatibility studies of thermoplastics and elastomers showed an overall improvement in compatibility of various materials ABS, phenolic, neoprene, NBR (Buna-N) with R471A and POE32 as compared to R134a and POE32.

NOMENCLATURE

HVAC&R	Heating, Ventilation, Air Conditioning, and Refrigeration
VLE	Vapor Liquid Equilibrium
POE	Polyolester oil
GWP	Global Warming Potential
ODP	Ozone Depletion Potential

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