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Dissolved Gas Analysis on POME-based Nanofluids

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***Abstract*—This paper investigates the dissolved gas analysis (DGA) of aged palm oil methyl ester (POME)-based nanofluids. Thermal stress is applied to nanofluids with various concentrations of TiO_2 and MWCNT nanoparticles. The experiments are conducted on a laboratory scale, with the POME-based nanofluids thermally aged at 130 ºC in the absence of solid insulating materials. The study aims to compare the gases generated in both nanofluids. Four different diagnosis methods are employed: Rogers Ratio, IEC Gas Ratio, Duval Triangle, and Duval Pentagon. The experimental results reveal nearly identical gassing behaviors between the two nanofluids after thermal ageing. These findings contribute to expanding investigations into transformer fault diagnosis, particularly in cases where various natural ester oils, including POME-based nanofluids, are used in power transformer. This helps diversify the growing body of research on DGA for natural ester oils, which has largely focused on commercially dominant types.**

***Index Terms*—Dissolved gas analysis, nanofluids, palm oil methyl ester, thermal aging, transformer.**

I. INTRODUCTION

USING methyl ester oil as an alternative insulating liquid has been increasingly investigated in recent years to explore the potential of alternative materials in replacing conventional mineral oil for transformer applications. In the past five years, scholarly investigations have predominantly focused on exploring chemically modified ones as transformer liquids. These investigations encompass various studies, with Terminalia catappa kernel oil [1], karanji oil [2], and jatropha curcas oil [3], showing a variety of sourcing options. Different chemical modification methods for converting triglycerides into methyl esters, including direct purification, transesterification, esterification, and epoxidation-esterification, are the prime candidates utilized and discussed in the literature.

The extensive literature on various natural ester oils has sparked significant interest among researchers globally, leading to studies on plant-based insulating liquids tailored to local resources. In Southeast Asia, particularly in Indonesia, Malaysia, and Thailand, extensive research has focused on palm oil (PO) as an alternative transformer oil. This focus is due to several factors: (i) these countries are the world's leading producer of PO, offering a sustainable and abundant source of biodegradable oils, (ii) empirical data has demonstrated PO's potential as a transformer insulation option, with numerous studies reporting remarkable and promising outcomes [4], [5], [6].

While various methyl ester oils have been explored as base fluids for nanofluid preparation, palm oil methyl ester (POME) has received less attention due to specific challenges. POME is prone to oxidative degradation and hydrolysis, particularly under high-temperature conditions, which can compromise its long-term stability. Furthermore, compared to other extensively studied biodegradable oils like rapeseed and soybean oils, POME research remains limited, likely because of its niche focus and the dominance of more established alternatives. However, our previous works [7], [8] have demonstrated that POME possesses significant potential as a viable and sustainable alternative to mineral oil. These study highlights the enhanced dielectric performance of POME-based nanofluids, particularly in terms of higher AC breakdown voltage (BDV), while their low viscosity, which is lower compared to mineral oil, improves heat dissipation. As a biodegradable and environmentally friendly insulating fluid, POME-based nanofluids offer a safer and more sustainable alternative to conventional mineral oil.

Building on this foundation, this study investigates the properties of POME-based oils and their applicability in transformer applications. An essential issue remains whether the chemically modify POME, enhanced with nanoparticles and surfactants, optimize its performance as an insulating liquid. Conducting an ageing study is essential to understanding how insulating liquids behave when subjected to thermal stress [9].

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The occurrence of thermal stress for a longer duration in transformers results in the generation of specific gases through the decomposition of the insulating liquid. Monitoring the presence of various gases in transformer liquid is crucial in predicting the transformers overall health. Therefore, examining Dissolved Gas Analysis (DGA) in insulating liquid is significant for assessing the operational state and preventing premature failures. The presence and composition of dissolved gases in the insulating liquids can provide valuable information and offer potential issues or abnormalities in the oils.

Several studies [10], [11], [12], [13] have reported a comparative analysis in the DGA results between mineral oil and various ester oils. In their groundbreaking research, Przybylek and Gielniak [10] conducted extensive analyses of gas generation in various synthetic and natural ester insulating liquids under thermal fault conditions. Their findings have significant implications for power transformer diagnostic, particularly in understanding the difference in gas generation and solubility between conventional mineral oil and newer ester-based alternatives. One notable finding was that in ester liquids, methane (CH_4) and ethane (C_2H_6) are created in similar amounts at higher temperatures, unlike in mineral oil, which is attributed to the longer hydrocarbon chain in ester liquids. The findings from this work offer valuable insights for optimizing the interpretation of DGA results for ester liquids employed in power transformer applications. A comprehensive study by Perrier et al. [13] demonstrated that during thermal faults, natural ester oils generated significantly higher levels of hydrogen (H_2) and ethane (C_2H_6), which is in agreement with results in [14], [15], compared to mineral oils or synthetic esters, with ethane emerging as the key indicator for natural esters. While Duval triangle 4 effectively identifies stray gassing phenomenon in mineral oils, its application to ester oils shows varying results, with some samples indicating overheating rather than stray gassing characteristics.

This study examines DGA, a diagnostic tool employed to assess ageing and degradation in insulating liquids. Despite the increasing interest in nanofluids, a research gap persists regarding the comprehensive DGA analysis of POME-based nanofluids containing titanium dioxide (TiO_2) and multi-walled carbon nanotubes (MWCNT), especially in the context of aged nanofluids. The aim is to investigate, on a laboratory scale, how thermal stress influence DGA analysis in ester oils and to enhance the interpretation of the DGA results. This contributes to the evolving understanding of methyl ester-based nanofluid for transformer insulating liquid. Additionally, this paper addresses the gap by exploring the thermal behavior and degradation of these nanofluids under ageing conditions.

