Dissolved Gas Analysis of Methyl Ester-Mineral Oil Mixture Under Partial Discharge

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Abstract **– This paper deals with the dissolved gas analysis (DGA) of methyl ester and mineral oil mixtures under partial discharge (PD). Experiments were conducted on pure methyl ester and mixtures containing 5%, 7.5%, and 10% mineral oil to simulate transformer retrofilling conditions. PD was generated using a needle-plane electrode pair immersed in the oil sample and subjected to AC high voltage. The gases produced were analyzed using gas chromatography to evaluate gassing tendencies and fault diagnostics using the Key Gas, Duval Triangle, Duval Pentagon, and IEC 599 ratio methods. Results indicate that residual mineral oil reduces the partial discharge inception voltage (PDIV) and increases gas production. Hydrogen, constituting approximately 85% of total gas production, indicates the applicability of the Key Gas method. The Duval Triangle method consistently identified PD faults, whereas the Duval Pentagon method underestimated PD as stray gassing. In contrast, the IEC 599 method was ineffective for fault diagnosis in methyl ester-based oils under PD. These findings suggest that up to 10% residual mineral oil in a retrofilling scenario does not significantly affect DGA-based fault diagnostics in methyl ester but enhances gas production due to increased PD activity.**

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1. Introduction

Dissolved gas analysis (DGA) is a well-recognized technique engineers and technicians use in condition monitoring and diagnostics of mineral oil immersed-power transformers. DGA involves the measurement and analysis of gases present in transformer oil, which can provide valuable information about the internal condition of the transformer. Analyzing the dissolved gases in transformer oil is a cost-effective and reliable way to diagnose problems, such as thermal or electrical faults, before they escalate and cause catastrophic failure [1], [2].

Recently, interest in using natural esters as alternative insulating fluids has steadily increased due to their biodegradability, low toxicity, and renewability [3], [4]. Its application mainly applies to distribution transformers and slightly less to power transformers [5]. Although it has deficiencies in high viscosity, this oil has other advantages in terms of high flame points and its compatibility with paper isolation which is also found in the transformer [6], [7]. Its water affinity causes the isolation paper in a transformer containing natural ester to dry out, reducing the rate of paper degradation triggered by moisture. Paper insulation aged thermally in natural ester extends its life expectancy to 5-8 times longer than in mineral oil [8], [9], [10].

Numerous studies have examined DGA in natural esters, revealing that while both natural esters and mineral oil generate the same gases, their gas profiles differ. Natural esters exhibit high levels of ethane and carbon oxides, but less methane than mineral oil, especially under low-temperature thermal faults [5], [11], [12], [13]. The ethane generation phenomenon applied mainly to natural esters having C=C bonds in their hydrocarbon chains [13].

It is important to mention that the natural esters in the tri-ester (triglyceride) structure inherently contain a hydrocarbon with a C=C bond [14], [15]. At elevated temperatures of thermal faults and under electrical faults, the gas profiles of both oils become more similar [11], [12], [16]. Generally, the DGA identification method, developed for mineral oil, needs adjustment before being used for natural esters [12], [16], [17], [18].

In addition to using a new natural ester-filled transformer, the oils are also implemented in retrofilling a mineral oil-filled transformer [19], [20]. The first distribution and power transformers were retrofilled by the natural ester in 1997 and 2001, respectively [21]. During the retrofilling process, about 4-7 % of the used mineral oil remains inside the transformer's tank, resulting in a mixture between the new natural ester and used mineral oil [9], [15], [22]. The effect of a relatively small amount of the residual used mineral oil on the DGA of natural ester needs investigation. This study examines the DGA techniques for methyl ester mixed with 5, 7.5, and 10% mineral oil, for simulating transformer retrofilling conditions, under PD. Fault identification techniques such as the Key Gas, IEC 599, Duval Triangle, and Duval Pentagon are evaluated. The DGA of pure methyl ester is also tested for comparison.

2. Methodology

To examine DGA techniques for methyl ester mixed with residual mineral oil in a transformer retrofilling scenario, a series of experiments - including sample preparation, PD tests, and gas analysis - were conducted. The experimental procedures are detailed below.

