Enhanced Coolant for Low Temperature

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In many applications, e.g. aerospace thermal conditioning, there is a need for liquid heat carriers or coolants, which are used at low temperatures. Perfluorocarbons often apply in such applications due to their low freezing points and low viscosities. However, thermal properties of perfluorocarbons such as heat capacity and thermal conductivity are unfavourable compared to other heat carriers. Fraunhofer UMSICHT developed phase change slurry designed as coolants for aerospace applications. Phase change slurries are dispersions of water as inner phase and perfluorocarbons as continuous phase. The water is dispersed into the perfluorocarbon and serves as phase change material, meaning that it absorbs and releases latent heat during the phase transition, while the perfluorocarbon remains in the liquid state. The latent heat of the phase transition increases the heat storage and heat transportation capability of the fluid. The phase transition of the water droplets changes the dispersion from an emulsion into a suspension and vice versa, but the dispersion remains viscous due to the continuous liquid phase of the perfluorocarbon. This paper describes the dispersion and provides measurements to determine the fluid properties. These are a Differential Scanning Calorimetry, measurements of thermal conductivity, viscosity, and density as well as microscopy pictures. The phase transition of the phase change material takes place in the temperature range between -25 °C and 0 °C. The expected operating temperature of the dispersion is in the range from -80 °C to 40 °C. In view of the potential use of the coolant for human spaceflight, toxicity and flammability have been two of the criteria in selecting the perfluorocarbon for the continuous phase.

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Nomenclature

Abbreviations
BBZ = Butyl benzene
DSC = Differential Scanning Calorimetry
FoM = Figure of Merit
Mo = Mouromtseff number
PCM = Phase Change Material
PCS = Phase Change Slurry
SMAC = Spacecraft Maximum Allowable Concentration

Symbols
c = specific heat capacity
h = enthalpy
K = empirical factor
n = empirical factor
m = mass
T = temperature
w = mass fraction
γ = thermal expansion
$\dot{\gamma}$ = shear rate
Δ = difference
η = viscosity
ρ = density
τ = shear stress
ϕ = volume fraction

Subscripts
G = Galden HT 80
D = dispersion
W = water
$0, n$ = calculation points

I. Introduction

PERFLUOROCARBONS often apply as heat carrier fluids in applications operating at low temperatures, e.g. heat rejection in aerospace applications, due to their low freezing point and low viscosity compared to other fluids like water/glycol or water/alcohol mixtures. However, thermal properties of perfluorocarbons such as heat capacity and thermal conductivity are worse in comparison to other heat carrier fluids [1]. Lower heat capacities of the fluids result in higher flow rates for the thermal energy transport as well as the lower thermal conductivities result in a lower heat transfer, which requires larger heat exchange areas and heat exchangers that are more complex. In most manned aerospace applications, there is a hydraulic separation between an external cooling loop with a perfluorocarbon operating at low temperatures outside the spacecraft, and an internal loop with a conventional heat carrier operating at moderate temperatures inside the spacecraft [2]. Such cooling systems are called dual-loop systems. However, a dual-loop system is more complex, heavier, and less efficient than a single-loop system without hydraulic separation [3]. For the realisation of a single-loop system, the challenge is to find a heat carrier fluid with a low freezing point and a low viscosity as well as high thermal capacity and high thermal conductivity. Furthermore, the fluid must not jeopardize the crew in case of a leakage, which results in additional requirements concerning safety, toxicity and flammability aspects [4].

Examples of heat carrier fluids based on perfluorocarbons are FC 72 and Novec 7200 of the company 3M as well as Galden HT 80 of the company Solvay. These fluids are usable in hydraulic loops at low temperature, but they have some poor thermal properties. This is why there are investigations to improve the thermal properties by dispersing solid nano-particles or liquid nano-droplets into perfluorocarbons. In [5] the author describes several so-called nanofluids, one is a water-in-FC 72 nanoemulsion with enhanced thermal properties. The idea is to combine the good thermal properties of water with the advantageous properties of FC 72, i.e. low freezing point, low viscosity and the inert chemical character. A positive effect is that water acts as a Phase Change Material (PCM). Due to the phase transition of the nano-droplets in the dispersion, it absorbs and releases latent heat, which augments the stored heat in addition to the sensible heat when changing the temperature of the fluid. In this way heat transfer and heat transportation capacity of the fluid are improved [6].

