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Comparison of Electric and Radiometric Methods for Liquid Dielectric Diagnostic

***Abstract**. This paper is focused on the description and comparison the alternative photometric and radiometric methods for liquid dielectric quality diagnostic and parameters measurement, long-term practical verification and comparison of obtained data with conventional methods. The proper function and accuracy of the measured methods are verified in the Light Laboratory and in the CVVOZEPowerLab in Brno University of Technology.*

Streszczenie.** W artykule porównanie metody fotometrycznej i radiometrycznej stosowanej do diagnostyki właściwości dielektrycznych płynów. Przedstawiono weryfikację długoterminową oraz porównanie otrzymanych danych w wynikami metody konwencjonalnej. **Porównanie elektrycznej I radiometrycznej metody diagnostyki właściwości dielektrycznych płynów.

Keywords: Liquid Dielectric, Mineral Oil, Silicone Liquid, Spectroradiometer, Spectrum, UV-vis, Spectroscopy, D-lamp
Słowa kluczowe: właściwości dielektryczne płynów, metoda fotometryczna, metoda radiometryczna

1. Introduction to the Topic

This paper is aimed at creating a comprehensive methodology for comparison the laboratory measured quality of liquid dielectrics by electrical and radiometric or photometric methods. Standard electric parameters diagnostic could be divided on partial discharge measurement according by IEC 60270; DC electric parameters (EN 60247); dissipation factor measurement (EN 61620) and for breakdown voltage measurement can be used the procedure according the standard EN 60156. Conversely radiometric measurement for determining liquid quality used only as an alternative method. An exception is the testing of plant oils intended for food industry. In this case, the oil quality, type of oil and the presence of other substances are determined by optical and radiometric methods. Liquid insulators used in power engineering and energetic are most often tested according to ISO 2049 and EN 60422. The ASTM scale according to standard ISO 2049 for the determination of color of insulating transformer oil or other petroleum product. However, very accurate measuring technology is nowadays offered by optical and light laboratories. The result may be spectral transmittance or absorption measurements in visible, IR and UV area of spectra. Especially the UV area has proved to be very sensitive to small differences in the change in the quality of liquid dielectrics. Also, luminescence measurement is a very interesting method for measuring the quality of liquid dielectrics, thermal degradation and the presence of additives and water content.

Liquid dielectrics are used in the energetic and power engineering as insulating and heat transfer medium. Insulating oils are therefore an irreplaceable substance in many energetic systems and equipments such as transformers, some bushings, capacitors, oil cables or power switches and circuit breakers. The quality of liquid dielectrics greatly affects the reliability of the operation of these equipments. For this reason, energetic devices are regularly revised and oil samples diagnostics.

The most common degradation of insulating dielectrics is due to their aging. However, most modern oils contain stabilizers that allow oils to function as insulators for 30-60 years. This is true, however, if the quality of the dielectric is not influenced in the long run by other external influences. The most important influence is the thermal degradation, which occurs mainly due to the local overheating of windings losses - heavy short circuits, intermittent short circuits and short circuits between the plates in the magnetic circuit of the transformer. Thermal degradation changes the chemical composition of oils, changes in the length of macromolecular chains, releases free carbon and gaseous components such as hydrogen, oxygen and methane. The result is a small change in oil volume, the formation of a gas pocket. There is also a change in oil color - according to the ASTM scale the value increases. The most important change is the lower level of electrical strength and increased dielectric losses.

The second external influence affecting the quality of the oil is the penetration of water and air moisture into the liquid dielectric. All types of oils are very hygroscopic. The problem is the poor hermeticity of the equipments container and leakage insulation by the bushings. The electrical strength of the oil is rapidly influenced by the presence of water. In addition to water, airborne oxygen or sulfur oxides is also dangerous. These substances degrade the oil chemically and in particular cause corrosion of the internal transformer installation.

The last negative effect is the effects of the electric field. An alternating electric field causes charging and discharging of oil domains. This causes the liquid dielectric to move and increases the homogeneity of the oil throughout the volume. However, the electric field causes constant loading of long macromolecular oil chains, which may lead to changes in their length during the long time period. However, the immediate degradation and change in the chemical composition of mineral oils occurs when the electric field strength exceeds value approx. 2 kV/mm (1.8 kV/mm for esters and 2.6 kV/mm for mineral oil [3]). Partial discharge in oil could be caused by increased electric field strength. For example, creepage currents may also occur at the interface of solid dielectric material of transformer installation and liquid oil. All phenomena that cause partial discharges, sparks, creep currents, or an arc have a negative impact to the oil quality.

All kinds of oils are tested for quality, usually periodically or as output control. Oils used in the food industry are tested for compositional quality, acidity and content of undesirable substances. An oil group used in the engineering and automotive industries is tested for compliance with physical properties such as kinematic viscosity, surface and interfacial tensions, flash point and amount of released carbon. The liquid diagnostic in the energetic sector can be divided into chemical, electrical and optical tests. The most used are tests, diagnostics and measurement of electrical parameters.

1.1 Measurement of Electrical Parameters

Measurement and diagnosis of the tested liquids electrical parameters is one of the most basic output quality checks and periodic inspections. The measured sample of liquid is usually taken without access to air (air humidity) and placed under the internal gas or vacuum (low pressure) atmosphere. A sample of the liquid dielectric is placed in an oil tester or a test cell for measuring liquid dielectrics. The CVVOZEPowerLab is equipped with the Tettex 2830/2831 Dielectric tester and the Tettex 2903 Liquid dielectric measuring cell. This system makes it possible to measure the dissipation factor up to 10^{-5} with the resolution 10^{-6} at 50 Hz or the diagnostics can be focused to the sample permittivity measurement depending on temperature. All these parameters are measured in the voltage range up to 2 kVAC and sample temperature up to 150 °C. The Tettex 2903 measuring cell also makes it possible to measure the value of DC resistivity of the samples at the applied voltage up to 2.5 kV while monitoring the polarization level. The dielectric sample in the cell can be placed in a vacuum or in an inert gas atmosphere.