II. Material and Methods

A. Materials

POME is produced by chemically modifying using the transesterification process from refined, bleached, and deodorized palm oil olein ($RBDPO_o$) after mixing some chemical additives, i.e., pure methanol purchased from Merck, potassium hydroxide (KOH) as a catalyst bought from J. T. Baker. Two types of nanofluids were prepared using semiconductive TiO_2 and conductive MWCNT nanoparticles purchased from Sigma Aldrich. Hexadecyltrimethylammonium bromide (CTAB) was employed as a surfactant to attain a uniform dispersion of nanoparticles in the base fluids.

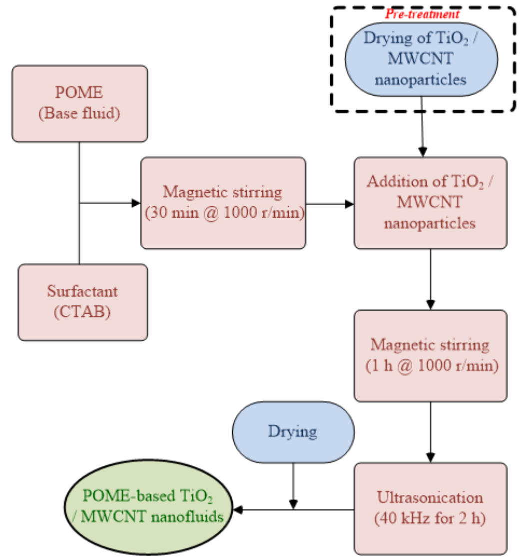

Fig. 1. Flowchart of POME-based nanofluids preparation in the laboratory.

B. Preparation of Nanofluids

Initiating the experimental research with nanofluids begins with the preparation of nanofluids. In this study, it was carried out using a two-step method, which involves the separate preparation of dried nanoparticles, followed by the chemical and mechanical treatments of the dried nanoparticles in the POME base fluids. Before initiating sample preparation, POME underwent a preprocessing step involving drying at 80 °C for 2 h to eliminate moisture. Subsequently, it was permitted to naturally cool to room temperature. The CTAB was incorporated into the POME base fluid and subjected to magnetic stirring for 30 min to achieve uniform dispersion. Based on the earlier investigation, the amount of mixing the CTAB was set at half of the weight percentage of the nanoparticles. However, the optimal concentration of the surfactant is still under investigation.

TiO_2 and MWCNT were selected as nanoparticles for this study due to their complementary properties that enhance the performance of POME-based nanofluids. TiO_2 is well-known for its excellent dielectric properties, chemical stability, and thermal conductivity, as reviewed in [16], [17], which contribute to improving breakdown voltage (BDV) and maintaining the stability of nanofluids under high electric stress. Its availability, cost-effectiveness, and high surface area further make it suitable choice for insulating liquid applications. On the other hand, MWCNTs offer exceptional electrical and thermal conductivity, and unique nanostructures that significantly enhance thermal stability and dielectric strength [18]. The separate investigation of oxide-based TiO_2 and carbon-based MWCNTs allows for comprehensive investigation of their individual effects on the thermal and dielectric performance of POME-based nanofluids, providing insights into their applicability in transformer insulation systems.

The nanoparticles were precisely measured and dispersed into POME to achieve the desired concentration. Nanoparticles were incorporated into the base fluid at concentrations of 0.01-, 0.02-, 0.05-, and 0.10-g/L, offering a varied range for exploring the enhancement of optimal insulating liquid properties in the nanofluids. Achieving a uniform dispersion of nanoparticles in the base fluids necessitates subjecting samples to energetic magnetic stirring and ultrasonication. Fig. 1 shows the flowchart of preparing POME-based nanofluids in the laboratory.

The mixture was heated at 60 ºC and magnetically stirred at 1000 rpm with a reaction time of 60 minutes. The cooled reaction mixture at ambient temperature was then transferred into a separatory funnel mounted on a stand to allow it to separate for 24 hours. Upon transesterification, glycerol and methyl ester become two distinct phases. The mixture of crude glycerol and KOH, which had settled at the bottom of the funnel, was discarded. The top layer, fatty acid methyl ester (FAME), was water-washed to remove excess KOH. The remaining impurities were then removed by heating and magnetically stirring the mixture for 30 minutes at 60 ºC. The FAME was then referred to as POME, which is the product of the transesterification of RBDPO olein and used as matrix oil or base fluid in the preparation of the NFs. This study expands our previous investigation [7], where the production process and methodology were referred to accordingly.

C. Accelerated Thermal Ageing Test

The nanofluid samples, once prepared, underwent the accelerated thermal ageing test in the laboratory, as shown in Fig. 2. This process was conducted at a single operating temperature for 1000 h under closed or sealed ageing conditions. A temperature of 130 ºC was strategically chosen to mitigate the risk of exceeding the liquid's flash point [19]. Furthermore, following the IEEE Std C57.147 [20], the thermal ageing temperature for natural ester oils is maintained at levels below 180 ºC to prevent fluid scorching. This aligns with the requisite temperature of at least 130 ºC for a meaningful comparison of the performance of the natural ester oil with the conventional mineral oil. The decision is supported by previous investigations that have been investigated by Carcedo et al. [21], Cong et al. [22], and Karambar and Tenbohlen [23].

While 130 ºC may not fully replicate the exact thermal conditions of real transformers, it serves as a standardized and controlled benchmark to compare the ageing behavior of different concentrations of the nanofluids. Furthermore, the consistency of the ageing behavior of POME-based nanofluids with varying nanoparticle concentrations at this temperature was observed and analyzed to ensure reliable and meaningful results.