The authors present a dispersion consisting of water dispersed into Galden HT 80. The dispersion forms a so-called phase change slurry (PCS), which is developed to be used as heat carrier fluid in the temperature range from -80 to 40 °C. PCS are dispersions consisting of a PCM as inner phase in a continuous, liquid phase. While absorbing heat, the PCS changes from a suspension into an emulsion and, while releasing heat, vice versa [7]. In this case, water is the PCM and Galden HT 80 is the continuous phase. One major challenge is the formulation of a stable dispersion. Especially perfluorocarbons like Galden HT 80 are very hydrophobic and without suitable dispersing methods and additives, it is not possible to produce respective dispersions [8].

Up to now, only a few tests were performed with the dispersion. Some measurements of the dispersion properties were conducted to enable the design of a single-loop cooling system. These measurements are Differential Scanning Calorimetry (DSC), measurements of the thermal conductivity via hot-wire method, measurements of viscosity by a rotary viscometer, measurements of the density by an oscillating u-tube, and microscopy pictures.

II. Water-in-perfluorocarbon dispersion as phase change slurry

Fraunhofer UMSICHT and Airbus Defence and Space made theoretical and analytical considerations to pre-select candidates for subsequent testing. The considerations include material compatibility, flammability, and safety aspects. Concerning safety aspects, also the harmfulness of the considered fluids was assessed considering the Spacecraft Maximum Allowable Concentrations for airborne contaminants (SMAC) value. The SMAC value allows the calculation of the maximum acceptable volume of a fluid in case of a leakage. Other than these considerations the thermo-hydraulic performance of the fluid was assessed considering the Mouromtseff number (Mo), as in [9], and the Figure of Merit (FoM) according to [1]. Whilst the Mouromtseff number characterizes the capability of a fluid to transport heat, the FoM characterizes the ability to store and move heat. However, neither Mo nor FoM does consider the melting/crystallization enthalpy of the PCM. Therefore, another figure of merit was defined to identify the Gain Ratio φ_{GR} due to the phase change. For this purpose, the sum of (absolute) energy of phase change and advection is set in relation to the advection term, as in equation (1).

$$\varphi_{GR,D} = \frac{m_D \cdot c_D \cdot \Delta T + m_{PCM} \cdot h_{PCM}}{m_D \cdot c_D \cdot \Delta T} \quad (1)$$

$$\varphi_{GR,W} = \frac{m_D \cdot c_D \cdot \Delta T + m_{PCM} \cdot h_{PCM}}{m_W \cdot c_W \cdot \Delta T} \quad (2)$$

For a further comparison, the sum of (absolute) energy of phase change is also set in relation to the advection of pure water, as in equation (2).

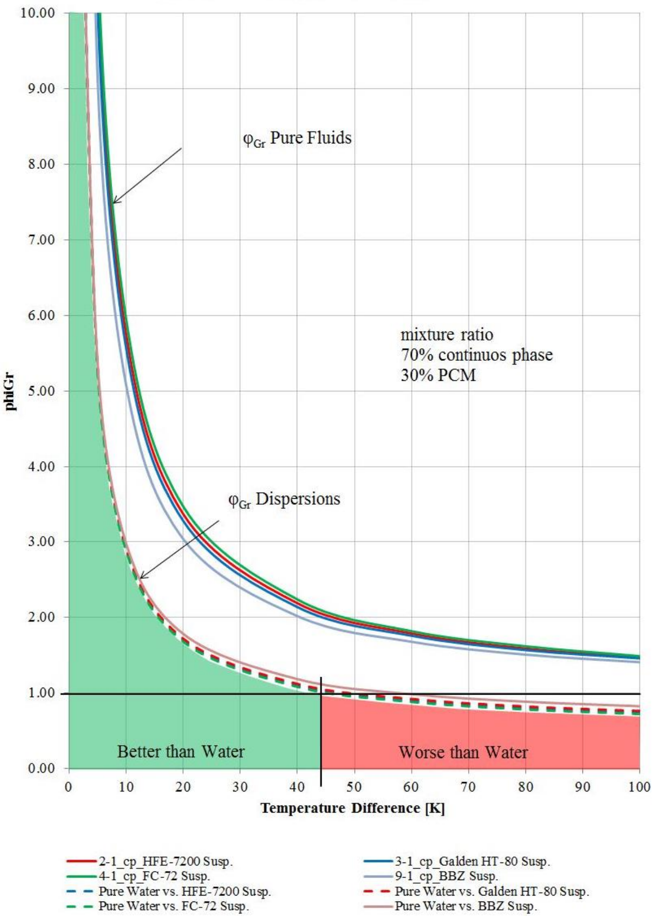

Figure 1. $\varphi_{GR,D}$ (continuous line) and $\varphi_{GR,W}$ (dotted line) for several fluid dispersions/suspensions

The results of both equations describe the heat storage and transport characteristics and are given in figure 1 with $\varphi_{GR,D}$ as continuous line and $\varphi_{GR,w}$ as dotted line. The value $\varphi_{GR,D}$ is always higher than 1, because the additional melting heat of the PCM increases the heat storage and transport characteristic of the pure fluid.

