Dielectric Thermoscopy Characterization of Water Contaminated Grease

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ABSTRACT

The temperature dependence on the dielectric properties of water-contaminated grease is investigated in this article. The purpose of this investigation was to identify the dielectric properties that could be measured to differentiate varying levels of water contamination in calcium sulfonate complex (CaS-X) grease with three different test cells with the purpose of eventually developing a grease condition sensor. Measuring the change in dielectric constant over a small temperature change yielded useful estimations of water content and amount of grease where the added water content ranged from 0.22% to about 5.5%. Additionally, other parameters such as incomplete fill/coverage of the sensors were investigated as a prestudy. The results from this set of experiments show that the measurement has potential to be developed into a condition monitoring sensor in the future.

EDITOR'S NOTE:

Measuring water content in hydrocarbons can be done a number of different ways, but few of them apply well to grease samples. This month's Editor's Choice paper investigates the use of three different dielectric cells (parallel plate, coaxial and interdigitated fringe) to estimate water content in greases. The results are very promising using calcium sulfonate complex greases, with further work required to determine suitability with different thickener technologies that are less hydrophilic, and different additive technologies. Should the technology work well on other greases, the technology could be considered for online monitoring.

Evan Zabawski, CLS
Editor

INTRODUCTION

Maintenance expenses can represent 15 to 60% of the total life cycle cost for machinery (Mobley (1)). For example, up to 25% of the costs of wind energy generation can be attributed to maintenance alone (Mumper (2)). Much of these expenses can be ascribed to the performance and reliability of the bearings, which are largely determined by the quality of the lubricant. This is often a lubricating grease, which will be further discussed later.

Knowing when to perform routine lubrication maintenance will reduce costs and downtime and possibly increase the usable life of the machine. Generally, the time for relubrication of a grease-lubricated bearing can be estimated if the lubrication conditions are known. In many applications, these conditions are not known, and continuous in situ monitoring of the lubricant condition is better for determining when relubrication is required.

The performance of lubricating grease is significantly affected by the presence of water. A 90% reduction in the service life of a journal bearing can be caused by the contamination of only 1% water in the lubricant (Noria Corporation (3)). Rolling bearings are even more susceptible to damage from contamination (Eachus (4)). Free water is detrimental, and knowledge of how water interacts with grease is therefore of great importance (Cyriac, et al. (5)). Aging of lubricants can be accelerated up to 10 times faster due to the depletion of additives and destruction of base oils, causing acid formation (Mortier, et al. (6)). These reasons and more make it apparent why detecting water is important because it is often the cause of false identification of failure (Duncanson (7)). Lubricated machine components often run into problems once contaminated with water. Water contamination can cause rust and corrosion, water etching, erosion, vaporous cavitation, and hydrogen embrittlement, among other problems (Cyriac, et al. (5); Dietz (8)). Water 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 (Agarwal (9)). 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 (Eachus (4)), which hastens the wear of the components. Corrosion is the electrochemical reaction of the metal surface due to the presence of oxygen. 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 (Braun and Hannon (10)). 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 (Eachus (4); Braun and Hannon (10)). Hydrogen embrittlement occurs when extreme conditions allow for the separation of the fundamental atoms. The hydrogen atoms can diffuse into the metal, making it brittle and more susceptible to high-pressure damage (Ray (11)). The hydrogen can also collect in cracks and in metal grains, resulting in crack propagation and spalling (Eachus (4)).

For a tribosystem (a system of lubricated components), 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, because oxidation of a lubricant is catalyzed by the presence of water. A higher water content increases the rate of lubricant degradation (Dietz (8); Fletcher and Edelson (12); Johnson and Ameye (13)). Water can also have an impact on grease-lubricated elastohydrodynamic lubrication contacts in the case of starvation (Cyriac, et al. (5)), which usually happens in grease-lubricated bearings.

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 called an emulsified mixture of oil and water and will become milky in appearance (Duncanson (7); Dietz (8)). 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. Freestanding separated water will be the most damaging of all water-lubricant mixtures (Eachus (4)). Because of the differences between the states of water in lubricants (dissolved, emulsified, and free water), water content alone could be useful to detect but in some cases may not be helpful because different lubricants have different saturation points. Knowing the water saturation points at the desired operating temperatures will help determine the upper limits for water content (Eachus (4)). These values will have to be determined for each type of grease. This topic will have to be thoroughly investigated because this information for grease is not widely known and will likely be very different from grease to grease, because there are so many varieties and variations of grease in use (Lugt (14)). It also could be that water will change the structure of grease.

Grease is a semisolid and therefore does not flow as lubricating oil does. Typically, grease will be tested by standardized ASTM mechanical or instrumental tests to classify the properties of the grease. For example, tests for weight loss after aging, dropping point (when the grease "melts"), cone penetration (how hard the grease is), infrared spectroscopy (how oxidized the grease is), rheometer tests (how viscous the grease is), pin-on-disc tests, and other friction and wear studies (how well it protects tribological interfaces from wear; Brown, et al. (15)). However, these methods cannot be used online because of the instrumentation involved.

Although grease composition can vary widely, according to Madius and Smets (16), grease is only a general description of thickened oil, not thick oil, and contains about 85% base oil, 10% thickener, and 5% additives. The base oil is either a synthetic or a mineral-based oil. 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. The difference in the thickeners will likely pose a problem for developing sensors because they range from metallic soaps (such as lithium or lithium–calcium), complex thickeners (which are functional lubricants in themselves and are combinations of metal salts and soaps such as lithium complex), to non-soap thickeners such as polytetrafluoroethylene or clay (Madius and Smets (16)). Additives can vary as well and can include different types of antioxidants, metal deactivators, corrosion inhibitors, extreme pressure, and antiwear additives (Johnson and Ameye (13)). 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. Only one grease will be examined in this article. It is a commercially available general purpose rolling element bearing grease with a calcium sulfonate complex thickener with poly-alpha-olefin and mineral base oil.

