

Investigation of Ethanol Based Secondary Fluids with Denaturing Agents and Other Additives Used for Borehole Heat Exchangers

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ABSTRACT

Ground source heat pumps are commonly used in Sweden. The dominating method of exchanging heat with the ground is by circulation of a secondary fluid through borehole heat exchangers inserted into energy wells. Due to the environmental reasons and relatively good thermophysical properties, aqueous solutions of ethanol are recommended and are commonly used for the borehole heat exchanger application in Sweden and other Nordic countries. The primary idea of using these fluids is to decrease the freezing point of the fluid since many borehole loops in Northern Europe operate near or below the freezing point of water. The available ethanol based commercial products consist of ethyl alcohol with up to 10% of denaturing agents without any corrosion inhibitors. The main denaturing agents used to prevent from drinking are mixtures of isopropyl alcohol, n-butanol, acetone, methyl ethyl ketone and methyl isobutyl ketone. These denaturing agents in commercial secondary fluids can influence the fluid's thermophysical properties and thereby affect the pressure drop and heat transfer in the borehole heat exchanger. These not well defined effects of additives can cause uncertainties in the ground source heat pump design and operation. The thermophysical properties of a few aqueous ethanol solutions are experimentally studied in this paper including a field sample, a pure mixture of ethanol and ethanol including additives are compared with reference data. The density of 20 wt-% solution of ethanol including additives and the field sample were higher than pure 20 wt-% ethanol sample. The additives increased the specific heat capacity by 2.3% at -10 °C and 1% at 0 °C. A decrement by 10.5% in the dynamic viscosity was observed. Presence of additives increased the thermal conductivity values by around 1.5% at -8 °C and 4.3% at +40 °C. Some implications in terms of pressure drop and fluid to pipe thermal resistance are observed when these properties are applied in borehole heat exchangers, as the occurrence for laminar or turbulent varies at given temperature levels. An alternative would be to replace the denaturing agents with corrosion inhibitors to decrease the concentration of additives, improve corrosion protection of the systems and still fulfill the market recommendations.

1. INTRODUCTION

A secondary fluid is a medium used for transporting heat as well as transferring heat between two bodies due to temperature difference. One of the most common secondary fluids is water, which cannot be used when the operating temperature is below +3 °C due to its freezing temperature at 0 °C as mentioned by Aittomäki and Kianta (2003). However, various freezing point depressants (antifreeze agents) can be added to water, e.g.; ethanol (ethyl alcohol), propylene glycol, ethylene glycol, chlorides, potassium formate, potassium acetate and potassium carbonate. An extensive study about this was presented in Melinder (2007).

One application for secondary fluids that has become very popular during the last decades is Ground Source Heat Pump (GSHP) systems. In Sweden there are about 500 000 small and 400 large GSHP systems using secondary loops, most of them in vertical borehole loops. Swedish GSHP systems deliver approximately 15 TWh energy per year, with a total installed capacity estimated to be about 4,6 GW as found in Antics et al. (2013). The typical set up of such a system consists of one or several borehole heat exchangers (BHE) vertically drilled in the ground to a certain depth (normally between 120 to 300m). The most common secondary fluid used for the borehole heat exchangers in Sweden is an aqueous solution of ethanol, and it is recommended by the Geological Survey of Sweden (SGU) and the Swedish Environmental Protection Agency (Naturvårdsverket), SGU (2008). The available ethanol based commercial products consist of denatured ethyl alcohol including up to 10 wt-% of denaturing agents without any corrosion inhibitors in 95wt-% ethanol solution. The content of these denaturing agents in commercial secondary fluids will with no doubt influence the fluid's thermophysical properties and likely also affect the pressure drop and heat transfer in, for example, borehole heat exchangers. The main denaturing agents (added to prevent drinking) found in these mixtures are isopropyl alcohol, n-butanol, acetone, methyl ethyl ketone and methyl isobutyl ketone, according to European Union regulations EUR-Lex (2013). In Sweden the most common additives found are isopropanol (isopropyl alcohol) and n-butanol. Ethyl alcohol is considered to be environmentally friendly secondary fluid due to the fact that it occurs naturally as a fermentation product. Ethyl alcohol is expected to biodegrade rapidly in all environments with predicted half lifetime ranging from 0.1 to 10 days, MassDEP (2011). Both isopropanol and n-butanol can be found in nature as well and have degradation time up to 28 days, Swed Handling Chemicals (2012).

Ethanol based secondary fluid concentration is normally not exceeding 30 wt-% (corresponding freezing point of -20.5 °C) due to safety regulation and flammability risks. The concentration is usually chosen to give a freezing point 8-10 K below the normal operating temperature to avoid freezing problem, Melinder (2009). In BHEs, it is common to use about 25 wt-% concentration, giving a freezing point of about -15 °C. This concentration is often higher than necessary. A BHE is one of many applications where it may be relevant to have good knowledge about the thermophysical properties of the secondary fluid in order to ensure efficient systems in terms of thermal and hydraulic performance. A small part of the total borehole resistance (i.e. inverse overall heat transfer coefficient) per meter in a BHE is due to the convective film in the secondary fluid. In U-pipe BHEs, it is usually desired to keep the flow within the turbulent region in order to keep a low contribution of this fluid-to-pipe thermal resistance as described in Acuña (2013). However, the pumping power required to induce turbulence must be controlled (the pumping power is

proportional to the pressure drop and the flow rate in the BHE pipes) so that the best heat transfer conditions are achieved in the borehole at the lowest cost, i.e. using as little pumping energy as possible to circulate the fluid.

