Effect of Ionizing Radiation on the Physical Characteristic of Mineral Insulating Oil

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Abstract
Understanding how ionizing radiation affects insulating materials is essential for maintaining the reliability and safety of high-voltage systems operating in radiation-prone environments that use mineral insulating oil. In this research, mineral insulating oil was irradiated with gamma rays from a cobalt source of Co60, with a radioactivity of 70 kcl. The irradiation process was carried out at a dose rate of 8.9 kGy/h, and doses up to 250 kGy were used. This study focused on density factor. Other parameters such as moisture content, acidity and breakdown voltage were measured according to internationally recognized standards. Fourier transform infrared spectroscopy (FTIR) was also used to detect changes in oil's chemical composition. Results indicated that gamma radiation caused a decrease in oil density, with the lowest value of 0.853 mg/cm^3. These changes were effectively modeled using polynomial regression, with a high correlation coefficient (R^2) of 0.982, demonstrating the model's reliability in capturing the relationship between radiation dose and density change.

Keywords: dielectric; density; gamma irradiation; mineral oil.

Introduction•

Mineral insulating oils are among the most widely used insulating fluids due to several factors, the most important of which are their economic cost and low price, in addition to other properties such as insulating durability. However, synthetic and natural esters are increasingly being chosen because of their biodegradability and better aging behavior than mineral oils [1, 2]. Exposure to electric fields is one of the most important types of stress in electrical equipment, since liquid insulating materials are used for insulation and cooling in transformers. Due to high electrical stress, partial discharges (PD) may occur in

•The abbreviations list is in page 331.

liquid insulating materials, potentially causing complete breakdown of the transformer's insulation system [3, 4]. The study of properties of insulating oils includes examining physical, chemical and electrical properties of the insulating fluid to estimate the oil's ability to withstand thermal stresses that occur during operation. Evaluation of oil condition through approved standard oil tests (SOT) is the traditional method, where measurement results are compared with specified values, mentioned in IEC 60422 standard [5]. Radiation is basically defined as emission or transfer of energy in form of waves or particles through space or a material medium. Electromagnetic radiation consists of radio waves, microwaves, infrared radiation, visible light, ultraviolet radiation, X-rays and gamma (γ) radiation. Particle radiation exists in the form of alpha (α), beta (β), proton and neutron radiations [6]. In addition to the use of nuclear energy to provide electricity, radioactivity has numerous applications in various fields. Therefore, it is important to study effects of exposure to gamma rays on electrical, physical and chemical properties of materials. Gamma irradiation is a well-established technique for enhancing electrical conductivity in polymer components [7]. The significance of researching these challenges became particularly evident following 1986 Chernobyl nuclear disaster, after which several incidents exceeding level 4 on International Nuclear Events Scale (INES) were recorded [8]. Previous researchers have studied both removal from Polychlorinated biphenyls using gamma radiation and properties of insulating oil after irradiation, with absorbed doses in the range from 29.7 to 237.6 kGy, and dose rates of 0.21 Gy/s [3, 4, 9]. Overall, studying the impact of ionizing radiation on mineral insulating oil is vital for enhancing reliability, safety and environmental integrity of electrical systems, particularly in radiation-exposed environments. This supports ongoing advancement of technology and materials used in energy sector and beyond.

Materials and methods

Materials

Experiments within the study employed a sample of used transformer insulation oil (China Kelamayi 45), which was sourced from a transformer operating within the range from 0.4 to 20 kV, being commonly used in Syrian power distribution networks.

Characterization electrical, chemical and physical properties

Mineral oil samples were tested in terms of their electrical, chemical and physical properties, including breakdown voltage, acidity, density, viscosity and moisture content, in accordance with standardized methods outlined by IEC, ASTM and ISO. 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. Relative density of oil is the weight of equal volumes of oil and water, tested at a temperature of 15 °C. The test was carried out according to ISO 3675 [11]. Acidity was experimentally measured according to IEC 62021 [12]. 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 occured 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 [13]. 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. Properties of studied sample are listed in Table 1.

Table 1: Properties of studied sample.

