

Mixing grease with water

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ABSTRACT

The purpose of this paper is to convey a simple and effective method to mix water with grease and verify the homogeneity of the mixture using Karl-Fischer (KF) titration as the analysis tool. This is required to better develop a future water in grease sensor for on-line condition monitoring. This paper will also attempt to investigate how well KF titration functions as a tool for measuring water content in grease. Additional investigations include how the water content varies with time in grease samples open to atmosphere at different temperature. This research verifies a simple grease mixing method and investigates the water transport in water contaminated grease samples.

Keywords: grease; water contamination; water evaporation; Karl-Fischer titration

1 INTRODUCTION

Continuous *in situ* monitoring is important because maintenance costs can represent between 15 and 30% of the total cost of the machine [1]. Up to 25% of the costs of wind energy generation can be attributed to maintenance alone [2]. Knowing when to do maintenance will reduce costs, downtime and possibly make the product last longer.

A lubricant's performance is greatly affected by the presence of water; therefore, an increased understanding of the mechanisms behind how lubricants and water mix is of great importance. A 90% reduction in life of a journal bearing can be caused by the contamination of only 1% water in the lubricant [3]. Rolling element bearings are even more susceptible to damage from water and water content as low as 0.02% (200ppm) will negatively affect the service life [4]. Water reduces film thickness, causes flash vaporization and the resulting erosive wear and hydrolysis will cause hydrogen embrittlement. Water can age lubricants up to ten times faster due to the depletion of additives and destruction of base oils causing acid formation. These are a few of the reasons why detecting water is important as it is often the cause of false identification of failure [5].

Maintenance is often related to changing a lubricant with the goal of increasing the machine life. The simplest maintenance intervals are solely determined by time alone. This would be called preventative maintenance but predictive maintenance would be based on heuristics or lab tests. Typically, the most sophisticated maintenance intervals are determined by heuristic algorithms which take operating and/or environmental conditions into account but do not actually measure the properties of the lubricant [6, 7]. Predictive maintenance has shown to have significant reductions in maintenance costs at 60 to 80% through longer machine life and reduced ancillary costs, such as personnel hours and lubricant costs [7]. Although various algorithms exist for determining lubricant change intervals [8], maintenance personnel and engineers would still benefit from making laboratory based tests to compile enough data to produce decisions.

For a tribosystem, water will contribute to the degradation of both the lubricant and surfaces. Lubricant oxidation has been shown to be strongly related to the content of water. The oxidation of a lubricant is catalyzed by the presence of water. A higher water content increases the rate of lubricant degradation [9–11]. Water can be dissolved in the lubricant in small quantities without a visible difference. To some extent, water in lubricants is analogous to the humidity in air. Until the relative humidity of air is 100%, water does not condense and does not form visible droplets and thus is not visible to the human eye over short distances. This is the same case for lubricants. The water molecules are individually dispersed between the lubricant molecules and will not be visible until either the concentration is increased or the temperature is dropped to cause the water molecules to “condense” and form a water “fog” within the lubricant. This is now called an emulsified mixture of oil and water and will become milky in appearance [5, 10]. Emulsified water is much more damaging than dissolved water so being able to detect when water becomes emulsified would likely be a good design requirement for a sensor. Free standing separated water will be the most damaging of all [4] because it could be that at some point, a quantity of water could completely displace a lubricant where the lubricant is normally supposed to be protecting components.

Because of the differences between the states of water in lubricants (dissolved, emulsified and free water), water content alone may not be helpful because different lubricants have different saturation points (e.g. 1% water in one grease may be completely emulsified yet may not be in another grease). Knowing the water saturation points at the desired operating temperatures will help determine the upper limits for water content [4]. These values will have to be determined for each type of grease. This topic will have to be thoroughly investigated as this information for grease is not widely known and will likely be very different from grease to grease, as there are so many varieties and variations of grease in use.