2. OBJECTIVES

The purpose of this paper is to investigate the effects of different additives used in the ethanol based secondary fluids by analyzing the most important thermophysical properties (dynamic viscosity, density, thermal conductivity, specific heat capacity and freezing point); compare these data with the reference literature values for pure ethanol solutions as well as with a field sample, including an implication of the use of different thermophysical properties in a ground source heat pump installation.

3. METHODOLOGY

3.1 Investigation of thermophysical properties

The most important thermophysical properties of secondary fluids when evaluating performance of secondary fluids in borehole heat exchangers are: freezing point, viscosity, thermal conductivity, specific heat capacity and density. A field sample from a BHE installation was collected and compared with aqueous solutions of pure ethanol, ethanol solution including same concentration of additives as the field sample, and with literature reference data retrieved from Melinder (2010). The pure ethanol sample with the same concentration was prepared using a volumetric dilution method and its concentration was verified using Abbe refractometer as 20 wt-% (24.47 vol-%) ethanol solution. The field sample was a denatured type of 20 wt-% solution of ethanol including 1.4-1.8 wt-% isopropanol and 0.4 wt-% n-butanol as additives. Field sample was made by diluting the "concentrate" (a concentrated mixture of ethanol including 7-9 wt-% isopropanol and 2 wt-% n-butanol additives); to the desired concentration of 20 wt-% solution of ethanol including 1.4-1.8 wt-% isopropanol and 0.4 wt-% n-butanol additives. In order to verify results, a control sample of 20 wt-% ethanol including the same concentration of both additives was made in the laboratory. Since the concentration of isopropanol additive was not exactly stated by the concentrate producer, it was decided to use the most common concentration recommended of 8 wt-% which is at the same time an average value for the range between 7 wt-% and 9 wt-%. Control sample of 20 wt-% solution of ethanol including additives was prepared in the laboratory by first creating a similar concentrate of ethanol including 8 wt-% isopropanol and 2 wt-% n-butanol that was later diluted to 20 wt-% solution of ethanol including additives (1.6 wt-% isopropanol and 0.4 wt-% n-butanol). Thus, the expression "20 wt-% ethanol including additives" should be understood in this case that 20 wt-% is the total concentration of the three different alcohols: ethanol, isopropanol and n-butanol. The only difference between the field sample and 20 wt-% ethanol sample including additives is presence of the green dye and slight difference in concentration of isopropanol. First ice crystals were observed at -10 °C and samples were tested down to -8 °C only.

3.1.1 Freezing point and concentration

To measure the freezing point plastic beakers were filled with 90 ml of the tested secondary fluid and placed in specially designed sample holder into an ultra-low temperature freezer with adjustable temperature. Thermocouples were placed in the center of each beaker and calibrated with an accuracy of +/-0.1°C. The temperature at which the sample freezes could be detected by looking at the temperature change profile over time. The ultra-low freezer temperature for all tests was set as -20 °C to avoid too high temperature difference between expected freezing point and freezer inner temperature. In order to validate the method, a pure 20 wt-% ethanol solution and 20 wt-% ethanol including additives (1.6wt-% isopropanol and 0.4 wt-% n-butanol) were tested in same conditions. All freezing tests were later compared to a reference value from Melinder (2010).

3.1.2 Density

Density measurements were performed in two different ways by using aerometers and a pycnometer. An aerometer is designed to measure the density of a liquid at +20 °C according to ISO 17025 with accuracy of +/-0.001 g/ml. The pycnometer or specific gravity bottle is another way to obtain accurate measurements of density. The pycnometer is a flask with a stopper that has a capillary tube through it. The specific volume of pycnometer (25.131 cm³) was determined according to DIN ISO 3507 with tolerance of +/-0.01cm³. The pycnometer uses a working liquid with well-known density, such as distilled water, to define the volume. By knowing the volume and measuring mass of empty as well as full pycnometer with Mettler Toledo high accuracy analytical balance (accuracy of +/-0.0001g), it is possible to determine the density of secondary fluid including additives at +20 °C. Later, all results are fitted to a function to extrapolate values in the desired range between -10 °C to +40 °C.

3.1.3 Dynamic viscosity

A Brookfield rotational viscometer DV-II Pro with special low viscosity adapter (UL-adapter) with temperature controlled water-ethylene glycol jacket was used to perform measurements in the temperature range -8 °C to +40 °C. The temperature accuracy for the viscometer is defined as +/-1°C and Brookfield refrigeration unit as +/-0.2 °C. The operation principle of the rotational viscometer is to drive a spindle (which is immersed in the test fluid) through a calibrated spring. The viscous drag of the secondary fluid against the spindle is measured by the spring deflection. The measurement range of a rotational viscometer is determined by the rotational speed of the spindle, the size and shape of the spindle, the container the spindle is rotating in, and the full scale torque of the calibrated spring. All viscosity measurements in temperature range between -8 °C and +40 °C for different ethanol based secondary fluids are done using UL-adapter and the same spindle to reduce the uncertainty in measurements.