The second group of electrical tests are withstand voltage test by alternately or impulse voltage. Alternatively, another type of tests is carried out to measure the electrical strength of the material. For this purpose it is possible to use the Hipotronics AC Test System with a measuring cell equipped with mushroom electrodes.

1.2 Measurement of Optical Parameters

Optical diagnostics of liquid dielectrics are based on the measurement of spectral absorption or transmission of the measured sample. The fast oil diagnostic methods based on the ASTM scale according to ISO 2049 are most commonly used in practice. The oil or liquid sample is placed in a cuvette and compared to the sampler, see Figure 1.

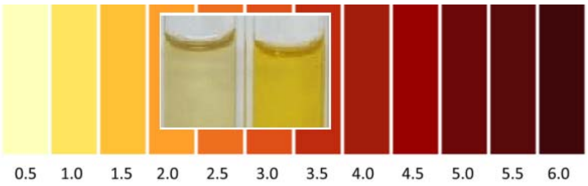

Fig.1. Liquid Sample Comparison to the Sampler Scale [1]

The result of this test is to quickly determine the degree of oil degradation. However, the disadvantage is the impossibility of sensitivity analysis and more precise identification of the type of oil, the character of the degradation and determination of the water content. Spectral measurements in both the visible and UV spectrum are suitable for determining [1]. Both methods are based on simple spectral transmission measurements. The measured oil sample spectra are always compared relative. The measured spectra are analyzed and compared with the previous state of the oil sample taken. The test chain of this analysis method is shown in Figure 2.

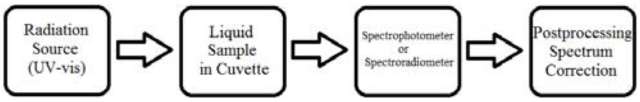

Fig.2. Spectrum Measurement Chain [1]

In addition to common methods, measurement can be extended by measuring sample luminescence. The method based on the oil sample luminescence is highly sensitive to the detection of impurities, low carbon and soot content.

Determining the dependence and difference between the dielectric liquid diagnostic results of electrical and optical methods is the main purpose of this article. Each of the newly used methods is distinctly sensitive and their combination can lead to increased measurement sensitivity, acceleration of the measurement process and the result of a reduction in price.

2 Input Knowledge for Diagnostics

Basic input is the question of what we want to measure and what should be the result. An example may be determining the type of oil. Types of dielectric liquids used:

- Mineral Oils - Made from oil. Saturated hydrocarbons are the most important - especially aromatic hydrocarbons, naphthalene and paraffin.
- Silicone Liquid - The main base of the oil molecules are silicon and oxygen with hydrocarbon compounds.
- Ecological and Food Oils - Natural oils such as sunflower, rapeseed or olive oil.
- Synthetic Esters Liquids [2].

Information about the aging of the oils, type or level of degradation is another diagnosed parameter. Aging of dielectric fluids can be divided into several types [2, 3]:

- Natural aging – Type of liquid aging at constant temperature and without liquid moving.
- Operational aging - Aging of the dielectric under the normal operating parameters of the equipment without exceeding the permitted limits.
- Artificial aging - Operational parameters of the oil are adjusted or exceeded for simulation of accelerated aging.

Various degradation processes affect the properties of the oil. More degradation processes operate simultaneously during operation. These processes can be divided into the following groups and their combinations [2, 4, 5, 6]:

- Natural aging.
- Electrical field degradation.
- Slow thermal degradation by operating temperature.
- Short-term overheating - short-circuit currents, inter-circuit short circuits, short circuits in the magnetic circuit.
- Degradation by the air humidity, moisture penetration and oxidation process.
- Degradation by changing the chemical composition of the oil, external contaminants, and degradation of stabilizers, oxidation, light or external radiation influence.
- Degradation by the soot, released carbon or other inorganic substances.

Table 1. Measured Oil Samples

No	Oil Type	Age, degradation type
1A	Mineral Exxon Univolt 60	New, fresh
1B	Mineral Exxon Univolt 60	Lab. aged, 180 °C, 15 min
1C	Mineral Exxon Univolt 60	Lab. aged, 180 °C, 60 min
1D	Mineral Exxon Univolt 60	Lab. aged, 230 °C, 15 min
1E	Mineral Exxon Univolt 60	Lab. aged, 230 °C, 60 min
2A	Mineral Paramo Mogul Oil	New, fresh
3A	Mineral Transf. Oil 90`s	Used, aged, approx. 20 years
4A	Mineral Transf. Oil 80`s	Used, aged, approx. 30 years
5A	Silicone Liquid - Oil Panoil	New, fresh
5B	Silicone Liquid - Oil Panoil	Lab. aged, 150 °C, 180 min
5C	Silicone Liquid - Oil Panoil	Used, aged, 1 year
5D	Silicone Liquid - Oil Panoil	Used, aged, 3 year with soot
5E	Silicone Liquid - Oil Panoil	New, with 10 ppm of water
5F	Silicone Liquid - Oil Panoil	New, with 100 ppm of water
6A	Ester Liquid – Midel 7131	Used, aged, approx. 3 years
7A	Natural Gas Motor Oil	New, fresh

Determining the correct diagnostic method depends on the type and character of the observed quantity. Accurate and correct measurement results can be achieved by using a method of measuring electrical parameters, using optical measurements or combinations thereof.

Measurement accuracy and repeatability is affected by the selected method. The same measurements are carried out repeatedly with another measuring sample to verify the repeatability of the measurement, the variance of the values and the homogeneity of the sample. The example is the use of 10 pieces per 30 ml of each sample of test liquids for electrical measurements and 10 pieces of 3 ml for optical measurement. Individual oil samples are deliberately chosen for different types (mineral, silicone, ester or rape liquid) and with different levels of degradation (new, aged 10 years, 30 years, laboratory aged, etc). Measured oil samples are listed in Table 1.