Fig. 2. Overheating of nanofluid samples.

D. Analysis of Dissolved Gas

In this DGA, the emphasis is placed on analyzing the gases present in the oil, contributing to its degradation. There are three sets of essential gases monitored that are associated with incipient fault types, which decompose on the application of thermal stress: (i) hydrocarbon gases include hydrogen (H_2), methane (CH_4), ethane (C_2H_6), acetylene (C_2H_2), and ethylene (C_2H_4); (ii) carbon monoxide (CO) and carbon dioxide (CO_2); and (iii) oxygen (O_2) and nitrogen (N_2) are non-fault gases which are analyzed to provide complementary information [24], [25].

The gas generation mechanism in POME-based nanofluids and natural ester oil follows the thermal decomposition of chemical bonds, dehydration, and free radical formation [12], producing the aforementioned gases. However, due to structural differences, POME — composed of triglycerides with abundant C-C double bonds and carboxyl (COOH) groups — undergoes different degradation patterns than mineral oil, which consists mainly of paraffinic and aromatic hydrocarbons. The specific blend of gases generated depends on the temperatures induced by the faults [9]. The typical gases produced under different temperature conditions are categorized as follows [26]:

(i) Low Temperature Thermal Decomposition ($T < 300$ ºC): Generates mainly H_2, CH_4, C_2H_6, and small amounts of C_2H_4.

(ii) Moderate Temperature Thermal Decomposition (300 ºC $< T <$ 700 ºC): Produces a higher amount of H_2, C_2H_4, and CH_4.

(iii) High Temperature Thermal Decomposition ($T >$ 700 ºC): Generates significant amount of H_2, C_2H_2, C_2H_4, CH_4, and H_2.

The DGA of the thermally aged nanofluid samples was sent to Tenaga Nasional Berhad (TNB) Research Sdn. Bhd., a wholly owned Malaysian subsidiary specializing in R&D, for assessment using an Agilent 6890 Gas Chromatograph as per IEC 60567 standard.

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E. Diagnosis Method

In this study, the gases are analyzed using the Rogers Ratio Method (RRM), the IEC Gas Ratio Method, the Duval Triangle Method (DTM) and the Duval Pentagon Method (DPM).

1) RRM and IEC Gas Ratio Method: RRM and IEC Gas Ratio Method used the concentration ratios of the key gases to recognize the incipient faults. The following ratios are computed: C_2H_2/C_2H_4, CH_4/H_2, and C_2H_4/C_2H_6, and the nature of the fault can be identified as shown in Table I.

TABLE I
FAULT TYPES IN THE RRM AND IEC GAS RATIO METHODS [27], [28]

Gas Ratio	Fault Types					
	Unit normal / Partial discharge	Discharge low energy	Discharge high energy	Thermal fault T < 300 ºC	Thermal fault 300 ºC < T < 700 ºC	Thermal fault T > 700 ºC
RRM						
C_2H_2/C_2H_4	<0.01	≥1.0	≥0.6, <3.0	<0.01	<0.1	<0.2
CH_4/H_2	<0.1	≥0.1, <0.5	≥0.1, <1.0	≥1.0	≥1.0	≥1.0
C_2H_4/C_2H_6	<1.0	≥1.0	≥2.0	<1.0	≥1.0–<4.0	≥4.0
IEC Gas						
C_2H_2/C_2H_4	Not significant	>1	0.6–2.5	Not significant	<0.1	<0.2
CH_4/H_2	<0.1	0.1–0.5	0.1–1	>1	>1	>1
C_2H_4/C_2H_6	<0.2	>1	>2	<1	1–4	>4

2) Duval Triangle Method: DTM is a visual tool that utilizes the relative percentages of three hydrocarbon gases: CH_4, C_2H_4, and C_2H_2, to depict the fault types. The relative percentage of the gases is used to plot coordinates within a triangle. This method categorizes faults into six zones: partial discharge (PD), discharge with low energy (D1), discharge with high energy (D2), a combination of thermal faults and discharges (DT), thermal fault < 300 ºC (T1), and thermal fault 300 ºC < T < 700 ºC (T2) or thermal fault > 700 ºC (T3).

The relative proportions are calculated using (1) and (2):

$$C_T = CH_4 + C_2H_4 + C_2H_2 \tag{1}$$

$$\begin{aligned} \%CH_4 &= CH_4/C_T \times 100 \\ \%C_2H_4 &= C_2H_4/C_T \times 100 \\ \%C_2H_2 &= C_2H_2/C_T \times 100 \end{aligned} \tag{2}$$

Duval and Lamare [28] introduced the Duval pentagon, which uses the relative percentages of five gases: H_2, C_2H_6, CH_4, C_2H_4, and C_2H_2 as five-digit coordinates to construct a pentagon. The Duval Pentagon Method (DPM) utilizes a regular pentagon with five fault zones. The zones are PD: corona partial discharge, D1: low energy discharges, D2: high energy discharges, T1: overheating (thermal faults < 300 ºC), and T2: arcing (thermal faults > 700 ºC).

The two versions of DPM are Duval Pentagon 1 (uses the regular pentagon with six zones) and Duval Pentagon 2 (uses a modified pentagon with seven zones corresponding to PD, D1, D2, T3-H, C, O, and S), where T3-H: thermal faults in oil, C: carbonization of paper, O: overheating, and S: stray gassing at temperature < 200 ºC in the laboratory.

