Bureau of Safety and Environmental Enforcement
Oil Spill Preparedness Division

Examination of Physical and Chemical Characteristics of Dielectric Fluids

Final Report

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Margarita Hillón, Kira Howard, Sophia White, Alexa Allen, Jesse George, Emma Lynch-Galvin, Glenn Frysinger, Gregory Hall

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Cover image by LT Brandon Aten, USCG. CG marine science technician collects sheen samples on the Mystic River on April 14, 2018. The CG and Massachusetts Department of Environmental Protection responded to a dielectric oil leak near Alford St. bridge.

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Dielectric fluids are electrically non-conductive liquids often found in underwater electrical cables and power generation equipment, such as associated with offshore wind turbines. To accurately model spills in oil spill trajectory models, such as WebGNOME, the material properties of available dielectric fluids are needed. Through a contract with the US Coast Guard Academy, Coast Guard Academy cadets characterized the physical and chemical properties of fresh and weathered mineral based, synthetic, and ester-based dielectric fluids. This contract was for a 17-month research effort, valued at $42,477.16.

The changes induced by photooxidation indicate that dielectric fluids will respond differently compared to previously studied crude oils or petroleum products. These results demonstrate that dielectric fluids must be modelled with relevant response technologies to gain proper insight into their effectiveness. Further research will provide responders with more accurate decision-making tools to aid in responding to an on-water dielectric fluid spill.

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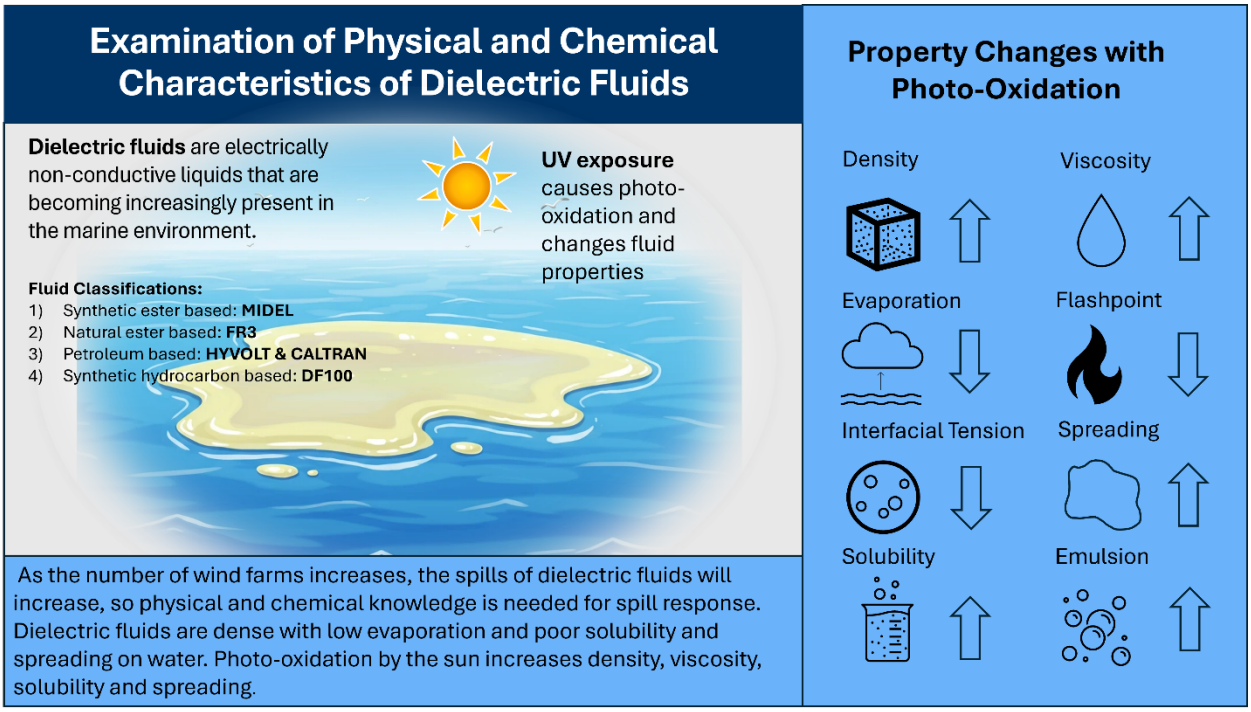
GRAPHICAL ABSTRACT



EXECUTIVE SUMMARY

Dielectric fluids are electrically non-conductive petroleum, synthetic, or plant-based liquids with strong resistance to electricity at high voltages. Dielectric fluids often flow through underwater electrical cables, are used in other parts of power generation equipment, and are used in offshore wind turbines. As more offshore wind farms and electrical transmission lines go into service, the risk of these fluids being released into the marine environment increases. When crude oil, refined oil, or the dielectric fluids that are a focus of this work, are introduced into the environment, a number of weathering and removal mechanisms including, evaporation, aerosolization, spreading, emulsification, tar-ball formation, dissolution, dispersion, sedimentation, microbial biodegradation and photo-oxidation control the fate on the oil. There are two main goals of this research which was performed at the U.S. Coast Guard Academy by cadet researchers. First, physical and chemical properties of the dielectric fluids will be measured that satisfy the required inputs to the NOAA ADIOS database which is used for oil spill modeling in programs such as NOAA WebGNOME or RPS OILMAP. The second is to investigate how exposure to sunlight might change these properties. Five different fluids representing three categories of dielectric fluids were examined in this work: Mineral oil-based fluids of petroleum origin – HYVOLT II NG and CALTRAN 60-30; Synthetic Oil – DF 100; and Ester-based Fluids – ENVIROTEMP FR3 and MIDEL 7131. This sample set spans most of the chemistries represented in the dielectric fluid market as utilized in this context. None of these fluids evaporate readily, with only the mineral oils evaporating by 5% after days of heating and the others showing no appreciable evaporative loss, results confirmed with Simulated Distillation analysis (SIMDIST). This comports with all fluids having very high flashpoints ranging from 128 °C to 221 °C. Densities range from 0.865 g/mL to 0.975 g/mL and were in all cases less dense at 20 °C than 10 °C. Viscosities range from 20 cSt to 210 cSt and in all cases more viscous at lower temperature. Pour points were generally in agreement with manufacturer information and range from -49 °C to -20 °C. Interfacial Tension ranged from 9 mN/m to 45 mN/m and were all higher at higher temperature, although not always statistically significantly so. Spreading and solubility of pure fluids were minimal or non-detected, and these fluids did not show a propensity to form an emulsion. These results paint a picture of fluids that when initially spilled will not evaporate, will probably float, but maybe only marginally so, and don’t present a fire hazard. Exposure to simulated sunlight created some significant changes. It was found that these fluids absorb light at environmentally relevant wavelengths between 300 and 400 nm. While the evaporation behavior did not change appreciably, increases in density and viscosity were observed. More significant changes were observed in that spreading behavior and solubility that increased tremendously by exposure to simulated sunlight. We believe that these observations mean these fluids need to be modelled in relevant response technologies specifically and should not be assumed to behave the same as crude oils, or petroleum distillate products.

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1 Introduction

Dielectric fluids are electrically non-conductive with strong resistance to electricity at high voltages. Dielectric fluids often flow through underwater electrical cables and are used in transformers for offshore wind turbines. The chemical composition of dielectric fluids is varied and includes petroleum, synthetic, or plant-based liquids. Waterborne spills of dielectric fluids are a growing concern. Dielectric fluid spills into waterways have occurred and the circumstances vary. For example, in May 2017, a New York City onshore power station had a 31,000-gallon dielectric fluid spill shutting down the Brooklyn side of the East River (Moore, 2017). In April 2018, a spill of dielectric fluids occurred in the Great Lakes when approximately 600 gallons of a mineral oil dielectric fluid leaked from an electrical cable near the Straits of the Mackinac after an anchor strike (Bergquist, 2018). In July 2021, Con Edison Energy stated that more than 1,000 gallons of dielectric fluid leaked from their underground transmission cables into New Rochelle Harbor near New York (CBS, 2021). In April 2024, Con Edison Energy stated that thousands of gallons of dielectric fluid spilled into a storm drain at Nereid Avenue from a Con Edison transmission feeder in Yonkers and spread to the Bronx (Dhaliwal, 2024). Information about the total number and volume of dielectric fluid spills into navigable waters on a national scale with yearly resolution does not seem to be readily available information. A 2021 study by the Pacific States/British Columbia Oil Spill Task Force estimated that there were 82 spills of mineral oil or transformer oil greater than 42 gallons amounting to 23,207 gallons spilled in the west coast states of the U.S. and in British Columbia (Stevens, 2022). Over the 20-year period from 2002 to 2021, the report identified 1,695 spills of mineral oil/transformer oil greater than 42 gallons with the total volume of 504,850 gallons spilled. This represents about 4% of the total volume of all oil types spilled in that region over that 20-year period (Stevens, 2022).