3 Practical Laboratory Measurements

Our practical measurements are based on the long-term activities of measurement liquid dielectric samples in the CVVOZEPowerLab. New liquid samples, aged, degraded samples and artificially aged oil samples were included in the tests. Oil tests are divided into electrical and optical applied measurement methods.

However, methods based on chemical changes or mechanical tests are also very often used. Examples are acidity test, oil flash point, or dynamic and static viscosity diagnostics. Unfortunately, this paper will not focus on these tests and their results will not be compared with other measurements.

3.1 Measurement of Electrical Parameters

All electrical parameters of the liquid insulating dielectrics were measured by the Tettex 2830 electronic bridge. Each measurement includes 10 tests of oil samples per 30 ml. The resulting values are the average value with the value scatter shown.

The Tettex 2904 Cell for Liquids allows test and heating up samples of liquids up to 150 °C and provides measurement with a voltage range of 0-2 kV AC. The maximum achievable electric field strength is 1 kV/mm in 2 mm sample gap. The air pressure in the cell was reduced to approx. 100 Pa by the vacuum pump. The result of these tests is the measurement of permittivity, dissipation factor and DC resistivity of the samples in dependence on voltage and temperature.

3.1.1 Oil Samples DC Resistivity Measurement

Resistivity of liquid dielectrics can be measured in dependence on time, temperature or voltage. The dependence of resistivity on the applied voltage or electric field strength is not very different for individual liquid samples. Measured resistivity value up to 1 kV/mm seems to be a constant. Partial discharges that affect the conductivity of the measured samples begin to show at higher values. All measured samples were tested at electric field strength value of 500 V/mm.

The time dependence of measured resistivity values is significant in the first few seconds. The value increases by approximately three orders. Then most polarization processes have already taken place and the value remains almost stable. For these reasons, each measured sample was measured 60 seconds after the applied temperature or voltage change.

Measurement of resistivity depending on the temperature of the sample is the most interesting and most relevant testing of individual dielectric liquid types. The results differ significantly for each type of liquid. The measurement results in Figure 3 were obtained by the test of 10 new samples of fresh Mineral oil, Silicone liquid and aged Ester liquid.

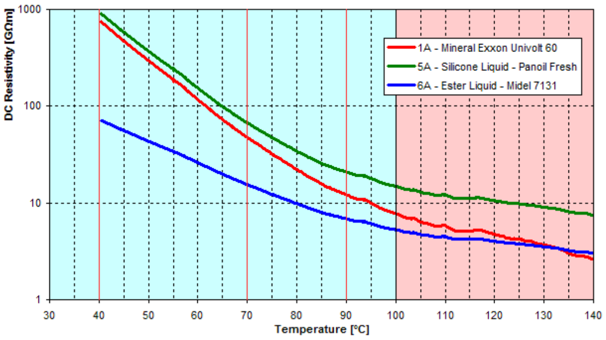

Fig.3. DC Resistivity of Liquid Samples

The result chart shows the logarithmic dependence of the resistivity values on the sample temperature and unequal behavior of different types of insulating liquids.

3.1.2 Measurement of Permittivity

Relative permittivity is another material parameter that significantly influences the behavior of the substance in the electric field. A material with a higher permittivity value puts greater resistance to the electric field when penetrating the material. The permittivity value is determined by the ability of material dipoles to react to the external electric field. The voltage drop value of mixed dielectrics and composite materials is inversely proportional to the permittivity of each material. Reaction of dipole moments to the electric field is poorly dependent on temperature, and with increasing temperature the permittivity decreases slightly [4].

However, relative permittivity values are different for different types of dielectrics. Values for liquid dielectrics are usually in the range from 2 to 4. And the samples of contaminated oils or oils with water content are higher.

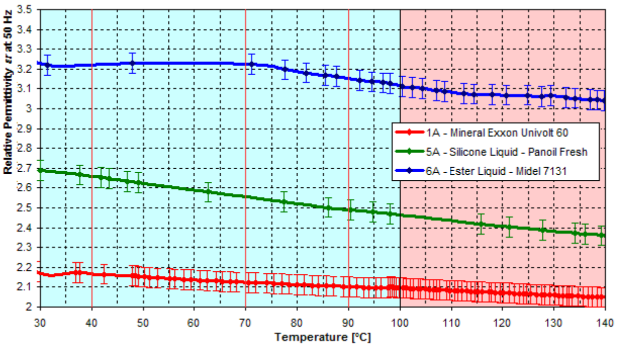

Fig.4. Relativite Permittivity of Liquid Samples

3.1.3 Measurement of Dissipation Factor

The dissipation factor (dielectric loss factor or tan delta) is the most used characteristic material parameter of solid and liquid dielectric, which is related to their quality, losses and degradations level.

The dissipation factor of the measured sample is very dependent on the intensity of the external electric field and the frequency of the current. Samples of dielectrics used in energetic are typically measured at a 50 or 60 Hz of line frequency. The dissipation factor achieves the lowest values at these frequencies. A lower current value by the capacity of the sample at a lower frequency causes a higher value of the loss factor, because the leakage resistive current is constant [4, 5].

The value of the loss factor also increases significantly with the sample temperature. Higher temperatures cause more dielectric losses. This dependence is highly exponential and may vary over the order in the operating temperature range. This dependence is shown in Figure 5 for different types of liquids. All samples were measured at a constant value of electric field 500 V/mm. The scattered values are also shown in the graph.