2.1. Sample Preparation

The samples utilized in this experiment were methyl ester and mineral oil. To simulate retrofilling conditions, the new methyl ester was combined with a small amount of used mineral oil for the oil mixture. Pure methyl ester oil was also investigated for comparison.

Three kinds of mixture samples were prepared, namely, the oil mixture containing 95.0% methyl ester and 5.0% mineral oil (ME95-MO05), the mix between 92.5% methyl ester and 7.5% mineral oil (ME92.5-MO7.5), and the mixture between 90% methyl ester and 10% mineral oil (ME90-MO10). The oil mixture was agitated with a magnetic stirrer at 250 rpm for approximately 1 hour before being exposed to PD stress.

2.2. PD Test

The experimental arrangement for the PD test is illustrated in Figure 1 [23]. PD was induced using a needle-plane electrode setup submerged in the oil sample. PD takes place when the local electric field exceeds a specific threshold, causing a partial breakdown of the surrounding insulating material. Employing a needle-plane electrode setup can generate a strong electric field locally [23]. Oil treatment processing is shown in Figure 2. The oil chamber and the electrode configuration are depicted in Figures 2a and 2b. The gap between the two electrode pairs is 10 mm. An acrylic with a thickness of 5 mm was put above the plane electrode to avoid damage in case a large current flow through the electrode pairs. The PD signal was detected by an RC detector equipped with a high-pass filter connected to an oscilloscope.

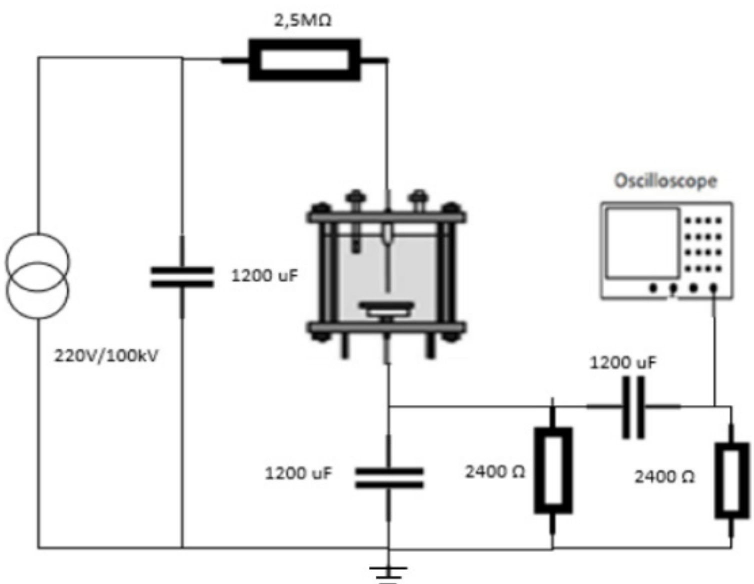


Figure 1. Experimental setup for PD test

The electrode was connected to an AC high-voltage source. The voltage was gradually increased until PD occurred. The voltage at which the first PD appears is known as the PD Inception Voltage (PDIV). After PD was initiated, the voltage was raised to 2 kV above the PDIV to perform the PD test. The following sample of the same mixture proportion was stressed by another 2 kV increase in the applied voltage. This procedure was repeated for other oil mixtures with different ratios between methyl ester and mineral oil.

2.3. Gas Analysis

The headspace method was employed to isolate the gases from the oil, using a syringe as depicted in Figure 2c. The detailed gas extraction procedure can be found in [24]. In short, a space was created above the oil inside the syringe. The syringe was shaken, and the extracted gasses were accumulated in the space before being injected into the gas chromatograph for gas analysis.