Standard	Value	Property
ASTM D6304-20	0.927	Water content
ISO 3675	0.869	Density
IEC6 2021	0.0561	Acidity
IEC60156	36	Breakdown voltage

FTIR analysis

IR absorption positions are generally presented as either wavenumbers (ν) or wavelengths (λ). 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:

$$\mathrm{v}(Cm^{-1}) = \frac{1}{\lambda(\mu m)} \times 10^{-4} \tag{1}$$

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

$$\mathrm{A} = log_{10}\frac{1}{T} = -log_{10}\mathrm{T} = -log_{10}\frac{I}{I_O} \tag{2}$$

where T is transmittance, I is the sample, Io 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 vary from infinity to zero [14-15]. In this study, analyzes were performed by placing 1 mL of the sample on a JASCO 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, and determining its structure and chemical composition. FTIR of the studied sample before irradiation is shown in Fig. 1.

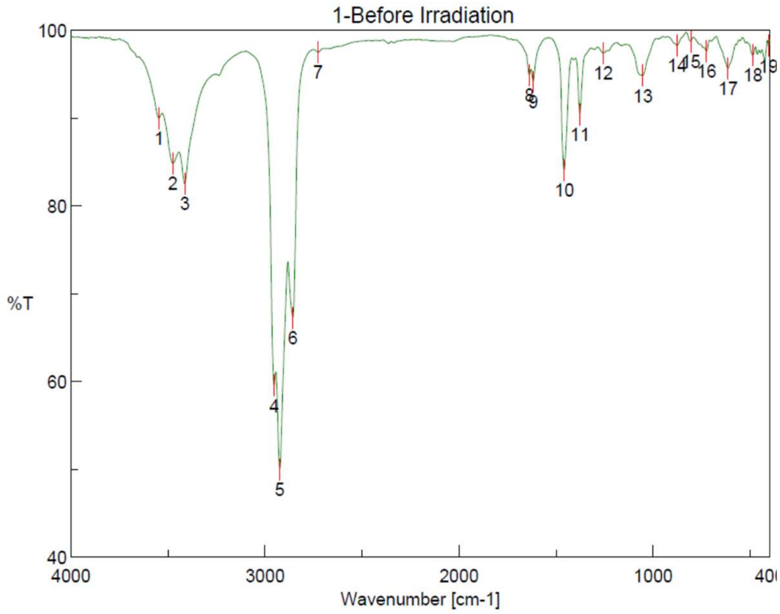

Figure 1: FTIR of studied sample before irradiation.

Gamma irradiation

Irradiation was conducted using a Russian-made ROBO irradiation station using a Co^{60} radioactive source with a radioactivity of 70 kcl. The sample was irradiated by placing it 10 cm from the source holder at a dose rate of 8.9 kGy /h. The source holder consists of cylindrical tubes with diameter of 12 mm and length of 1 m, within which cobalt radioactive sources are placed. Therefore, the source can be considered approximately rectangular in shape, and the law for calculating the abovementioned absorbed dose does not apply to it. Thus, the experimental method was used to calculate the absorbed dose rate, and calibration was carried out using chemical dose meters such as chlorobenzene ethanol, which are approved according to international standard ISO/ASTM: 51538.2017. Samples were placed in sealed polyethylene bags, where the sample took the shape of a rectangular parallelepiped (2*10*10 cm) at room temperature, and in air, at a dose rate of 8.9 kGy /h, with successive doses of 50, 100, 150, 200 and 250 kGy.

Polynomial regression

Regression analysis is a statistical method used to model dependence between variables. In the context of forecasting, it estimates the target variable based on known input parameters. Its advantages include ease of implementation and interpretation of results. Polynomial regression is a form of regression analysis in

which the relationship between independent and dependent variables is modeled using a polynomial of nth degree. The result of polynomial regression is a non-linear function. Although it could be said that this is a non-linear regression, polynomial regression is a special type of multiple linear regression, because multiple independent variables are used to estimate regression parameters. Regression coefficients are generally computed similarly as in linear regression, using the method of least squares [16-19].
In general, a polynomial regression is represented by the following equation:

$$Y = \beta_0 + \beta_1 X + \beta_2 X^2 + \ldots + \beta_p X^p + \varepsilon \qquad (3)$$

where Y is predicted variable, X and X^p are multiples of independent variables (β_0), β_1 and β_p are regression coefficients and ε is error. Coefficient of determination, commonly referred to as R-squared (R^2) or residual sum of squares, represents squared correlation between predicted values generated by a regression model and observed experimental values. R^2 value of 0 indicates that regression model has no predictive power over experimental data, while that of 1 signifies a perfect fit, meaning the model predicts experimental values without error. When R^2 falls between 0 and 1, it implies that the model can explain only a certain proportion of variability in experimental data [20,21]. This coefficient was computed using the following equation:

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(y_i - y_i^{\wedge})^2}{\sum_{i=1}^{n}(y_i - y_i^{-})^2} \qquad (4)$$

where y^1 is experimental values of each electrical property, $y_i^{\wedge}$ is correlated values obtained by regression analysis for each electrical property and y_i^{-} is the mean of six test values for each point. R^2 was 0.982.

Results and discussion

Fig. 2 shows variations in density under applied gamma doses.

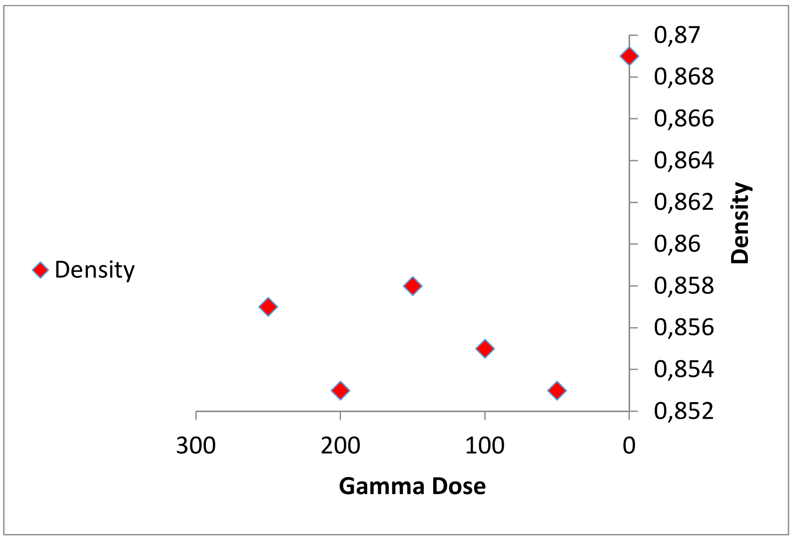

Figure 2: Variations of density under applied gamma doses.

FTIR of the studied sample after irradiation with 50 kGy is shown in Fig. 3.