Depending on the temperature difference of the fluid between inlet and outlet of a loop over one cycle, $\varphi_{GR,w}$ is higher or lower than 1. Whilst one corresponds to the heat transport capability of water, values higher than one mean that the heat transport capability is φ_{GR}-times higher than that of pure water (green area). Vice versa, values lower than one mean that the heat transport capability is φ_{GR}-times lower than that of pure water (red area). The pre-selection of fluids based on the theoretical and analytical considerations are described before. Out of nine candidate coolants, three were selected for the further characterization and tests. These are FC 72, Novec 7200, and Galden HT 80. Finally, water dispersed in Galden HT 80 was chosen for the development of the coolant.

The water-in-perfluorocarbon dispersion appears like a “milky” fluid. Water is dispersed into the Galden HT 80 by an ultra turrax. The speed of the ultra turrax was 20.000 rpm. The dispersion is liquid disregarding the water droplets are frozen or not. Figure 2 is a picture of one of the first dispersion samples. Surfactants and additives stabilize the dispersion, which means that they avoid a separation of the water phase and Galden HT 80. Figure 3 is a microscopy picture of the dispersion.

Figure 2. Picture of a water-in-perfluorocarbon dispersion

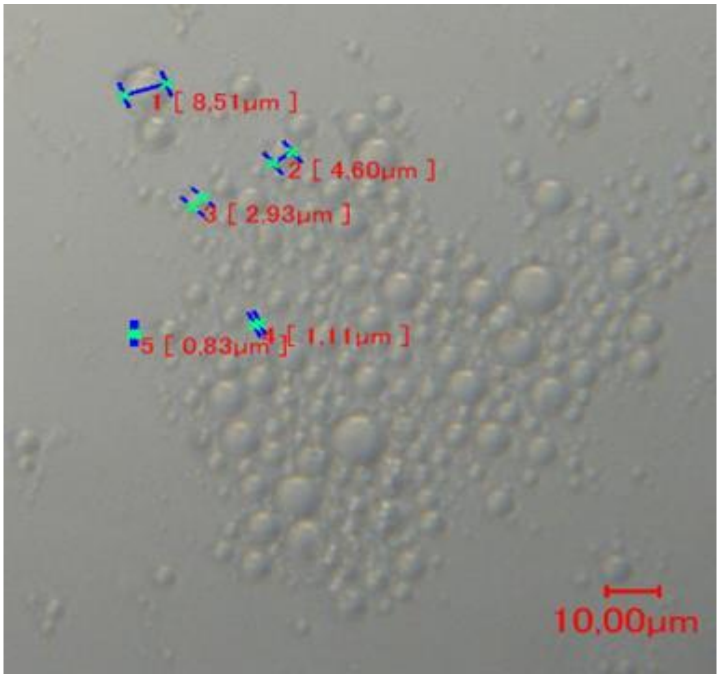

Figure 3. Microscopy picture of the dispersion

The size of the water droplets is between 0.8 and 10 µm. The dispersion was exposed to five thermal cycles, which means the dispersion temperature was reduced and increased in the way that the water droplets undergo a phase transition, and were observed under the microscope again. There is no change in the droplet size, what means that agglomeration of the droplets seems not to take place. The dispersion was stored at room temperature for more than four months without any visible change. Further stability tests are pending.

III. Thermal properties of the dispersion

The thermal properties of the dispersion are investigated by Differential Scanning Calorimetry (DSC) and hot-wire measurements.

DSC measurements apply to determine the heat of fusion and the temperatures of the freezing as well as of the melting process. A DSC device of the company Netsch type 204 F1 with intercooler was used. The DSC is a thermoanalytical technique. The measurement plot of a DSC provides the heat flux absorbed or released of a sample while changing its temperature. These plots directly indicate the temperature of the phase transition in form of peaks, see Figure 4. The area under the peaks represents the heat of fusion. Negative values mean that the sample releases heat, positive values mean that the sample absorbs heat.

