

# Spectroelectrochemical Tracking of Transformer Oil Aging Dynamics (Case Study)

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## Abstract

The preservation of insulating oil's system integrity is a key strategy for maintenance planners to curtail the incidence and progression of internal faults. This imperative highlights the essential role of accurate diagnostic methodologies in transformer oil (electrical insulating oil) condition monitoring and analysis. This study aims to evaluate long-term operational aging effects on molecular and dielectric properties of insulating oils used in power transformers within Syrian electricity grid, employing visible spectroscopy and Fourier Transform Infrared (FT-IR) spectroscopy. A comparative analytical approach was adopted, focusing on spectroscopic changes in conjunction with electrical property measurements. Four oil samples of the same origin were analyzed: fresh unused oil, oil used for three years, oil used for ten years - taken from the bottom- and oil used for ten years - taken from the top. Additionally, relative permittivity (dielectric constant) of all samples was measured at multiple frequencies (1, 2, 3, 10, 100 and 200 kHz). Results identified a two-stage aging process: an initial decrease in IR transmittance (first 3 years), followed by a significant increase after 10 years, particularly in the bottom sample, indicating accumulated polar oxidation products. Dielectric properties varied accordingly. The study confirms FT-IR as an effective tool for monitoring oil degradation.

**Keywords:** Transformer oil; aging; FT-IR spectroscopy; dielectric constant.

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## Introduction\*

Many researchers have studied properties of mineral insulating oil and the factors that can improve its behavior [1]. Liquid dielectrics not only provide electrical insulation, but also play a vital role in thermal management by dissipating heat generated during power transformers operation. The choice of dielectric liquid is influenced by several factors, including the transformer's rated voltage, operating

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\*The abbreviations list is in page 734.

environment, load conditions and geographical location of installation. Various types of transformer oils serve as liquid dielectrics, each with unique properties and applications [2]. Mineral oils are the most commonly used, due to their excellent electrical insulating properties, availability and cost-effectiveness. Ester oils, which can be either natural or synthetic, are gaining popularity as alternatives to mineral oils, owing to their biodegradability and higher flash point [3]. Dielectric breakdown tension, which expresses the oil's ability to withstand tensile stresses without failure, is measured. It is the stress at which breakdown occurs between the electrodes under specific test conditions. This test primarily indicates the presence of electrically conductive contaminants within the oil, such as water, dirt, wet cellulose fibers or conductive particles. A high breakdown tension does not necessarily indicate the absence of contaminants. ASTM D877 specifies electrodes as smooth, flat discs, which are not representative of electrodes found in transformers. Although circular electrodes described in ASTM D1816 do not perfectly match characteristics of insulated transformer electrodes, they are closer to those used in transformer devices. However, the electrodes described in the former are more responsive to particulate matter and oil-dissolved substances, and they are detrimental to the transformer's electrical durability.

Therefore, test results according to the former provide a better assessment of potential changes in the oil caused by the transformer. The following are methods for measuring breakdown stress of insulating oils: ASTM D877 [4] recommends a routine acceptance test of untreated oil obtained from the supplier and used in circuit breakers. This test method uses flat-faced cylindrical electrodes spaced 2.5 mm apart. Sensitivity decreases when its effective value exceeds kVr.m.s. (25); ASTM D1816 [5, 6] recommends tests on fluids that have been treated for use in transformers or contained within them or in conversion converters; ASTM D924 recommends a test which is used to check for oil degradation or contamination due to its sensitivity to ionized pollutants.

Loss factor (power factor) [7] is a measure of energy losses in liquid dielectrics subjected to alternating current (AC) fields, which are thermal within the liquid. A low loss factor means the liquid will cause minimal power losses.