As more offshore wind farms and electrical transmission lines go into service the risk of release of these fluids increases. Planning for these possible releases is conducted by assuming the worst-case discharge from wind farms. One such study was completed by the Cape Wind Project in New England after becoming the first federally approved facility for offshore energy production (Gunter, 2014). They were required to conduct a systematic review and identify previous spills in the Northeast of similar volume and location as their capabilities for production (Gunter, 2014). The potential volume is calculated by understanding the type of central connection installation, the electric service platform that moves the electricity. Four transformers with several types of dielectric oil have the potential to spill approximately 40,000 gallons of dielectric fluid (Gunter, 2014). Additionally, each of the 130 wind turbine generators will utilize approximately 214 gallons of various oils for operation (MMS, 2008).

When crude oil, refined oil, or the dielectric fluids that are the focus of this work are introduced into the environment, several weathering and removal mechanisms including, evaporation, aerosolization, spreading, emulsification, tar ball formation, dissolution, dispersion, sedimentation, microbial biodegradation and photo-oxidation control the fate on the oil (NAS, 2022). Measurable physical and chemical factors that control, influence, or are related to these removal processes include evaporation rate and flashpoint, density, viscosity, pour point, interfacial tension, spreading, solubility and emulsification. Moreover, many of these factors

change during the evolution of a spill as an oil is degraded.

There were two main goals of this research. First, physical and chemical properties of the dielectric fluids were measured to satisfy the required inputs to the NOAA ADIOS database that is used for oil spill modeling in programs such as NOAA WebGnome or RPS OILMAP. Without inputs specific to the various types of dielectric fluids used, the models must choose other oils such as crude oil, motor oils, or lube oils as a proxy. Second, oil will chemically change immediately with release into the environment. Environmental processes such as wave turbulence, high temperatures and exposure to ultraviolet rays from the sun change the chemical composition and physical properties of the oil (Liu, 2012). This experiment aims to examine the impact of photo-oxidation from simulated sunlight on dielectric fluids. We hypothesize that exposure to sunlight will impact the bulk behavior of these fluids and control evaporation and flashpoint, density, viscosity, pour point, interfacial tension, spreading, solubility and emulsification. Whether photo-oxidation will be the primary driver of the fate of these fluids will need further research, especially into possible biodegradation.

2 Dielectric Fluids

2.1 Dielectric Fluids Studied

Five different fluids representing several categories of dielectric fluids were examined in this work. All samples were sourced from BSEE through Ohmsett – The National Oil Spill Research & Renewable Energy Test Facility in Leonardo, NJ, USA. The fluids are summarized in Table 1.

The first category is mineral oil-based fluids. The first mineral oil included is HYVOLT II NG (HYVOLT) transformer oil manufactured by Ergon Specialty Oils, Jackson, MS, USA. HYVOLT is described on its Safety Data Sheet (SDS) as a mix of hydrotreated light naphthenic and paraffinic and dewaxed paraffinic oil with a small quantity of BHT preservative. Using alternate chemistry terms, that means that the mineral oil consists of many branched alkane chain isomers and also substituted cyclohexane, cyclopentane, or fused saturated rings with alkyl chain substituents. Dewaxed means that straight chain alkanes have been removed. The second mineral oil is CALTRAN 60-30 (CALTRAN) electrical insulating oil manufactured by Calumet, Indianapolis, IN, USA. CALTRAN is described similarly in its SDS as light naphthenic hydrotreated petroleum distillates with a BHT blend preservative. While the HYVOLT sample is a new product, the CALTRAN sample is a used fluid.

The second category is synthetic oils including DF 100 (DF100) insulating cable oil manufactured by Soltex Inc, Houston, TX, USA. It is described in its product brochure as a synthetic oil consisting of alkylate/polybutene. The SDS differently describes it as a “benzene, mono-C10-13 alkyl derivatives, distillation residues”. These seemingly conflicting descriptions leave uncertainty about the chemical composition of DF100

The last category is ester-based fluids. The first is ENVIROTEMP FR3 (FR3) natural ester dielectric fluid manufactured by Cargill, Minneapolis, MN, USA. The SDS describes FR3 as containing > 99% soybean oil which is expected to contain triglycerides with 16 and 18-carbon saturated, mono and polyunsaturated chains. The second is MIDEL 7131 (MIDEL) that is a

synthetic ester dielectric liquid manufactured by Shell. The SDS describes it as “fatty acids, C5-10 (linear and branched), mixed esters with pentaerythritol”. Pentaerythritol is a tetra-ester backbone substituted with mix of shorter linear and branched alkyl substituents.

Table 1 Dielectric Fluid Inventory

Fluid Trade Name	Short Name	Status	Type
HYVOLT II NG	HYVOLT	New	Mineral oil
CALTRAN	CALTRAN	Used	Mineral oil
DF100	DF100	New	Synthetic
ENVIROTEMP FR3	FR3	New	Natural ester
MIDEL 7131	MIDEL	New	Synthetic ester

2.2 Photo-oxidation

Photo-oxidation of dielectric fluids was done with a Q-Lab Inc. LX-5090B-TM Q-SUN Xe-1-B Xenon Test Chamber. The instrument has a Daylight Q Filter used in combination with a xenon lamp to produce an accurate simulated solar spectrum. The Daylight Q filter has a cut-on wavelength of 295 nm. The test chamber irradiance level was set to 0.34 W/(m^2 nm) measured at 340 nm wavelength. The exposure duration for the dielectric fluids was 24 hours. The chamber temperature was 35 °C. The total exposure is equivalent to about 3 days of summer exposure at northern U.S. latitudes.

A 7.75 mL volume of a dielectric fluid was added to a glass container with bottom dimensions of 15.2 cm × 10.21 cm to create a 0.05 cm fluid thickness. After simulated sunlight exposure, the container contents were transferred to a vial. Considering minor evaporation and incomplete container scraping, fluid recovery was typically greater than 90%. Photo-oxidation was done with the dielectric fluids spread on a glass surface only. The photo-oxidation of fluids on water surface and the possible indirect photo-oxidation caused by the water and dissolved salts was not studied.

The ultraviolet-visible spectra of the fluids were measured and the specific extinction coefficients (α) were calculated to determine what thickness of the fluid is effectively irradiated with the simulated solar radiation. Solutions of each fluid were diluted with cyclohexane solvent for target concentrations of ~2 g/L. Figure 1 shows the spectra of the five dielectric fluid solutions plotted with a 400 to 300 nm wavelength range on the *x*-axis and α with units (L/g cm) on the *y*-axis. The fluid with the highest UV absorbance at 300 nm was CALTRAN with α_{300} = 0.355 L/g cm. For the 0.05-cm thick pure dielectric fluid film in the glass container, the absorbance is estimated as 1.78 and the percent transmittance is 1.7%. This means that 98.3% of the light was absorbed by the 0.05 cm film. The least absorbing fluid, MIDEL, had a α_{300} = 0.016 L/g cm, A = 0.08, and %T = 83% for 17% absorbed. Therefore, depending on the fluid, the surface of the fluid experienced significantly more light exposure than the bottom of the fluid. However, this was a compromise between uniform irradiance of the whole sample and the need for larger photo-oxidized volumes for physical and chemical property measurement.

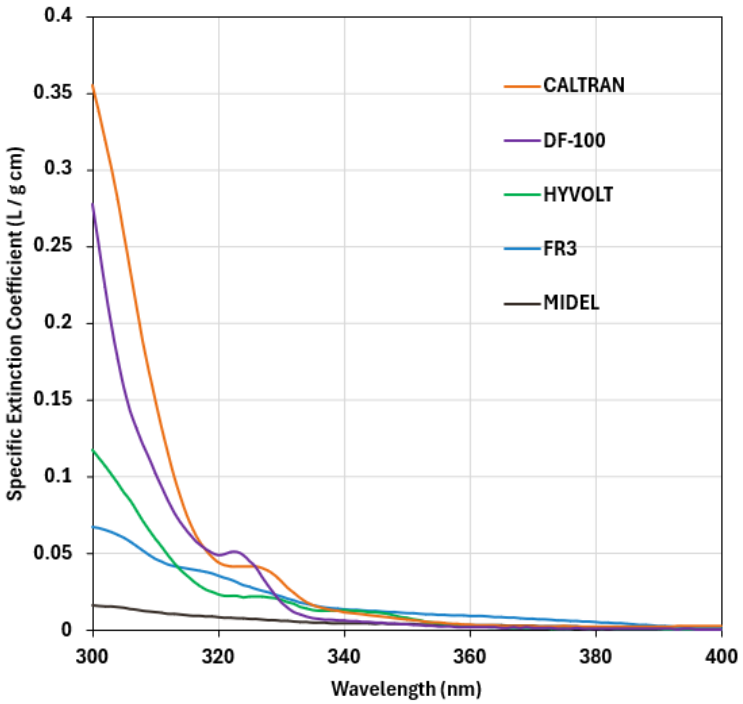

Fig. 1 UV-Spectra of dielectric fluids measured from 300-400 nm.