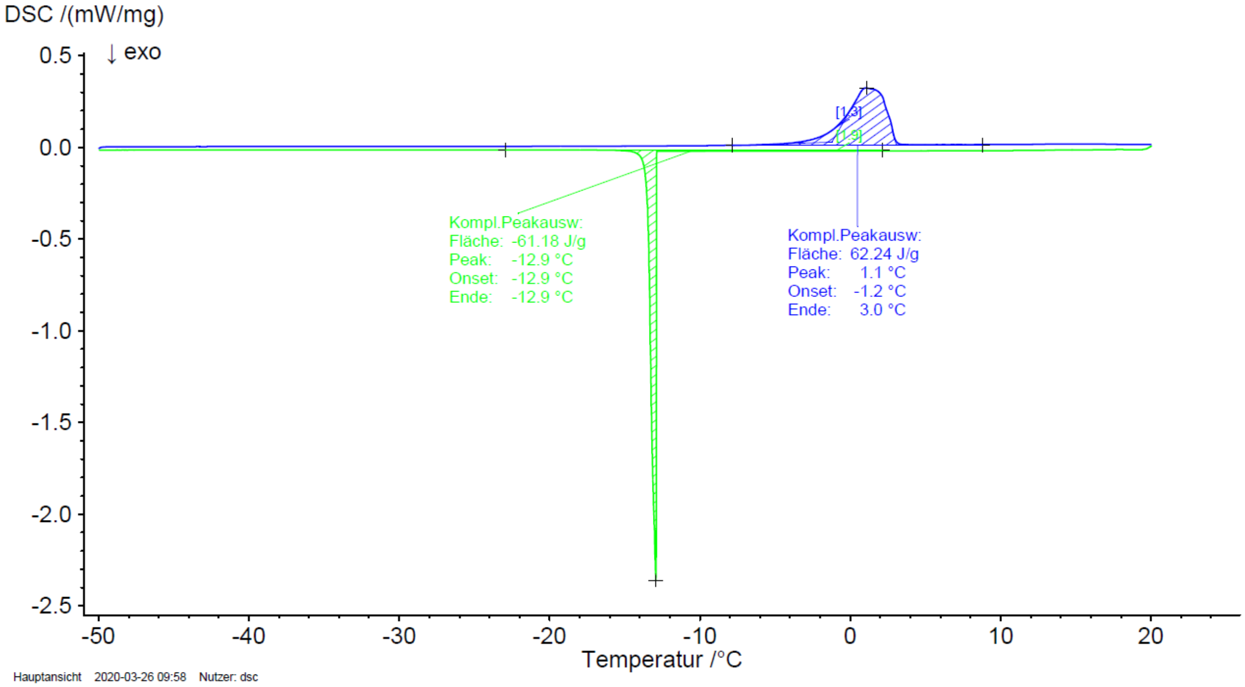

Figure 4. DSC measurement of a 19 wt.% dispersion

A dispersion with 19 wt.% of water is used as sample for the DSC. The weight of the sample is 41 mg. The temperature during the calorimetric scanning changed from 20 °C to -50 °C and back to 20 °C. The measurement speed is 1 K/min. Freezing of the water droplets occurs at -12.9 °C and the melting in the temperature range between -1.2 °C and 3 °C. The heat of fusion is approximately 62 J/g and is consistent with the expected value in view of the water content. It can be used in addition to the sensible heat of the dispersion and increases the heat transfer capability of the dispersion compared to the pure Galden HT 80. The temperature difference between melting and freezing peak is mainly related to supercooling or hysteresis effects. This effect must be considered while designing respective cooling loops.

The specific heat of dispersion c_D is calculated using an equation (3) based on the specific heat of water c_w as well as Galden HT 80 c_G, both as functions of the temperature, and taking into account the mass fraction of water w. Further additives of the dispersion besides water, which are less than 3 wt %, are not considered for the calculation.

$$c_D(T) = w \cdot c_w(T) + (1 - w) \cdot c_G(T) \qquad (3)$$

In Figure 5, the calculated values as well as measured values of a dispersion sample and of a Galden HT 80 sample are plotted in a diagram. Additionally, the reference values of Galden HT80 and propylene glycol/water mixture (47 vol.%, antifreeze max. -30 °C) taken from [10] and [11] are also indicated. The determination of the specific heat capacity is performed via DSC measurements, applying the sapphire-method. The analysis of the DSC measurement as well as interpretation and calculation of the specific heat capacity is made by NETZSCH Proteus Software. To obtain an evaluable measurement signal, the measurement speed is increased to 10 K/min. The sample for this measurement is not taken from the same batch as the sample used before for determining the heat of fusion, but it has the same composition.