Fourier Transform Infrared (FT-IR) spectroscopy identifies unknown materials; it determines the quality or composition of a sample and the quantity of components in a mixture. The insulating oil used in power transformers consists of saturated hydrocarbons such as paraffins and naphthenes, and it cannot conduct electric current or dissolved water. The oil's conductivity depends on its type, and increases over time. Contaminants, such as from refining residues and aging/oxidation products, enable the oil to conduct ionic current. Oxidation/degradation products of the oil are divided into soluble (dissolved) and insoluble (suspended) products [8]. The utility of FT-IR spectroscopy in detecting oxidation depends on the type of base oil used in the fluid's formulation. Synthetic fluids often contain ester compounds with a distinct peak in the infrared spectral region where the oxidation level of mineral oils is

measured. For this reason, it is important not to use FT-IR results alone for diagnosis, but to study the trend of these results and present them in conjunction with other oil-related criteria such as viscosity [9].

## Materials and methods

### Materials

Present investigations were focused on the measurement of based electrical and chemical properties from mineral insulating fluids. Four types of fluids were used as mineral oils: fresh unused oil (A), oil used for three years (B), oil used for ten years, taken from the top (C), and oil used for ten years, taken from the bottom (D).

### Characterization of electrical and chemical properties

Mineral oil samples were tested in terms of their electrical and chemical properties, including breakdown voltage, acidity, moisture content, loss factor and relative permittivity, in accordance with standardized methods outlined by IEC and ASTM. Moisture content was measured using a Precisa XM 60 meter, according to ASTM D6304 [10]. Vessel temperature was set to 105 °C. The meter completed calibration without any movement or vibration. The result was displayed as a percentage.

Acidity was experimentally measured according to IEC 62021 [11]. In this experiment, acid concentration in oil was determined by the volume of a KOH solution required to neutralize it. The solution was loaded into a burette, while the oil sample was placed in a flask beneath it. The stopcock of the burette was carefully opened, allowing KOH to flow gradually into the oil sample. Titration continued until a visible color change occurred in the oil, indicating equivalence point where acid has been completely neutralized.

Breakdown voltage was measured using an automatic Megger device, according to IEC 60156 [12]. The test was conducted at room temperature. The distance spacing between electrodes was fixed to 2.5 mm, and the sample was placed in the container. A gradual voltage of 2-5 kV/s was applied, and six consecutive tests were conducted until the occurrence of electric shocks. Dissipation factor (loss tangent:  $\tan\delta$ ) and dielectric permittivity ( $\epsilon_r$ ) were measured by Schering bridge. Samples were placed in the electrode system, and temperature was controlled from 20 to 100 °C. Applied voltage was changed from 0.1 kV up to 2.0 kV by step 0.2 kV. First measurement was taken at room temperature. After that, temperature was increased up to 100 °C by step 10 °C [13]. Properties of the studied sample are listed in Table 1.

**Table 1:** Properties of studied samples.

| Sample | Moisture% | Acidity(mgKOH/g) | BDV(Kv) | $\epsilon_r$ | $\tan \delta$ |
|--------|-----------|------------------|---------|--------------|---------------|
| A      | 0.615     | 0.0461           | 44      | 3.2          | 0.007         |
| B      | 0.821     | 0.0517           | 33      | 3.244        | 0.008         |
| C      | 0.923     | 0.0522           | 31.33   | 3.283        | 0.01          |
| D      | 0.919     | 0.0498           | 31.83   | 3.488        | 0.03          |

*FT-IR analysis*

IR absorption positions are generally presented as either wavenumbers ( $\nu$ ) or wavelengths ( $\lambda$ ). Since wavenumbers define the number of waves per unit length, they are directly proportional to frequency. Wavenumber and wavelengths can be interconnected using the following equation:

$$\nu(\text{cm}^{-1}) = \frac{1}{\lambda(\mu\text{m})} \times 10^{-4} \quad (1)$$

Transmittance is the ratio of radiant power transmitted by the sample to radiant power incident on it. Absorbance is the logarithm to base 10 of reciprocal of transmittance, which was given by the following equation:

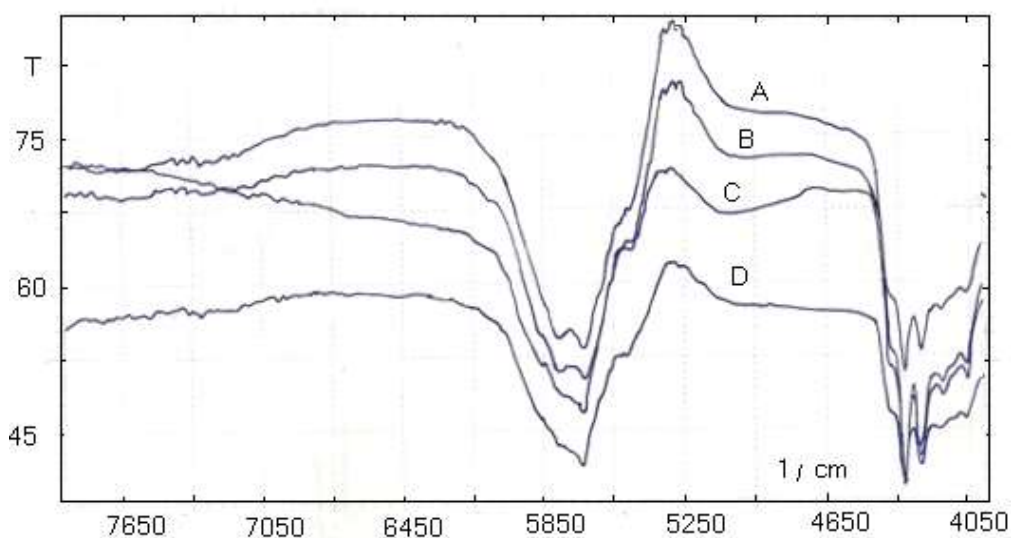
$$A = \log_{10} \frac{1}{T} = -\log_{10} T = -\log_{10} \frac{I}{I_0} \quad (2)$$

where T is transmittance, I is radiative power transmitted by the sample,  $I_0$  is radiant power incident on the sample and A is absorbance.

Transmittance spectra provide better contrast between intensities of strong and weak bands, because it ranges from 0 to 100% T, whereas absorbance varies from infinity to zero [14-15]. In this study, analyzes were performed by an IR prestige – 21 spectrometer. Spectroscopic analysis is based on the difference in vibrational frequencies between different chemical bonds in compounds. Infrared radiation, of which wavelengths range from 700 nanometers to 1 micrometer, is able to affect bonds in a compound's molecules, generating molecular vibrations. This helps determining the type and number of chemical bonds connecting the molecules of the compound, as well as its structure and chemical composition.