3.1.4 Thermal conductivity

Thermal conductivity measurements were done using Transient Plane Source (TPS) method by means of a Hot Disk Thermal Constants Analyzer TPS-2500. The method is based on the use of a heated plane sensor. The Hot Disk sensor consists of an electrically conducting pattern in the shape of a double spiral, which has been etched out of a thin metal (Nickel) foil. This spiral is sandwiched between two thin sheets of an insulating material (Kapton). When performing a thermal conductivity measurement, the plane Kapton sensor is fitted between two pieces of the sample holder filled with liquid. By passing an electrical current high enough to increase the temperature of the sensor between a fraction of a degree up to several degrees, and at the same time recording the resistance (temperature) increase as a function of time, the sensor is used both as a heat source and as a dynamic temperature sensor. The typical liquid sample size is 10 ml and 25mm inner diameter of sample holder. It is important that the size

of the sample surfaces is appreciably larger than the diameter of the sensor, thus Kapton sensor 7577 with radius 2,001 mm was chosen.

3.1.5 Specific heat capacity

The specific heat capacity was measured using the Differential Scanning Calorimetry (DSC) method. It is a thermo analytical technique in which the difference in the amount of heat required to increase the temperature of sample and reference is measured. Both sample and reference are maintained at the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned. To perform tests, a microDSC evo7 from Setaram Instrumentation was used. The tests were performed in Cp continuous mode with heating scanning rate of 0.05 K/min in temperature range between -15 °C and +40 °C. The tested sample volume was 580 µl and the tests were repeated three times.

3.2 Some implications for a borehole heat exchanger

The variations in thermophysical properties of the secondary fluids tested in this paper according to the methods explained in section 3.1 are put in perspective in terms of calculated secondary fluid-to-pipe thermal resistance and pressure drop in a U-pipe BHE of type PE 40x2.4 mm and with depth of 140 m. The pressure drop ΔP in the pipes occurs mainly due to friction and it is estimated assuming fully developed flow according to eq. (1)

$$\Delta P = f \frac{\rho \cdot w^2}{2} \cdot \frac{L}{D} \quad (1)$$

The calculated pressure drop does not include the losses in the U-bend of the BHE. The estimation of the friction factor f depends on whether the flow is laminar or turbulent. In this paper, this is solved for laminar flow (for $Re \leq 2300$) with eq. (2) and for turbulent flow (for $Re \geq 2300$) with the correlation suggested by Gnielinski (1976), eq. (3)

$$f = \frac{64}{Re} \quad (2)$$

$$f = \frac{1}{(0,79 \cdot \ln Re - 1,64)^2} \quad (3)$$

where the Reynolds number is calculated as stated in eq. (4)

$$Re = \frac{w \cdot D \cdot \rho}{\mu} \quad (4)$$

The internal convection resistance at a given temperature inside the U-pipe shanks can be estimated as a function of an average internal heat transfer coefficient, h , as expressed in eq. 5.

$$R_{f-p} = \frac{1}{\pi \cdot h \cdot D} \quad (5)$$

where h is obtained using eq. (6)

$$h = \frac{Nu \cdot k}{D} \quad (6)$$

and the Nusselt number is calculated using the correlation for the turbulent case given by Gnielinski, eq. (7)

$$Nu = \frac{(f/8)(Re_D - 1000)Pr}{1 + 12.7(f/8)^{1/2}(Pr^{2/3} - 1)} \quad (7)$$

For laminar flow (assuming constant heat flux and fully developed conditions), $Nu = 4.36$.

4. RESULTS

4.1 Thermophysical properties

Table 1 presents the results of the freezing point test for all samples. The freezing point of 20 wt-% ethanol according to data from Melinder (2010) is -10.92 °C. Experiments showed lower freezing points for three different samples tested in same conditions. The difference in the freezing point for the reference fluid (Melinder 2010) and pure 20 wt-% ethanol solutions could be related to the experimental conditions. A subcooling effect of 3-4 K was observed for all three tested samples. Moreover, very similar freezing points were obtained for both the field sample and corresponding 20 wt-% ethanol including additives, which confirmed the concentration of the field sample with information stated by the ethanol producer.

Figure 1, presents the dynamic viscosity for pure 20 wt-% ethanol; 20 wt-% ethanol including additives and field sample. As seen, the pure ethanol 20 wt-% results were slightly higher than the reference values from Melinder (2010). Moreover, the field sample

and 20 wt-% ethanol including additives had about 10 % lower dynamic viscosity than the pure 20 wt-% ethanol. Results indicate that the presence of additives can lead to lower dynamic viscosity. Thus, isopropanol and n-butanol additives decreased the dynamic viscosity. This can have practical consequences in terms of pressure drop as will be shown for a U-pipe BHE later in this article.