Figure 2 shows a photograph of the five dielectric fluids. The fluids with the highest ultraviolet light absorbance between 300 and 320 nm wavelengths, CALTRAN, HYVOLT, and DF100, appear most yellow to the eye. FR3 has the highest light absorbance across longer 330 to 390 nm wavelengths, which may be why the fluid has a light green color.

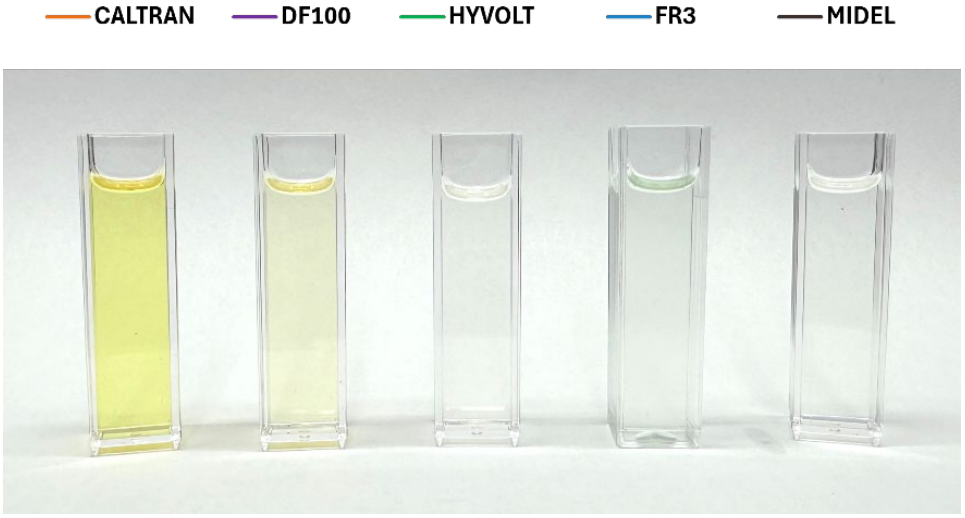

Fig. 2 Photograph of dielectric fluids, CALTRAN, DF100, HYVOLT, FR3, MIDEL.

Figure 3 shows the CALTRAN, DF100, and HYVOLT fluids before and after simulated solar irradiation. These three showed significant darkening while the FR3 and MIDEL fluids had no significant change in appearance. For the mineral oil-based CALTRAN and HYVOLT samples, irradiation caused reddish-brown particulates to make the fluid cloudy. Over time the dense particulates settle as seen in the Figure 3 photo. The color change and particulate formation suggests significant chemical modification and perhaps polymerization of the dielectric fluid samples.

For petroleum-based oils, UV-induced photooxidation is known to be responsible for altering the chemical composition of oil. Chromophores present in the oil absorb sunlight, initiating a sequence of photochemical reactions (Shankar, 2015). According to (Mill 1980), the oils form an excited state when exposed to solar radiation. This excited state causes the oil to form radicals which react with oxygen. Photooxidation causes low molecular weight molecules to become high molecular weight molecules (Dutta, 2000).

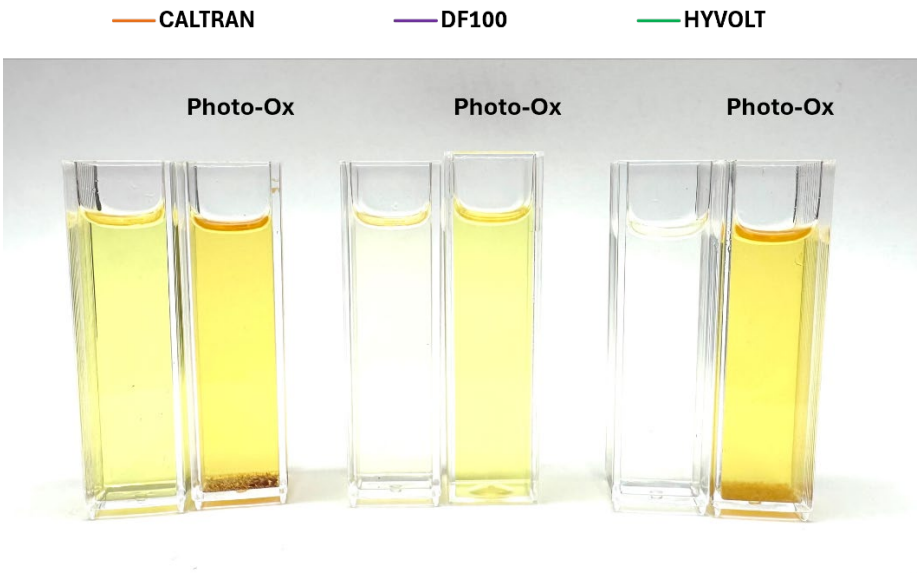

Fig. 3 Photograph of dielectric fluids CALTRAN, DF100, and HYVOLT before and after photo-oxidation.

3 Physical & Chemical Characteristics

3.1 Evaporation

Evaporation of spilled dielectric fluids will be influenced by the chemical composition. Fluids with smaller molecules (carbon numbers < 15) will evaporate and lose mass to the atmosphere more rapidly than fluids that have large molecules (carbon numbers > 15). In the environment, factors such as a large slick area, warm temperatures, and high wind or waves will promote evaporation (NAS, 2022).

3.1.1 Methods

Experiments were conducted to see how well the dielectric fluids evaporate. Four of the dielectric fluids were heated to increasingly high temperatures of 110, 115, and 180 °C for times of 2 days (48 hours) to 14 days (336 hours). Samples of untreated dielectric fluids with a known mass were added to open glass test tubes and heated on a hot plate in a copper shot bath. Samples were periodically removed and weighed to monitor mass loss by evaporation. The target was to see what conditions lead to 5% mass loss.

3.1.2 Results

None of the dielectric fluids were easily evaporated. Table 2 shows that the mineral oil-based fluids CALTRAN and HYVOLT were most volatile, but it took elevated temperatures and 2 to 4 days to evaporate 5% of each. The ester-based fluids FR3 and MIDEL only showed measurable evaporation after 14 days at 180 °C. As a result, it is expected that under environmental conditions, significant evaporative loss of dielectric fluids is not expected.

Table 2 Dielectric Fluid Evaporation

Dielectric Fluid	Time/Temperature		
	48 hours @ 110 °C	96 hours @ 115 °C	336 hours @ 180 °C
MIDEL	-	0.13%	0.54%
FR3	-	0.40%	1.59%
CALTRAN	5.57%	-	-
HYVOLT	-	5.66%	-

3.2 Simulated Distillation

Simulated distillation is an instrumental method to model evaporation or distillation with a gas chromatograph to assign boiling points to the distribution of the hydrocarbon chemical components.

3.2.1 Methods

Simulated distillation (SIMDIST) for the dielectric fluids was measured by method ASTM D2887-22 Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography. A gas chromatograph (Agilent 6890) was configured with a PTV inlet programmed 60°C (0.1min) – 400 °C @ 300 °C/min. Injection volume was 0.5 µL splitless. Detector was a flame ionization detector (FID) at 350 °C. The column (Restek MXT-1 SIMDIST, 10 m x 0.530 mm x 0.88 µm) was temperature programmed (40 °C (1 min) – 430 °C @ 10 °C/min) for a 40 min analysis time. Helium was the carrier gas at 15.0 mL/min constant flow. Samples were diluted 10/1 in chloroform for analysis. Retention time was calibrated to boiling point with an alkane standard (AccuStandard DRH-002S-R1). GC-FID data was converted to CSV format using OpenChrom (Wenig, 2010) for import into MS Excel. GC-FID data was baseline subtracted with a blank run and integrated to determine percent of total area or percent mass of each dielectric fluid. Data is reported as boiling point (°C) for IBP (initial boiling point 0.5% mass), 5, 10, 20, 30, 40, 50, 60, 70, 80, 90, 95%, and FPB (final boiling point 99.5 % mass).