This article looks at the dielectric properties of grease, which is in the field of condition monitoring. To the authors' knowledge, there is no published research on dielectric parameters of lubricating grease. With the previously mentioned problems related to water contamination and the challenges associated with the many compositions of grease in mind, we can obtain a better picture of why making a grease condition sensor could be useful in expensive grease-lubricated machines and why a dielectric sensor could potentially be suitable to measure water in grease. Because this is a methodology article, no applications are directly examined.

METHOD

The experiment and mixing method will be discussed in this section. The grease used in this investigation was a commercially available calcium sulfonate complex grease with polyalphaolefin and mineral base oil. This grease will be referred to as CaS-X. Sample preparation, water measurement, and the experimental measurement will be explained in this section.

Water-contaminated grease sample preparation

The grease mixing setup is composed of two 50 ml syringes connected end to end with a short length of vinyl tubing such that they are sealed and airtight. The pumping action between the two syringes is intended to homogenously mix grease with water. This method was chosen because of the low material cost and the simplicity of use. How well it mixes water and grease was previously studied and it was found to be sufficiently homogenous (Dittes (17)).

A jig, shown in Figure 1, was made to hold the syringes to a table to allow for easier operation. Once attached to a table, this jig prevents the syringes from coming apart and holds it in place, making it easier to pump the plungers.

The samples were prepared on a standard four-digit scale on a stand (not shown) to hold the syringe to be filled. Another syringe was used to inject fresh grease into the syringe on the scale. Thirty grams of grease were added with different amounts of water. Water was added with a 3 ml disposable pipette, drop by drop, to control the quantity of water.

Thirty grams of grease corresponds to approximately 30 ml 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 10 ml of air inside. The air was found to be of benefit in mixing the samples.

Alternating cycles of pushing twice and pulling twice were used with a total number of 50 cycles. When the plunger was pulled, the air bubble assisted in making a more homogenous mixture. This is apparent when a negative gauge pressure is pulled and the 10 ml 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 is possible that the water in the mixture is evaporating and condensing over a larger surface area in the process, thus facilitating the mixing of the two components.

Table 1 shows the water content in the samples used in the experiment. The range in quantity of water was chosen because several drops of added water ended up increasing the water content of the 30 g sample of grease by 0.27%. This was thought to
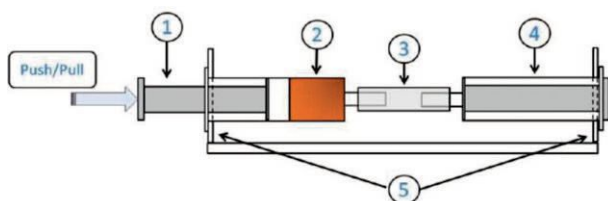
Table 1. Karl-Fischer determined water content of grease samples. The "new (pumped)" sample was "pumped" in the syringes as the water-contaminated samples were but with no added water. This was intended to act as a control to expose any dependence on the mixing process in the measurements. Due to slight variations in the manufacturing process, the new grease water content appears to vary slightly.

CaS-X grease samples	Added water set 1 (%)	Total water set 1 (%)	Added water set 2 (%)	Total water set 2 (%)
New	0	0.310 ± 0.06	0	0.326 ± 0.06
New (pumped)	0	0.310 ± 0.06	0	0.326 ± 0.06
1	0.27	0.58 ± 0.05	0.22	0.54 ± 0.05
2	0.76	1.07 ± 0.03	0.47	0.79 ± 0.03
3	1.4	1.71 ± 0.03	1.12	1.44 ± 0.03
4	3.05	3.36 ± 0.20	3.25	3.58 ± 0.20
5	6.29	6.60 ± 0.35	5.37	5.69 ± 0.35

be a rational starting point, because many bearings can contain that order of magnitude of grease, and several drops of water subjectively seemed like a good starting point to be able to distinguish, which could easily be caused by either water ingress or condensation. The amounts were increased arbitrarily until it was apparent that the saturation point of the grease was surpassed and the visible appearance changed. In addition, anything higher than the 6.6% water content of sample 5 would likely not provide useful information, because it is already a very significant level of contamination and bearing damage could follow shortly.

Figure 2 is a photo of the physical appearance of the grease with the varying amounts of water contamination used in this investigation.

Karl-Fischer titration

After the grease samples were prepared, the precise water content measurement was made with a Karl-Fischer (KF) titration instrument to ensure that the water content was precisely known. The instrument used in this experiment was a coulometric type water titration instrument. This analytical technique has several variations in principle, but the instrument used was coulometric. An oil evaporator was used in this experiment (this is a common accessory for KF instruments). The oil evaporator vessel uses about 30 ml of pure dry base oil that is heated
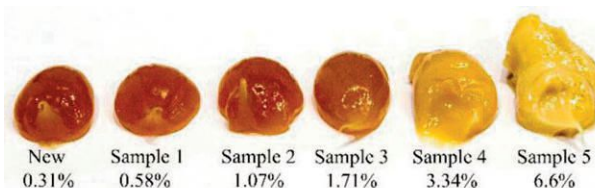

Figure 1. **Grease mixing jig: (1) syringe plunger, (2) grease within syringe, (3) tubing connecting syringes, (4) syringe housing, and (5) jig holding syringes.**