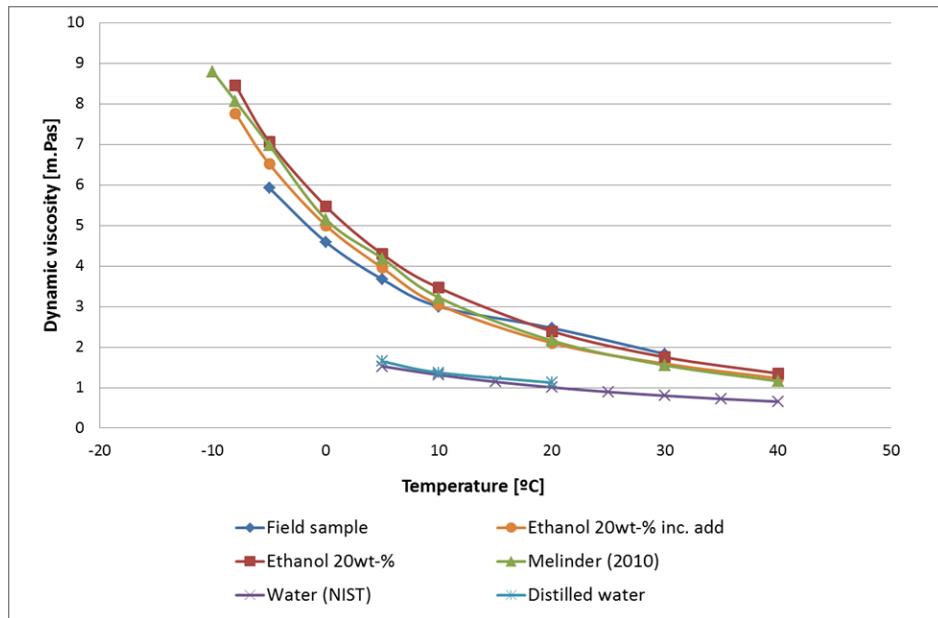


Figure 1: Dynamic viscosity results.

Figure 2, presents the results from density measurements. Results for temperatures +20 °C were fitted to the curve to obtain density as a function of temperature. As seen, the reference density was higher than pure 20 wt-% ethanol sample by 0.52% which could again be related to the testing method. Reference values were determined only by means of aerometer at +20 °C while the experimental results were performed as well using the pycnometer which gives higher accuracy. The density of field sample in comparison to 20 wt-% including additives was slightly higher (0.05%). The reference value for 20 wt-% was very close to both field and 20 wt-% ethanol including additives sample. The results showed that the additives were increasing the density by 0.4% which could be explained by the fact that both additives are alcohols. The measurement results preformed for distilled water using same testing method was having an error below 0.14% when compared to reference results.

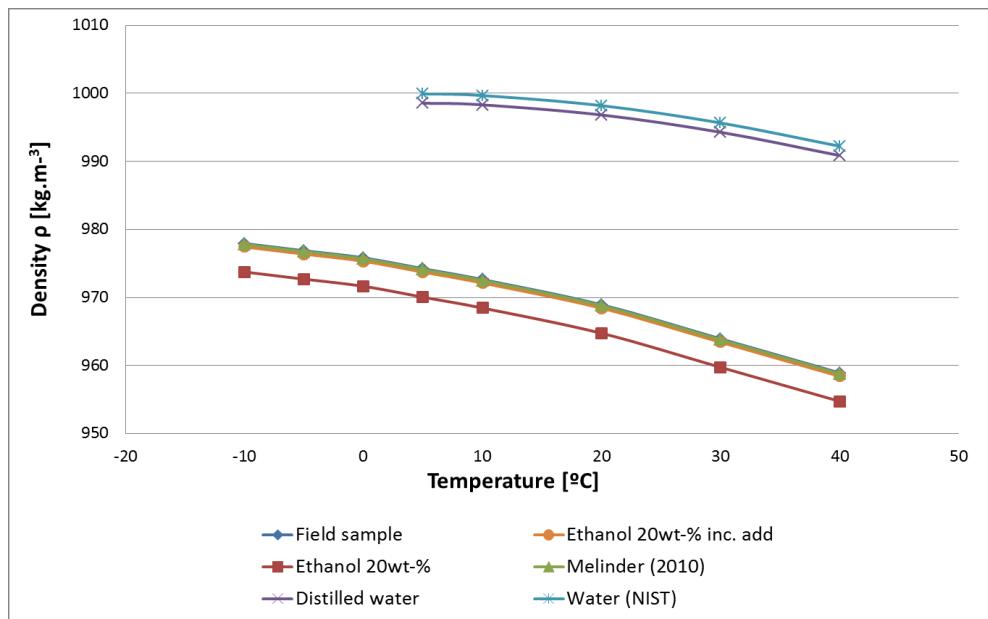


Figure 2: Density results.

Figure 3, presents the results of the specific heat capacity. Note that the ethanol based secondary fluids at concentrations lower than 25 wt-% have higher specific heat capacity than water. With increasing ethanol concentration the specific heat capacity value is decreasing as shown in Melinder (2007). All measured series showed a different tendency than literature values in ICT (1928-29),

Carrier (1965), Plank (1988), Melinder (1998), and Melinder (2007). These differences could be explained by the fact that the new testing method was applied for liquid samples based on the concept of very slow (0.05 K/min) continuous heating process instead of cooling mode. Same testing method was applied to test distilled water and an error of 1.4% was obtained.