3.2.2 Results

The simulated distillation chromatograms for the 5 untreated dielectric fluids are shown in Figures 4a-e. Each chromatogram has a retention time (RT) axis, a calibrated simulated distillation boiling point axis (BP), and a linear alkane carbon number (carbon #) axis. These chromatograms can provide more chemical insight into the manufacturer's description of each dielectric fluid introduced in Section 2.1. The HYVOLT and CALTRAN mineral oil-based fluid chromatograms show a hump of chromatographically unresolved petroleum hydrocarbons isomers with sizes from 10 carbons to 36 carbons. The hydrocarbon isomers include thousands of different saturated branched alkanes and akyl-substituted cycloalkanes. The petroleum origin of the mineral oil fluids is evident by prominent labeled peaks for pristane and phytane petroleum branched chain isoprenoid biomarkers (He, 2018). The CALTRAN fluid is a used product, which may explain why the smallest, lowest molar mass hydrocarbons in the 10-13 carbon region may be already evaporated or degraded when compared to the HYVOLT sample. The DF100 synthetic fluid also has a hump of unresolved organics, but the range is shifted to 17 to 40 carbons. It is expected that the larger, higher molar mass, molecules in DF100 will lead to less evaporation under environmental conditions and elevated flash point. The ester-based fluid FR3 chromatogram shows peaks for the soy oil triglycerides (labeled TG) but it also shows more abundant peaks at lower retention times that may be for mono or diglycerides either natively present in the FR3 fluid or perhaps generated in the high temperature inlet and column of the gas chromatograph. For this reason, the SIMDIST results for the FR3 fluid are not validated and may not be applicable to oil spill modeling. The synthetic ester-based fluid MIDEL has a chromatogram with many peaks that elute late in the chromatogram at high simulated distillation boiling point temperatures. The many clustered peaks in the chromatogram is consistent with the chemical description that MIDEL contains tetraesters substituted with 5 to 10-carbon linear and branched chains.

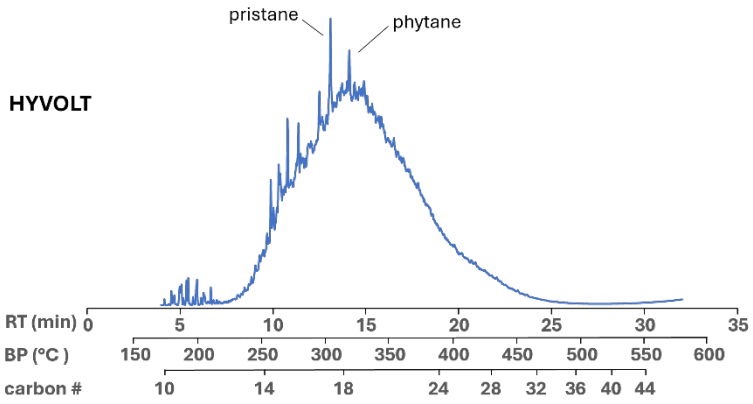

Fig. 4a Simulated distillation chromatograms for untreated HYVOLT

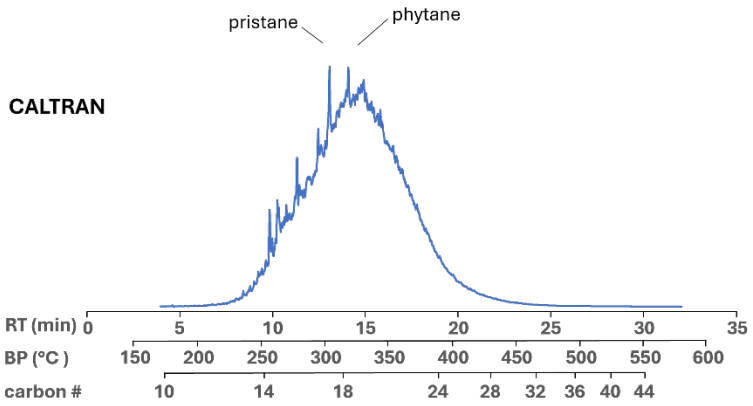

Fig. 5b Simulated distillation chromatograms for untreated CALTRAN

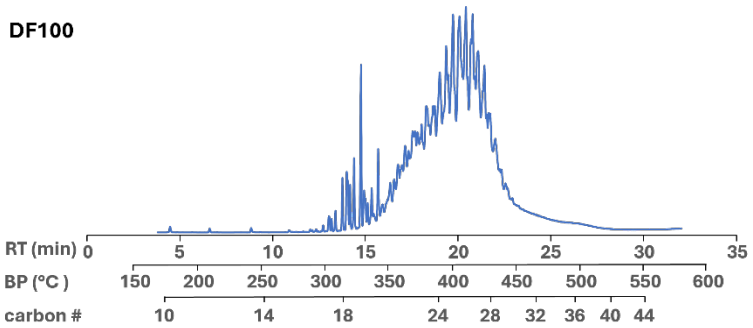

Fig. 6c Simulated distillation chromatograms for untreated DF100

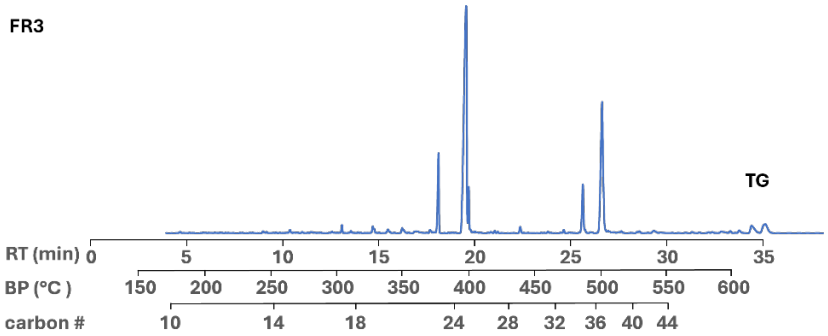

Fig. 7d Simulated distillation chromatograms for untreated FR3

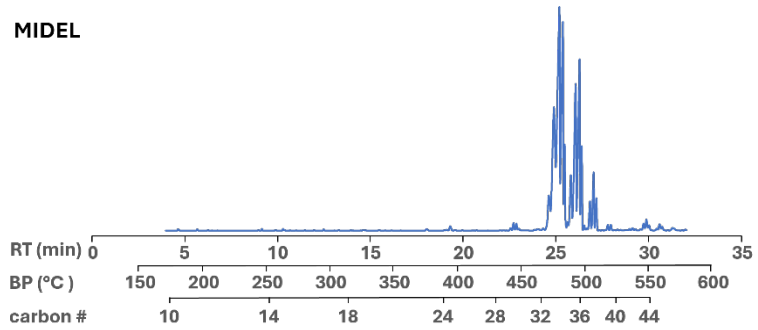

Fig. 8e Simulated distillation chromatograms for untreated MIDEL

Figure 5 shows the plotted integrated SIMDIST results. The data plotted in the figure is in Table 3. The initial boiling point (IBP) represents the temperature where 0.5% of the total mass of the fluid has evaporated. The fluid with the lowest IBP is HYVOLT mineral oil with an IBP of 198 °C (Table 3). Of all fluids, HYVOLT had the smallest molecules that will be most readily evaporated (see Figure 4a). The photo-oxidized HYVOLT lost some of the smallest molecules through evaporation of degradation so the IBP is higher at 256 °C. Compared to the mineral oil fluids, the synthetic DF100 and the ester fluids FR3 and MIDEL require much higher temperatures for significant evaporation.

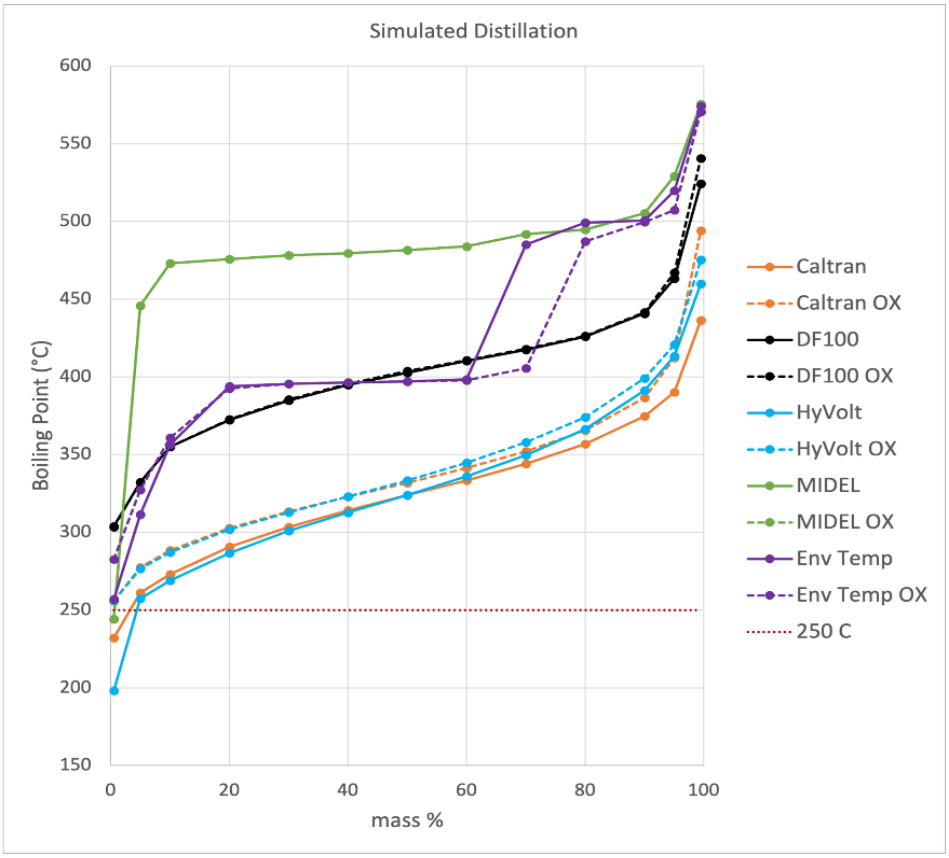

Fig. 9 Simulated distillation (SIMDIST) results for untreated and photo-oxidized dielectric fluids.