Lubricants begin to have many problems once contaminated with water. Water contamination can cause rust and corrosion, water etching, erosion, vaporous cavitation, hydrogen embrittlement and also a reduced hydrodynamic film thickness [5, 12]. Water also degrades or consumes some additives and causes oils to degrade faster and cause the formation of acids, particularly synthetics. It also depletes oxidation inhibitors and demulsifiers, causes the precipitation of some additives (which then contributes to sludge) and competes with polar additives for metal surfaces [12]. Depletion of additives can indicate a chemical failure of the lubricant and can be used to judge when replacement is necessary [11]. When water becomes adsorbed onto metal surfaces displacing the oil and its additives, it causes further exposure to harsh environments and even direct metal on metal contact [4]. This hastens the wear of the components. Corrosion is the electrochemical reaction of the metal surface due to the presence of oxygen. Corrosion can be caused by the increasing total acid number (TAN). Once the TAN is above a certain value, it can cause corrosion of machine components and the lubricant [9, 13]. The amount of corrosion is also dependent on materials and environmental or operating conditions. Water etching can be caused by the formation of hydrogen sulfide and sulfuric acid from the lubricant degradation. Erosion occurs when water flash vaporizes on hot metals causing pitting [14]. Vaporous cavitation is common in pure clean lubricants as well but water causes the lubricant to be much more susceptible to this process. The implosion caused by the near instantaneous vaporization and condensing implosion of water can cause micropitting [4, 14]. Hydrogen embrittlement occurs when extreme conditions allow for the separation of the fundamental atoms. The hydrogen atoms can absorb into the metal surfaces making it brittle and more susceptible to high pressure damages [15]. The hydrogen can also collect in cracks and in metal grains resulting in crack propagation and spalling [4].

Grease is only a general description of thickened oil, not thick oil [16]. It typically consists of a base oil, thickener and additives. The thickener can be thought of as a sponge that holds the oil and the additives. Additives can be used for a multitude of reasons from increasing oxidation stability to improving the extreme pressure performance. Grease is a semi-solid so it does not flow as lubricating oil does because it requires an external shearing force to cause displacement, as long as the grease is used in its normal operating temperature range. Most types of grease will essentially “melt” if used above their “drop point” temperature and may not return to the original condition. Typically, grease will be tested by standardized ASTM mechanical or instrumental tests to establish the properties of the grease. These tests could include weight loss after aging, dropping point (when it “melts”), cone penetration (how hard it is), infrared spectroscopy (how oxidized it is), rheometer tests (how viscous it is), pin on disc tests and other friction and wear studies (how well it protects metals) [17].

Grease typically contains about 85% base oil, 10% thickener and 5% additives, though this varies significantly between different varieties [16]. The difference in the thickeners will likely pose a problem for developing sensors as they range from metallic soaps (such as lithium or lithium-calcium), complex thickeners (which are combinations of metal salts and soaps such as lithium complex), functional thickeners (where the thickener contributes to the lubricating properties, such as calcium-sulphonate) to non-soap thickeners such as polytetrafluoroethylene (PTFE) or clay [16]. Additives can vary as well, as they can include antioxidants, metal deactivators, corrosion inhibitors, extreme pressure, and anti-wear additives [11]. There are two types of antioxidants: primary and secondary. A lubricant can contain one or both. Primary antioxidants are radical scavengers and include amines and hindered phenols. Secondary antioxidants eliminate hydroperoxide decomposers (which form non-reactive products) and include zinc dithiophosphates or sulphurized phenols [11]. More research will have to be carried out to verify which additives will pose any problems for developing sensors due to the often different mechanical, electrical and optical properties.

The base oils are typically mineral or synthetic oils. The choice criterion are similar to when deciding which lubricating oil to use. Viscosity, oxidation stability and other properties are equally as important. The difference between base oils is likely to be smaller than the difference between thickeners, so base oil will likely contribute to fewer sensor design difficulties than thickeners will.

Research will have to be carried out to see which affects the various base oils, thickeners and additives will have on sensor design. Though not investigated in this paper, the oxidation of grease takes place on every component of the grease [11]. Now that it is known that water increases the oxidation rate, the oxide products of grease components may be interesting to investigate in future research.

1.1 Method

The experiment and mixing method will be discussed here.

1.1.1 Karl-Fischer Titration

This analytical technique has several variations in principle. For the purposes of this paper, only the coulometric principle will be discussed. Coulometric Karl-Fischer titration uses a two part reagent system where 1 mole of water will

stoichiometrically react with 1 mole of iodine. Additionally, a known amount of electric charge is required to cause the titration to occur so the amount of water present in the reagents can be very precisely measured [18].