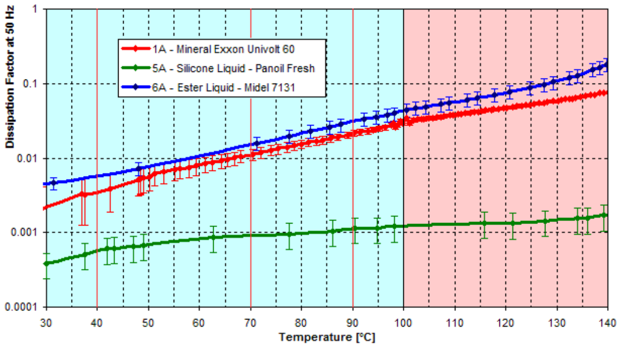

Fig.5. Measured Dissipation Factor of Oil and Liquid Samples

The dissipation factor is also dependent on the intensity of the external electric field. This dependence is less pronounced compared to temperature dependence; therefore, it only occurs at low temperatures. The measured dependence of the mineral oil dissipation factor on the temperature and on the electric field strength is shown the Figure 6.

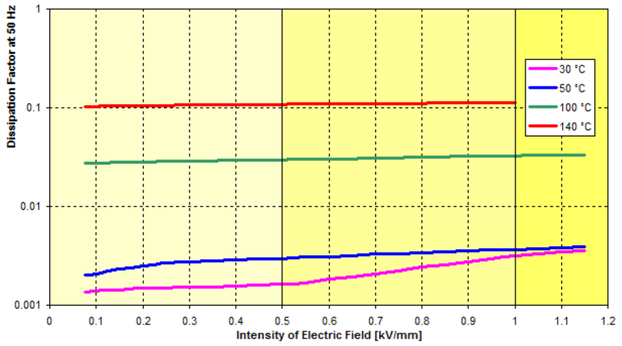

Fig.6. Dependence of Dissipation Factor of Mineral Exxon Oil on Intensity of Electric Field and Temperature

3.2 Measurement of Optical Parameters

Optical measurement of the properties of liquid dielectrics can now be regarded as an alternative to measuring electrical parameters. However, optical diagnostics may be faster and significantly cheaper. For example, it is very common to use a relative method of measuring oil samples using the ASTM scale according to ISO 2049 for the determination of the color of the insulating transformer oil or other petroleum product. Liquids are diagnosed by simply comparing the ASTM pallet with the oil sample in the cuvette. Liquid samples are compared with the palette in daylight. An example of a mineral oil scale is shown in Figure 7. Various pallets are used for different oils, insulating liquids and petroleum products [1].

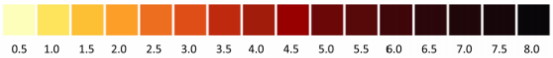

Fig.7. ASTM Color Scale for Mineral Oils according ISO 2049:2015

Comparative optical tests for liquid dielectrics are very fast, but they are less precise and they are only suitable for relative comparison of samples with the sampler or the determination of wear level.

Optical methods based on spectrometry are significantly more accurate. Differentiation between different types of liquids, determination of the degradation character, determining the degree of degradation, determination of water content or other chemical substances allows spectrophotometry and spectroadiometry of insulating liquid samples.

Spectrophotometry is the easiest and cheapest way to quickly measure oil samples. The method is based on the measurement of spectral absorption or transmittance in the visible part of the spectrum. A sample of the liquid is placed in a small cuvette (typically 1-3 ml). The source of light is a full spectral xenon lamp. The light passes through the sample and impinges on the CCD spectral sensor, and using the computing technique and correction the transmittance spectrum of the sample is calculated. In out case the samples of the liquids are measured by the Konica Minolta Spectrophotometer CM-3600D. The oil is placed in a 3 ml PMMA cuvette. The result of the measurement is a transmittance spectrum from 380 to 780 nm with a bandwidth of 5 nm. The results are more accurate than the ASTM method, but in addition to determining the degradation - darkening of the sample, it is not possible to determine anything else. Simple spectral measurements in the visible area are most commonly used in the chemical industry and, above all, in the food industry for measuring the quality of used edible oils [1, 8, 9, 10].

The use of spectradiometry brings significantly better results, which can already be compared with the electrical methods of measuring oil samples. The principle of spectroradiometry is the measurement of the spectra of transmittance and absorption of liquid samples in the UV-B, UV-A and blue part of the spectrum, i.e. in the range of 280 to 450 nm. Unfortunately, this method requires expensive measuring techniques, and the cost of testing is already comparable to conventional electrical parameter measurement tests. The source of radiation is a special deuterium discharge lamp "D-Lamp". A continuous spectrum over the entire UV range of the spectrum is produced by this lamp and only D-Lamp is therefore suitable for these spectral measurements. All measurements in this paper were performed by D-Lamp D2E Narva. The spectroradiometer Jeti Specbos 1211 with a bandwidth of 4.5 nm and a resolution of 1 nm in the range of 230-1000 nm was used for spectrum measurement. Experimental measurements in IR spectra were also implemented, however, without satisfactory results. Samples of oils are placed in small PMMA or quartz glass cuvettes. PMMA cuvettes are cheap and disposable, but Quartz glass cuvettes are very expensive. However, the differences in parameters are significant and for the purpose of spectrum measurement are decisive. Quartz glass cuvettes are usable from 180 nm of wavelength and can be used also in the UV-C range of the spectrum. However, cheap PMMA cuvettes are usable only from 260 nm. All the measurements in this paper were performed using quality quartz glass cuvettes [1].

The third area of spectroadiometry, which is suitable for measuring the properties of liquid dielectrics samples, is luminescence measurement. Luminescence is a complex process whereby the absorbed high energy radiation in a material evokes the excitation of electrons of individual substances, and these substances emit a portion of the radiation back with specific spectral distribution. Detection of the presence of certain substances (chemical pollution, breakdown products and water) and determination of the chemical composition of the measured oils can be accomplished by measuring luminescence spectra.