Table 3 Simulated Distillation (SIMDIST) Results

		m/m%												
Dielectric Fluid		IBP 0.5	5	10	20	30	40	50	60	70	80	90	95	FBP 99.5
MIDEL	Untreated	244	446	473	476	478	479	482	484	492	495	505	529	575
	Photo-Ox	244	446	473	476	478	479	482	484	492	495	505	529	575
FR3	Untreated	257	311	356	394	396	396	397	398	485	499	500	520	574
	Photo-Ox	283	327	361	393	395	396	397	398	405	487	500	507	571
CALTRAN	Untreated	232	261	273	291	303	314	324	333	344	357	375	390	436
	Photo-Ox	256	278	288	303	313	323	332	341	352	366	387	412	494
HYVOLT	Untreated	198	257	269	287	301	313	324	336	350	367	391	413	460
	Photo-Ox	256	277	287	302	313	323	333	345	358	374	399	421	475
DF100	Untreated	304	332	355	372	385	395	403	410	417	426	441	463	524
	Photo-Ox	303	332	355	373	385	395	404	411	418	426	442	467	541

3.3 Flash point

The flash point of a fluid is the lowest temperature at which it produces enough vapor to ignite when exposed to a spark or flame. Liquids with flash points less than 60 °C are considered flammable. Many freshly spilled crude oils also have low flash points until the lighter components have evaporated or dispersed (Hollebone, 2010). The SIMDIST initial boiling point and the ability of a fluid to evaporate are correlated with flash point.

3.3.1 Methods

Flash point was measured using method ASTM D93-20 Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester. The instrument was a Setaflash Series 8 ActiveCool Closed Cup Flash Point Tester. A 5-mL volume of each fluid was deposited in the closed cup and heated from 90 °C to 120 °C while an ignitor periodically opened the cup to test for vapor ignition. The maximum flash point test temperature of the device is 120 °C.

Flash point was also calculated using method ASTM D7215-22 Standard Test Method for Calculated Flash Point from Simulated Distillation Analysis of Distillate Fuels. The method uses the SIMDIST data to calculate flash points comparable to method D93 for fluids that have flash point temperatures above the Setaflash test limit. The method was developed and applicable to petroleum diesel with an IBP between 103 °C and 163 °C, plus 5 % recovery temperatures between 144 °C and 210 °C, and 10 % recovery temperature between 159 °C and 236 °C as measured in ASTM D2887. The simulated distillation shows what temperatures are needed for a significant 5% or 10% mass percent of the most volatile molecules of the dielectric fluids to evaporate to create a flammable mixture with air. Since ASTM D7215 is calibrated for diesel range petroleum distillates and all the dielectric fluids have SIMDIST data above that range, the calculated flash points must be considered rough estimates.

3.3.2 Results

In experimental testing, all dielectric fluids had measured flash points greater than 120 °C, the maximum test temperature of the Setaflash instrument. Therefore, calculated flashpoints from SIMDIST data for each fluid are listed in Table 4. The mineral oil fluids HYVOLT and CALTRAN have the lowest calculated flashpoints while the DF100 and the ester-based FR3 and MIDEL have the highest calculated flashpoints. For HYVOLT, the manufacturer published specification lists a 145 °C flash point compared to the calculated 128 °C flash point calculated from the SIMDIST data.

Even considering the uncertainties, the spill response considerations derived from the flashpoint data tell that all dielectric fluid spills have minimal fire danger and in situ burning may not be possible, but would take further research utilizing operational conditions to establish it's feasibility.

Table 4 Flash Point of Dielectric Fluids

Dielectric Fluid	Untreated (°C)	Photo-Ox (°C)
MIDEL	221	221
FR3	179	193
CALTRAN	143	158
HYVOLT	128	143
DF-100	201	201

3.4 Density & Viscosity

Viscosity is a measure of how readily a fluid flows. According to (Hollebone, 2010), the viscosity of an oil is a function of its chemical composition. Therefore, crude oil from different locations with different chemical composition may display a wide range of viscosities. In general, the greater the fraction of saturates and aromatics and the lower the amount of asphaltenes and resins, the lower the viscosity. As with other physical properties, viscosity is affected by temperature, lower temperatures oils have higher viscosities. As oil weathers, the evaporation of the lighter components leads to increased viscosity. Dielectric fluid densities help in predicting how readily the fluids will float on the surface by either making a sheen across the water or being more likely to form smaller blobs (NAS, 2022). Heavier oils, including heavy crudes and residual fuel oils, will contract as temperature decreases, increasing the density, potentially causing them to sink in freezing waters. Furthermore, as the smallest molecules of the oil evaporate off and increase density, weathered oil may be prone to sinking or over washing, even long after the spill where the fresh oil may have floated (Hollebone, 2010).

3.4.1 Methods

Dielectric fluid density and viscosity was measured with an Anton Paar SVM 3001 Viscometer following method ASTM D7042-21a Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity). A Stabinger viscometer is a rotational viscometer with a cylinder geometry that is based on a modified Couette principle where the test fluid that is placed between a rapidly rotating outer tube and an

inner cylinder causes a measurable viscous torque on the inner cylinder. In the experiment, density and viscosity was measured at 10 °C and 20 °C. Prewetting of the instrument was done with 1 mL of dielectric fluid, followed by 1 mL of dielectric fluid for measurement. Density (g/mL) and kinematic viscosity with SI units (m^2/s) or centistokes (cSt) are reported. Dynamic viscosity with units of centipoise (cP) can be calculated from kinematic viscosity by multiplying the kinematic viscosity (cSt) by the density (g/cm^3).

3.4.2 Results

The density and viscosity results are shown in Figures 6 and 7 with the corresponding data in Appendix Tables 7 and 8. Error bars represent 95% CI for three replicates. At both 10 °C and 20 °C the ester-based fluids MIDEL and FR3 had both higher densities and kinematic viscosities than the mineral oil-based fluids CALTRAN and HYVOLT. The synthetic hydrocarbon, DF100 had a lower density than CALTRAN and HYVOLT but a higher kinematic viscosity similar to MIDEL and FR3.

Densities and viscosities are temperature dependent. All fluids had higher densities and higher viscosities at the lower 10 °C temperature than the warmer 20 °C temperature. Spill response considerations could be that some of the dielectric fluid spills will have high density and poor flotation. The 10 °C MIDEL fluid has a density of 0.975 g/mL which is approaching the fresh water density of about 1.00 g/mL or seawater density of about 1.030 g/mL. The density of MIDEL is almost as high as Bunker C (0.989 g/mL at 15 °C). Fortunately, while the ester-based MIDEL and FR3 have the 10 °C highest viscosities at 120 and 110 cSt respectively, those are considerably less viscous than Bunker C (~8600 cSt at 15 °C) (Jokuty, 1996).

The densities and viscosities also increased when the fluids are photo-oxidized. For all fluids, the density at both 10 °C and 20 °C increased when the fluid was photo-oxidized. For all fluids, the viscosity at both 10 °C and 20 °C increased when the fluid was photo-oxidized. The spill response considerations are that higher density increased viscosity with photo-oxidation may impact recovery and skimming operations.

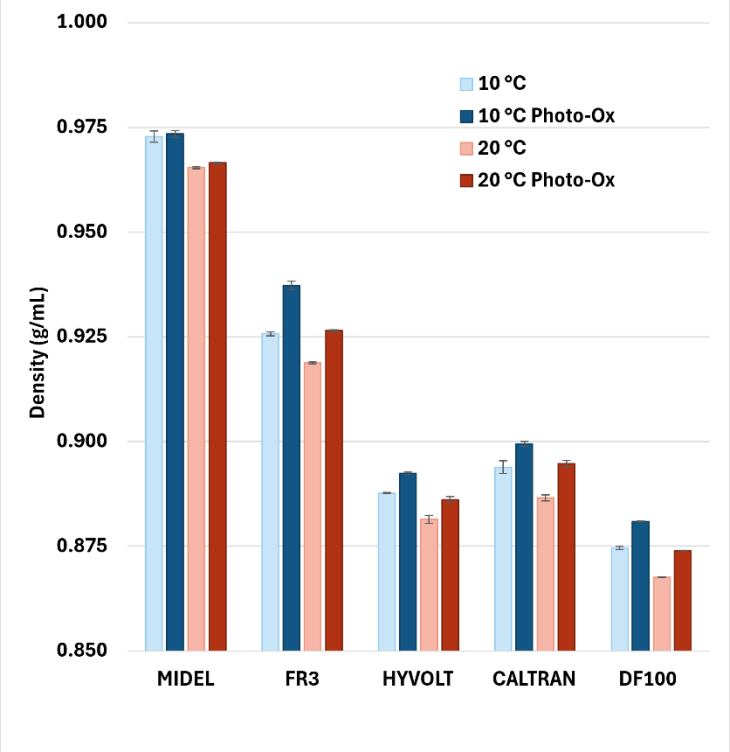

Fig. 10 Density of untreated and photo-oxidized dielectric fluids at 10 and 20 °C.