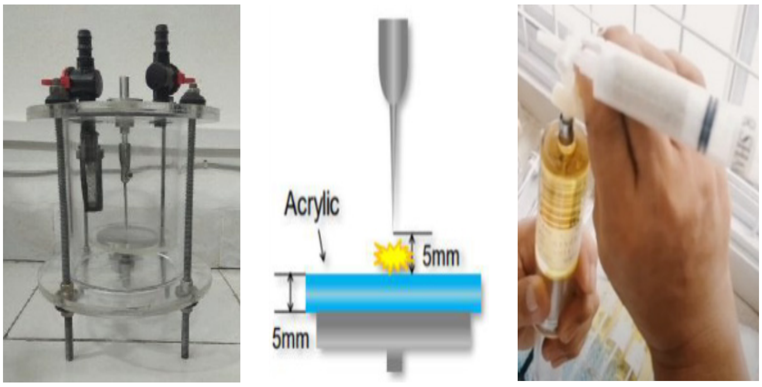


Figure 2. Oil treatment; (a) oil chamber, (b) electrode configuration for PD generation, and (c) syringe for gas-in-oil extraction.

3. Results and Discussion

By following the procedure mentioned in 2.2, it is found that the PDIV of the pure methyl ester and the oil mixture are 18 and 15 kV, respectively. Then, the PD test was conducted for pure methyl ester at the applied voltages of 20 and 22 kV, whereas those for the oil mixtures were at the applied voltages of 17 and 19 kV. It should be noted that the mineral oil used in the experiment was taken from a transformer that has operated for more than 30 years. The oil should have impurities from the oil and the solid insulation degradations during the service period. It is thus understandable that the PDIV of the oil mixture is considerably less than that of pure methyl ester. The decrease in the PDIV due to impurities in the oil is confirmed by the literature [25].

3.1. Gas Pattern

Table 1 shows the gas generation in pure methyl ester and oil mixture samples containing mineral oil of 5.0, 7.5, and 10.0%, respectively. All cases offer similar gas production patterns. The gas concentration is higher at the higher applied voltage. Gas production in oil mixtures is higher than in the pure methyl ester, although the PD tests were conducted at the lower applied voltage to the oil mixture than to the pure methyl ester. An increase in the applied voltage results in a modest rise in gas concentration.

Moreover, a greater proportion of mineral oil in the oil mixture correlates with an increased concentration of gases. Hydrogen (H_2) is the most prevalent gas observed, followed by methane (CH_4), carbon monoxide (CO), ethane (C_2H_6), and ethylene (C_2H_4). Acetylene (C_2H_2) was not observed in all oil samples. The higher generation of hydrogen and methane in natural and synthetic esters under PD is also reported by [26], [27].

Table 1. Gas production by PD in the pure methyl ester and methyl ester-mineral oil mixtures

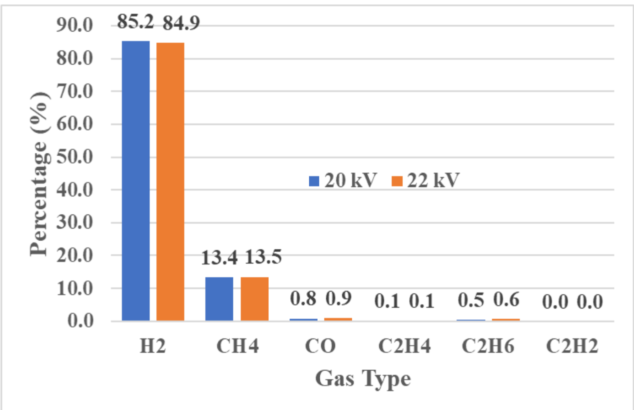
Gas Type	Gas Concentration in Oil (ppm)							
	Pure Methyl Ester		ME95 – MO05		ME92.5 – MO7.5		ME90 – MO10	
	20 kV	22 kV	17 kV	19 kV	17 kV	19 kV	17 kV	19 kV
H_2	835	837	910	923	979	988	1015	1037
CH_4	131	133	145	147	158	159	160	164
CO	8	9	7	8	8.7	8.9	10	10
C_2H_4	1	1.46	1.7	2	1.5	2	2	2.2
C_2H_6	5	5.7	4	5	4	4	5	5.7
C_2H_2	0	0	0	0	0	0	0	0