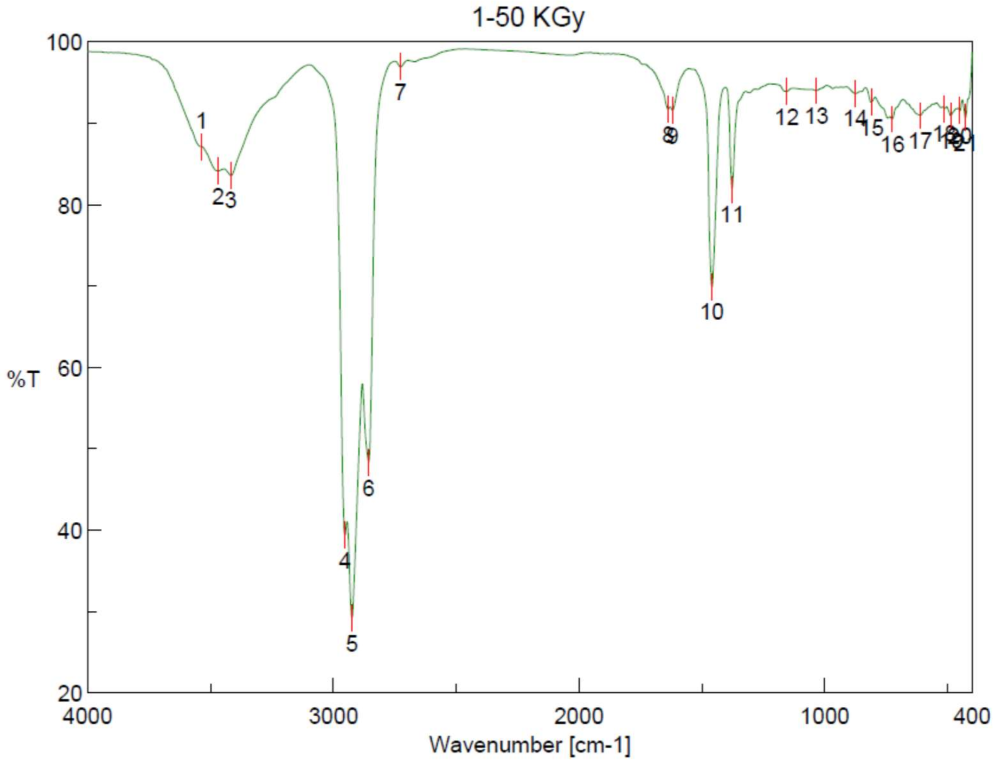

Figure 3: FTIR of studied sample with 50 kGy gamma dose.

FTIR of the studied sample after irradiation with 100 kGy is shown in Fig. 4.

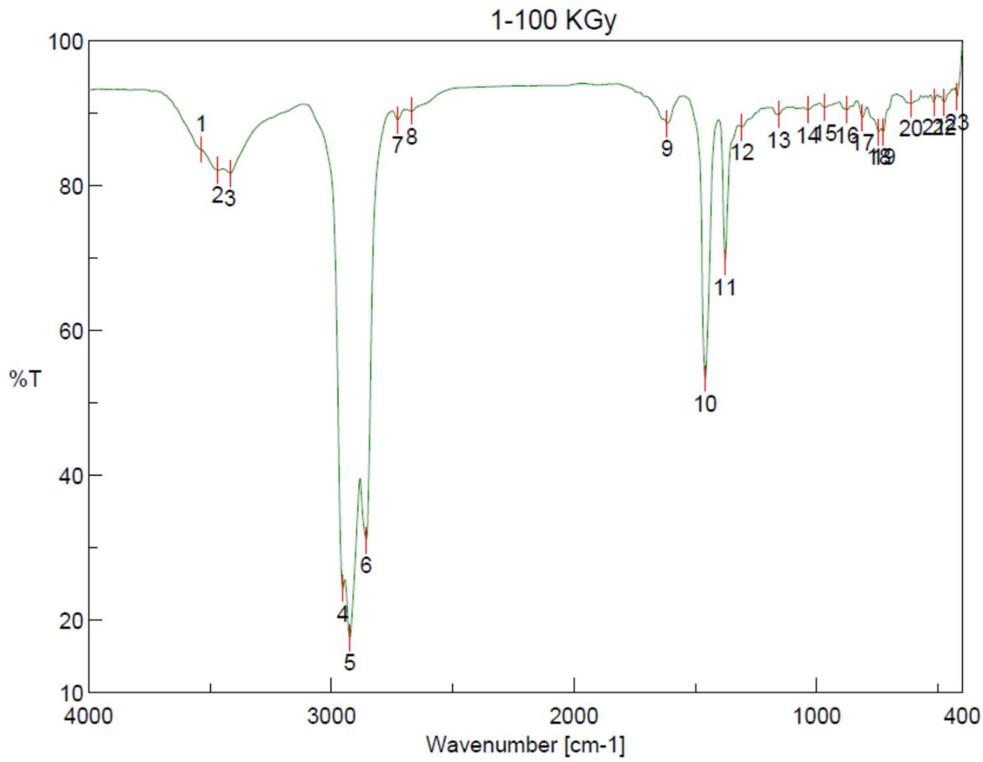

Figure 4: FTIR of the studied sample with 100 kGy gamma dose.

FTIR of the studied sample after irradiation with 150 kGy is shown in Fig. 5.