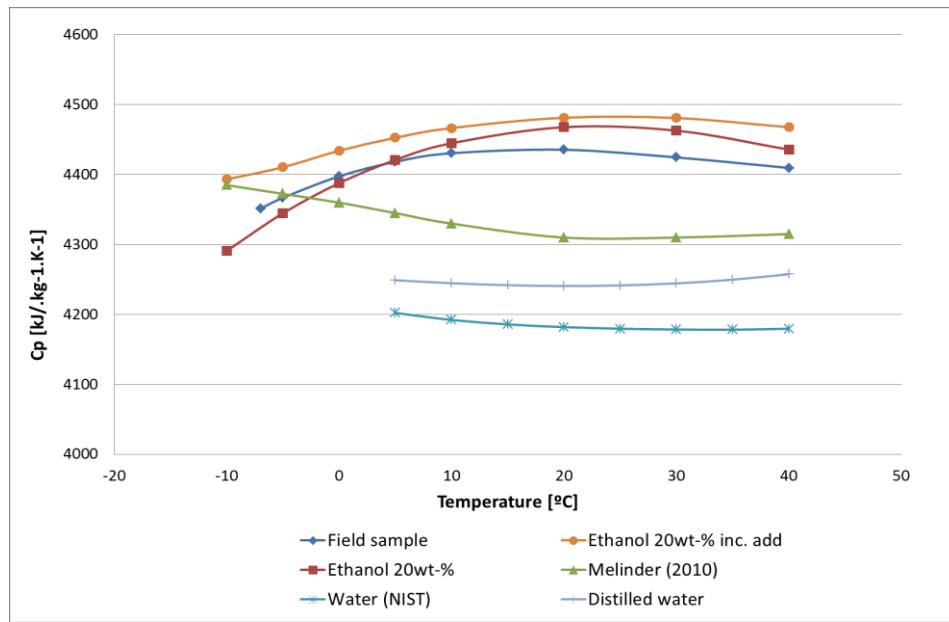


Figure 3: Specific heat capacity results.

In contrast to the reference data, the same slope or tendency of results was observed for all three ethanol samples which give more confidence in obtained results. The sample of 20 wt-% ethanol including additives showed 1 – 2% higher specific heat capacity than pure 20 wt-% ethanol and the field sample. This slight difference in results between the field sample and 20 wt-% ethanol including additives could be related to slight difference in concentration of isopropanol in both samples. The increment in the specific heat capacity was observed especially for temperatures between -10 °C and +0 °C by 2.3% and 1%, respectively. Additionally, a slight increment in specific heat capacity was observed in temperature range +30 °C to +40 °C. Isopropanol and n-butanol have higher specific heat capacity than ethanol and this can explain the observed increment in specific heat capacity value of tested samples.

Figure 4, presents the results of the thermal conductivity tests. One important input value for post processing of thermal conductivity result is use of density and specific heat capacity value. Knowing ρC_p it was possible to decrease the measurements error below 2%. The higher values of C_p and density could explain the steeper slopes of curves for three different samples. As seen, the additives had a positive effect and a higher thermal conductivity could be observed in comparison to pure 20 wt-% ethanol solution. The increment in thermal conductivity was around 1.5% at -8 °C and 4.3% at +40 °C. The difference between distilled water results and reference data was found to be less than 1.15%.

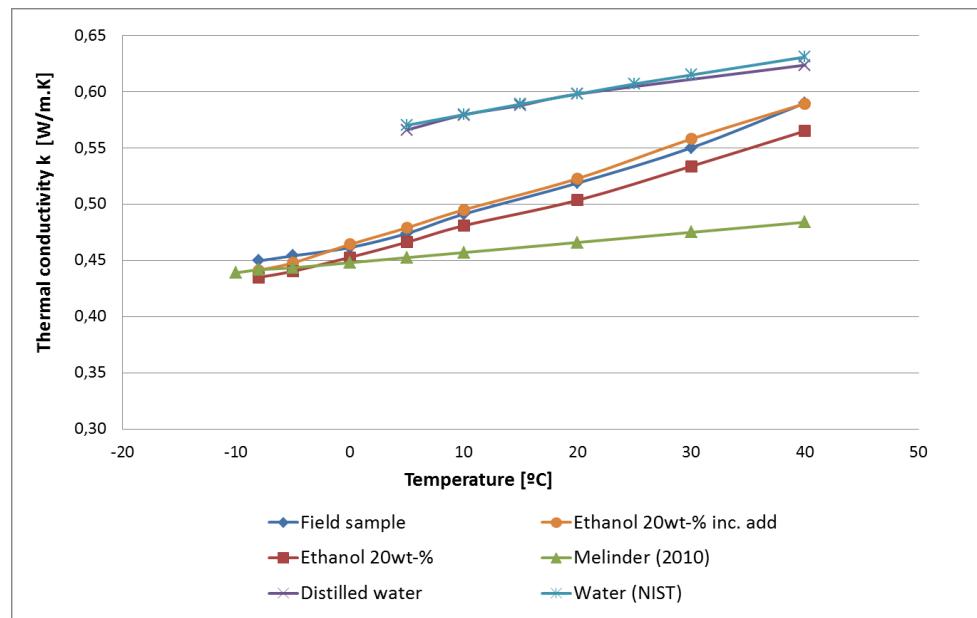


Figure 4: Thermal conductivity results.