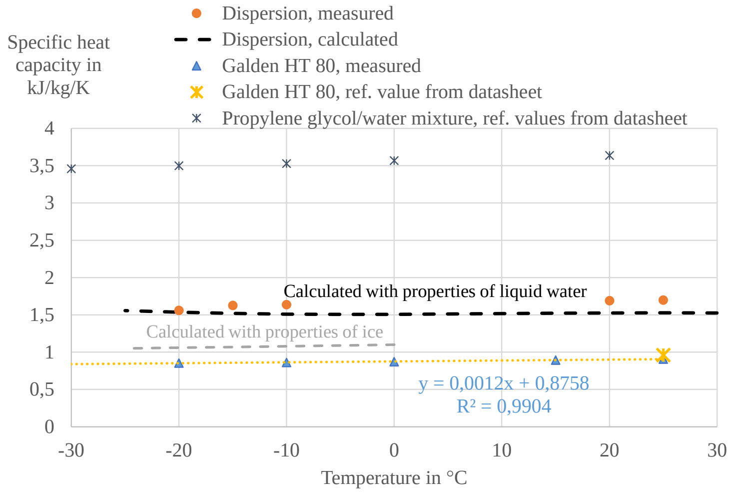

Figure 5. Specific heat capacity of 19 wt.% dispersion

The measured value of the specific heat capacity of the dispersion sample is ranging from 1.7 kJ/kg/K at 25 °C to 1.56 kJ/kg/K at -20 °C. A freezing of the water droplets does not seem to take place. This might be related the measurement speed. The calculated specific heat of the dispersion with frozen water particle is expected to be around 1.1 kJ/kg/K. The reference value for Galden HT 80 is 0.96 kJ/kg/K at 25 °C related to the datasheet [10]. The measured values of Galden HT 80 are ranging from 0.91 kJ/kg/K at 25 °C to 0.85 kJ/kg/K at -20 °C. One important result is that the water content in the dispersion increases the specific heat capacity of the dispersion by factor 1.8 compared to pure Galden HT 80 as long as water droplets are liquid. The specific heat capacity of pure water is about 4.2 kJ/kg/K for the liquid state and about 2 kJ/kg/K for the solid state.

The hot-wire method applies to measure the thermal conductivity. Type of hot-wire sensor is TC-18 of the company East 30 Sensors. The data acquisition system is an ADAM Ethernet-Module of the company Advantech. Measurements are conducted for the temperature range below and above the melting point of the PCM in the dispersion as well as during the phase transition, in which Fourier approach is considered for heat conductivity. The measurement results of a 19 wt.% dispersion sample as well as of a Galden HT 80 sample are given in the diagram of Figure 6. Additionally, the reference values of Galden HT 80 and propylene glycol/water mixture (47 vol.%, antifreeze max. -30 °C) taken from [10] and [11] are also indicated, too.

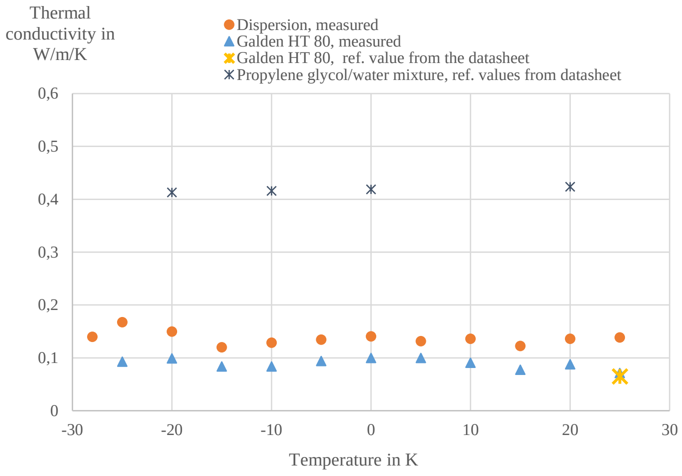

Figure 6. Thermal conductivity of a 19 wt.% dispersion

The thermal conductivity of the dispersion sample is about 0.14 W/(m·K) and higher than the thermal conductivity of pure Galden HT 80. At 25 °C, the thermal conductivity of the dispersion sample has a value, which is twice the value compared to pure Galden HT 80 with 0.07 W/(m·K). Nevertheless, the value is clearly lower than the values of propylene glycol/water mixture.