3.2.1 Spectrophotometry in Visible Spectrum Area

Measurement of the absorption and transmittance spectrum of the liquid dielectrics in the visible spectrum does not bring any new information. Practically all fresh, new oil samples have a spectral transparency index of 90-99 % across the entire visible spectrum (380-780 nm). The exception is mineral oil samples. Absorption in the blue part of the spectrum (400-450 nm) reaches about 10-15 % due to yellow oil coloration. Determination of oil type, water content and degradation level of individual oil samples can not be determined in this way. Spectroradiometry in the UV area and luminescence measurements are methods that are more sensitive and accurate to measure the parameters of liquid dielectrics. That is why we will look at the results of these methods in more detail.

3.2.2 Spectroradiometry in UV and Blue Spectrum Area

Measuring spectral transmittance in the UV spectrum is a great tool for determining the type of liquid dielectric. Differences between oil samples are striking. The measurement and evaluation results are shown in Figure 8.

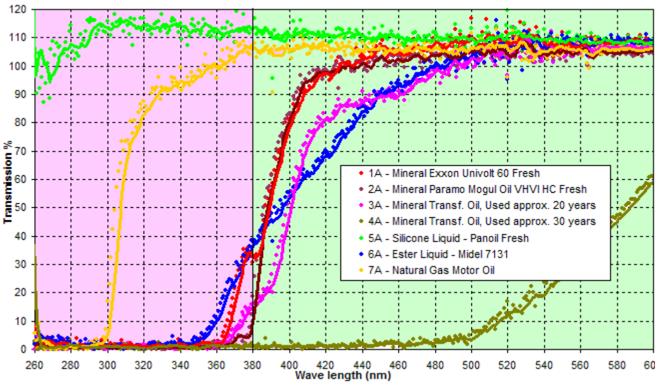

Fig.8. Differences in Spectral Trans. for Different Types of Liquids

Most of the decisive and specific changes in the spectrum are on the edge of the visible spectrum or already in the UV area. The 300-450 nm range is the most important for liquid analysis. There are three parameters, mathematical procedures that can be applied to the resulting spectra and obtain numerical results: Ultimate absorption wavelength, Slope of the spectral transmittance and the third parameter is the sharp changes in spectral distribution and specific spectral peaks. The ultimate absorption wavelength is wavelength when the value of transmittance is less than 10 % of the transmittance at wave length of 10 nm higher. A more detailed description is given in [1, 7, 8].

Table 2. Mathematical Processing of Spectral Transmittance

Oil Type	Ultimate Abs.Wavelength (mm)	Slope Value of S.Trans. (mm / 60 %)
1A	365.28	32
2A	373.32	15
3A	365.87	34
4A	398.08	108
5A	193.15	44
6A	353.49	73
7A	298.02	14

The mathematical significance of the "spectral transmittance" parameter is the slope of the spectral transmittance curve between 20% and 80% of the relative transmittance value. The results of the mathematical processing of the measured spectra of the individual oil samples are shown in Table 2.

Value ranges for identical liquids do not differ much. The measurement deviations in our laboratory tests reached a maximum of 10 to 20 %. These values can therefore be considered as reference for further evaluation and relative comparison of individual types of liquids. Further use of spectroradiometry is to determine oil degradation, wear level or aging. Aging may be natural (influence of electric field, heat, natural processes in chemical changes in oil) or it can be created artificially, in laboratory. The examples of measurement results are shown in the chart in Figure 9.

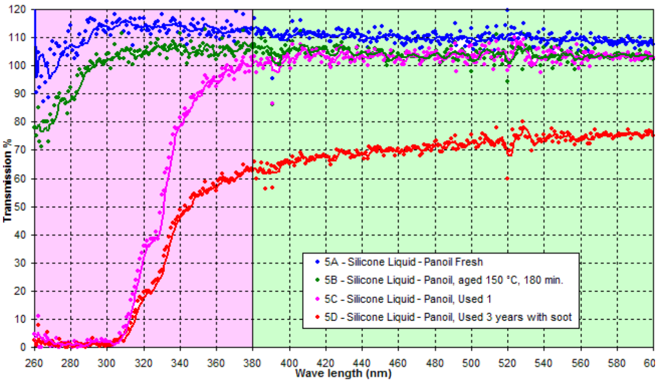

Fig.9. Differences in Spectral Trans. for Different Degradations

The results in Figure 9 show differences in spectral transmittance of mineral oil samples. The new and fresh "5A" mineral oil achieves a constant level in the area of visible radiation. The values over 100% of transmittance are partly due to luminescence in UV radiation. Artificially degraded sample "5B" or naturally aged "5C" and "5D" samples show a certain decrease in visible light transmission. However, the main changes occur in the field of UV area. Results show that the analysis of oil samples in the UV range of the spectrum can be used to measure relative or absolute degree of degradation.

And the last analysis of the water content and other soluble substances in the oil can be performed gain by spectroradiometry transmittance spectral measurements.

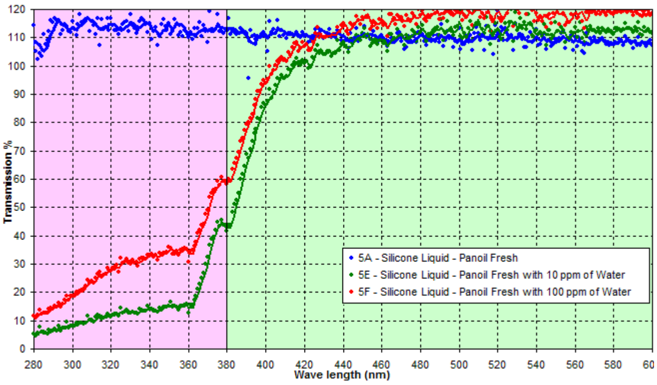

Fig.10. Spectral Trans. of Silicone Liquids with Water Content

Water, alcohol or salt in the oil sample can not be detected in the visible area of the spectrum. All of these substances are transparent and do not exhibit significant absorption in the range of 380 to 780 nm. However, primarily water and the dissolved sodium, potassium and calcium salts cause significant absorption of measured oil samples in the range of about 300-400 nm. A specific feature is the steep change in the spectrum. Occasionally, even very sharply varying parts of the spectrum or significant peaks are due to the specific chemical composition of the dissolved salts. Examples are spectra of measured spectral transmittance of mineral oil with a water content of 10 ppm "5E" and 100 ppm "5F" in Figure 10.