Normally, the sample is added directly into the reagent chamber but another accessory can add greater functionality. It is called an oil evaporator. It uses a small vessel of pure dry base oil that is heated to around 150°C. It has an accurate temperature control. The vessel is airtight and uses a precisely metered flow of dry nitrogen gas. The nitrogen bubbles up through the hot base oil. The sample is dropped into this oil evaporator chamber so that the water can be turned into vapor and carried by the nitrogen gas into the reagent chamber. Since the reagents are extremely dry and very hydrophilic (it contains dry alcohols), the water vapor is very quickly absorbed and the instrument is able to titrate out the water.

The GR Scientific Coulometric KF instrument with oil evaporator will be studied to verify how consistent the results can be so that later measurements have a baseline of how consistent the data can be expected to be. It cannot likely be expected a mixed sample to have a lower standard deviation than new uncontaminated grease.

1.1.2 *Mixing grease with water*

The test setup is essentially two 50 ml syringes connected end to end such that they are sealed. The pumping action between the two syringes is intended to homogeneously mix grease with water. This method was chosen because of the low material cost and the simplicity of use. Therefore, it was desired to investigate how effective the method is for mixing water with grease.

A jig was made to hold the syringes to allow for easier operation. Once fastened to a table, this jig prevents the syringes from coming apart and holds it in place making it easier to pump the plungers. A short length of vinyl tubing of appropriate diameter is used to join the two syringes such that it is tight to a friction fit and does not leak. The system is airtight.



Figure 1. Grease mixing jig.

The samples were prepared on a standard four digit scale on a stand. Another syringe was used to inject fresh grease into the syringe on the scale. Thirty grams of grease were put in and different amounts of water were added in different amounts, ranging from approximately 0.1 grams to 3 grams to end up with 0.3% to 10% water. Water is added with a 3ml disposable pipette, drop by drop to meter the quantity of water. To arrive at smaller quantities of water in a sample, a portion of the already prepared grease samples could be added to fresh grease and mixed accordingly.

Thirty grams of grease corresponds to approximately 30 milliliters in the syringe. Before joining the syringes with the vinyl tubing, the plunger was put into the syringe with the grease and water inside and pushed in to read 40 ml. This leaves approximately 10ml of air inside.

Alternating cycles of pushing twice and pulling twice were used. When the plunger was pulled, the air bubble facilitated in making a more homogenous mixture. This is apparent when a negative gauge pressure is pulled and the 10ml air bubble is suddenly released, making a “pop” and distributing the grease and water mixture within the syringe being pulled upon. The grease may expand and become more like a foam as well, due to the reduced pressure. It appears to go back to its original state and does not retain the gas bubbles formed by the reduced pressure. It may be able to be assumed that the water in the mixture is evaporating and condensing over a larger surface in the process, thus facilitating the mixing of the two components.

Three types of greases were used, as shown in Table 1. These were chosen because calcium-sulphonate and lithium greases are common high performing greases used in bearings. Two calcium sulphonate greases were used because the mineral base oil was supposedly more water resistant.

Table 1. The greases used in this paper.

<i>Grease</i>	<i>Thickener</i>	<i>Base Oil</i>
A	Wide Temp Range Calcium-Sulphonate Complex	Mineral + PAO
B	High Temp Calcium-Sulphonate Complex	Mineral (water resistant)
C	Low Temp Lithium Complex	Mineral

1.1.3 Homogeneity of water mixed with grease

Several samples were made only to investigate how homogenous the sample became after mixing. For approximately 30 grams of grease in the syringe, ten samples were taken and measured in the Karl-Fischer instrument plus two more, representing the very first and the very last grease in the syringe.

Small samples of grease were taken with small strips of low-density polyethylene (LDPE) taken from disposable pipettes. This solved the problem of measuring very small samples of grease and placing them into the oil evaporator. Using syringes to inject the samples and other methods (such as with a spatula) proved very difficult, as it was easy to accidentally apply the grease to the wall of the oil evaporator chamber opening and consistent sample sizes were next to impossible.