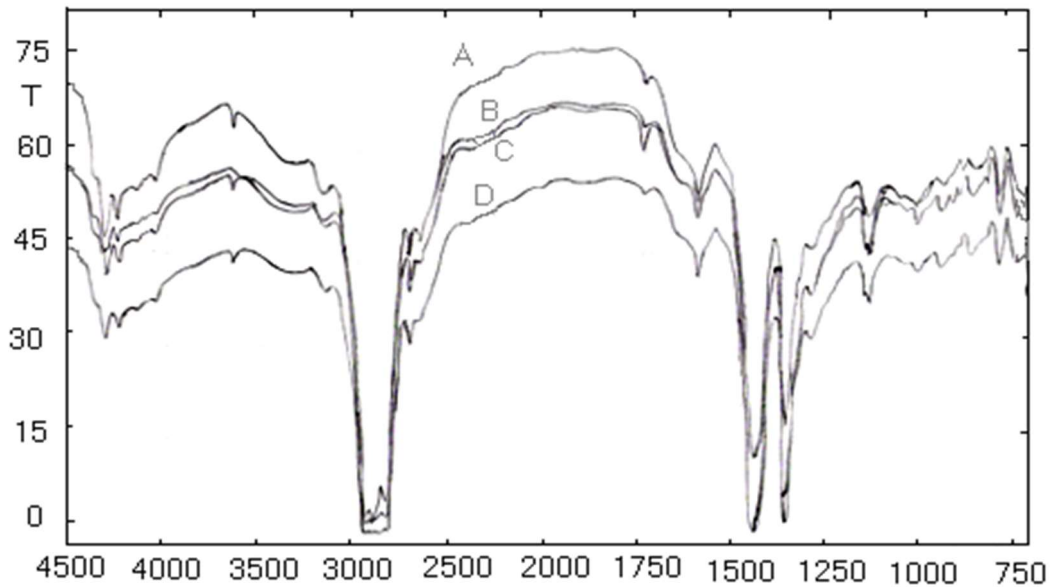
**Results and discussion**

Infrared spectral characteristics of electrical insulating oil change with the effect of usage time, as shown in Fig. 1.



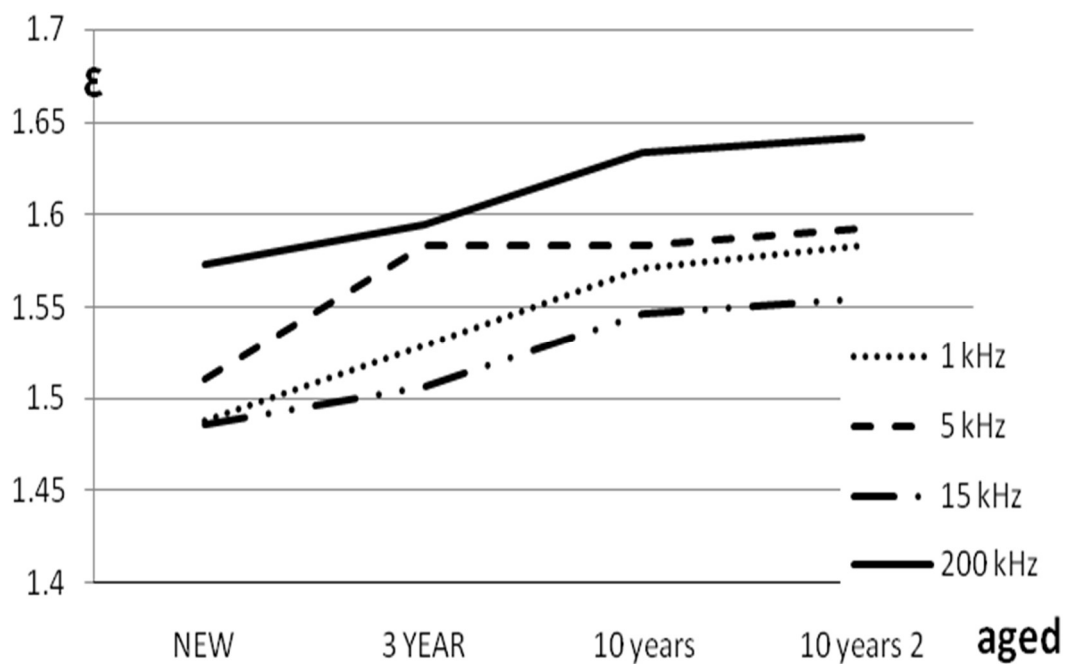
**Figure 1:** FT-IR of studied sample change with the effect of usage time.

Infrared spectral characteristics of electrical insulating oil change with the effect of usage time (another spectral range) are shown in Fig. 2.



**Figure 2:** FT-IR of sample change with the effect of usage time (another spectral range).

The effect of changes in relative electrical permittivity of the different samples taken with the change in usage time (Fig. 3) was studied in the frequency range shown above.