3.2.3 Oil Samples Luminescence Spectral Measurement

Luminescence is very complex and for describes the complicated phenomenon that occurs with most known substances. It is also present in all tested oil samples. Each wavelength of high-energy radiation (UV radiation) affects the result at longer wavelengths with less energy. Each chemical component of liquid and other test substances behaves specifically and exhibits different spectral peaks or absorptions. However, the test substance can only be tested in complexity with the overall chemical composition. Also, the spectrum of radiation source can be only one specific wavelength (laser, UV diode) or can be implemented as a continuous light source (in our case for example L-lamp) [13, 14].

Based on this knowledge, specimens can be collected for the oil samples and can be compared relatively comparatively in the future as images. Any chemical changes, degradation of chemical impurities, stabilizers, etc. may be determined by this analysis. Measured spectra of liquid samples for a continuous source of radiation are shown in Figure 11.

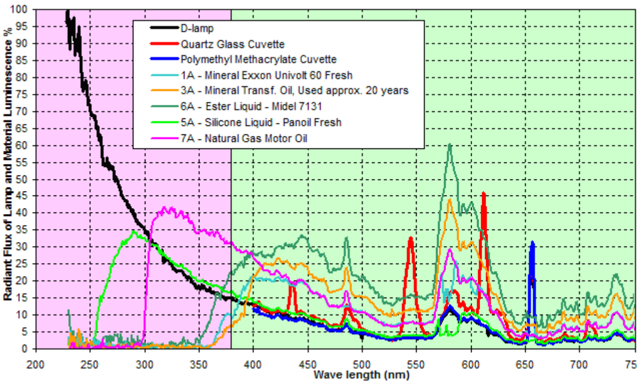

Fig.11. Liquid Luminescence - Continuous Radiation Source

And the spectrum of radiation produced by an oil samples for a discrete radiation source is shown in Figure 12. UV diode with a peak value of 278 nm was used as discontinuous radiation sources.

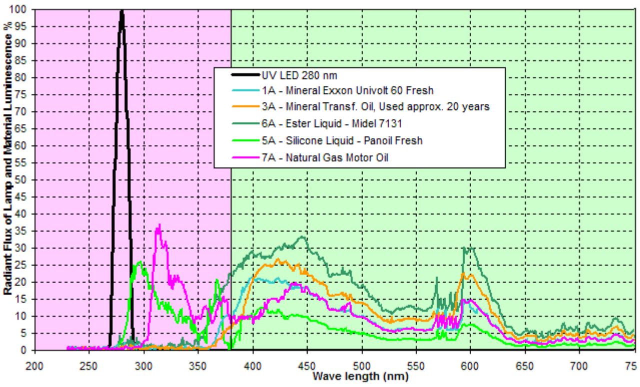

Fig.12. Liquid Luminescence - Discrete Radiation Source

Spectral distribution of radiated radiation is very similar for different types of oils. This is related to a similar chemical composition of individual samples. The difference is the initial value of wavelength, but above all, the summary intensity of the individual spectra. Specific spectral peaks are indicative of the admixture of various substances, contaminants, stabilizers, but also for determination of water, sulfur or free carbon content.

The difference in the spectral distribution of radiated radiation in the case of a discontinuous radiant source is mainly in lower intensity at longer wavelengths and the loss of some specific peaks. A more detailed description is complicated and inappropriate for the purposes of this paper. Our research in this field will focus on creating a database system for collecting images of individual oil samples and comparing these images with knowledge of specific degradation.

4 Comparison of Individual Methods

The measurement of standard electrical parameters of liquid dielectrics has demonstrated its objectivity, accuracy and, above all, the repeatability of measurement. However, even the results from optical and radiometric measurements were very beneficial.

It is not possible to carry out a complex comparison of the individual methods only on the basis of the measured samples. Therefore, the evaluation will only be classified if the method is capable of a particular measurement and which method is more sensitive or which method ensures the better repeatability of the measurement. The final evaluation of the methods is given in Table 3.

In addition, the individual parameters considered will be discussed in detail. However, current results would require further discussion. First, only selected electrical parameters, photometric and radiometric parameters are measured. Further parameters such as kinematic viscosity, surface and interfacial tensions, acidity and electrical strength should also be measured in the future research. Secondly, only a small number of samples were measured. Higher reporting value would result from multiple measured samples with different types of degradation, age, composition, manufacturer and type.

Table 3. Comparison of Electrical and Radiometric Methods

Parameter	Electric Par. Meas.	Radiometric Meas.
Soot content	D*	A
Water content	A	B
Acidity of oil	C	D
Dissipation factor	A	D
Parameters depen. on temperature	E	A
Identification of impurities	D	A
Thermal degrade	C	A
Ageing	B	A
Type oil identification	C	A

* Note: A – the best, F - the worst

4.1 Soot and Free Carbon Content in Oil Samples

Small particles of soot and carbon are formed by thermal degradation and discharges in oil. Particle sizes are in the range of about 1 to 100 µm. Measurement of the dissipation factor is not very sensitive to this degradation because it is done at low electrical field intensities. Only the electrical strength measurement of the oil is sufficiently sensitive to show the presence of soot. Conversely, optical measurements are the most appropriate for qualitative and quantitative measurements of this degradation. Spectral transmission in the UV and visible area of the spectrum linearly decreases with the presence of soot. However, the most appropriate method is microscopy with special software that determines the number of particles and size in a given volume.