The KF instrument operates best within a certain range of measured water. This means that dry samples require a larger sample size than wet samples with larger quantities of water. Since the exact water content of each sample was not known before testing, trial and error needed to be used such that each sample contained between 500 and 3000 micrograms of water. For the very dry Grease C, this was not possible to be within that range as it required such a large sample that it was simply too big to fit inside the opening of the oil evaporator. Because of this, a sacrifice of accuracy had to be made. This meant that grease samples with high water content only required samples as small as ~40 mg and the very dry samples required over 600mg and still contained less than 300 micrograms of water. This will be discussed more in detail in the results.

LDPE was chosen because it is a dry plastic with a low melting temperature. It is also easy to cut and work with. Grease samples were taken by dragging the small strip of LDPE into the grease. It was weighed before and after to find a precise weight. The KF instrument is then started and the sample is dropped into the oil evaporator.

1.1.4 Time dependence of water content in grease open to atmosphere

Grease samples mixed with water were left open to the atmosphere and later measured to see how well the water stays mixed. Scraping tools were made using a milling machine from square aluminum tubing with one and five mm cutouts to allow for spreading the grease at different thicknesses.

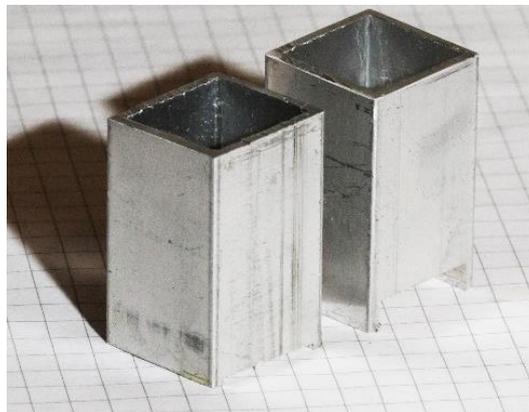


Figure 2. Aluminum scraper tools to apply grease in a layer. Left: 1mm cutout. Right: 5mm cutout.

The grease from the syringe was injected into the aluminum scraper as it was dragged along an aluminum plate. The layers were approximately one and five mm thick.

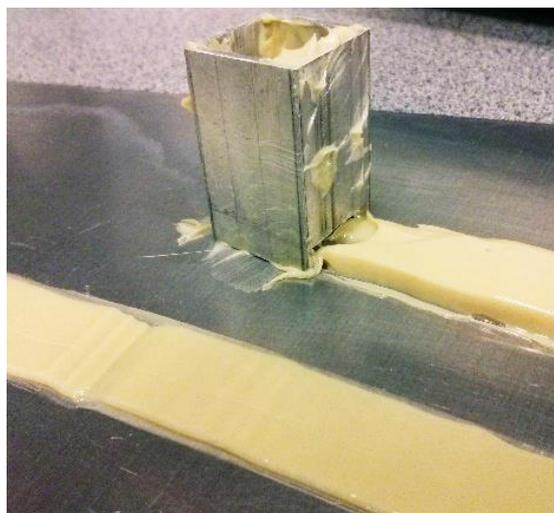


Figure 3. Example of the grease directly after application.

The samples were initially measured at least once per day to try to understand the water evaporation trend. Measurement samples were taken in the same way as described in the previous section with strips of LDPE. For the one mm thick layer, the strip was dragged through the entire layer. The five mm layer required scraping off the top 4mm with the scraping tool to expose the grease underneath, which was measured.

1.1.5 Temperature dependence of water content

Grease samples contaminated with water were placed in an oven at 65 and 90°C to see how quickly water can be evaporated out of the grease.

2 RESULTS

The measurement results will be given here.

2.1 Results from KF titration

Table 2 contains the approximate amount of water added to the grease, according to the scale. Grease A was supposed to be a grease naturally containing a little bit more water than other greases, therefore less water was added.

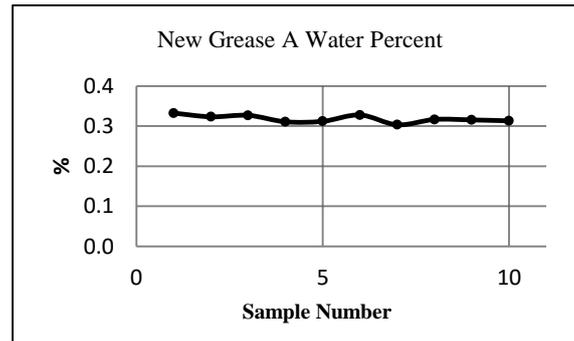
Table 2. The approximate amount of added water to the grease samples.