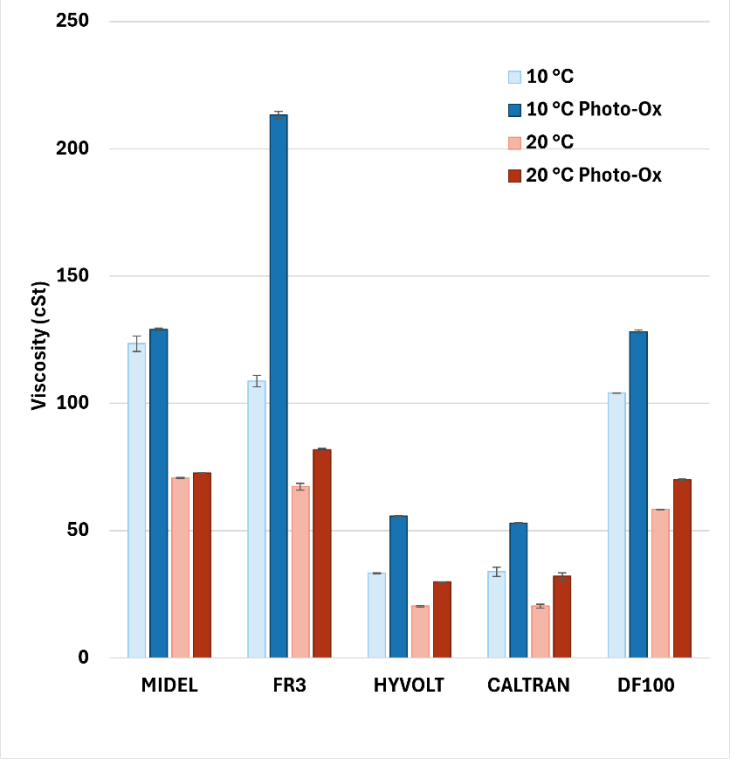

Fig. 11 Kinematic Viscosity of untreated and photo-oxidized dielectric fluids at 10 and 20 °C.

3.5 Pour point

Pour point is the minimum temperature at which a liquid will still flow by gravity.

3.5.1 Methods

Pour point was determined using method ASTM D97 Standard Test Method for Pour Point of Petroleum Products. Each dielectric fluid was added to a pour point test tube to the fill line. A thermometer was inserted so that the bulb was positioned 3 mm below the surface of the fluid sample. After heating to 45°C, the sample was cooled. Given low expected pour points, a final cooling bath with dry ice / isopropanol at -80 °C was used. Observations began 9°C above the expected pour point. For every 3 °C decrease, the container was removed from the jacket and tipped to observe if the sample flows. If the sample moves, it was returned to the cooling jacket to continue cooling. The test ended if the sample did not move. The temperature 3°C before the sample did not flow was recorded as the pour point. Because the test requires a large volume of fluid, the pour point for the photo-oxidized fluids was not measured.

3.5.2 Results

The pour point results are shown in Table 5. The expected pour points are data found in the fluid manufacturer's product information brochures or in the Safety Data Sheet (SDS) for the product. In general, there was good agreement between manufacturer's reported values and our measurements. The pour points for all the fluids are low for all environmentally relevant temperatures so all dielectric fluids are expected to be dense, viscous fluids, but not solids at cold winter temperatures

Table 5 Pour Point of Dielectric Fluids

Sample	Manufacturer Pour Point (°C)	Experimental Pour Point (°C)
MIDEL	-56	-43
FR3	-21	-20
CALTRAN	-64	-46
HYVOLT	-40	-49
DF100	< - 40	-43

3.6 Interfacial Tension

The spreading of an oil on water depends on the surface tension of the water/air interface ($\gamma_{w/a}$), the surface tension of the oil/air interface ($\gamma_{o/a}$) and the surface tension of the oil/water interface ($\gamma_{o/w}$). If the sum of $\gamma_{o/a}$ and $\gamma_{o/w}$ are less than $\gamma_{w/a}$ the oil will spread (Winoto, 2015). The focus of this work is to measure the interfacial tension between the water and the dielectric fluids.

3.6.1 Methods

The interfacial tension between the dielectric fluid and artificial seawater at 35 ppt was measured with a Du Nouy ring with the Zuidema-Waters equation (Zuidema, 1941). The instrument used was the DST60 Series Surface Tension Analyzer. A 30-mL volume of seawater chilled to either

10 °C and 20 °C was added to a clean sample container. The container was placed on the tensiometer platform, and a flame-cleaned Du Nouy ring was lowered below the water surface. A 30-mL volume of the dielectric fluid chilled to either 10 °C and 20 °C was carefully poured to create a layer on the water surface. The tensiometer platform was lowered at a fixed rate and the maximum force on the ring as it was pulled across the interface and the water meniscus ruptured was measured.

3.6.2 Results

The dielectric fluid – water interfacial tension ($\gamma_{o/w}$) is shown in Figure 8. Error bars represent 95% CI for three replicates. The corresponding numerical data is in Appendix Table 9. The FR3 fluid has the lowest interfacial tension suggesting that it will spread on water better than the other fluids. The interfacial tension is only moderately influenced by temperature, with lower temperatures having lower interfacial tensions. Because the test requires a large (20 – 30 mL) volume of fluid, the interfacial tension for the photo-oxidized fluids was not measured.

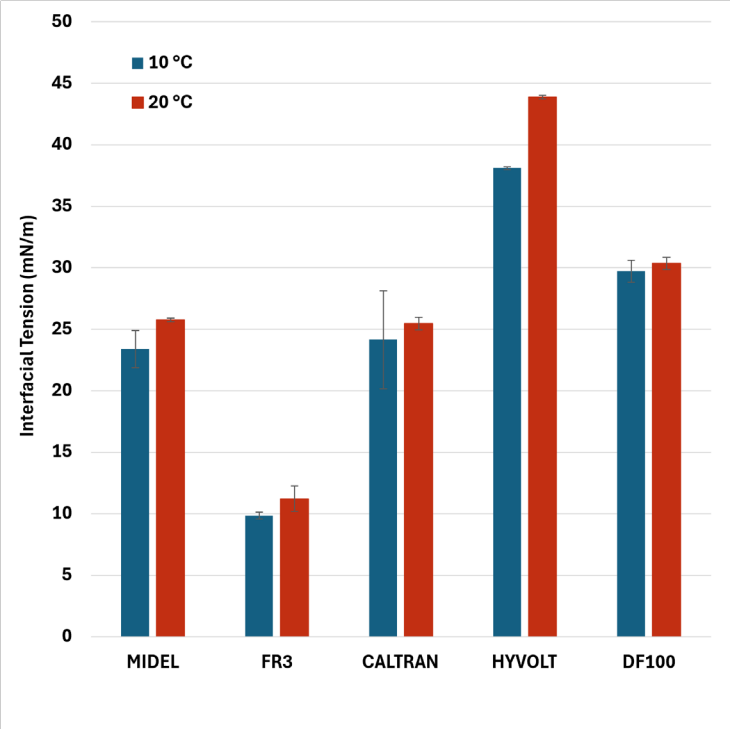

Fig. 12 Interfacial tension of untreated dielectric fluids on 35 ppt artifical seawater at 10 and 20 °C.

3.7 Spreading

As a follow on to interfacial tension, the dielectric fluids with the lowest fluid – air interfacial tension are expected to spread the best on the water surface. Because only small quantities of fluid are required in this spreading experiment, the effect of photo-oxidation can be assessed on spreading because interfacial tension of photo-oxidized fluids was not possible.

3.7.1 Methods

To determine the ability of dielectric fluids to spread on seawater, a known amount of each fluid was directly deposited on a seawater surface. Artificial seawater at 35 ppt was added to a 12 by 18-inch tray. The water surface was lightly dusted with either lycopodium or talcum powder. A small 10 μL volume of neat untreated or photo-oxidized dielectric fluids was dropped onto the center of the water surface using an Eppendorf pipette. The fluid spreads and pushes the powder to make a nearly circular film and a visible ring of the powder. The diameter of the spread fluid film was measured on three different axes with a ruler, averaged, and a surface area was calculated. The thickness of the fluid film was calculated using the diameter and the 10 μL deposited volume. The uncertainty in the results is large because it is difficult to reproducibly dispense the small 10 μL volume because of the fluid viscosity. In addition, the area is difficult to measure because surface water movement can distort the circular spread.