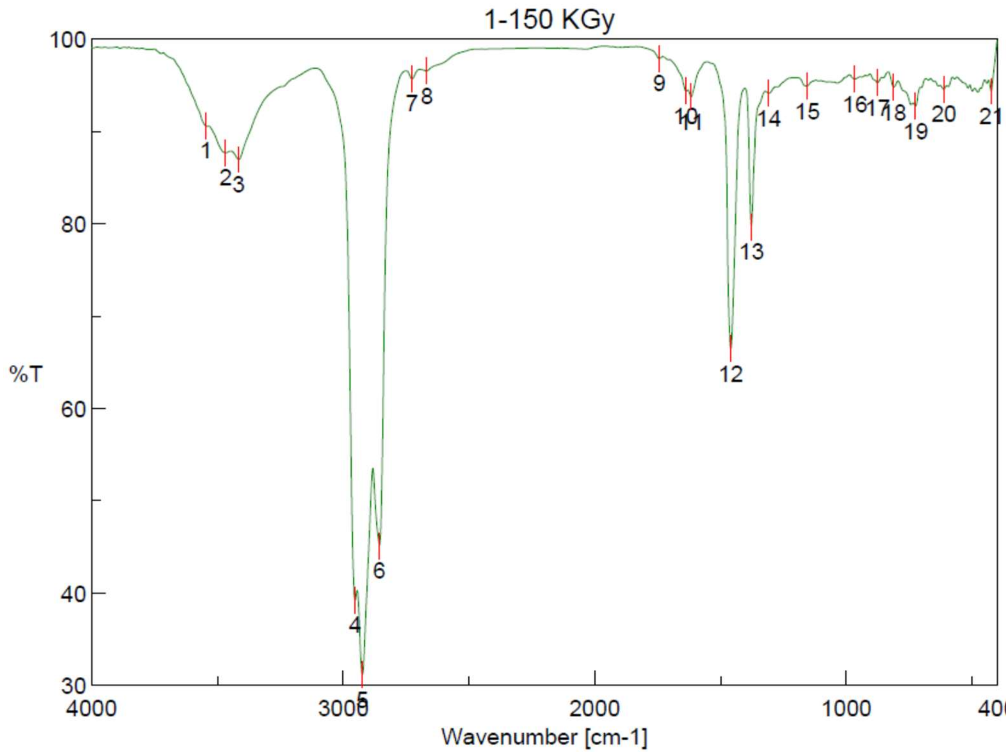

Figure 5: FTIR of the studied sample with 150 kGy gamma dose.

FTIR of the studied sample after irradiation with 200 kGy is shown in Fig. 6.

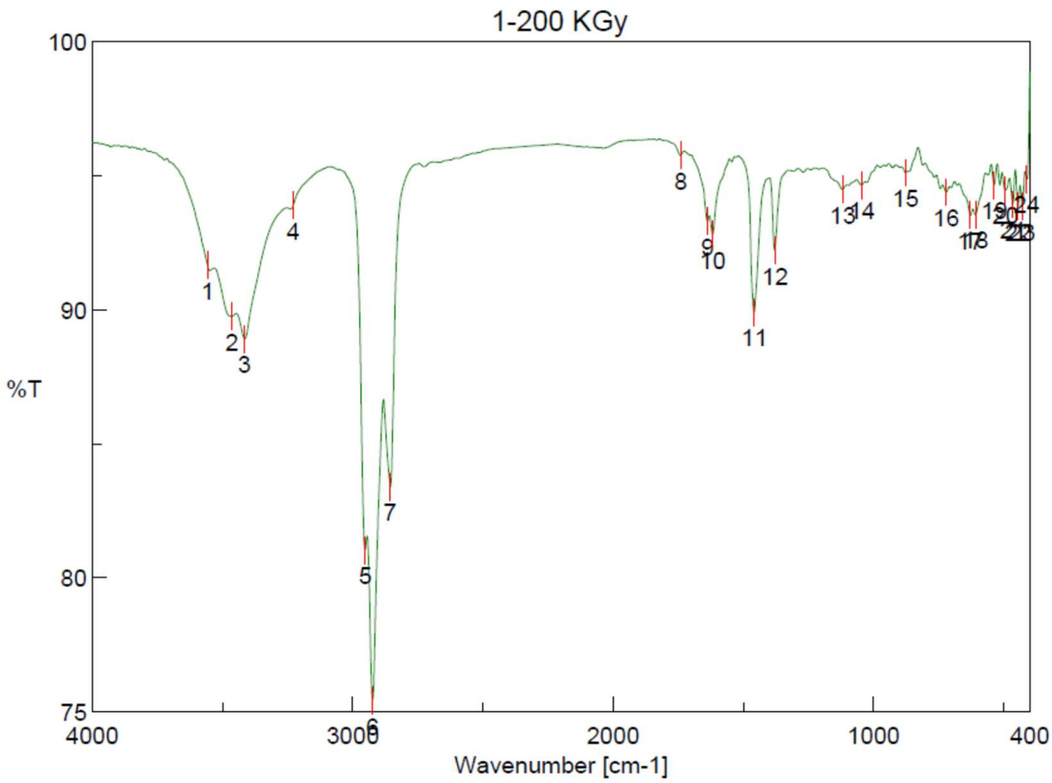

Figure 6: FTIR of the studied sample with 200 kGy gamma dose.