4.2 Water and Moisture Content in Oil Samples

Liquid dielectrics always contain water, typically in the range of tens to hundreds of ppb. Significant decreases in electrical strength and increase of loss factor occur only from 1 ppm. Increasing the water content from 1 ppm to 100 ppm causes an increase in the dissipation factor by about one order. So measurement of the loss factor is the most sensitive method for determining the water content in the liquid dielectric. Increased content of water also causes a small increase in the permittivity of the measured sample.

Optical and radiometric methods are not very sensitive to the measurement of water content in the liquid dielectric because pure water is not absorbed in the spectral range of 250 to 1000 nm. Only in the IR area is absorption significant, and the method of spectral transmittance measurement in IR radiation is sufficiently sensitive for water content measurements below 1 ppm. However, measurements from 250 to 450 nm were successful and sensitive in our case. Samples of liquid with a water content of approx. 1, 10 and 100 ppm were measured. Absorption in the 250 to 450 nm region is proportional to the water content. However, this absorption does not cause by water alone, but dissolved Na, K, Ca and Mg ions from oil or added water. This optical method is therefore dependent on the type of oil and salt content. And also it can be used for determining how oil contamination with water, because water from air moisture does not usually contain the salt ions.

4.3 Acidity of Oil Samples

The acidity of the measured oil sample is one of the main indicators of quality, age and level of degradation. Acidity according to ASTM D 947, DIN 51558 and EN 14104 can be expressed as the amount of KOH needed to neutralize the acidity of the oil in mg KOH per one gram of oil. Measurements of dissipation factor or dc resistance are methods that can alternatively be used. However, the change in conductivity of the sample may not be due to its acidity but also to thermal degradation, water or other admixtures. Unfortunately, it is not possible to decide between these degradations by electrical measurement methods.

Photometric and radiometric methods based on spectral permeability or absorption are not sensitive enough and can not distinguish the nature of degradation. A possible alternative is to measure the luminescence of the oil sample. Small changes in the chemical composition, including the acidity level, can be measured sensitively by this method. Unfortunately, this measurement is complicated and can only be done by comparing spectra.

4.4 Dissipation Factor

Dissipation factor is a variable that can be directly measured using methods of electrical measurement. The dissipation factor of oils in the energy sector is measured at temperature of 40, 70 and 90 °C. The value is usually 0.001 and lower for new oil samples; the value of ageing and contaminated oils is higher. And the maximum allowable limit for the operation of energetic equipments is 0.005.

Methodology for measuring dissipation factor dielectric liquid samples is still in the development stage. The method is based on the measurement of the initial wavelength where the spectral absorption (300-500 nm) and the slope of the spectral characteristic occur. This behavior for each type of oil is directly proportional to its loss factor at a given temperature. However, for the creation of a useful methodology, a significant number of differently degraded oil samples have to be measured.

4.5 Measured Parameter Dependence on Temperature

The standard measured electrical parameters of liquid dielectrics - dissipation factors, resistivity, permittivity and electrical strength, as well as viscosity and surface tension parameters are very strongly dependent on the temperature of the measured sample. Above all, repeatability and comparability of measured samples place great demands on the knowledge of dependence on the temperature of the measured quantities or the necessity of measuring at one precise specific temperature.

However, this dependence was not confirmed in radiometric and photometric measurements. And most spectral measurements and measurements of oil samples luminescence are temperature independent in the range of 30 to 90 °C. The dispersion of measured values does not exceed 10 % of the mean value. This is one of the main advantages of optical measurements, as there is no need for expensive equipment for heating and temperature stabilization of the oil sample. Optical methods are therefore significantly faster and cheaper in design.

4.6 Impurities Measurement and Identification

The contaminants and impurities entering to the liquid dielectric from its manufacture. During the transport and operation in facilities where they penetrate from the surrounding space or they are released from the internal construction of the devices. Thermal degradation of oils also causes impurities and chemical compounds to degrade oil. Impurities in the oil release free ions that increase conductivity. Conventional oil measurement methods, based on the measurement of loss factors, can very easily detect the presence of impurities. However, the identification of specific pollutants is virtually impossible. Impurities and contaminants in oils optical method can be demonstrated by changing the spectral transmittance in the visible radiation. However, the method is significantly more sensitive in the UV area. Identification of specific contaminants is possible based on specific measured spectral peaks for a given chemical component. Another sensitive method is based on luminescence measurement. There is great scope for research because the response is also dependent on the spectral distribution of UV-emitting sources.

4.7 Thermal Degradation of Oil Samples

Thermal degradation of liquid dielectrics involves the release of soot, the release of contaminants and the chemical change of oil molecules in the distance and properties. The process of thermal degradation also accelerates oil oxidation and sulfur-oil reaction to metallic instrument components.

This process is natural and occurs at low temperatures (normal ambient) for a long time (decades). The result is a darkening of oil according the ASTM Color Scale.

However, local and total oil overheating speeds up this process. In particular, thermal degradation by high temperature of discharges causes chemical contamination of all the oil contained in the device. Measurement of the dissipation factor is again a suitable method for determining the level of degradation, however, without distinction of the character. Optical methods are considerably more sensitive to determine the nature of the degradation. Contamination of oil soot causes a decrease in uniform spectral transmittance in the UV and visible spectrum. The change in chemical composition is reflected by the formation of specific absorption peaks in the absorption spectrum. A more sensitive method for determining the degradation character may be the use of luminescence measurements in UV and visible spectrum.

4.8 Ageing of Oil Samples

Aging is a natural process that can be partly compared to previous thermal degradation. However, in addition to thermal degradation, natural processes of degradation of oil preservatives and stabilizers are included. The aging rate is different for different types of oils. The most common mineral and silicone oils are aging. Esters and bio-organic oils, on the contrary, are less stable and their degradation over time affects many factors - oxidation, UV radiation, visible radiation, electric fields, thermal degradation and degradation of stabilizers.