4.2 Pressure drop and fluid to pipe thermal resistance in a U-pipe borehole heat exchanger

The pressure drop and the fluid to pipe thermal resistances in a U-pipe BHE have been calculated for the ethanol mixtures at three different volumetric flow rates (typical flow rates in BHE installations today), using the thermophysical properties presented in section 4.1 for three temperatures within the typical operating range of BHEs in northern Europe during heat extraction mode: +5, 0 and -5 °C. The idea is to identify the implications of using some of these properties in borehole heat exchanger design for typical flow conditions.

A compulsory first step before carrying out the calculations is the determination of the Reynolds number, which decides whether the flow regime for each case is laminar, transitional or turbulent, and indicates the correlations that should be used in the design process. The result of this calculation, introducing the laboratory density and viscosity measurements in equation (4), is presented in Figure 5. An axis line has been on purpose drawn at $Re = 2300$ in order to delimit the theoretical transition between laminar and turbulent flow.

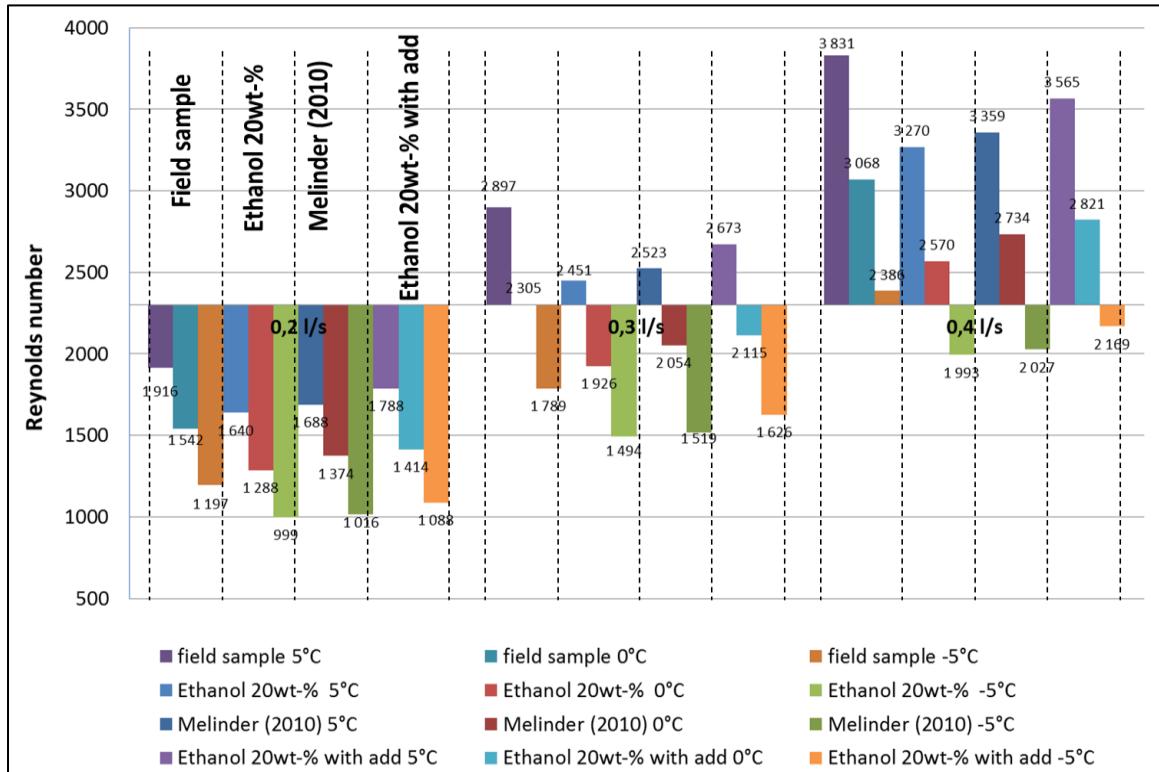


Figure 5: Reynolds number for different fluid properties at different flow rates in a PE40X2.4mm U-pipe BHE.

For all fluids, Reynolds number decreases with decreasing temperature level, related to the shape of the density and viscosity curves shown in the previous section of this paper. The properties of the field sample and the ethanol solution including additives give the highest Reynolds number when compared to pure ethanol and to the reference data. This is valid for all flows and temperature levels. In some cases when the flow is 0.2 and 0.3 l/s, the properties of the field sample results in turbulent flow while the pure mixture and the values from Melinder (2010) indicate laminar flow, for example.