3.7.2 Results

The surface areas for waterborne films of untreated and photo-oxidized dielectric fluids are shown in Figure 9. In general, the untreated dielectric fluids formed thick lenses on the water surface. The ester-based dielectric fluid FR3 that was found in Section 3.6 to have the lowest fluid – water interfacial tension is expected to have the greatest spread. Examining the blue bars in Figure 9, the FR3 spread out on the water with a film diameter of 3.6 cm which is 4 to 6 times greater diameter than the other dielectric fluids which had higher interfacial tensions.

For all photo-oxidized fluids, there was significant spread compared to the untreated fluids. The photo-oxidized fluids had spread film diameters from 3 to 30 times larger than the untreated fluid. The mineral oil-based fluids CALTRAN and HYVOLT and the synthetic DF100 showed the biggest change. Despite the large uncertainty in the fluid spread areas, all photo-oxidized fluids showed a significant increase in spreading.

The thickness of the films is reported in Table 6. The thickest lenses for the untreated fluids are 300 μm / 0.3 mm thick. The photo-oxidized dielectric fluids spread readily to form thin films and the thinnest are about 0.3 μm / 300 nm thick. Films that are 300 nm thick are still thicker than monomolecular films which can be expected to be as thin as 2 nm (Langmuir, 1917). It is clear that the photooxidation promotes spreading of the fluids. Spreading is likely driven by light-driven incorporation of oxygen into the fluids and the formation of polar molecules that are attracted to the water and likely reduce the interfacial tension of the fluids to promote spreading on the water surface. Unfortunately, the measurements in section 3.7 Interfacial Tension do not include the photo-oxidized fluids but the spreading data suggest that their interfacial tension may be significantly reduced.

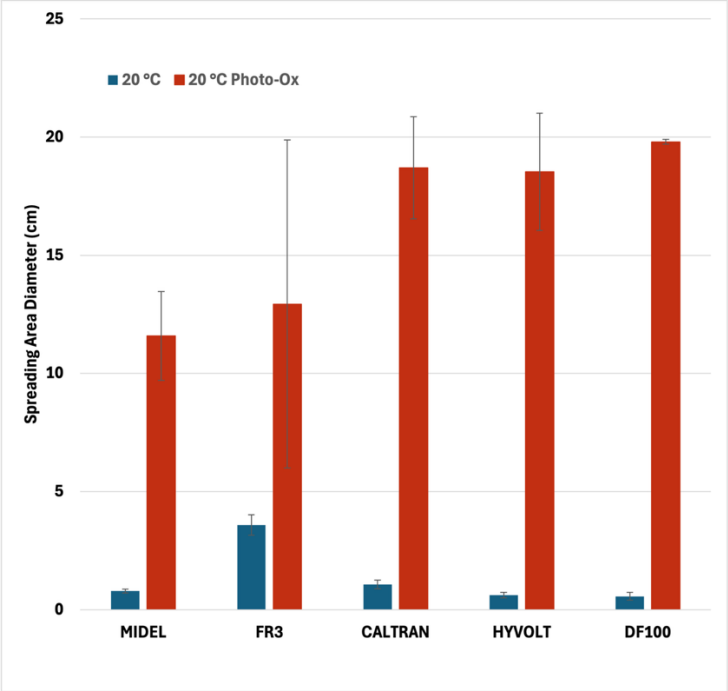

Fig. 13 Surface spreading of untreated and photo-oxidized dielectric fluids against 35 ppt artificial seawater at 20 °C.

Table 6 Spreading of Dielectric Fluids

Dielectric Fluid	Average Diameter (cm) Untreated	Thickness (μm)	Average Diameter (cm) Photo-Ox	Thickness (μm)
MIDEL	0.8	203.2	11.6	0.95
FR3	3.6	9.9	12.9	0.76
CALTRAN	1.1	111.9	18.7	0.36
HYVOLT	0.6	334.8	18.5	0.37
DF100	0.6	396.5	19.8	0.32

3.8 Solubility

3.8.1 Methods

To measure dielectric fluid solubility in seawater, approximately 300 mg of each untreated and photo-oxidized dielectric fluid was equilibrated with 125 mL seawater at 20 °C in the dark on a temperature-controlled shaker table for 24 h at 100 rpm. To remove entrained water-insoluble oil droplets, each seawater leachate was filtered through a 0.22-μm pore filter into a 40 mL glass vial. Samples of the remaining dissolved organics were acidified with trace-metal grade concentrated hydrochloric acid and then dissolved organic carbon (DOC) was quantified as

carbon dioxide after high-temperature combustion using a Shimadzu TOC-L CPH Total Organic Carbon Analyzer.

The instrument reports concentrations of DOC as μM concentrations of carbon. Solubility can be reported as mass of dielectric fluid dissolved per volume of seawater by multiplying the concentration by the molar mass of carbon and then dividing by the estimated fraction of carbon in the dielectric fluid. The mineral oil-based fluids, CALTRAN, HYVOLT, and the synthetic DF100 were estimated to have 0.85 carbon fraction. The ester-based fluids, FR3 and MIDFEL were estimated to have 0.77 carbon fraction.

3.8.2 Results

The mg/L solubility of untreated and photo-oxidized dielectric fluids are shown in Figure 10. Error bars represent 95% CI for three replicates. The corresponding numerical data is in Appendix Table 10. The untreated dielectric fluids were sparingly soluble with three of them having no measurable solubilities (ND = not detected). The two that were slightly soluble were CALTRAN and MIDEL. The CALTRAN sample tested is a used dielectric fluid. Due to its field use, it may have been chemically degraded to produce polar molecules that will slightly increase the solubility of CALTRAN relative to HYVOLT, the other mineral oil-based fluid that had no detected solubility. MIDEL is a synthetic tetra-ester compared to FR3 which is a soy oil-based ester; the additional ester in MIDEL may make it slightly more soluble.

All of the photo-oxidized samples significantly increased solubility. The change in solubility is related to the modified chemical makeup of the fluids. The mineral oils, CALTRAN and HYVOLT, increased the most of all the fluids. The photograph in Figure 3 shows that these fluids changed color significantly and formed insoluble materials that clouded the fluids. These photo-oxidation chemical changes produced an increase in polarity that drive increased solubility as well as increased surface spreading documented in section 3.7. While all fluids increased solubility with photo-oxidation, the mineral oil-based fluids reached a level of solubility that is significant to spill response. Photo-oxidized HYVOLT and CALTRAN were calculated to lose 0.61 and 0.93% of their mass to the seawater.

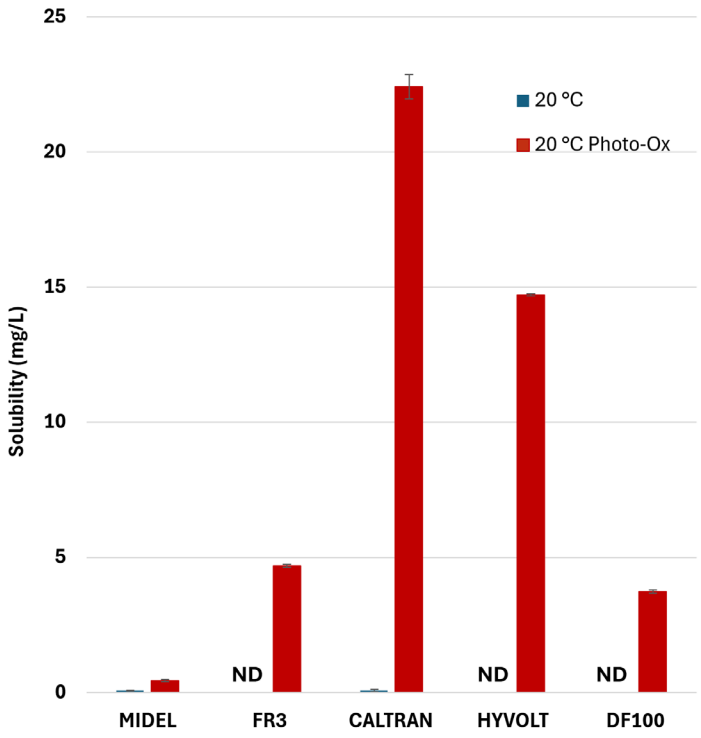

Fig. 14 Solubility of untreated and photo-oxidized dielectric fluids in natural seawater at 20 °C.