The coordinates of each of the five points are calculated based on (3) and (4) [29], [30]:

$$\begin{cases} x_0 = \%H_2 \cos\frac{\pi}{2} \\ x_1 = \%C_2H_6 \cos\left(\frac{\pi}{2}+\frac{2\pi}{5}\right) \\ x_2 = \%CH_4 \cos\left(\frac{\pi}{2}+\frac{4\pi}{5}\right) \\ x_3 = \%C_2H_4 \cos\left(\frac{\pi}{2}+\frac{6\pi}{5}\right) \\ x_4 = \%C_2H_2 \cos\left(\frac{\pi}{2}+\frac{8\pi}{5}\right) \end{cases} \quad \begin{cases} y_0 = \%H_2 \sin\frac{\pi}{2} \\ y_1 = \%C_2H_6 \sin\left(\frac{\pi}{2}+\frac{2\pi}{5}\right) \\ y_2 = \%CH_4 \sin\left(\frac{\pi}{2}+\frac{4\pi}{5}\right) \\ y_3 = \%C_2H_4 \sin\left(\frac{\pi}{2}+\frac{6\pi}{5}\right) \\ y_4 = \%C_2H_2 \sin\left(\frac{\pi}{2}+\frac{8\pi}{5}\right) \end{cases} \tag{3}$$

$$\begin{aligned} C_x &= \frac{1}{6A}\sum_{i=0}^{n-1} (x_i + x_{i+1})(x_i y_{i+1} - x_{i+1} y_i) \\ C_y &= \frac{1}{6A}\sum_{i=0}^{n-1} (y_i + y_{i+1})(x_i y_{i+1} - x_{i+1} y_i) \\ A &= \frac{1}{2}\sum_{i=0}^{n-1} (x_i y_{i+1} - x_{i+1} y_i) \end{aligned} \tag{4}$$

where x_i and y_i are the coordinates of the five points, C_x and C_y are the coordinates of the centroid, and A is the surface of the pentagon.

III. EXPERIMENTAL RESULTS

This section presents the dissolved gas generation and the fault diagnosis of thermally aged POME-based nanofluids with TiO_2 and MWCNT nanoparticles.

A. Dissolved Gas Generation

Fig. 3 compares the presence and distribution of H_2, CH_4, C_2H_4, C_2H_6, and C_2H_2 gases in the two types of nanofluid samples after 1000 h of ageing, excluding CO and CO_2. It is clear that these two nanofluids have almost similar gassing behavior for thermal faults. However, the production of hydrocarbon gases does not show significant variation between the two types of aged nanofluids.

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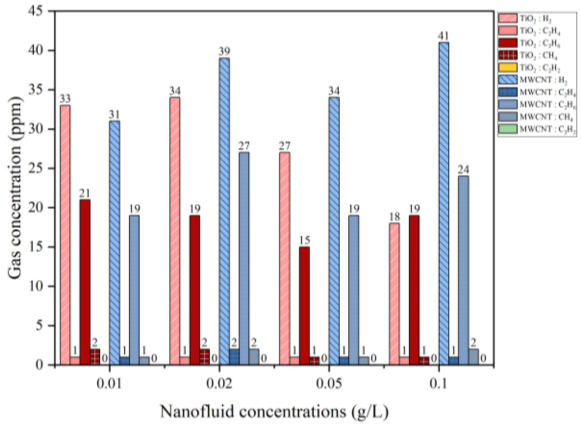

Fig. 3. Distribution of hydrocarbon gases in 1000-h-aged POME-based nanofluids.

B. RRM and IEC Gas Ratio Methods

Table II shows gas concentration ratios for the nanofluids and their respective fault diagnoses based on the RRM and the IEC gas ratio methods. Although the gas ratios for the two methods are identical in their classification as shown in Table I, the actual values of the gas ratios are different.

TABLE II
GAS CONCENTRATION RATIOS BASED ON RRM AND IEC

Nanofluids	Gas Ratios			Fault Diagnosis	
	C_2H_2/C_2H_4	CH_4/H_2	C_2H_4/C_2H_6	RRM	IEC
TiO_2 0.01-g/L	0	0.061	0.048	Normal	Partial discharges
TiO_2 0.02-g/L	0	0.059	0.053		
TiO_2 0.03-g/L	0	0.037	0.067		
TiO_2 0.10-g/L	0	0.056	0.053		
MWCNT 0.01-g/L	0	0.032	0.053		
MWCNT 0.02-g/L	0	0.051	0.074		
MWCNT 0.03-g/L	0	0.029	0.053		
MWCNT 0.10-g/L	0	0.049	0.042		

According to IEEE standard C57.104, the RRM suggested that the nanofluid samples do not show any signs of fault or degradation due to thermal ageing. However, according to IEC 60599-2022, the IEC ratio method suggested that the nanofluid samples had a partial discharge fault. The discrepancy between the two methods highlights how diagnostic approaches differ in their interpretation of the same data set. This underlines the importance of using complementary diagnostic techniques to ensure a more comprehensive fault analysis.

C. Duval Triangle and Pentagon Methods

1) Duval Triangle Method (DTM): The DTM is based on the relative percentage of CH_4, C_2H_4, and C_2H_2, gases. The Duval Triangle 3, explicitly designed for non-mineral oils, is applicable for fault types diagnosis in vegetable oils with some modifications on the boundaries of fault zones in Duval Triangle 1. Table III shows the relative proportion of gases. For instance, based on the calculated relative percentage of CH_4 = 66.7%, C_2H_4 = 33.3%, and C_2H_2 = 0%, for POME-based TiO_2 at a concentration of 0.01-g/L, the point (yellow dot) falls in the T1 fault zone in the Duval Triangle 3, indicating overheating (thermal faults < 300 ^oC), as shown in Fig. 4(i). Overall, according to the interpretation using DTM of POME-based TiO_2 and MWCNT nanofluids as illustrated in Figs. 4 and 5, all nanofluid samples were at the T1 zone in Duval Triangle 3, indicating overheating of thermally aged samples < 300 ^oC.