In [12] equation (4) is considered to calculate the thermal conductivity for the dispersion. Applying this equation on the dispersion sample – disregarding the additives in the dispersion with less than 3 wt.% – it is where λ_D is the thermal conductivity of the dispersion, λ_G thermal conductivity of Galden HT 80, λ_W thermal conductivity of the water content in the sample, ϕ water volume fraction, and n is an empirical scaling factor, which takes into account how different particle shapes affect thermal conductivity.

$$\lambda_D = \lambda_G \left[\frac{\lambda_W+(n-1)\lambda_G-(n-1)\phi(\lambda_G-\lambda_W)}{\lambda_W+(n-1)\lambda_G+\phi(\lambda_G-\lambda_W)}\right] \qquad (4)$$

Equation (4) is used to calculate the thermal conductivity of the dispersion sample by taking the thermal conductivities of water from [13] and by using the equation of a trend line (yellow line) based on the thermal conductivity measurements of Galden HT 80. The results of the calculation are plotted in Figure 7.

Equation (4) describes the measured thermal conductivity. The empirical scaling factor n is set on 2 for the calculation in order to adjust the calculation results to the measurement data. The trend line corresponding to the thermal conductivity of the pure Galden HT 80 seems to be slightly too high, which has also an impact on the calculation results.

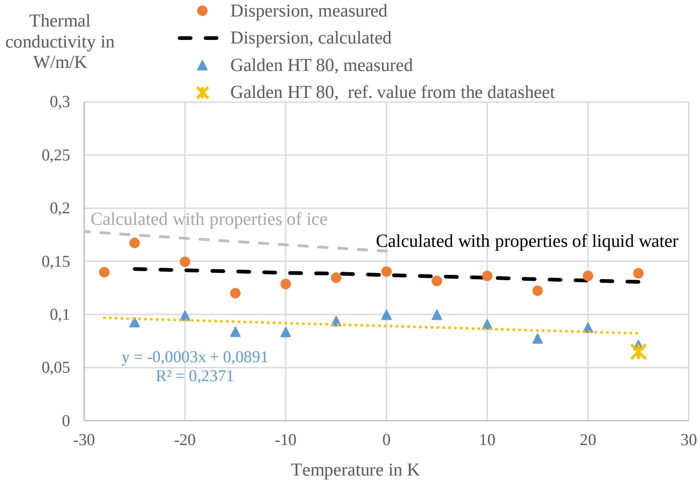

Figure 7. Comparison of calculated and measured thermal conductivity of a 19 wt.% dispersion sample

IV. Viscosity and density

In view of the rheological behaviour of the dispersion, measurement of the viscosity by a rotary viscometer and density by an oscillating u-tube were accomplished.

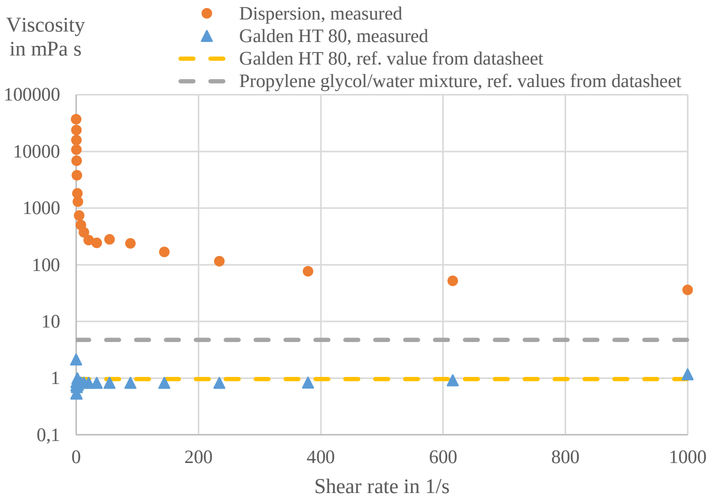

Figure 8. Viscosity of a 19 wt.% dispersion at 25 °C

A rotary viscometer type MCR 302 of the company Anton Paar measures the viscosity. In the diagram of Figure 8, the viscosity of the dispersion sample and pure Galden HT 80 are plotted against the shear rate at 25 °C. Additionally, the viscosity of Galden HT 80 is measured as well as dotted lines indicate the reference values of Galden HT 80 and propylene glycol/water mixture (47 vol.%). Both reference values are not provided with shear rates in the datasheets [10] and [11].

The viscosity of the dispersion is clearly higher than the viscosity of pure Galden HT 80 as well as of the propylene glycol/water mixture due to the multiphase composition of the fluid. The water content of the dispersion as well as the additives have a strong influence on the viscosity. Further investigations of the dispersion are still necessary focusing on the concentration of additives and their impact on the viscosity. The dispersion have a shear thinning behavior.