Figure 2. **Photo of grease samples showing the different physical appearance of heavily water-contaminated grease and grease with less water content. Notice that up to 1.71% water contamination, the grease appears essentially the same as new grease.**
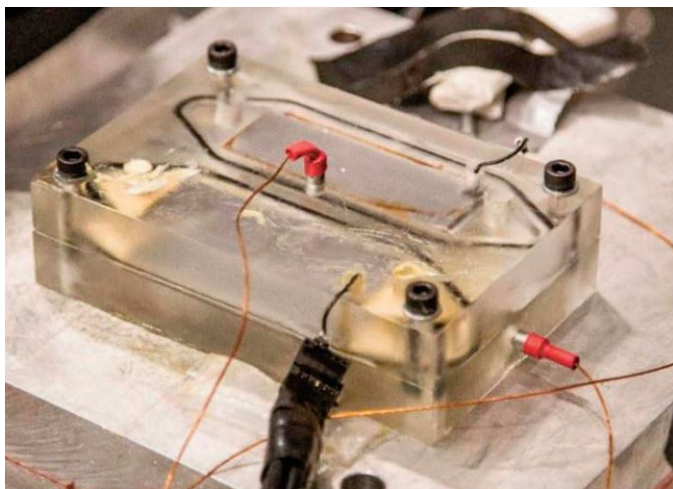
Figure 3. **Test cell used in the experiment. Three thermocouples are used to measure the temperature of the cell, one on the top and two on the sides. The aluminum block underneath is a water block attached to a heating and cooling unit. Only one of the cavities was used in this experiment.**

to a set temperature, in this case 140°C. The oil evaporator was modified to accommodate a high-speed stirrer to allow the use of greases, because the grease tended to stay clumped together and caused measurement errors due to a long measurement duration. Thin strips of low-density polyethylene strips were used to provide an easier method of placing the very small grease sample (between 30 and 150 mg depending on water content) into the oil evaporator. Experimentally, lowdensity polyethylene was found to be very dry and melts at a low enough temperature such that it melts in the oil evaporator and does not contribute to the water content of the measurement.

Experimental setup
The dielectric measurements were taken manually with a bench-top resistance, capacitance. Data weremanually recorded from
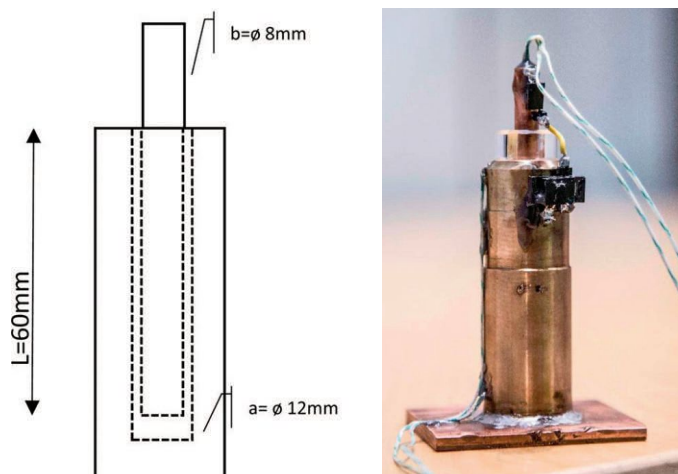

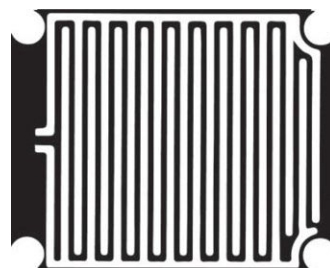Figure 4. **Sketch and photo of the coaxial test cell.**

Figure 5. **Diagram of fringe field capacitor plate used in the experiment. The traces are 1.2 mm wide and are approximately 0.01 mm thick. The traces on the board measure 38 × 41.5 mm.**

the resistance, capacitance, inductance (RCL) bridge. There was no internal averaging set in the instrument due to acceptably low levels of noise. Calibration of the unit was carried out using the measurement cables with connectors that allowed for an open and closed circuit calibration. The capacitance and the dissipation factor were recorded from the instrument for this investigation.

Temperature measurements were taken with a generic type-K thermocouple reader with generic thermocouple wire. Three cells were used and were built to have approximately the same empty capacitance such that the order of magnitude of the measurements would be similar. Three different test cells (a parallel plate test cell in Figure 3, a coaxial test cell in Figure 4, and an interdigitated fringe field capacitor in Figure 5 were used in this experiment to verify whether different configurations would work with this measurement principle.

The experiment order of operation is as follows:

1. Prepare grease samples.
2. Fill test cell(s) with grease sample.
3. Allow time for stabilization of temperature throughout the test cell.
4. Take measurements of temperature, capacitance, dielectric constant, and resistance.
5. Change settings on temperature control device and measure at next desired temperature and allow for temperature stabilization again.
6. Measure again.

New grease samples were prepared for each test cell because they were tested at different times and consumed enough grease to require this. However, the same grease and sample preparation techniques were used to make the experiment as repeatable as possible.

Parallel-plate capacitor test cell
The test cell was a rectangular parallel-plate type capacitor in a two-part polymethyl methacrylate (PMMA) enclosure. The plates were uncoated steel with dimensions of 30 × 85 mm. The separation distance was 3 mm. The test cell was made with two sides, though only one was used in this experiment. Cables were made from 50 Ohm coaxial BNC cables and attached with 0.1-in. standoff pins.

The test cell sits on top of a water block that is connected to

a heating and cooling unit. It has a useful temperature range between –18 and 100°C. The test cell is shown in Figure 3.

The capacitance of this configuration can be approximated with

$$C \cong \frac{k\varepsilon_o A}{d},$$

as given by Serway and Jewett (18) for a parallel-plate capacitor, where ε_o is the permittivity of free space ($8.85 * 10^{-12}$), k is the dielectric constant, A is the area, and d is the distance between the plates. The cables used were standard 50 Ohm coaxial cables with bayonet connectors to plug in directly to the RCL bridge. The cables were connected directly to the test cell with standard 0.1-in. header pins.