FTIR of the studied sample after irradiation with 250 kGy is shown in Fig. 7.

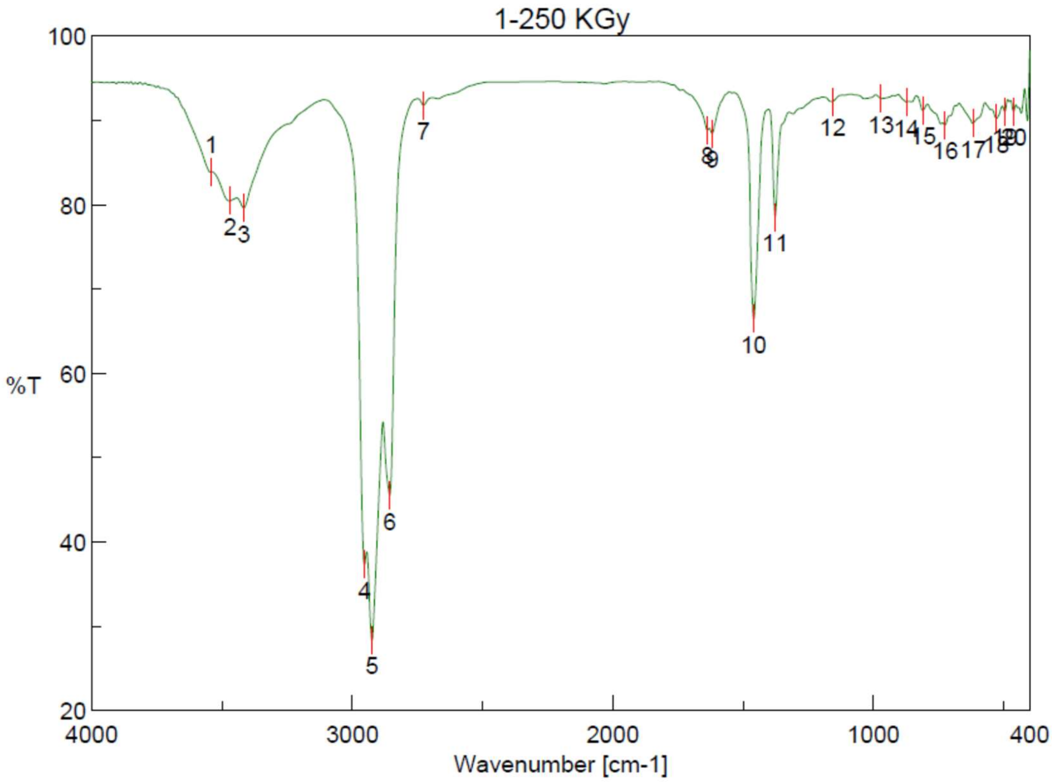

Figure 7: FTIR of studied sample with 250 kGy gamma dose.

Before irradiation

The sample recorded a moisture content of 0.927%, which is relatively high compared to internationally recognized standard (ASTM D6304-20).

The appearance of distinct peaks at a wavelength of 3546.45 cm^{-1}, with an intensity (%T) of 90.0, indicated the presence of free moisture (O-H). The appearance of distinct peaks at a wavelength of 3413.39 cm^{-1}, with an intensity (%T) of 82.6, indicated the presence of bound moisture (H-bonded).

Measured acidity of 0.0561 mgKOH/g is low compared to internationally recognized standard (IEC62021), demonstrating that oxidation was not progressing. The presence of a peak at 1637 cm^{-1} (C=C) (C=O) and another at 1257 cm^{-1} (C-O) indicated the onset of oxidation processes. The absence of a peak (1700 cm^{-1}), which shows the presence of carboxylic acids, confirms that oxidation was in its early stages and not advanced.