The impact of the temperature on the viscosity is investigated with additional measurements. Unfortunately, the amount of the original sample was not sufficient enough, so that a new sample had to be prepared. Then, viscosity is measured versus shear rate for four different temperature levels ranging from -25 C° to 25 °C. The measurement results are presented in the double logarithmic diagram of Figure 9. The viscosity at -10 °C is measured, when the water droplets are frozen. Thus, the viscosity measurements at -25 °C and -10 °C are related to the suspension state of the fluid, and 5 °C and 25 °C are related to an emulsion state of the fluid.

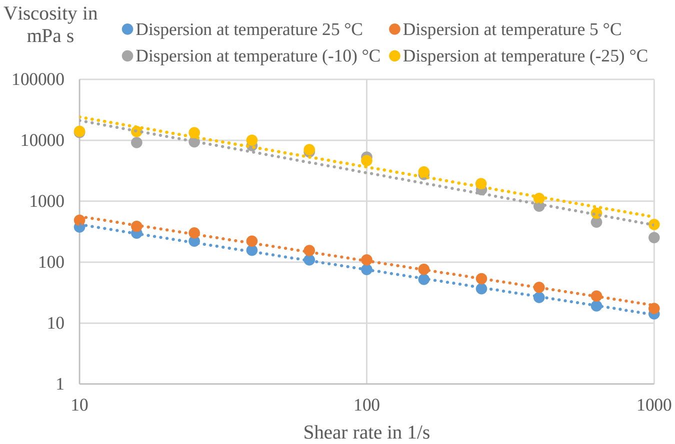

Figure 9. Viscosity as function of shear rate on different temperature levels / water particles frozen and liquid

The dispersion have a shear thinning behavior. This means, that the viscosity is not constant, but itself a function of the shear rate.

The Ostwald–de Waele relationship for shear stress τ and shear rate $\dot{\gamma}$ is proposed to describe the behaviour of the dispersion:

$$\tau = K \cdot \dot{\gamma}^{n} \qquad (5)$$

The viscosity η can be described as function of the shear rate $\dot{\gamma}$:

$$\eta = K \cdot \dot{\gamma}^{n-1} \qquad (6)$$

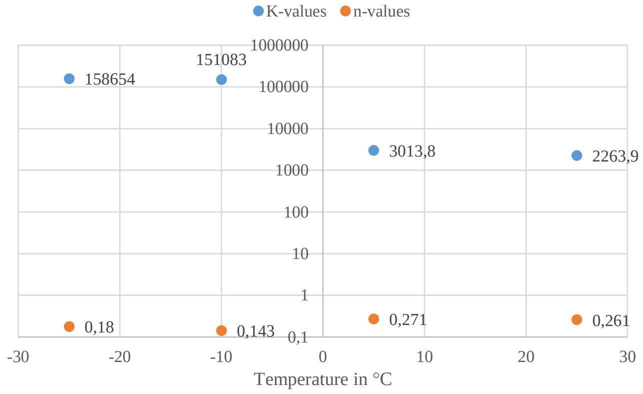

Figure 10. Coefficients K and n for the different temperature levels / water particles frozen (left handside) and liquid (right handside)

The coefficients K and n in equation (5) and (6) can be determined through the measurements with the rotational viscometer. The consistence of the fluid has impact on the coefficient K, which increases in case of higher viscosity of the fluid. The coefficient n characterize the degree of non-Newtonian behaviour of the fluid: n < 1 pseudo plastic behaviour, n > 1 dilatant behaviour, and n = 1 Newtonian fluid.

For the determination of the coefficients K and n, the measurement data from the double logarithmic diagram of Figure 9 are used and exponential functions are derived that describes the measurement curves. Based on the exponential functions the coefficients K and n to calculate the viscosity are found as depicted in Figure 10.

The change of the viscosity due to the phase transition is much higher than due to the temperature change. Therefore it is suggested to calculate the viscosity rather regarding its respective physical condition, means suspension or emulsion, than other changing parameters.

An oscillating u-tube type DMA HPM of the company Anton Paar measures the density. The diagram in Figure 11 provides the results of this measurements. The density of the dispersion sample is increasing with lower temperature and ranging at about 1.5 g/cm³. It is higher than the density of a propylene glycol/water mixture (47 vol.%), but lower than the values of pure Galden HT 80. It is remarkable that a step in the density curve related to any phase transition of water is not visible. This might be caused by the measurement methods and needs further investigations.