Additionally, a thermally and electrically shielded enclosure (not shown) was fabricated to reduce electrical noise and help insulate the test cell. It was made out of polystyrene foam and stainless steel fiber cloth.

Type-K thermocouple probes were manufactured and embedded into the PMMA enclosure. Because a small temperature variation was inevitable in a measurement cell made of thick plastic, the temperature of the top was averaged with the sides to get an approximate temperature of the inside.

Coaxial capacitor test cell

Another test cell was manufactured to reduce time for heating and cooling, shown in Figure 4. The body is a solid piece of bronze and the base is a solid copper plate. This configuration was also chosen because it was easier to reduce air bubbles in the internal cavity, which could help reduce experimental variations. It also required a much smaller sample size.

The capacitance of the coaxial test cell configuration can be approximated as follows:

$$C \cong L \frac{2\pi k\varepsilon_o}{\ln\left(\frac{b}{a}\right)},$$

as given by Serway and Jewett (18) for a coaxial capacitor, where ε_o is the permittivity of free space, k is the dielectric constant, b is the outside diameter of the inner conductor (8 mm), a is the inner diameter of the outer conductor (12 mm), and L is the length of the portion inside the inner diameter (60 mm). The cables used were standard 50 Ohm coaxial cables with BNC connectors to plug in directly to the RCL bridge. The cables were connected directly to the test cell with standard 0.1-in. header pins. Standard type-K thermocouples were glued to the bronze housing and onto the end of the inner copper element.

Fringing field capacitor test cell

An additional test cell was used that is capable of measuring with only a surface, though it does additionally measure a certain volume. It is called a fringe field capacitor and is similar in concept to what is used on many of today's capacitive touch-screen devices (3M Company (19)). This concept was chosen

as part of the investigation because research (Dean, et al. (20)) showed that the response of such a sensor is the same as that with other typical capacitive sensor designs, such as the parallel plate and coaxial variation described in this article.

The calculation for an approximation for this configuration of an interdigitated fringing field capacitor, where one "line" is as follows (Abu-Abed and Lindquist (21)):

$$C_{line} = \varepsilon_0 \left(\varepsilon_{pcb} + \varepsilon_g\right)\frac{K(k_1)}{K(k_2)} + 2\varepsilon_0\varepsilon_g\frac{t}{s},$$

where ε_o is the relative permittivity of free space ($8{:}85 * 10^{-12} F/m$), and ε_{pcb} and ε_g are the dielectric constants of the PCB material and grease, respectively

$$k_1 = \sqrt{1 - \left(\frac{w}{2(w+s)}\right)^2}, \quad k_2 = \frac{w}{2(w+s)},$$

where w is the width of the digits, s is the distance between the traces (edge to edge), and t is the thickness of the digits (the copper traces on the PCB). The capacitance of one pair of traces can then be calculated:

$$C_{line} = \varepsilon_0 \left(\varepsilon_{pcb} + \varepsilon_g\right)\frac{K(k_1)}{K(k_2)} + 2\varepsilon_0\varepsilon_g\frac{t}{s}.$$

K represents the complete elliptic integral of the first kind calculated with the estimate in the following equation, using the arithmetic–geometric mean calculated to the fourth order where k_1 and k_2 are substituted into the equation and used, respectively.

$$K(k) = \frac{\frac{\pi}{2}}{agm\left(1, \sqrt{1-k^2}\right)}.$$

Finally, the total capacitance can be calculated using

$$C_{total} = C_{line}(N-1)L,$$

where L and N are the length and number of digits. Temperature measurement was carried out by placing a standard type-K thermocouple into the grease on top of the sensor.

The required minimum thickness of the grease sample was determined experimentally by reducing the thickness of the sample until the capacitance of the sensor was reduced. A plastic plate was raised and lowered until it was certain that a certain thickness had no effect on the measured capacitance. This was determined to be approximately 1 mm thick and an extra millimeter was added to ensure that only the grease properties were being measured, not those of the plastic cover. The grease layer was thus approximately 2 mm thick in this experiment.

▶▶▶

Theoretical background

The objective of this article is to investigate the development of a method using dielectric measurements to detect water in grease. For this experiment, understanding the chemistry of water is important to learn what is actually being detected because the dissociation of water plays an important role in almost every case where it is present (Trout and Parrinello (22)). One limitation of a sensor of this type could be that it may not function well below 0°C. Once water freezes, it has significantly different dielectric properties due to the more limited movement of the molecules (Lewowski (23)). However, this was not investigated in this work and will be left for future studies.

With the parallel-plate and coaxial test cells that measure a volume in an enclosed space, air bubbles posed a problem with keeping the results consistent and linear. However, due to the nature of the dielectric thermoscopy method, it is not as sensitive to the quantity of the mixture inside the test cell as other dielectric measurement methods are, such as merely measuring the value of the dielectric constant. This is because the overall capacitance at a given temperature is entirely proportional to the amount of material within a test cell, where the dielectric thermoscopy measurement is measuring the change in properties that correlate to water content.

Frequencies of 20 Hz, 90 Hz, and 100 kHz were chosen for the dielectric measurements because they have no close higher order multiples with either 50 or 60 Hz, which should reduce measurement noise regardless of the local electric network where future measurements could be made because some countries use a different network frequency. One hundred kilohertz was chosen as a significantly higher measurement frequency to better understand the relationships between frequency, temperature, and the dielectric constant but is not used in any estimations in this investigation at this time. There are a number of advantages to using a low-frequency measurement. The test cell geometry and configuration are not as important as with radio frequency dielectric measurements (Wu, et al. (24)), reducing possible errors with design and manufacture of the measurement device. It was also stated in Wu, et al. (24) that low-frequency measurements are cheaper and easier to implement.