**Figure 3:** Changes in relative dielectric constant of different samples with changing usage time.

That effect was calculated relatively as an absolute value by dividing electrical capacitance of a plane capacitor with an oil dielectric by capacitance of the same capacitor insulated with air (real and imaginary parts of electrical permittivity of electrical dielectric oil samples were not calculated).

Relative permittivity ( $\epsilon_r$ ) value increases with operational years of transformer oil within the transformer. Fig. 3 illustrates that this increase follows a linear pattern, though it is not symmetrical at different voltage levels. For new transformer oils [16], measured value was approx. half the reference values, indicating that moisture content exceeded specifications' allowable limits. It is scientifically established that reaching a moisture content of 0.06% reduces electrical permittivity by nearly half. Additionally, the insulating oil may interact with certain transformer components, with reaction products accumulating over time, as the oil circulates between cellulose-insulated copper windings and the transformer's cooling fins while heating up during operation.

Consequently, optical absorption spectrum of transformer oil, which indicates the intensity of its transmittance across wavelengths in the visible range, decreases as the oil's service life within the transformer increases. This is observable from the color change of samples, where absorptivity increases with prolonged use.

Transformer oil spectrum possesses known primary absorption peaks characteristic of paraffinic oils [17, 18], which generally indicate carbon-hydrogen bonds ( $-\text{CH}_3$ ,  $-\text{CH}_2$ ) with their various vibrational modes such as bending, stretching, spatial distribution and rotation. Spectral details in Fig. 1, within the wavenumber range from 4000 to 400  $\text{cm}^{-1}$ , generally align with samples studied by researchers [19]. The effect of transformer oil service time on the spectrum can be observed by examining infrared spectra at the following points: in the mid-infrared range (4000–400  $\text{cm}^{-1}$ ), high absorption peaks (low transmittance) expected for aliphatic organic structures are present in two regions (3100–2650  $\text{cm}^{-1}$ ), corresponding to vibrations of  $-\text{CH}_3$  and  $-\text{CH}_2$  groups and carboxylic acid ( $-\text{COOH}$ ) groups. Notably, transmittance depth decreases with increasing years of service. Characteristic minor absorption peaks appear at both ends of this range, consistently across all samples- at 3130  $\text{cm}^{-1}$ , representing  $-\text{CH}_3$  and  $\text{CH}_2$  vibrations, and at 2750  $\text{cm}^{-1}$ , representing aldehyde or aromatic and sulfonate compound vibrations; the second region (1530–1340  $\text{cm}^{-1}$ ) contains two prominent troughs- at 1458.7  $\text{cm}^{-1}$ , indicating ester bonds,  $\text{CH}_2$  deformation and  $\text{C}-\text{O}-\text{C}$  stretching, and at 1376.8  $\text{cm}^{-1}$ , representing carbon bonds of the type ( $-\text{C}-\text{CH}_3$ ),  $-\text{O}-\text{C}-\text{C}$ ,  $-\text{SO}_2$ ,  $-\text{SO}_3$ , and  $\text{CH}_2$  deformation. Since transmittance increases for samples used for ten years, this suggests that vibrations of these bonds become less probable due to various factors.

Two consistent absorption values also appear at the edges of this region: at 1606.3  $\text{cm}^{-1}$ , characteristic of  $\text{N}-\text{N}$  bond vibrations; and, at 1310  $\text{cm}^{-1}$ , intrinsic (per ASTM) to  $-\text{O}-\text{CH}_3$ ,  $\text{CH}_3$ , acid radical,  $\text{SO}_4$  and  $-\text{NH}_2$  vibrations.

Absorption peak group (1000–700  $\text{cm}^{-1}$ ), while not particularly deep, is highly characteristic of electrical insulating oils (transformer oils), as documented in

multiple references. These describe vibrations involving bonds between N, S, P and O elements, which are generally few. Characteristic peaks include 720, 830 and 890  $\text{cm}^{-1}$ , indicative of aromatic structures and acid radicals.