As expected, the Reynolds number increases with increasing flow rate for a given temperature, being laminar or turbulent depending on the fluid properties. The effect of more viscous flows at lower temperatures for a given fluid sample becomes clear. At the lowest flow rate, the regime is laminar in all cases. At 0.3 l/s the regime is strictly dependent on the viscosity values while at the highest flow rate (0.4 l/s); the flow is almost always turbulent. It is suspected, however, that good heat transfer conditions cannot be guaranteed at such flow rates since it is known that the Reynolds number even varies from inlet and outlet along the U-pipe.

The flow regime has implications for a U-pipe BHE in terms of pressure drop and fluid-to-pipe thermal resistance, R_{fp} . The pressure drop and R_{fp} calculated for the conditions from Figure 5 are shown in Figure 6 and Figure 7, respectively. These results were obtained using the approach presented in section 3.2. The pressure drop indicates that, in a few cases, the result falls outside what could be intuitively estimated, although it is normal to expect that ΔP increases with decreasing temperature level as the viscosity and density become higher.

The internal fluid-to-pipe thermal resistance shown in Figure 7 shows that the convective layer inside the pipes can significantly affect the resistance if the flow is laminar, as expected. Laminar flow conditions become harmful and the highest flow is certainly preferred in U-pipe BHEs. Most of the conditions with the low and medium flow rates, independently of the fluid properties are unfavorable for heat transfer and for a proper thermal performance of the U-pipe. Even at the higher flow rate (0.4 l/s), laminar conditions are obtained at the lowest temperature levels. It seems, though, that the characteristics of the field sample representing the commercial products are more favorable than pure ethanol solutions, implying that installations may probably perform slightly

better than expected. It can be seen that, within each pipe, the resistance is normally lower than 0.02 Km/W for turbulent flow. If the pipes are symmetrically placed inside the borehole, the contribution of the fluid to pipe resistance to the total borehole resistance is, in this case, no more than about 0.01 Km/W. This number can be put in perspective as compared to the total local borehole resistance in a groundwater-filled borehole (which also includes the contribution the pipe properties and thickness, and the convection outside the pipes), typically ranging from 0.06 to 0.09 Km/W, see Acuña (2013), i.e. R_f -p is a small fraction of the total resistance. On the other hand, R_f -p would have a dominant weight at laminar conditions, as interpreted from Figure 7.

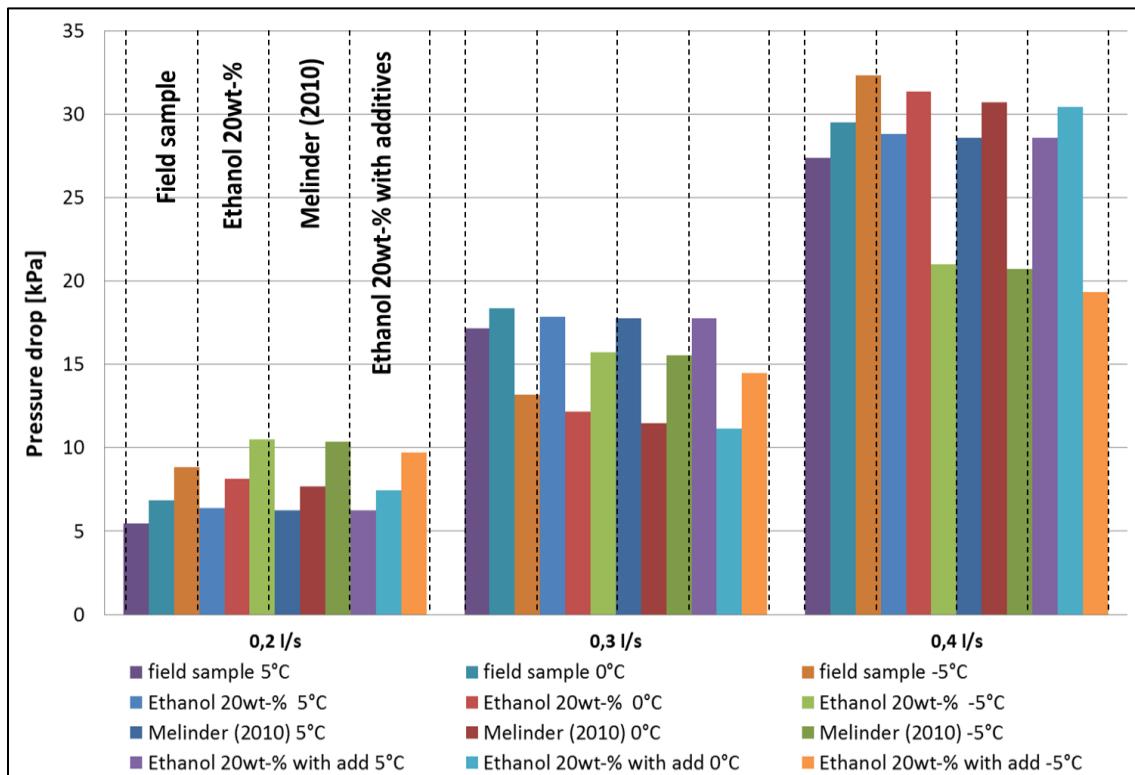


Figure 6: Pressure drop for different fluid properties at different flow rates.