Dielectric thermoscopy functionality

The term "dielectric thermoscopy" was coined to describe a measurement that quantifies the change in the dielectric constant over a change in temperature. Typically, in dielectric measurements, a constant temperature and frequency are used to quantify dielectric properties but sometimes lead to nonlinear

or poor relationships with estimating the water content of samples (Wu, et al. (24)).

There is more than one physical property that contributes to the functionality of this measurement. Research shows that the dielectric constant of water rises rapidly as the frequency of measurement becomes lower (Angulo-Sherman and Mercado-Uribe (25)). This is due to two properties of water: the first results from the ability of water to separate into ion pairs that can move toward the charged electrodes, decreasing reactance and increasing capacitance. Reactance is the imaginary part of impedance, meaning that a smaller magnitude capacitive reactance results in a higher opposition of the capacitor to a change in voltage or current. The ions increase ionic conduction, allowing electrons to move more easily and, additionally, with a lower frequency the ion pairs can separate farther resulting in more stored energy ("Ion Association" (26)). The second involves the dipole pairs of water molecules that polarize according to the direction of the electric field (Lewowski (23)). This happens only with low frequencies (in the range of tens or hundreds of Hertz, not megahertz or higher) because there is a greater time between alternating electric fields, thus allowing for a charge to be stored in either polarized dipoles or ion pairs due to it being a "slow" process (Trout and Parrinello (22)). Normally, dielectric properties are measured at frequencies above 1 MHz (i.e., radio frequency–based techniques) to reduce the mentioned increases in dielectric constant but are more complicated than low-frequency measurements (Dean, et al. (20)). In other words, more typical dielectric measurements use high frequencies (in the range of megahertz to gigahertz), so the contribution to the impedance of the measurement from ionic conduction is less than that of the impedance due to the capacitance (Lewowski (23); Bonanos, et al. (27); Flaschke and Trankler (28)).

Additionally, an increase in temperature results in a corresponding increase in the dielectric constant due to an increased propensity to separate into ion pairs. This results in a decrease in the pH of distilled water (Shiue and Matthewson (29)). This means that water has a much greater temperature dependence on the dielectric constant at low frequencies, which enables the measurement outlined in this article. An example of the effects is given in Figure 6 with grease samples from set 1 in Table 1. At the measurement frequency of 90 Hz, it can be seen that the temperature dependence on the capacitance is much greater than that with the higher measurement frequency of 50 kHz.

It was also shown in Angulo-Sherman and Mercado-Uribe (25) that increased levels of contamination through ions (i.e., salt) only amplify the outlined effects, which indicates that this is a reasonable measurement principle for lubricant condition monitoring due to the fact that increased conductivity causes a greater rate of oxidation of metal components and is thus more damaging to the mechanical system (Craig (30)).

RESULTS AND DISCUSSION
Measurements from the experiment that varies the amount of water contamination, frequency, and temperature are given and explained in this section. Repeat experiments with reapplica-
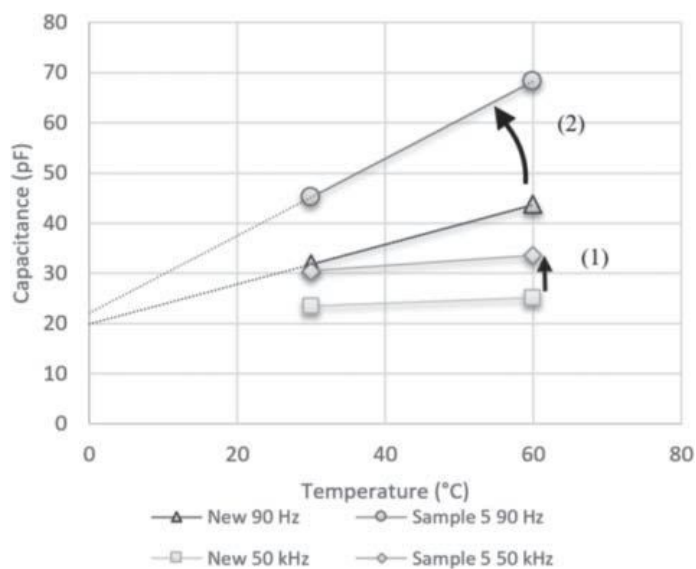

Figure 6. **Example of the relationships between capacitance, temperature, frequency, and water content in the dielectric thermoscopy measurement (actual measurement data used in representative plot). (1) Represents the change at 50 kHz when the water content is increased. The slope is practically unchanged, but the values are shifted upwards. (2) Represents the change at 90 Hz, where not only does the average value change but the slope changes as well.**

tions of the same grease samples have been shown to be within approximately 3%, probably due to slight inhomogeneities in the prepared samples of water and grease. Subsequent measurements of the same undisturbed grease sample on/in the test cells have an error of less than 1%. This is likely an indication that the measurement is reproducible, but it is impossible to guarantee that the water and grease composition and position is the same with each subsequent sample preparation and application on or into the test cell. The experiments have shown that a larger difference is caused by sample inhomogeneities and sample preparation instead of within the measurement itself.

Influence of frequency
As stated earlier, low frequencies give a higher dielectric constant measurement with water because of the unique properties of water. Figure 7 shows the difference in the capacitance temperature slope relative to water content and frequency.

Influence of temperature
It has been established that an increase in temperature results in an increase in dielectric constant for water; Figure 8 shows this relationship from experimental data. The capacitance follows a line very close to linear, which for the purposes of this article shows that it is acceptable for the outlined measurement.