Acidity decreased in the early stages of oxidation, as intermediates (such as ketones or ethers) accumulate before acids are formed. This is consistent with spectroscopic and laboratory results.

Peaks of 2923 and 2856 cm^{-1} for C-H stretching in CH2/CH3 are typical of non-oxidizing mineral oils. Peaks below 1000 cm^{-1} show hydrocarbon ring vibrations, supporting the traditional insulating oil structure.

Density of 0.869 g/mL is within the normal range for insulating oils (ISO 3675), enhancing the stability of hydrocarbon structure. Breakdown stress was 36 kV, which was within acceptable range according to IEC 60156 standard. This low value was caused by high humidity (as in experimental measurement) which may reduce breakdown stress over time due to the formation of conductive impurities.
In comparison with approved standards for spectroscopic analysis (ASTM D2144) [22], for C-H stretching, peak position of 2923.56 cm^{-1} was within normal range (2950-2850 cm^{-1}), indicating that the hydrocarbon structure conformed to the standard. For water content, peaks at 3413-3546 cm^{-1} indicate a negative deviation from the expected standard (<3300 cm^{-1}) which is weak, confirming the presence of excess moisture. The appearance of a 1637 cm^{-1} (C=O) peak versus its absence in fresh samples supports the hypothesis of early oxidation.

After irradiation

After irradiation, it is observed that, with a decrease in density with increased doses, a shift occurred in O-H bond vibrations towards lower wave numbers (shift towards longer wavelengths). Additionally, oxidation reactions and chemical decomposition of oil-forming molecules occurred, which led to the formation of oxygen compounds. In fact, compounds belonging to C-O group appeared at a wavelength of 1155 cm^{-1}, which indicates the presence of carboxylic acids. Also, at a wavelength of 3469 cm^{-1}, compounds belonging to hydroxyl group O-H indicate the presence of alcohols or moisture. On the other hand, as a result of irradiation process, compounds containing ketone or aldehyde groups were formed, belonging to carbonyl group that appeared after irradiation at a wavelength of 1730-1739 cm^{-1}. This group was not visible before irradiation. These results are due to gamma rays having a high energy able to penetrate materials and cause changes in atomic structure. This led to disintegration of oil molecules and formation of new ones. Gamma rays broke molecular structures of the oils studied, resulting in production of lighter molecules, which explains lower density value.

Conclusions

This study systematically investigated the influence of Gamma irradiation, at doses up to 250 kGy, on physical and chemical properties of insulating oils, with a particular focus on density variation. Characterization of oil properties was conducted in accordance with established international standards: physical properties were assessed by density measurements following ISO3675; chemical properties, as moisture content and acidity, were evaluated using ASTM D6304-20, IEC62021; electrical properties were investigated by breakdown voltage testing based on IEC60156. Furthermore, FT-IR spectroscopy was employed before and after irradiation to assess potential modifications in molecular structure.

Experimental results demonstrated that Gamma irradiation induced molecular fragmentation within the oil, leading to formation of lighter hydrocarbon chains. This structural alteration manifests as a measurable decrease in oil density. Notably, despite the observed reduction, all post-irradiation density values remained within permissible range defined by ISO3675, which specifies a maximum limit of 0.895 g/cm^3.
A robust non-linear relationship between radiation dose and density reduction was established, with a correlation coefficient of R^2 = 0.982, indicating strong predictive capability of the applied model polynomial regression. These findings offer valuable insights for the development of radiation-tolerant insulating materials, and support implementation of predictive maintenance strategies in high-voltage equipment operating under radiation exposure. Consequently, this work contributes to enhancing operational reliability and longevity of electrical systems in radiation-intensive environments.

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Authors' contributions
Mothana Aljassem: performed analysis, wrote the paper. **Ali Alsayed**: collected data. **Ahmad Falah**: analysis tools.

Abbreviations
ASTM: American Society for Testing and Materials
FTIR: Fourier transform infrared spectroscopy.
IEC: International Electrotechnical Commission
INES: International Nuclear Events Scale
ISO: International Organization for Standardization
KOH: potassium hydroxide
PCB: Polychlorinated biphenyls
PD: partial discharges
SOT: standard oil tests

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