Three minor peaks are influenced by the oil's service time in the transformer: at 3651.2  $\text{cm}^{-1}$ , an absorption peak for  $-\text{OH}$  (carboxylic),  $-\text{NH}$  or sulfur-containing bond vibrations. This peak is notably prominent in the 10-year-old sample taken from the top of the transformer, and is also found in the sample of the same age taken from the bottom; at 1740  $\text{cm}^{-1}$ , corresponding to esters, carboxamides, amides, hydrated sulfonic acid radicals and  $-\text{C}=\text{O}$  bonds. Transmittance depth weakens with increased oil use, indicating reduced absorption corresponding to these bond vibrations; and at 1150  $\text{cm}^{-1}$ , indicative of sulfonate salts,  $-\text{C}-\text{SO}_2-$  bonds,  $\text{P}=\text{O}$  and  $\text{P}=\text{N}$  bonds. Absorption of the new oil is strong compared to the used one.

These last three values are particularly useful for studying the effect of transformer oil's service time on infrared spectral properties. Possible interpretations include reduced vibrations of  $\text{C}-\text{O}-\text{C}$ ,  $-\text{C}=\text{O}$ ,  $\text{P}=\text{N}$ ,  $\text{C}-\text{S}$ ,  $-\text{NH}_2$  and  $-\text{ON}$  bonds, due to their conversion into other compounds, or detachment from the main carbon chains of paraffinic oil compounds.

This explains the decrease in transmittance across the entire studied wavenumber range up to three years of use. However, continued use alters the nature of transformations to reduce absorption, either through precipitation of compounds with high vibrational activity or by their conversion into less vibrationally active compounds.

Subsequently, transmittance increases again until samples used for ten years exhibit higher transmittance compared to the new oil. Moreover, transmittance of oil taken from the bottom of the transformer is higher than that of both new oil and 10-year-old oil taken from the top. This indicates that bonds transformed into other types result in materials with lower density compared to the main oil components.

## Conclusions

The study concluded that: over time, bonds in electrical insulating oil compounds change in two stages: a decrease in infrared permeability within the first three years of use; and an increase in permeability until, after ten years, the permeability of the insulating oil under the transformer exceeds that of new oil. The study concluded that, over time, bonds in electrical insulating oil compounds change in two stages: its infrared permeability decreases within the first three years of use; and then, it increases until, after ten years, it exceeds that of the new oil.

Chemical activity involves  $\text{C}-\text{O}-\text{C}$ ,  $-\text{C}=\text{O}$ ,  $\text{P}=\text{N}$ ,  $\text{C}-\text{S}$ ,  $-\text{NH}_2$  and  $-\text{ON}$  bonds, requiring further spectroscopic studies such as magnetic resonance and chromatography.

The aging of mineral insulating oils is not a linear process, as it occurs in two key phases: initial stabilization or consumption phase followed by a progressive accumulation of advanced degradation products. The observed increase in

infrared absorption after long-term use serves as a critical indicator of advanced chemical aging, potentially signaling a decline in dielectric reliability. Therefore, regular monitoring of these chemical linkages through FT-IR spectroscopy, supported periodically by Nuclear Magnetic Resonance and chromatography, is recommended for predictive maintenance and assessment of the remaining useful life of transformer oil. This integrated approach allows for a more accurate assessment of the oil condition beyond traditional electrical tests, facilitating timely reclamation or replacement decisions to ensure grid reliability.

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### **Authors' contributions**

**Mohammed Elayoubi and Sinan Aljalali:** performed analysis. **Mothana Aljassem:** wrote the paper and collected data.

### **Abbreviations**

**AC:** alternating current

**ASTM:** American Society for Testing and Materials

**BDV:** breakdown voltage

**FT-IR:** Fourier transform infrared spectroscopy.

**IEC:** International Electrotechnical Commission

**KOH:** potassium hydroxide

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