Grease	Sample	Added Water
A	High water contamination	3.00%
B	Low water contamination	0.26%
B	High water contamination	3.50%
C	Low water contamination	0.29%
C	High water contamination	3.5%

The KF instrument operates best when approximately the same quantity of water is titrated for each measurement. Thus, mixtures of grease and water with higher concentrations of water will need to be measured in smaller sample sizes. The instrument operates well when measurements contain between 500 and 3000 micrograms of water for each sample. If too much water is present, the titration takes too much time and much more reagent is consumed. Too little water and the measurement can become less accurate. This was apparent in grease C that had about 90% less water in uncontaminated grease. All measurements for grease A, B and the high water contaminated grease C were all intentionally within the known reliable range of 500-3000 micrograms of water.

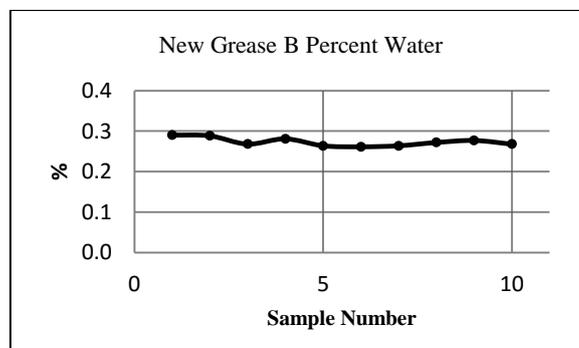
First, it was necessary to test the machine to verify the repeatability of the results so that it can be determined if the tested samples have a larger spread than the inherent error of the instrument. If there is a larger spread, it is important to know how much.

Figure 4-6 show the results of the machine test. For comparison, the data was normalized by dividing each data point by the average and the standard deviation was taken so it becomes comparable between data sets.



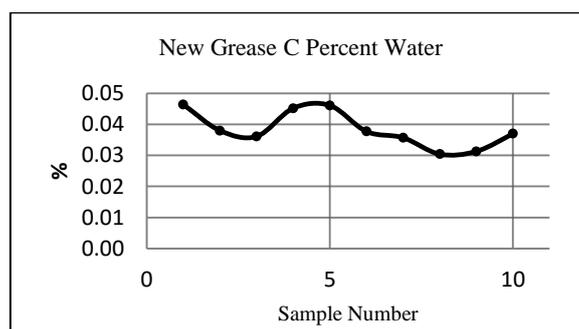
Average	0.318
Standard Deviation of Normalized Data	0.029

Figure 4. Plot of new grease A water content and statistics.



Average	0.273
Standard Deviation of Normalized Data	0.038

Figure 5. Plot of new grease B water content and statistics.



Average	0.038
Standard Deviation of Normalized Data	0.150

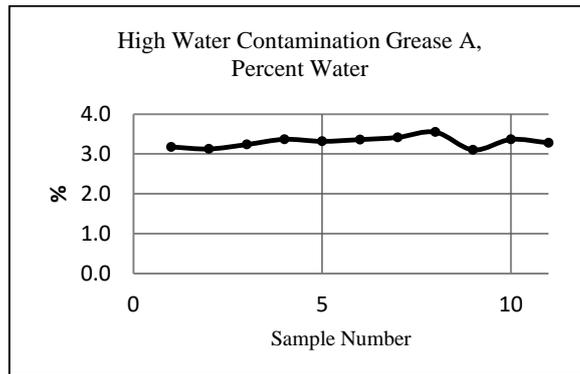
Figure 6. Plot of new grease C water content and statistics.