3.2. Fault Identification

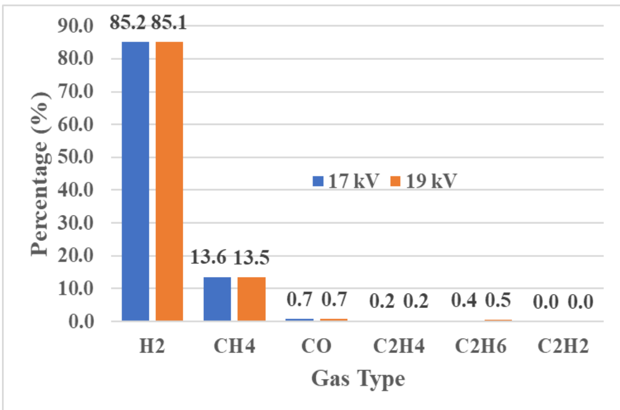
Four types of fault identification methods are evaluated based on the DGA data. The aim is to assess the effect of the different concentrations of mineral oil in the methyl ester-mineral oil mixture on the fault identification results. The methods are the Key Gas, the Duval Triangle, the Duval Pentagon methods, and the IEC 599 Ratio.

3.2.1. Key Gas Method

The Key Gas method for detecting faults involves a specific gas that is highly concentrated among other gases. When the acetylene concentration is 30% or higher, it indicates a fault caused by arcing. Hydrogen, on the other hand, is generated by PD and is typically present at concentrations of 85% or more. If the ethylene concentration exceeds 63%, accompanied by about 20% hydrogen, the fault will be attributed to oil overheating [28]. Carbon monoxide, with a concentration of up to 92%, is linked to overheating cellulose [29].



(a)



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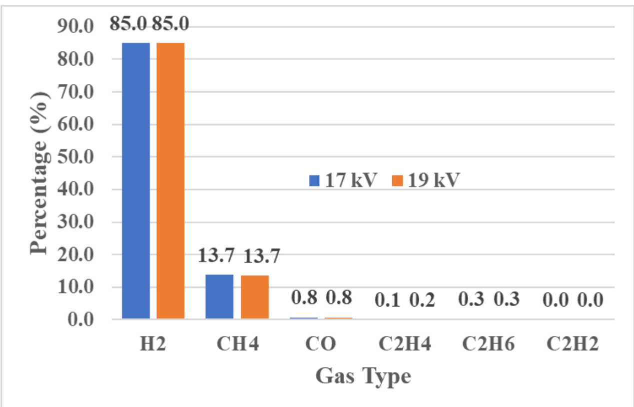

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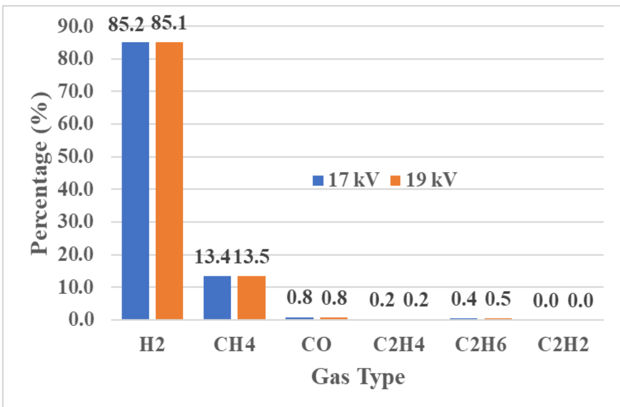


(d)

Figure 3. Proportion of a particular gas to the total combustible gases in oil samples: (a) pure methyl ester, (b) ME95-MO05, (c) ME92.5-MO7.5, (d) ME90-MO10

Figure 3 shows the proportion of a particular gas to the total combustible gases in all oil samples. Figure 3a represents the pure methyl ester sample, whereas Figures 3b, 3c, and 3d depict the mixtures between methyl ester and mineral oil samples at different compositions, namely, ME95-MO05, ME92.5-MO7.5, and ME90-MO10, respectively. It is evident in all Figures that hydrogen constitutes the most dominant gas with a percentage of 85% or larger, suggesting the relevance of this key gas to the methyl ester and methyl ester mixed by the used mineral oil up to the percentage of 10%. Methane was the second largest gas, with a percentage of about 13%. It is also clear that as the applied voltage increases, the percentage of hydrogen decreases, while the percentages of methane and ethane rise. The similar gassing tendencies are reported in [26].