One other consideration is how large of a temperature change is required to ensure that a good measurement can be taken. It was found that if the temperature has stabilized after a reasonable amount of time (around 30 min in the case of the fringe field capacitor), the dielectric thermoscopy method could
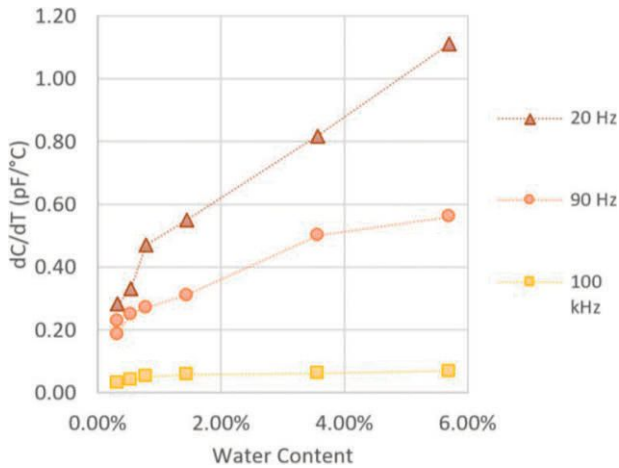
Figure 7. **Frequency considerations for measurement. This plot displays the dramatic difference between 20 Hz, 90 Hz, and 100 kHz despite the large comparative separation between 90 and 100 kHz. This is due to the low-frequency effects outlined in Angulo-Sherman and Mercado-Uribe (25). The fringe field capacitor was used in this example.**
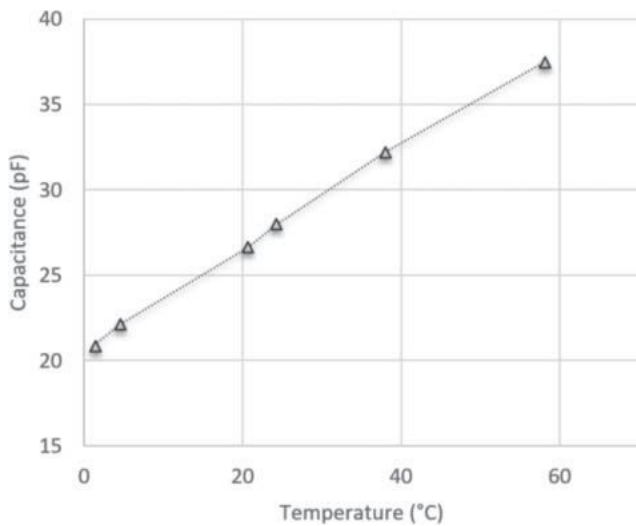

Figure 8. **Relationship between capacitance and temperature with a water-contaminated grease sample on the fringe field capacitor at a measurement frequency of 90 Hz. The capacitance rises linearly with temperature. The measurement was with sample 5 from set 1 of the grease samples.**

be used over a small or large temperature difference. Table 2 gives one example of sample 2 from the fringe field capacitor experiment. The capacitance temperature slope is essentially the same, even with a small temperature change of only 2.5°C.

The capacitance–temperature slope is calculated as $CT = \frac{C_1 - C_2}{T_1 - T_2}$, which represents the term dielectric thermoscopy.

In the initial experiment with the PMMA capacitor plate setup, a frequency of 90 Hz and temperatures of 30 and 60°C were used. Small deviations from the desired temperature did not seem to contribute significantly to error, because the meth-
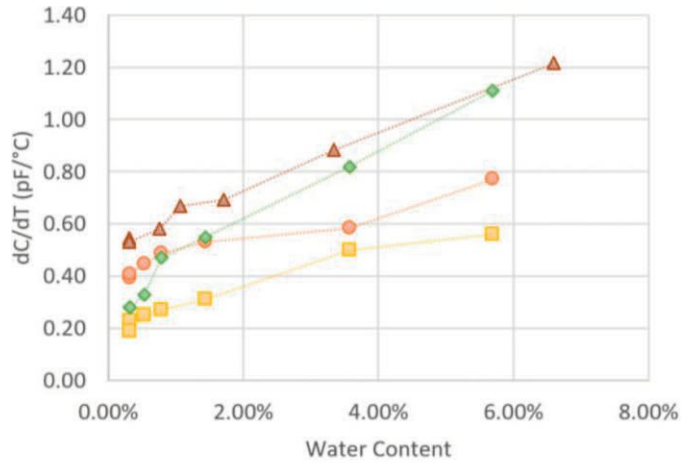

Figure 9. **Capacitance–temperature slope given over the known water content of the tested samples.**

Table 2. **Example of robustness of measurement in the fringe field capacitor. A small temperature change of 2.5°C and a large temperature change of 29.1°C were used. The measurement was from sample 2 from set 2 of the grease samples.**

Temperature change (°C)	dC/dT
27.5–30.0	0.48
30.0–59.1	0.47

od itself relies on the change in capacitance over a temperature change. Due to the significant thermal mass and low thermal conductivity of the test cell, a time of approximately 3 h was required to ensure a reasonably uniform temperature.

The results from each tested sample are given in Figure 9. A trend can be seen of increasing *dC/dT* with increasing water content.

Temperature-adjusted capacitance

The data in Figure 10 on Page 70 and Tables 3 and 4 on Page 71 are adjusted by creating a line fit between the data points and estimating what the capacitance should be at 30°C. This is to make the data comparable due to the strong temperature dependence on the measurements and the fact that it was found to be impossible to ensure that every measurement was exactly the same temperature.