3.9 Emulsions

3.9.1 Methods

The emulsion experiment follows Enviro Canada's oil/brine emulsion formation stability and tension method for petroleum products (Fingas 2004). 200 mL of artificial seawater (35ppt) and 10 mL of dielectric fluid was placed into a 1-liter Teflon-lined vessel. The vessel was sealed and placed it into a rotary mixer with an end-over-end rotation. After equilibrating for 4 hours, the vessel was mixed at 35 rpm for 12 hours. At the end of the 12-hour period, the vessel was taken out of the mixer and opened to view whether an emulsion formed. (Fingas, 2004). Because the test requires a large volume of fluid, the pour point for the photo-oxidized fluids was not measured.

3.9.2 Results

None of the untreated dielectric fluids created a stable emulsion after 24 hours. HYVOLT, CALTRAN, and DF100 formed bubbles on the top layer that dissipated within minutes of observation. According to Fingas, this is classified as an unstable water-in-oil state (Fingas, 1996). If the test can be adapted for small volumes of material, it is expected that the photo-oxidized dielectric fluids will form emulsions. Since this method takes a large sample, photo-oxidized samples were not analyzed, but given the photo-oxidation increased surface spreading caused by an increase in oxygen uptake and creation of polar functional groups, we expect that will lead to increased emulsion formation as well.

4 Conclusions and Response Considerations

This work provides preliminary insight into the chemical and physical properties of dielectric fluids and how they might act during an unintended release into the environment. It is clear that the studied products do not evaporate to any significant extent. Evaporation should not be assumed to be a primary fate for them. The fluids low evaporation and high flashpoints probably means that the fire hazard of a spill is low. This low fire hazard might also mean that in situ burning is not a viable response option, but further research would be required to say that definitively. Initially these fluids do not seem to form stable emulsions. However, we have shown that these fluids do absorb environmentally relevant wavelengths of light, and while this work was unable to test the emulsion behavior of samples exposed to simulated sunlight, the visual changes in some of the fluids, as well as the remarkable spreading and solubility changes could indicate a change in emulsion behavior that ought to be a topic for further research. A deeper investigation into the interfacial tension, spreading and solubility behavior of these photo-exposed samples, observed for the first time here, is also warranted. Finally, this work was motivated by providing the bulk property data required of operational spill models. Since the properties shown here depart from normal crude oil and petroleum products, modelling planning-relevant scenarios with this data would provide insight into how response to them might be different when the product is a dielectric fluid. Finally, this work did not drill down to a molecular level understanding of these properties, or of the changes introduced by sunlight exposure. Any conclusions of impacts on toxicity or final fate of these products would require more work in both the molecular composition of them and how that changes, along with traditional toxicological studies.

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Appendixes

Appendix A: Technical Summary

REPORT TITLE: Examination of Physical and Chemical Characteristics of Dielectric Fluids

CONTRACT NUMBER(S): IAA E23PG00033

FISCAL YEARS(S) OF PROJECT FUNDING: FY2024

CUMULATIVE PROJECT COST: $42,477.20

COMPLETION DATE OF REPORT: 28 May 2025

BSEE COR(S): Steven Buschang, Kevin Cabaniss

BSEE CO(S): Deja Bracey

PROJECT MANAGER(S):CAPT Gregory Hall, Ph.D.

AFFILIATION OF PROJECT MANAGER: U.S. Coast Guard Academy

ADDRESS: 27 Mohegan Ave, New London, CT 06320

PRINCIPAL INVESTIGATOR(S)*: CAPT Gregory Hall, Ph.D., Glenn Frysinger, Ph.D.

KEY WORDS: Dielectric Fluid, Density, Viscosity, Solubility, Evaporation, Flash Point, Pour Point, Interfacial Tension, Spreading, Emulsion, Photo-oxidation

* The affiliation of the Principal Investigators(s) may be different than that listed for Project Manager(s).

Appendix B: Figure Data

Table 7 Density Data for Figure 6

	Density (g/mL) 10 °C	95% CI	Density (g/mL) 10 °C Photo-Ox	95% CI	Density (g/mL) 20 °C	95% CI	Density (g/mL) 20 °C Photo-Ox	95% CI
MIDEL	0.9728	0.0013	0.9735	0.0008	0.9654	0.0003	0.9665	0.0002
FR3	0.9258	0.0005	0.9373	0.0009	0.9188	0.0002	0.9265	0.0002
HYVOLT	0.8877	0.0001	0.8925	0.0003	0.8815	0.0010	0.8862	0.0006
CALTRAN	0.8939	0.0015	0.8994	0.0006	0.8866	0.0007	0.8947	0.0009
DF-100	0.8746	0.0004	0.8809	0.0001	0.8676	0.0001	0.8740	0.0001

Table 8 Viscosity Data for Figure 7

	Viscosity (cSt) 10 °C	95% CI	Viscosity (cSt) 10 °C Photo-Ox	95% CI	Viscosity (cSt) 20 °C	95% CI	Viscosity (cSt) 20 °C Photo-Ox	95% CI
MIDEL	123.4	3.0	129.0	0.6	70.7	0.3	72.6	0.1
FR3	108.7	2.2	213.2	1.5	67.3	1.3	81.8	0.5
HYVOLT	33.2	0.2	55.7	0.2	20.4	0.3	29.8	0.0
CALTRAN	33.8	1.7	52.9	0.1	20.3	0.7	32.0	1.5
DF-100	104.0	0.1	128.1	0.6	58.4	0.0	70.0	0.4

Table 9 Interfacial Tension Data for Figure 8

	IFT (mN/m) 10 °C	95% CI	IFT (mN/m) 20 °C	95% CI
MIDEL	23.4	1.5	25.8	0.1
FR3	9.8	0.3	11.2	1.0
HYVOLT	38.08	0.1	43.9	0.1
CALTRAN	24.1	4.0	25.4	0.5
DF-100	29.7	0.9	30.3	0.5

Table 10 Solubility Data for Figure 10

	Solubility (mg/L) 20 °C	95% CI	% Solubility	Solubility (mg/L) 20 °C Photo-Ox	95% CI	% Solubility
MIDEL	0.073	0.019	0.0031	0.45	0.04	0.02
FR3	ND	ND	ND	4.69	0.05	0.20
HYVOLT	ND	ND	ND	14.70	0.04	0.61
CALTRAN	0.076	0.044	0.0032	22.41	0.45	0.93
DF-100	ND	ND	ND	3.74	0.06	0.16

6 Abbreviations and Acronyms

ADIOS	NOAA Automated Data Inquiry for Oil Spills
BP	Boiling Point
BSEE	Bureau of Safety and Environmental Enforcement
CI	Confidence Interval
DOC	Dissolved Organic Carbon
IBP	Initial Boiling Point
µM	Micro Molar
ND	Not Detected
NOAA	National Oceanic and Atmospheric Administration
OSPD	Oil Spill Preparedness Division
OSRR	Oil Spill Response Research
Photo-Ox	Photo-Oxidation
rpm	Revolutions per Minute
RT	Retention Time
SDS	Safety Data Sheet
SIMDIST	Simulated Distillation
UV	Ultraviolet

Department of the Interior (DOI)
The Department of the Interior protects and manages the Nation's natural resources and cultural heritage; provides scientific and other information about those resources; and honors the Nation's trust responsibilities or special commitments to American Indians, Alaska Natives, and affiliated island communities.

Bureau of Safety and Environmental Enforcement (BSEE)
The mission of the Bureau of Safety and Environmental Enforcement works to promote safety, protect the environment, and conserve resources offshore through vigorous regulatory oversight and enforcement.

BSEE Oil Spill Preparedness Program
BSEE administers a robust Oil Spill Preparedness Program through its Oil Spill Preparedness Division (OSPD) to ensure owners and operators of offshore facilities are ready to mitigate and respond to substantial threats of actual oil spills that may result from their activities. The Program draws its mandate and purpose from the Federal Water Pollution Control Act of October 18, 1972, as amended, and the Oil Pollution Act of 1990 (October 18, 1991). It is framed by the regulations in 30 CFR Part 254 – *Oil Spill Response Requirements for Facilities Located Seaward of the Coastline*, and 40 CFR Part 300 – *National Oil and Hazardous Substances Pollution Contingency Plan*. Acknowledging these authorities and their associated responsibilities, BSEE established the program with three primary and interdependent roles:

- Preparedness Verification,
- Oil Spill Response Research, and
- Management of Ohmsett - the National Oil Spill Response Research and Renewable Energy Test Facility.

The research conducted for this Program aims to improve oil spill response and preparedness by advancing the state of the science and the technologies needed for these emergencies. The research supports the Bureau's needs while ensuring the highest level of scientific integrity by adhering to BSEE's peer review protocols. The proposal, selection, research, review, collaboration, production, and dissemination of OSPD's technical reports and studies follows the appropriate requirements and guidance such as the Federal Acquisition Regulation and the Department of Interior's policies on scientific and scholarly conduct.