3.2.2. Duval Triangle Method

The Duval Triangle method relies on three types of gases - ethylene (C_2H_4), acetylene (C_2H_2), and methane (CH_4) - to identify faults. Each gas's concentration as a percentage of the total gaseous concentration is calculated and plotted on one of the triangle's three sides (the Duval Triangle). Graphical interpretation is obtained by inputting the results of the three plotted lines at their intersection point, resulting in various fault identifications.

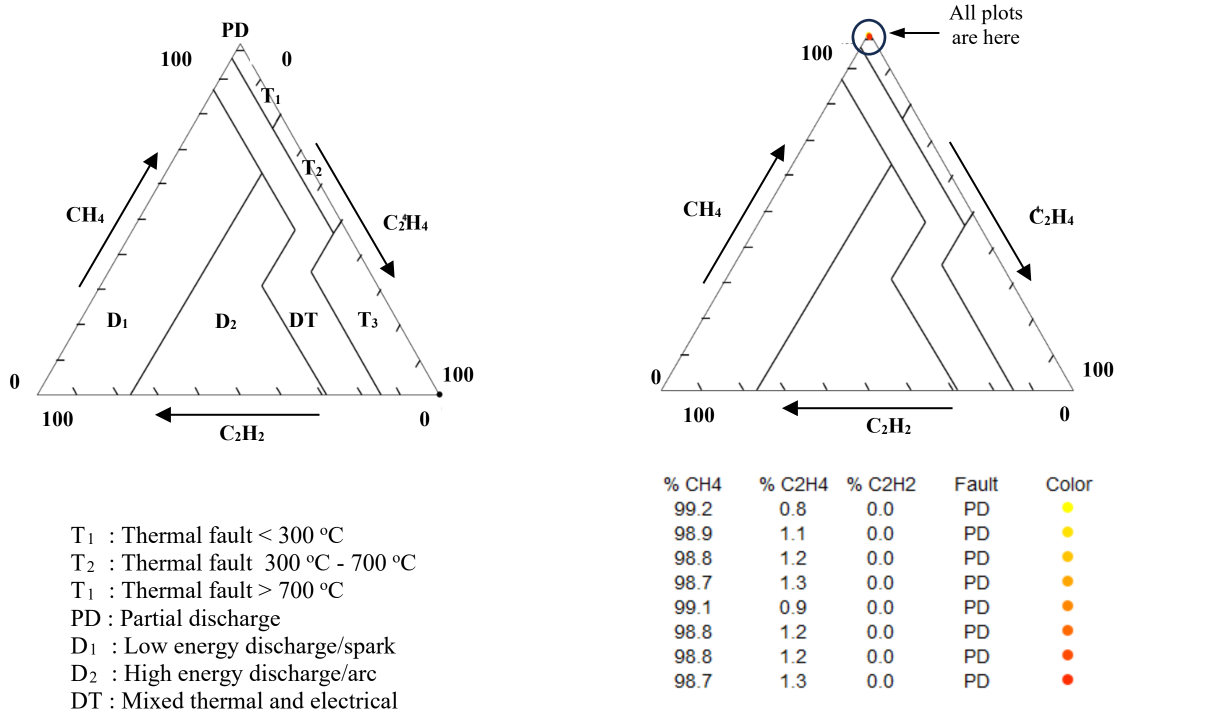
These identifications include thermal faults, with temperatures below 300 ºC (T_1), between 300 and 700 ºC (T_2), or above 700 ºC (T_3); electrical faults, with partial discharge (PD), discharge of low energy (D_1), or discharge of high energy (D_2); or a combination of electrical and thermal faults (DT) (Figure 4a) [30].

Table 2 shows the proportion of a particular gas to the total Duval Triangle's gases in pure methyl ester oil at two applied voltage levels. At 20 kV, for instance, the proportions of ethylene (C_2H_4), acetylene (C_2H_2), and methane (CH_4) are 1.0, 0.0, and 131 ppm, respectively, and the corresponding percentages of each gas are 0.76, 0, and 99.24%, respectively. The resulting plot is located inside the small triangle area at the top of the Duval Triangle (Figure 4b). Thus, the Duval Triangle method makes a correct fault identification.