TABLE III
DUVAL TRIANGLE 3 COORDINATES CALCULATION

Nanofluids	Gas percentages			Fault Type
	$\%CH_4$	$\%C_2H_4$	$\%C_2H_2$	
TiO_2 0.01-g/L	66.7	33.3	0	T1
TiO_2 0.02-g/L	66.7	33.3	0	T1
TiO_2 0.03-g/L	50	50	0	T1
TiO_2 0.10-g/L	50	50	0	T1
MWCNT 0.01-g/L	50	50	0	T1
MWCNT 0.02-g/L	50	50	0	T1
MWCNT 0.03-g/L	50	50	0	T1
MWCNT 0.10-g/L	66.7	33.3	0	T1

2) Duval Pentagon Method (DPM): Fig. 6 illustrates the DGA diagnosis results of the 1000-h-aged POME-based nanofluids using the DPM. Specifically, the calculation of coordinates for the pentagon with a concentration of 0.01-g/L is exemplified. To illustrate, for the coordinate on the H_2 axis, the angle between the H_2 axis and the *x*-axis is $\pi/2$ degree. Thus, the coordinates are $x_{H_2}=(33/57\times100)\cos\frac{\pi}{2}=0$, $y_{H_2}=(33/57\times100)\sin\frac{\pi}{2}=57.9$. Similar calculations are executed for the remaining four coordinates. The coordinates for C_2H_6, CH_4, C_2H_4, and C_2H_2 are (-35.0, 11.4), (-2.1, -2.8), (1.1, -1.5), and (0, 0), respectively. The centroid's (*x*, *y*) coordinates, computed using (3), are (-7.2, 13.0). Based on this calculation, the fault type falls within the 'S' zone. Overall, the DPM method consistently identified an 'S' status, signifying the presence of stray gassing of oil at a temperature < 200 ^oC for all nanofluid samples. This observation infers that natural ester oils might produce combustible gases under thermal stress [31].

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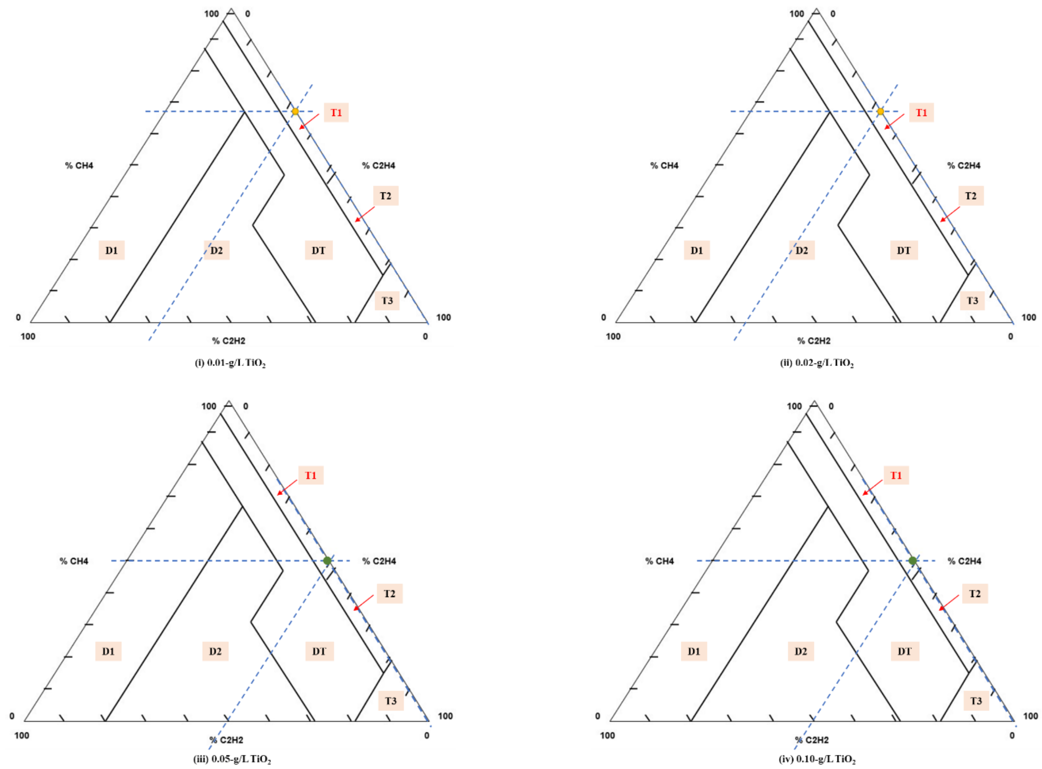