2.2 Homogeneity of syringe prepared samples

Results in this section verify how consistent the mixing is from the syringe mixing method. Each syringe has approximately 30 grams of grease within. The density is 0.9 g/ml. Twelve samples were taken out of the syringes in approximate quantities of 3ml at a time. The first sample is measured on each end of the extruded sample of grease since it

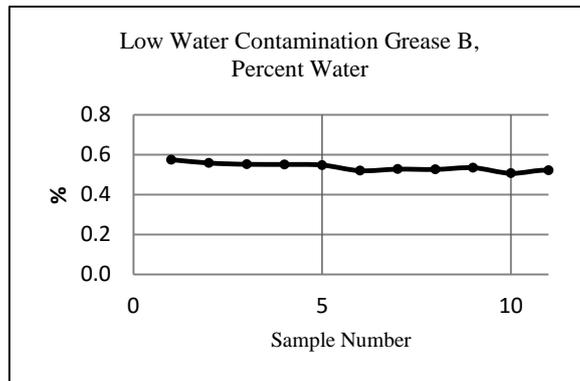
was desired to be known if the grease within the very end of the syringe was the same. The twelfth sample was taken from the inside the syringe nozzle to provide additional information as well.

Figure 7 -Figure 11 represent the water contaminated syringe samples.



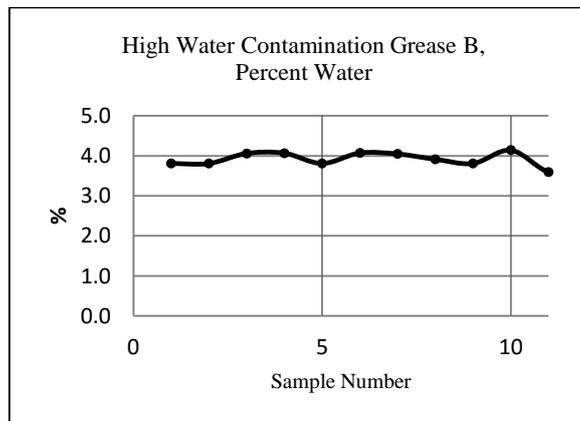
Average	3.290
Standard Deviation of Normalized Data	0.039

Figure 7. Plot of water contaminated grease A water content and statistics.



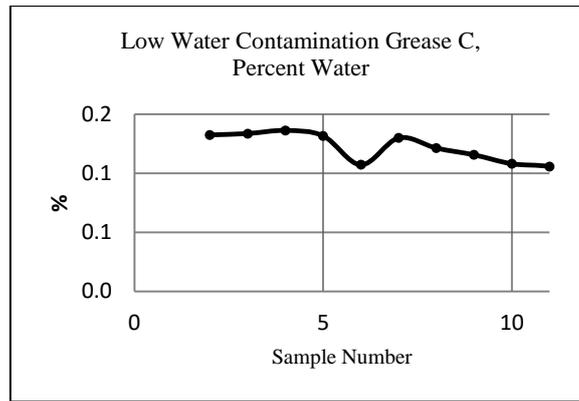
Average	0.533
Standard Deviation of Normalized Data	0.038

Figure 8. Plot of water contaminated grease B and statistics.



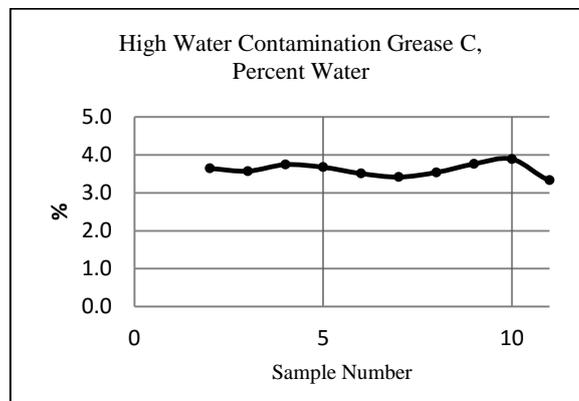
Average	3.886
Standard Deviation of Relative Data	.043

Figure 9. Plot of water contaminated grease B and statistics.



Average	.117
Standard Deviation of Relative Data	.120

Figure 10. Plot of water contaminated grease C and statistics.



Average	3.293
Standard Deviation of Relative Data	.057

Figure 11. Plot of water contaminated grease C and statistics.

Note that the high water contaminated grease C has the first data point omitted due to poor sealing on the cap. The data from all of the measurements shows that the instrument is capable of consistently measuring very dry to highly water contaminated grease samples and provide useful data. The standard deviation was comparable between datasets, except for the samples for grease C. There is an additional source of error when using this grease.