Table 2. Proportion of a particular gas of pure methyl ester (an example)

Gas Type	Proportion at (%)	
	20 kV	**22 kV**
C_2H_4	0.76	1.09
C_2H_2	0.00	0.00
CH_4	99.24	98.91

A similar procedure is conducted for the oil mixtures, and the resulting plots lay in the same area of the PD fault (Figure 4b). The presence of up to 10% used mineral oil does not deviate from Duval Triangle's identification of the PD fault in the methyl ester.



(a) (b)

Figure 4. Duval Triangle: (a) each area in the triangle represents a specific fault type, (b) resulted plots of all cases

3.2.3. Duval Pentagon

The method of Duval Pentagon involves the use of two types of pentagons to interpret faults with five different gases: ethane (C_2H_6), ethylene (C_2H_4), acetylene (C_2H_2), methane (CH_4), and hydrogen (H_2). The first pentagon (Figure 5a) plots each gas's percentage concentration compared to the five gases' total concentration. The acetylene and ethylene axes are angled at 18° and -54° relative to the positive x-axis, respectively, while the ethane and methane axes are positioned at -18° and 54° relative to the negative x-axis, respectively. The hydrogen axis, however, is parallel to the y-axis. The two-dimensional Cartesian coordinate of each gas in Pentagon 1 is determined, and the coordinates of all gasses are used to calculate the corresponding centroid's coordinate in Pentagon 2 for fault identification. The calculation of the centroid coordinate is detailed in [31]. The mark "S" in the figure refers to stray gases, whereas other marks are similar to those in the Duval Triangle.

For instance, the methyl ester sample subjected to the applied voltage of 20 kV contains methane of 131 ppm. Its relative percentage and Cartesian coordinate are 13.48 % and (-7.92, -10.90), respectively. Other gasses are calculated similarly and are tabulated in Table 3. The coordinate of the resulting centroid is (-0.58, 23.58), plotted in the Pentagon 2 in the region S (Figure 5b). Other cases are represented similarly, and the coordinates of their centroids are listed in Table 4. All centroids are placed in the S region in the Pentagon 2, very close to the PD area.

Hence, the Pentagon methods slightly underestimate the PD fault in methyl ester and oil mixtures as stray gassing (a phenomenon at normal operation conditions, but the oil generates a significant amount of gasses).

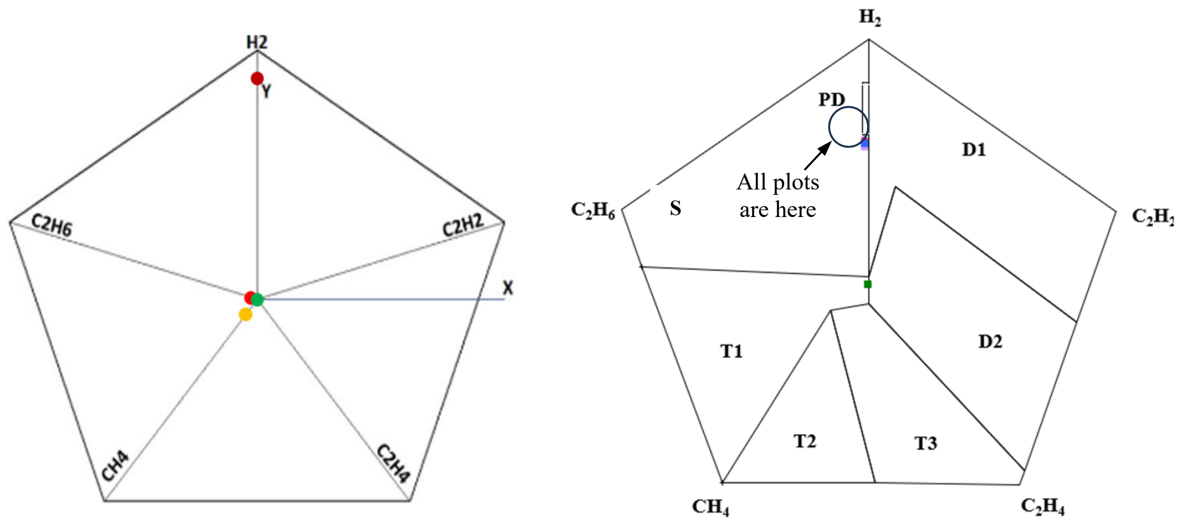
Hence, the presence of mineral oil up to 10% does not alter the Duval Pentagon's prediction on the PD fault in methyl ester oil.