Fig. 4. Result analysis using DTM for 1000-h-aged POME-based TiO_2 nanofluids

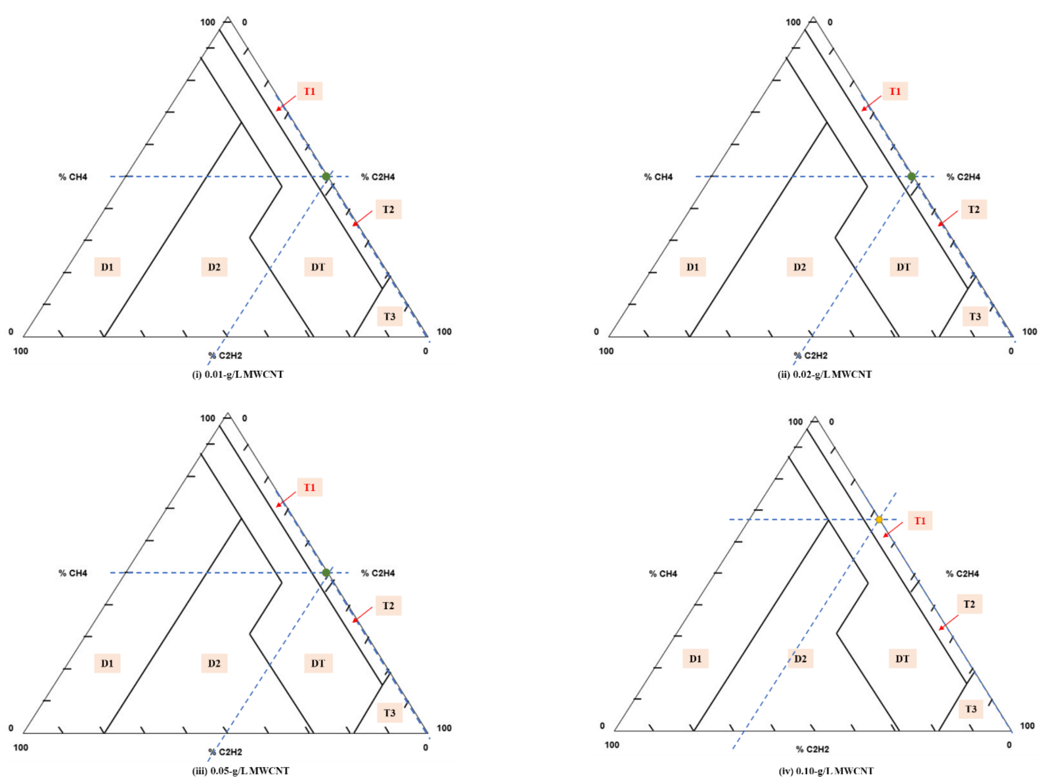

Fig. 5. Result analysis using DTM for 1000-h-aged POME-based MWCNT nanofluids.

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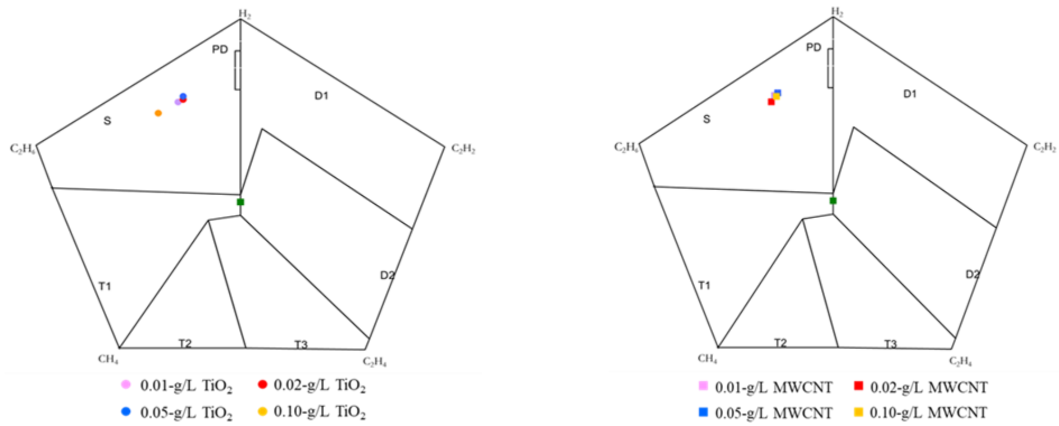

Fig. 6. Result analysis using DPM for 1000-h-aged POME-based nanofluids indicating stray gassing at temperature < 200 ºC.

IV. Discussions

The prediction of faults based on different types of DGA diagnosis techniques allows the study of gas formation patterns, fault diagnosis, and the reliability of diagnostic methods in identifying transformer faults.

A. Dissolved Gas Formation

The test results for thermally aged POME-based TiO_2 and POME-based MWCNT samples enable a comparison between these two types of nanofluids. The study of dissolved gas generation clearly shows that these two nanofluids have almost similar gassing behavior for thermal faults. The production of hydrocarbon gases does not show significant variation between the two types of aged nanofluids. This suggests that the presence of nanoparticles may not a strong impact on the formation of hydrocarbon gases during thermal ageing. Since hydrocarbon gases are primarily derived from the thermal decomposition of the POME base fluid itself, it is likely that the base fluid plays a dominant role in gas formation rather than the types of added nanoparticles.

Among the hydrocarbon gases, it is found that H_2 is the dominant characteristic gas in POME-based nanofluids for thermal faults under low temperature (below 300 ºC), followed by C_2H_6. This observation aligns with findings in other types of natural ester oils, such as overheating in soy seed-based oil investigated by Muhamad et al. [32], overheating in natural ester FR3 studied by Przybylek and Gielniak [10], and thermally stress camellia oil studied by Li et al. [14], further supporting the similarity to the results obtained in this study.

A significant amount of H_2 is generated for both types of nanofluids, accounting for more than 50% of the five types of hydrocarbon gases, the main characteristic gas of partial discharge. Furthermore, C_2H_2 was almost not generated in all nanofluids samples during the entire ageing experiment at 130 ºC. The thermal fault in POME-based nanofluids dissipated insufficient energy for producing these particular gases. The absence of significant C_2H_2 in all samples further supports findings from natural ester studies [12], [15].

B. Fault Diagnosis Analysis

Table IV summarizes the fault prediction for 1000-h-aged POME-based nanofluids after thermal stress using various methods. Using different fault prediction methods enabled the diagnosis of incipient faults under thermal stress in the insulation. Most diagnostic methods are suitable for POME-based nanofluids; however, some do not yield consistent results.