The equation (7) is proposed to calculate the density of a mixture ρ_D related to the density ρ_W and weight concentration w_W of water.

$$\rho_D = \frac{1}{\frac{w_W}{\rho_W} + \frac{(1-w_W)}{\rho_G}} \qquad (7)$$

This equation only considering the components water and Galden HT 80. Due to the low weight concentration of the additives, which totals less than 3 wt.-%, the additives are neglected in the calculation of the dispersion density. The density of water ρ_W related to temperature is taken from [13]. Additionally, the density of the dispersion is calculated with the density of ice. The density of ice is about 0.91 g/cm³. The density of Galden HT 80 ρ_G can be calculated for each temperatur T_n with the thermal expansion γ according to equation (8):

$$\frac{\rho_{G,0}}{\rho_{G,n}} = 1 + \gamma \cdot (T_n - T_0) \qquad (8)$$

The thermal expansion γ is 0.0011 1/K and the density ρ_0 of Galden HT 80 is 1.69 g/cm³, both values are found in the datasheet [10] and are determinated at 25 °C. The results of the calculation are also plotted in the diagram of Figure 11.

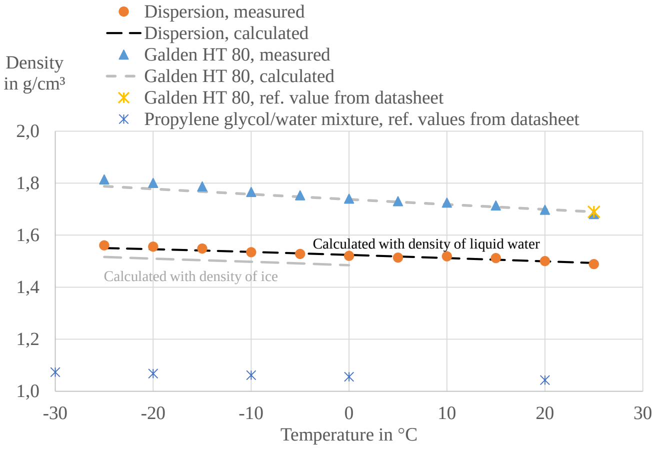

Figure 11. Density of a 19 wt.% dispersion at different temperatures

The calculated density of the dispersion as well as the density of Galden HT 80 fit well with the measured data, besides the phase transition of water is not visible.

V. Summary and Conclusion

The paper presents a PCS as heat carrier fluid for cooling systems. The thermal properties and the viscosity of the carrier fluid govern the effectiveness of any hydraulic cooling loop. In aerospace applications, perfluorocarbons are often used for heat rejection to space, because of their low toxicity, low freezing point, and low viscosity. However, perfluorocarbons do have some unfavourable thermal properties and are only used in a dual-loop system outside of the spacecraft. PCS could combine fluidity at low temperature and high thermal capacity as well as high thermal conductivity. Such a fluid permits the realization of a single-loop cooling design, which is technically less complex and more efficient. Furthermore, the fluid does not jeopardize the crew in case of a leakage.

One possibility to create a PCS is to disperse water into the common used perfluorocarbons. This forms a dispersion with enhanced thermal properties in which water acts as phase change material. The phase change material increases the heat transfer capability of the dispersion compared to pure perfluorocarbon. A DSC measurement with a 19 wt.% water-in-Galden HT 80 dispersion shows this effect and measures a heat of fusion 62 J/g. Freezing occurs at -12.9 °C and melting at about 0 °C. The specific heat capacity of the dispersion is ranging from 1.7 kJ/kg/K at 25 °C to 1.56 kJ/kg/K at -20 °C and is higher than of pure perfluorocarbon, especially if the water droplets are liquid. The hot-wire method is applied to measure the thermal conductivity of the dispersion, which is about 0.14 W/(m·K) and thus two times higher than the thermal conductivity of pure perfluorocarbon at 25 °C.

One disadvantage of the dispersion is the higher viscosity compared to that of pure perfluorocarbon. The viscosity is rising with decreasing temperature and have a shear thinning behavior. Especially, the amount of water as well as the additives to stabilize the dispersion have a big influence on the viscosity. In this context, the dispersion needs further optimization by finding a balance of the necessary concentration of additives as well as acceptable increase of the viscosity. The density of the dispersion is about 1.5 g/cm³ and increases with lower temperature.

The paper provides equations to describe the measured properties of a dispersion, which is developed to be used as heat carrier fluid. Application tests of the dispersion in a downscaled thermo-hydraulic circuit under typical operation conditions are the next step to investigate and further assess the applicability of the dispersion. The objectives of these tests are to gain further insights in the interaction of the PCS with other components in a hydraulic loop, e.g. pumps, filters, valves, orifices etc. and to determine the possible heat transfer as well as pressure drops.

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