Table 3. Coordinate of gases in pure methyl ester and its centroid at 20 kV

GAS	**Concentration**		**Coordinate**	
	ppm	**%**	**X**	**Y**
H_2	835	85.91	0.00	85.91
C_2H_6	5	0.51	-0.49	0.16
CH_4	131	13.48	-7.92	-10.90
C_2H_4	1	0.10	0.06	-0.08
C_2H_2	0	0.00	0.00	0.00
CENTROID			-0.58	23.57

Table 4. Centroid coordinates and predicted fault in oil samples

Sample, Applied Voltage (kV)	**Coordinate**		**Predicted Fault**
	X	**Y**	
Pure Methyl Ester, 20 kV	-0.58	23.57	S
Pure Methyl Ester, 22 kV	-0.62	23.25	S
ME95-MO05, 17 kV	-0.61	22.66	S
ME95-MO05, 19 kV	-0.63	22.73	S
ME92.5-MO7.5, 17 kV	-0.60	22.73	S
ME92.5-MO7.5, 19 kV	-0.63	22.32	S
ME90-MO10, 17 kV	-0.60	22.83	S
ME90-MO10, 19 kV	-0.62	22.84	S



(a) (b)

Figure 5. Duval Pentagon; (a) Duval Pentagon 1, dots represent the percentage of each gas in the pure methyl ester under the PD at 20 kV; (b) Duval Pentagon 2, dots represent the resulted centroids of all samples.

3.2.4. IEC 599 Ratio

Identifying a fault using the IEC 559 method involves three gas ratios, namely, C_2H_2/C_2H_4, CH_4/H_2, and C_2H_2/C_2H_4 [32]. A predetermined set of limits for each gas ratio shown in Table 5 is utilized to estimate the type of fault. In the table, NS denotes that the value does not substantially influence the diagnosis outcome.

The resulting ratios of C_2H_2/C_2H_4, CH_4/H_2, and C_2H_2/C_2H_4 for pure methyl ester and oil mixture are listed in Table 6. No set of ratio limits is fulfilled by any samples; hence, no fault prediction can be made based on the recorded data. It can be concluded that the IEC 599 technique is not applicable for methyl ester under PD, and the presence of mineral oil of up to 10% does not change the results.

Table 5. Fault identification based on the IEC 599 ratio method.

Type of Fault	C_2H_2/C_2H_4	CH_4/H_2	C_2H_4/C_2H_6
Partial discharge (PD)	NS	<0.1	<0.2
Low energy discharge (D1)	>1	0.1-0.5	>1
High energy discharge (D2)	0.6–2.5	0.1–1.0	>2
Low temp. thermal fault (T1)	NS	>1	<1
Medium temp. thermal fault (T2)	<0.1	>1	1 – 4
High temp. thermal fault (T3)	0.2	>1	>4

Table 6. Fault identification results based on the IEC 599 ratio method.

Sample, Applied Voltage	C_2H_2/C_2H_4	CH_4/H_2	C_2H_4/C_2H_6	Predicted Fault
Pure Methyl Ester, 20 kV	-	0.16	0.20	NP
Pure Methyl Ester, 22 kV	-	0.16	0.26	NP
ME95-MO05, 17 kV	-	0.16	0.43	NP
ME95-MO05, 19 kV	-	0.16	0.40	NP
ME92.5-MO7.5, 17 kV	-	0.16	0.38	NP
ME92.5-MO7.5, 19 kV	-	0.16	0.50	NP
ME90-MO10, 17 kV	-	0.16	0.40	NP
ME90-MO10, 19 kV	-	0.16	0.39	NP

NP: No Prediction

3.3. Discussion

The results show that the methyl ester mixed with the used mineral oils of up to 10% has a similar gassing tendency with the pure methyl ester, although the concentration of gas production expansion increases as the proportion of used mineral oil in the oil mixture rises.