According to the RRM, the transformer is considered to be in a 'normal' condition, indicating that the POME-based nanofluids did not show any signs of faults or degradation due to thermal ageing. In contrast, the IEC ratio gas methods predicted partial discharges, while the DTM and DPM predicted the signs of overheating (thermal fault < 300 ºC) and stray gassing at temperatures < 200 ºC for all aged POME-based nanofluids, respectively. All tasks involving the plotting of triangular and pentagon diagrams of the nanofluid samples were completed using a small algorithm, freely provided by Duval [28] .

By comparing various DGA techniques (RRM, IEC gas ratio, DTM, and DPM), the results indicate that the DTM is a highly dependable diagnostic tool for evaluating faults in POME-based nanofluids, particularly for identifying thermal fault below < 300 ºC. However, its accuracy depends on the gas concentration levels present in the insulating liquid. In this study, some gases, particularly CH_4, C_2H_4, and C_2H_2, were detected at extremely low concentrations, which raises concerns about the reliability of the gas ratio calculations in DTM.

To assess the impact of measurement uncertainty, an uncertainty analysis was conducted by introducing a ±5% error margin based on the instrument accuracy. The results showed that minor fluctuations in gas concentrations could lead to different fault classifications, particularly when the gas levels were near the threshold between fault categories. For instance, a small variation in the measured concentrations of CH_4 (0.94 ppm) and C_2H_4 (1.06 ppm), giving relative percentage of CH_4 = 47%, and C_2H_4 = 53%, could shift the diagnostic from T1 (thermal fault < 300 ºC) to T2 (300 ºC < T < 700 ºC), which impacts the result of fault prediction. This highlights the limitation of relying solely on the DTM method and need for further diagnostic accuracy analysis in future studies.

The application of DPM diagnosed all nanofluid samples to be in stray gassing zone, which confirming the conclusion from Maiti and Chakraborty [15], where a different type of natural ester was used.

TABLE IV
RESULTS OF FAULT PREDICTIONS USING VARIOUS METHODS

DGA diagnosis methods	Fault prediction	Suitability to POME-based nanofluids
RRM	Normal	Suitable; not highly accurate
IEC Ratio Gas Method	Partial discharges	Suitable; not highly accurate
DTM	Overheating (thermal fault < 300 ºC)	Suitable; highly dependable
DPM	Stray gassing at temperature < 200 ºC)	Suitable; highly dependable

V. CONCLUSION

There are several conclusions derived from the experimental results in this study:

(i) The dissolved gas analysis results indicate that POME-based nanofluids exhibit similar gas formation trends to commercially available natural ester (FR3) under thermal ageing.

(ii) Employing fault prediction techniques using these DGA diagnostic methods enables the gaining of diagnoses related to partial discharges, overheating, and stray gassing in the nanofluids. These initial findings indicate the feasibility of obtaining incipient fault types for POME-based nanofluids based on the collected DGA data. The Duval Triangle Method (DTM) consistently predicted overheating at low temperatures, where CH_4 and C_2H_4 being the dominant of the total fault gases generated. Notably, acetylene (C_2H_2) was almost not generated during the entire overheating ageing experiments.

(iii) With the DTM, all predictions indicating overheating are correct for POME-based nanofluids. The accuracy of the DTM identifying overheating faults aligns closely with findings reported by other researchers with different types of ester oils, further validating its reliability for POME-based nanofluids. The application of DPM identified all the aged nanofluids to be in stray gassing indicating thermal fault < 200 ºC, aligns with [15]. In contrast, the RRM and IEC Gas Ratio Method failed to provide correct fault predictions.

However, it is important to note that this preliminary study focused on two types of nanoparticles and a specific POME as the base fluid. Additional research is required before these results can be considered conclusive and useful, especially considering factors such as variations in nanoparticle types and concentrations, the presence of surfactants, and temperature settings. Future research will investigate these aspects to establish a broader applicability of this study, which should be interpreted within the scope of the tested nanofluid formulations.

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REFERENCES

[1] C. M. Agu, M. C. Menkiti, J. T. Nwabanne, and O. D. Onukwuli, “Comparative assessment of chemically modified Terminalia catappa L. kernel oil samples – A promising ecofriendly transformer fluid,” *Industrial Crops and Products*, vol. 140, no. 111727, 2019.
[2] M. Maharana, S. K. Nayak, and N. Sahoo, “Karanji oil as a potential dielectrics liquid for transformer,” *IEEE Trans. Dielect. Electr. Insul.*, vol. 25, no. 5, Oct. 2018.
[3] H. B. H. Sitorus, R. Setiabudy, S. Bismo, and A. Beroual, “Jatropha curcas methyl ester oil obtaining as vegetable insulating oil,” *IEEE Trans. Dielect. Electr. Insul.*, vol. 23, no. 4, Aug. 2016.
[4] N. A. M.Jamail, N. A. Azali, E. Sulaiman, Q. E. Kamarudin, S. M. N. S. Othman, and M. S. Kamarudin, “Breakdown Characteristic of Palm and Coconut Oil with Different Moisture,” *IJEECS*, vol. 12, no. 1, Oct. 2018.
[5] N. A. Mohamad, N. Azis, J. Jasni, M. Z. A. Ab. Kadir, R. Yunus, and Z. Yaakub, “Experimental Study on the Partial Discharge Characteristics of Palm Oil and Coconut Oil Based Al2O3 Nanofluids in the Presence of Sodium Dodecyl Sulfate,” *Nanomaterials*, vol. 11, no. 3, Mar. 2021.
[6] A. A. Suleiman, N. A. Muhamad, N. Bashir, N. S. Murad, Y. Z. Arief, and B. T. Phung, “Effect of moisture on breakdown voltage and structure of palm-based insulation oils,” *IEEE Trans. Dielect. Electr. Insul.*, vol. 21, no. 5, Oct. 2014.
[7] S. M. W. Masra *et al.*, “Assessing Electrical and Physicochemical Performance of Chemically Modified Palm Oil as an Alternative Transformer Liquid,” *J. Oil Palm Res.*, vol. 35, no. 1, pp. 33–44, 2023.
[8] S. M. W. Masra, Y. Z. Arief, S. K. Sahari, A. R. H. Rigit, M. R. Rahman, and N. S. Suhaimi, “Thermal Aging Effects on the Electrical Breakdown Voltage of TiO and MWCNT Nanofluids Based on POME <sub/>,” *IEEE Trans. Dielect. Electr. Insul.*, vol. 30, no. 6, Dec. 2023.
[9] N. Baruah, S. S. Dey, and S. K. Nayak, “Evaluation of dissolved gas analysis and long-Term performance of non-edible natural ester,” *IEEE Trans. Dielectr. Electr. Insul.*, vol. 27, no. 5, pp. 1561–1569, 2020.
[10] P. Przybylek and J. Gielniak, “Analysis of Gas Generated in Mineral Oil, Synthetic Ester, and Natural Ester as a Consequence of Thermal Faults,” *IEEE Access*, vol. 7, pp. 65040–65051, 2019.
[11] A. M. Salah-Eldin, K. Helal, D.-E. A. Mansour, and R. A. A. El-aal, “Dissolved Gas Analysis of Oil-Based TiO_2 Nanofluids under Thermal Faults,” in *2022 IEEE 21st International Conference on Dielectric Liquids (ICDL)*, Sevilla, Spain: IEEE, May 2022, pp. 1–4.