The level of oil aging can be easily determined by measuring electrical strength. The electrical strength of new oils is 60 to 100 kV/2.5 mm. The lowest limit for aged oils is 10 kV/2.5 mm. Measurement of the loss factor can also be used. An interesting method is to determine the polarization processes in DC resistivity measurements. The time dependency of a change in DC current passing through an oil sample can provide important information about the level and nature of the degradation. The applicable measurement procedure is currently being developed in our laboratory.

Photometric and radiometric methods are usable as well as thermal degradation measurements. Measurement results in spectral absorption at specific wavelengths that correspond to a particular type of degradation process.

4.9 Identification of Oil Type

Determining the type of oil by conventional electrical methods is based on the knowledge of the approximate values of permittivity and dissipation factor for a particular type of new oil sample. However, the combination of different types of oil degradation and temperature dependence of new and aged oil samples makes this method less accurate.

The alternative is again an optical method that is highly sensitive and independent of temperature, especially with the new oil samples. The wavelength of spectral absorption in the UV area is specific and unique for each type of oil. This method is highly reliable even for slightly thermally degraded and aged oil samples and achieves high value of repeatability.

Conclusion

The current results of the insulating liquid diagnostics are summarized in Chapter IV. At present, our research is focused on increasing the sensitivity of luminescence samples and eliminating external influences such as the influence of the spectral source or the luminescence of the cuvette.

The results of the diagnostics have shown the possibility of using radiometric methods of liquids diagnostics as a suitable and complementary alternative to conventional measurement of electrical parameters. Current measurements are focused on the analysis of modern silicone liquids, synthetic esters "Midel 7131" or rape and palm oil “FR-5” samples.

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REFERENCES

[1] Krbal M., Stepanek J., Nekvapil J., Analysis of Liquid Dielectrics by Photometric Instruments, *In Proceedings of the 2018 VII. Lighting Conference of the Visegrad Countries (Lumen V4)*. 2018. ISBN: 978-1-5386-7923-4.

[2] Heatcote J.M., The J & P Transformer Book, Great Britain: *Reed Educational and Professional Publishing*, 1998. 12. ISBN 07506 1158 8.

[3] Marci M., Kolcunová I., Kumimský J., Dielectric Properties of Natural Esters, Dept. of Electric Power Engineering, FEI TU of Košice, Košice, Slovak Republic, 978-1-4244-8782-0/11/©2011 IEEE

[4] Blodgett R.B., Influence of Absorbed Water and Temperature on Tan Delta and Dielectric Constant of Oil-Impregnated Paper Insulation, *IEEE Transactions on Power Apparatus and Systems* 81(3):93 – 98, May 1962, DOI: 10.1109/AIEEPAS.1962.4501273

[5] Peng D., Yang D., Wang F., Research of the Dielectric Loss factor tan delta of Transformer Oil by Multi-Parameter Regression Analysis, Changsha University of Science and Technology Changsha, China, 978-1-4244-2487-0/09/$25.00 2009 IEEE

[6] Ullah R., Rashid A., Haider A., Ullah Z., Ahmad N., Sulemani M. S., Rahman N. U., Performance Evaluation of Power Transformer under Different Diagnostic Techniques, COMSATS Institute of Information Technology, Abbottabad,Pakistan,978-1-5386-1370-2/18/$31.00, 2018 IEEE

[7] Norazhar A.B., Abu-Siada A., A Novel Method of Measuring Transformer Oil Interfacial Tension Using UV-Vis Spectroscopy, *IEEE Electronic Insulation Magazine*, IEEE Jan/Feb Vol..32, No. 1, 0883-7554/16/©2016/IEEE

[8] Norazhar A.B., Abu-Siada A., Islam S., El-Naggar M. F., A Novel UV-Vis Spectroscopy Application to Measure Interfacial Tension of Transformer Oil, 978-1-4673-8040-9/15©2015 IEEE

[9] Hussain K., Karmakar S., Condition Assessment of Transformer Oil using UV-Visible Spectroscopy, National Institute of Technology Rourkela, India, 978-1-4799-5141-3/14©2014

[10] Sing L.Y., Ker P J., Jamaludin M.Z., Mun L.H., Shukri C.N., Determining the Color Index of Transformer Insulating Oil Using UV-Vis Spectroscopy, Universiti Tenaga Nasional Selangor, Malaysia, 978-1-5090-2547-3/16/©2016 IEEE

[11] Kalathiripi H., Karmakar S., Fault Analysis of Oil-Filled Power Transformers Using Spectroscopy Techniques, Department of Electrical Engineering, National Institute of Technology Rourkela, India, *19th IEEE International Conference on Dielectric Liquids (ICDL)*, Manchester, United Kingdom, 978-1-5090-4877-9/17/©2017

[12] Alshehawy A.M., Mansour D.A., Impact of Thermal Aging of Transformer Oil on UV-Vis Optical Spectrum and Dielectric Properties, High Voltage and Superconductivity Laboratory, Department of Electrical Power and Machines Engineering, Tanta University, Egypt, 978-1-4673-9063-7/16/©2016 IEEE

[13] Chen P., Li Y., Signal Processing of Oil Detection in Port Area Based on UV-induced Fluorescence, Navigation College, Dalian Maritime University, Dalian, China, *2017 4th International Conference on Information, Cybernetics and Computational Social Systems (ICCSS)*, 978-1-5386-3257-4/17/©2017IEEE

[14] Panigrahi S.K., Thakur S., Sarathi R., Mishra A.K., Understanding the Physico-chemical Properties of Thermally Aged Natural Ester Oil Adopting Fluorescent Technique, Department of Electrical Engineering, Department of Chemistry, Indian Institute of Technology Madras, India, DOI: 10.1109/TDEI.2017.006592