Impurities produced during the service period of the used mineral oil intensify PD activity in the oil mixture, which subsequently enhances gas production. The effect of the PD activity on gas production is reported in [4]. The higher gas generation at the higher applied voltage is related to the higher energy resulting from the higher applied voltage, and the correlation between discharge energy and gas production is confirmed by [33].

All samples produce mainly hydrogen which constitutes about 85% of total gas production. The next gasses are methane, carbon monoxide, ethane, and ethylene with percentages of about 13, 0.8, 0.4, and 0.1, respectively. Except for the presence of CO, the gassing tendency mentioned above is similar to the typical gas generation in mineral oil under PD [34]. However, the higher generation of CO in ester under PD is also reported in [26], [27]. The CO generation may be linked to the presence of C-O bonds in the ester molecules (RCOOR). Natural esters generate more CO than mineral oil during electrical breakdown, which was also reported in [35].

For hydrocarbon-based oils, in general, when the oils are subjected to a fault, the fault's energy breaks the carbon-hydrogen and carbon-carbon bonds, resulting in radicals of C, H, CH, CH_2, and CH_3. Recombination of these radicals leads to the formation of H-H (hydrogen), CH_3-H (methane), CH_3-CH_3 (ethane), CH_2-CH_2 (ethylene), and C≡C (acetylene). Hydrogen, methane, and ethane are formed under PD or thermal faults with temperatures below 500 ºC, whereas ethylene and acetylene are included at the higher fault's energy [36].

For fault identification, the applicability of identification techniques to all samples is summarized in Table 7. The Key Gas and Duval Triangle techniques correctly identify the PD fault with a success rate of 100%. The Duval Pentagon underestimated fault as stray gassing (S), whereas the IEC 599 ratio failed in predicting the fault (NP). The correct prediction of the Key Gas and Duval Triangle and the failure in the prediction of the IEC 599 on the creepage discharge in natural ester are also reported in [4].

Although the identification methods evaluated show differences in identifying PD fault, each method consistently shows similar results for different samples. The Duval Pentagon method, for instance, identifies PD in methyl ester as stray gassing, but the phenomenon also applies to all oil mixtures. Thus, the presence of mineral oil up to 10% does not affect the fault identification results of PD by all evaluated methods in methyl ester oil. For practical implications, the remaining mineral oil of up to 10% in a transformer retrofilled with the methyl ester does not alter the fault identification by all evaluated methods under PD.

Table 7. Applicability of fault identification techniques to methyl ester and methyl ester mixed by used-mineral oils under PD.

Identification Technique	Methyl Ester		ME95 – MO05		ME92.5 – MO7.5		ME90 – MO10	
	20 kV	22 kV	17 kV	19 kV	17 kV	19 kV	17 kV	19 kV
Key Gas	+	+	+	+	+	+	+	+
Duval Triangle	+	+	+	+	+	+	+	+
Duval Pentagon	-	-	-	-	-	-	-	-
IEC 599 Ratio	-	-	-	-	-	-	-	-

+ Applicable
- Not applicable

4. Conclusion

The DGA in methyl ester and the methyl ester mixed with the mineral oil of 5, 7.5, and 10% by volume under PD has been studied. The results show the applicability of the Key Gas and Duval Triangle techniques for PD in all oil samples, whereas the Duval Pentagon underestimates the PD as the stray gassing. In contrast, the IEC 599 Ratio technique fails to predict the PD fault. These behaviors are valid for both pure methyl and methyl ester mixed with the used mineral oils, indicating that the presence of the mineral oil up to 10% does not substantially alter the DGA profile of methyl ester under PD conditions. This finding has practical implications for transformer retrofilling, as residual mineral oil at these concentrations does not impair the DGA diagnostics for PD in methyl ester. The main benefit of this study is that the current oil evacuation technique, which can remove residual oil up to 4-7%, does not require modification to ensure the effective application of the DGA technique when a mineral oil-filled transformer is retrofilled with methyl ester. However, further research is needed to evaluate DGA behavior for other fault types, such as low and high-energy discharges, and to investigate long-term aging behavior of methyl ester-mineral oil mixtures under varying operational conditions.

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