2.3 Time dependence of atmosphere exposed water contaminated grease samples

Time was a significant factor when samples were left open to the atmosphere at room temperature and humidity.

A curve fit was made using a custom least-squares regression curve following the form of Equation 1 where w_i is initial water content, w_{ss} is steady state water content after a significant amount of time, t is time and τ is a time constant (representing evaporation rate). It is a standard exponential curve fit allowing for a time constant and a y-axis offset.

$$\% \text{ water} = (w_i - w_{ss}) * e^{-\frac{t}{\tau}} + w_{ss}$$

Equation 1. Assumed curve fit function.

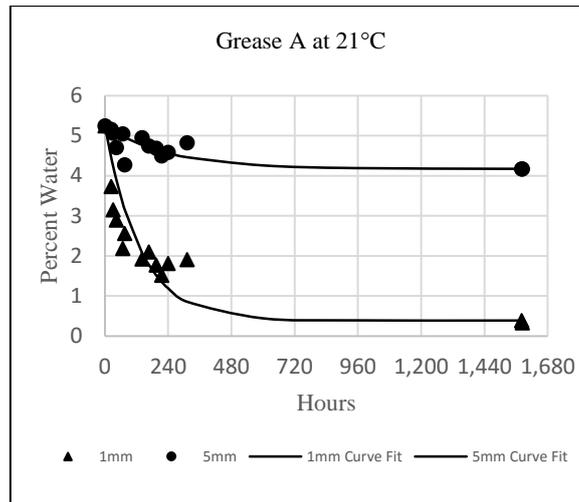


Figure 12. Water content at room temperature.

The r^2 is 0.822 for the 1mm layer and 0.500 for the 5mm layer. Two measurements were made at the very end of the test to represent what the end value will be. A least squares curve fit was then applied. The 5mm layer was the most difficult to measure, due to the highly user dependent nature of the measurement. If the layer was slightly thinner or thicker or the sample was taken slightly differently due to human error, the result would contain more scatter in the data. This explains the poor r^2 value. The 5mm curve fit is not especially good but at least shows what the expected trend can be. The average humidity at each measurement time was approximately 18% throughout the test and the average temperature was 21 °C. It was clear that the grease formed a dry layer on the outside, which acted as a barrier for further water transport to the atmosphere.

2.4 Temperature dependence of samples placed in oven

Temperature had a significant factor in the water content of the grease. A higher temperature resulted in a significantly different curve.

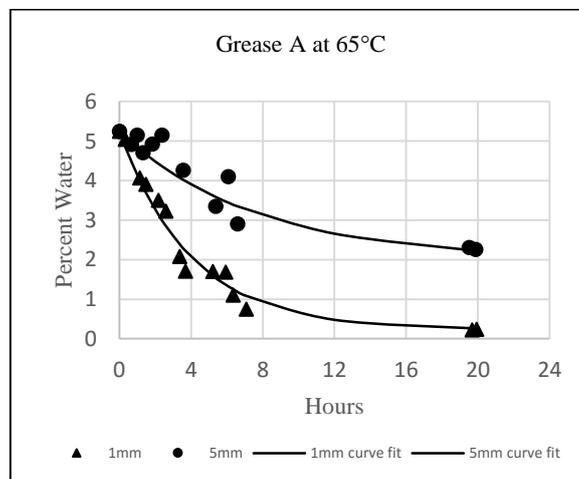


Figure 13. Water content in oven at 65°C

The time scale is obviously significantly different compared to the room temperature test. The measurements were made during the day and two follow-up measurements were made the following morning. The r^2 for the 1mm layer is 0.970 and 0.861 for the 5mm layer, indicating a reliable fit.