Figure 10 shows the temperature-adjusted values of the capacitance in the plot. There appears to have been a measurement error in the coaxial test cell, because it can be seen that increasing water content does not lead to a higher absolute value for the capacitance. Despite this error, however, the dielectric thermoscopy measurement as shown in Figure 9 still clearly ▶▶▶
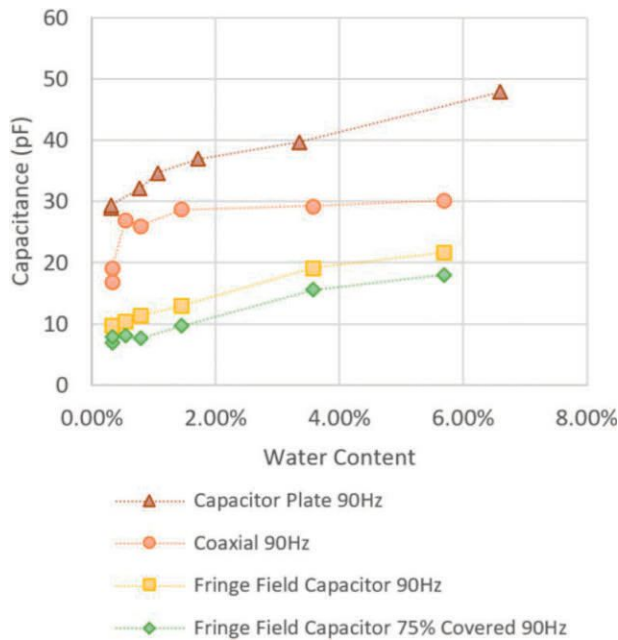
Figure 10. **Capacitance scaled to 30°C. Note that the coaxial test cell likely had problems with air bubbles in at least the last two measurements, because the capacitance does not increase with increasing water content.**

shows an increase in the measurement, which corresponds to an increase in the water content. One possible measurement error could have been air bubbles within the coaxial test cell. This would reduce the capacitance of the measurement.

Air pocket and incomplete coverage considerations

The fringe field capacitor was used to show that even with air in place of grease, the dielectric thermoscopy method can still be used to provide information about how much water is present in the mixture. This sensor was chosen because of the comparative ease in providing an incomplete fill. The two other configurations would be more challenging to reproducibly apply air pockets inside.

This experiment scraped a portion of the grease off the surface of the sensor. Approximately 25% of the grease was removed. This resulted in a change in capacitance as given in Table 3 and 4. Figure and show the difference between the fringe field capacitor fully and partially covered. It can be seen in Figure 11 that the absolute value of the capacitance measurement is scaled roughly accordingly to the amount of grease on the surface. However, the dielectric thermoscopy measurement in Figure 12 is not as significantly changed, indicating that this could be a reliable measurement that would help differentiate between grease loss and small gains in water. Merely measuring the dielectric constant would not only lead to problems with

temperature as a variable, but significant grease loss coupled with a very small addition of water could be indistinguishable from a properly operating system.

It can be seen from Table 3 and 4 that the approximate amount of coverage on the sensor can be estimated with the given data. Because the $C_{100\%}$ value may not be known, a lookup table would likely be required. Based on the capacitance temperature slope, the $C_{100\%}$ value could be determined and used in
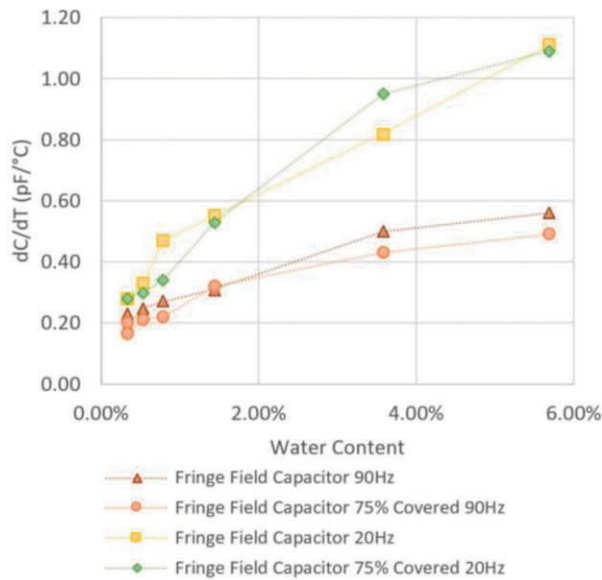
Table 3. **Capacitance measurement at 90 Hz scaled to 30°C with the sensor completely covered and 75% covered. The percentage covered is calculated as follows: Covered % =** $\frac{C_{75\%} - C_{empty}}{C_{100\%} - C_{empty}} * 100\%$**, where C represents capacitance.**

CaS-X samples	Total water (%)	100% Covered capacitance (pF)	75% Covered capacitance (pF)	Calculated % covered
	Empty	12.69	—	
New	0.31	22.36	19.65	72.0
New (pumped)	0.31	22.51	20.57	80.3
1	0.58	23.2	20.74	76.6
2	0.76	24.07	20.31	67.0
3	1.41	25.62	22.36	74.8
4	3.89	31.85	28.77	81.2
5	6.08	34.39	30.7	83.0
Average				76.3

Table 4. **Capacitance measurement at 20 Hz adjusted to 30°C with the sensor completely covered and 75% covered.**

CaS-X samples	Total water (%)	100% Covered capacitance (pF)	75% Covered capacitance (pF)	Calculated % covered
	Empty	10.96	—	
New	0.31	24.47	20.54	66.6
New (pumped)	0.31	24.56	20.43	65.6
1	0.58	26.2	22.67	73.9
2	0.76	27.7	22.53	65.6
3	1.41	29.85	27.3	85.1
4	3.89	40.8	34.85	78.8
5	6.08	45.29	39.9	83.5
Average				74.8