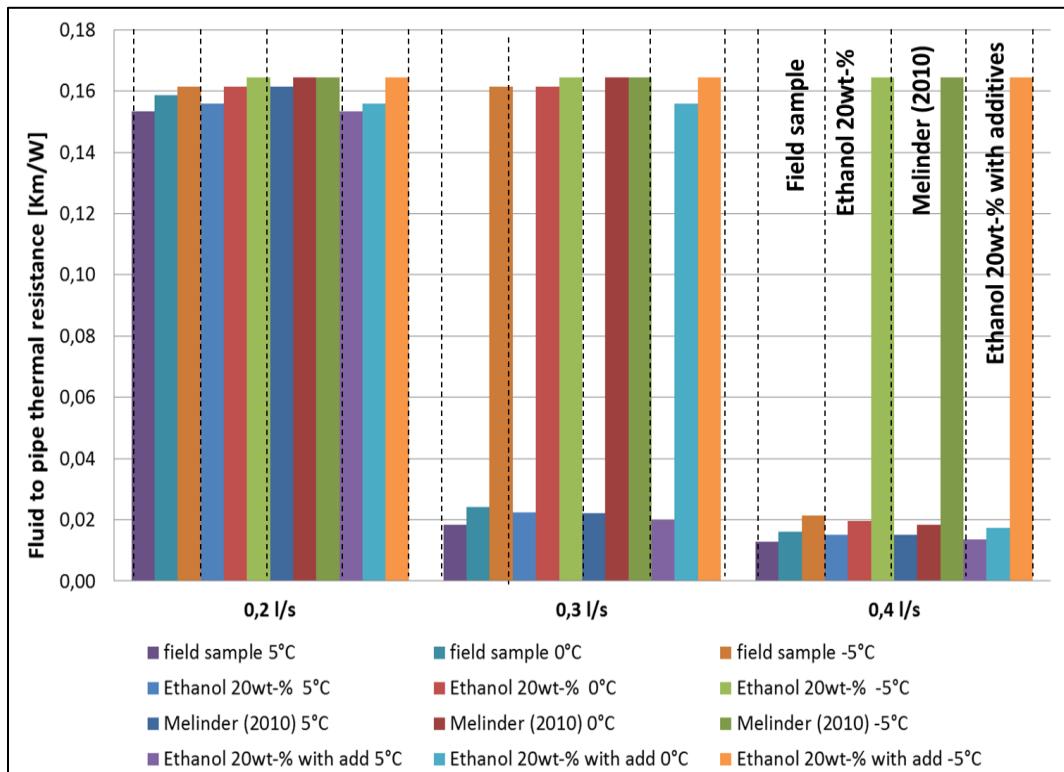


Figure 7: Fluid to pipe thermal resistance for different fluid properties at different flow rates.

5. CONCLUSIONS

Summing up, isopropanol and n-butanol did not have a significant effect on the freezing point despite the chemical nature of both additives. Results showed that presence of additives was not affecting so much the density (less than 0.4%) due to the similarity in chemical structure of alcohols used as the denaturing agents. Dynamic viscosity results showed that both samples containing isopropanol and n-butanol additives were having up to 10% lower dynamic viscosity than pure 20 wt-% ethanol. Specific heat capacity results showed a different tendency for 20 wt-% ethanol comparing to previously reported results. Sample of 20 wt-% ethanol including additives showed higher specific heat capacity than pure 20 wt-% ethanol and field sample. The increment in the specific heat capacity was observed especially for temperatures between -10 °C and +10 °C which are the common operating temperatures for secondary fluid used in borehole heat exchangers. A slight increment in specific heat capacity was observed in temperature range +30 °C to +40 °C as well. Isopropanol and n-butanol have higher specific heat capacity than ethanol and this fact can explain the increment of specific heat capacity value. Additionally, the additives present in field sample and 20 wt-% ethanol were having positive effect and higher thermal conductivity could be observed in comparison to pure 20 wt-% ethanol solution. The increment in thermal conductivity was around 1.5% at -10 °C and 4.4% at +40 °C.

In a U-pipe borehole heat exchanger, laminar conditions and low temperature levels should be avoided. The thermophysical properties must be carefully chosen in order to avoid errors in the design when the operation conditions are close to the transition between laminar and turbulent flow.

6. NOMENCLATURE

BHE	Borehole Heat Exchanger
C_p	Specific heat capacity [$\text{kJkg}^{-1}\text{K}^{-1}$]
D	Hydraulic diameter [m]
f	Friction factor [-]
h	Internal heat transfer coefficient [$\text{Wm}^{-2}\text{K}^{-1}$]
k	Thermal conductivity [$\text{Wm}^{-1}\text{K}^{-1}$]
L	Pipe length [m]
Nu	Nusselt number [-]
PE	Polyethylene
Pr	Prandtl number [-]
Re	Reynolds number [-]
$R_{f,p}$	Fluid to pipe wall resistance [mKW^{-1}]
TPS	Transient Plane Source
w	Velocity [m.s^{-1}]
wt-%	Weight concentration [-]
ΔP	Pressure drop [Pa]
μ	Dynamic viscosity [mPas]
ρ	Density [kgm^{-3}]

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