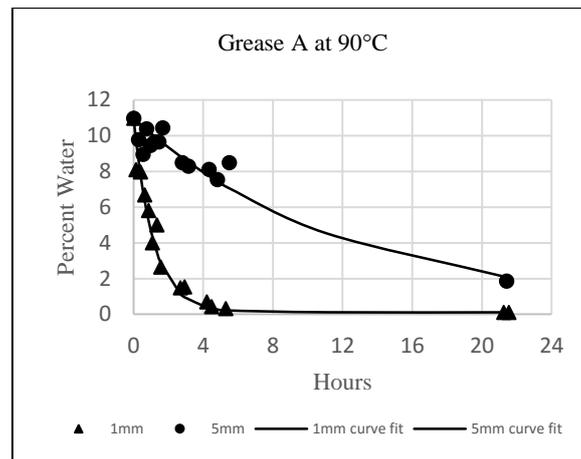


Figure 14. Water content at 90°C.

The curve fit has an r^2 of 0.967 and 0.909 respectively for the 1mm and 5mm samples. This again indicates a very good fit to the data. The higher temperature led to an even greater difference between the thin and thicker layers of grease. Again, a dry layer formed on the outside and appears to prevent water from being released into the surrounding atmosphere.

The grease visibly formed a dry layer on the surface of the water contaminated grease, as can be seen in Figure 15- Figure 16.

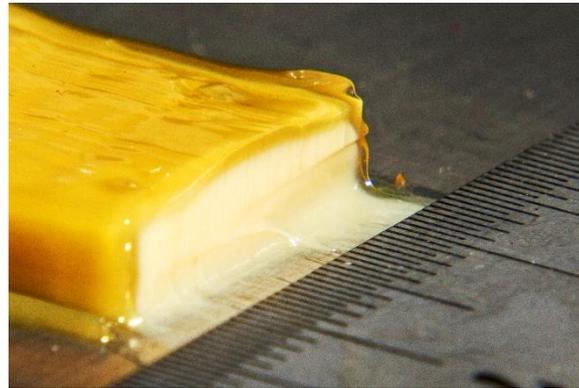


Figure 15. Example of water-contaminated grease after one week at room temperature. Approximately 5mm thick layer.

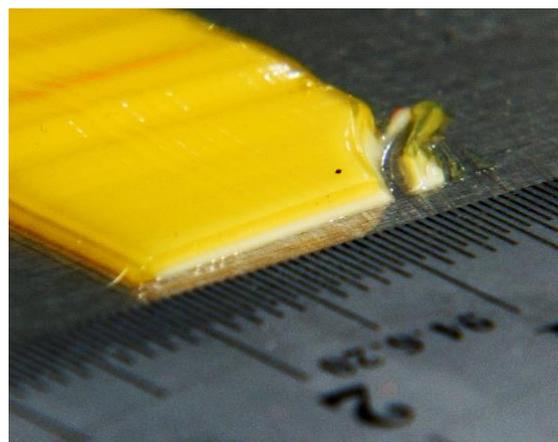


Figure 16. Example of water-contaminated grease. Approximately 1mm thick layer.

The dry layer in the 5mm thick sample is very visible and appears to be approximately as thick as the 1mm thick layer of grease. These photos were taken approximately 20 days after the grease sample was left open to the atmosphere at room temperature.

3 SUMMARY

This grease mixing method is suitable for use in continued development of sensor design. The normalized standard deviation of the water content of new grease was shown to be 0.029 to 0.038 for calcium-sulphonate complex grease, the latter being the water resistant grease with a slightly lower uncontaminated water content. The lithium complex grease was an order of magnitude dryer, thus leading to error that is more significant than with samples of higher water content. It had a normalized standard deviation of 0.150. Samples that were mixed with very low water content (approximately one drop of water per 30 grams of grease) had a higher normalized standard deviation and appeared to have a slightly downward slope. The samples contaminated with water led to a similar result, all of which otherwise had a standard deviation of 0.120 or less. The experiment has shown that this mixing method can be used to provide homogeneously mixed samples for further sensor development. This is an important step because it must be known if our sensors are measuring reliably produced samples.

Furthermore, knowledge of the water transport activity within grease may be important for sensor design in the future. This research has shown that this subject is complicated and warrants further research in the future. It also shows that grease may not release water into the surrounding air, even when heated to high temperatures and completely open to a dry atmosphere. It cannot be assumed that grease in a bearing will release water, even if it is heated to a high temperature.

4 ACKNOWLEDGMENTS

A special thanks goes out to SKF for their continued support and advice, especially Per-Erik Larsson and Piet Lugt.

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