Figure 11. **The temperature-adjusted absolute value of the capacitance is roughly scaled accordingly to the amount of grease on the surface of the sensor in this plot. The temperature of this measurement is 30°C.**
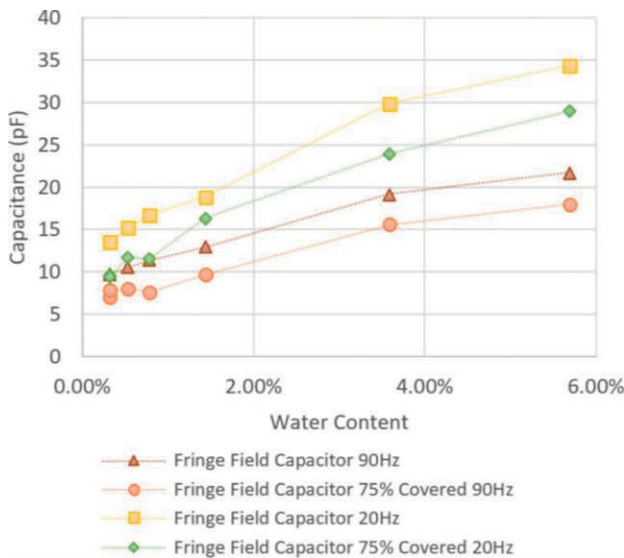

Figure 12. **Plot of the capacitance–temperature slope shows that the difference between fully covered and partially covered is fairly small.**

the calculation. The deviations in the data are likely caused by human error in the quantity of grease scraped off the surface. Because grease has an affinity to stick to surfaces, the grease could have smeared more on some samples than others due to slight variations in human error, leading to a higher or lower measurement. However, the average of the measurements for both frequencies was remarkably close to the goal of 75%.

Possible measurement errors
A contribution to measurement error that was discovered after the experiments is outlined in Figure 13 on Page 72. The process was shown to be nonreversible. After approximately 1 h, the measurement was repeatable during the rapid heating and cooling cycles that simulated the measurement. However, during the first hour, the measurements followed the trend in Figure 13 while the temperature was being held constant.

CONCLUSION
In this article, water-contaminated calcium sulfonate complex grease with a water content ranging from approximately 0.3 to 6% has been investigated with a measurement method called dielectric thermoscopy. This measurement quantifies the change in dielectric constant over a temperature change. As an initial study, a temperature difference of approximately 30°C was used, but it was also found that smaller temperature changes (2.5°C) also pro-

vided similar results. The capacitance temperature relationship in the studied range (above 0°C and below 60°C) was found to be sufficiently linear to be able to rely on it for this measurement.

The method was shown to be reasonably accurate using three different test cells, parallel plate, coaxial, and interdigited fringe field capacitor plate. Because the method appears to function in these three significantly different configurations, it shows promise to work in other configurations as well.

The interdigitated fringe field capacitor is a likely candidate for implementation within a grease-lubricated component where water contamination is detrimental to machine health. Research would have to be carried out to discover the most advantageous locations where water is likely to collect, either from condensation from heating and cooling cycles or from ingress due to failed seals. The fringe field capacitor can be configured in a multitude of different ways to either optimize for sensitivity or measure a larger volume. A number of different shapes could be used, and this will need to be researched as well to optimize the yet unknown advantages or drawbacks to the different possibilities. Electronics would be limited and could potentially be a component of existing condition monitoring hardware due to the very limited resources required for this concept.

Because incomplete coverage/fill in sensors was a consideration, this was also investigated and the measurement was found to not only continue providing useful data on water content but would also give an idea on how much grease is remaining on the sensor. A fringe field capacitor was used for this experiment and 75 and 100% coverage was used. Though the goal was to give 75% coverage, the sensor provided a calculated 74.8 and 76.3% with 20 and 90 Hz, respectively, using the average of all the test samples in the experiment.

Between the water content range of approximately 0.3 and 6% water, the dielectric thermoscopy measurement provided a useful correlation to water content with the chosen frequencies in the measurement. The correlation was likely good enough to be able to develop a model in the future.

Future work could include studying different types of grease with dielectric sensors. The CaS-X grease used in these experiments is quite hydrophilic, meaning that it seems to absorb large amounts of water in emulsion (even up to 50%), where other greases can only absorb a small percentage. These properties could change how the sensor functions considering that the greases will behave differently once exposed to water. Additionally, other greases will likely have different thickeners and different quantities of polar additives, which may change the sensor properties. Although preliminary experiments have not shown this to be a significant problem because water remains the dominating component of the measurement, it will still be necessary to ensure that there are no grease incompatibilities. Miniaturizing and testing a sensor in situ would also be valuable future research.

Other sensor techniques should be investigated as well, such as optical methods. Optical sensors are used in many industries to detect water and other contaminants or oxidation products. It might be useful to explore these methods to make sure that it
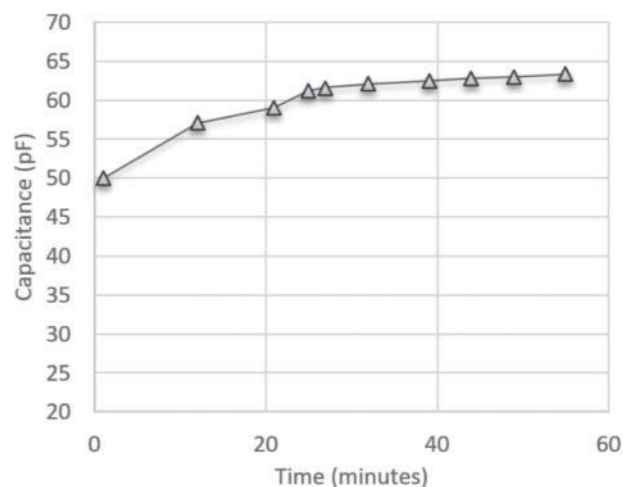

Figure 13. **After first applying grease on the fringe field capacitor, it was discovered that the capacitance slowly rises with time. This measurement was taken immediately after application and at approximately 60°C.**

is known whether there are better options available and how the different challenges of different methods compare.

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