[12] M. Meira, R. E. Alvarez, C. J. Verucchi, L. J. Catalano, and C. R. Ruschetti, "Comparison of gases generated in mineral oil and natural ester immersed transformer's models," in *2020 IEEE Electrical Insulation Conference (EIC)*, Knoxville, TN, USA: IEEE, Jun. 2020, pp. 114–117.

[13] C. Perrier, M. Marugan, and A. Beroual, "DGA comparison between ester and mineral oils," *IEEE Trans. Dielect. Electr. Insul.*, vol. 19, no. 5, Oct. 2012.

[14] J. Li, J. Zhou, C. Xiang, Z. Huang, J. He, and M. A. Mehmood, "Novel Approaches of DGA to Transformers Filled with Natural Ester Based Insulating Oils," in *2018 International Conference on Diagnostics in Electrical Engineering (Diagnostika)*, Pilsen: IEEE, Sep. 2018, pp. 1–5.

[15] P. K. Maiti and M. Chakraborty, "Dissolved Gas Analysis of Thermally Aged Mineral Oil and Vegetable Oil Based Nanofluids," in *2021 IEEE International Conference on the Properties and Applications of Dielectric Materials (ICPADM)*, Johor Bahru, Malaysia: IEEE, Jul. 2021, pp. 53–56.

[16] E. J. Kadim, Z. A. Noorden, Z. Adzis, and N. Azis, "Nanoparticles Application in High Voltage Insulation Systems," *IEEE Trans. Dielect. Electr. Insul.*, vol. 28, no. 4, Aug. 2021.

[17] S. N. Suhaimi, A. R. A. Rahman, M. F. Md. Din, M. Z. Hassan, M. T. Ishak, and M. T. B. Jusoh, "A Review on Oil-Based Nanofluid as Next-Generation Insulation for Transformer Application," *Journal of Nanomaterials*, vol. 2020, pp. 1–17, Feb. 2020.

[18] B. Bakthavatchalam, K. Habib, C. D. Wilfred, R. Saidur, and B. B. Saha, "Comparative evaluation on the thermal properties and stability of MWCNT nanofluid with conventional surfactants and ionic liquid," *J Therm Anal Calorim*, vol. 147, no. 1, Jan. 2022.

[19] T. Münster, P. Werle, K. Hämel, and J. Preusel, "Thermally accelerated aging of insulation paper for transformers with different insulating liquids," *Energies*, vol. 14, no. 11, 2021.

[20] IEEE Std C57.147-2018, *IEEE Guide for Acceptance and Maintenance of Natural Ester Fluids in Transformers IEEE Power & Energy Society*. 2018.

[21] J. Carcedo, I. Fernández, A. Ortiz, F. Delgado, C. J. Renedo, and C. Pesquera, "Aging assessment of dielectric vegetable oils," *IEEE Electrical Insulation Magazine*, vol. 31, no. 6, pp. 13–21, 2015.

[22] H. Cong, H. Shao, Y. Du, X. Hu, W. Zhao, and Q. Li, "Influence of Nanoparticles on Long-Term Thermal Stability of Vegetable Insulating Oil," *IEEE Transactions on Dielectrics and Electrical Insulation*, vol. 29, no. 5, pp. 1642–1650, 2022.

[23] S. Karambar and S. Tenbohlen, "Compatibility study of silicone rubber and mineral oil," *Energies*, vol. 14, no. 18, 2021.

[24] O. E. Gouda, S. H. El-Hoshy, and H. H. El-Tamaly, "Proposed three ratios technique for the interpretation of mineral oil transformers based dissolved gas analysis," *IET Generation, Transmission and Distribution*, vol. 12, no. 11, pp. 2650–2661, 2018.

[25] C. Perrier, M. Marugan, and A. Beroual, "DGA comparison between ester and mineral oils," *IEEE Transactions on Dielectrics and Electrical Insulation*, vol. 19, no. 5, pp. 1609–1614, 2012.

[26] J. Golarz, "Understanding Dissolved Gas Analysis (DGA) techniques and interpretations," in *2016 IEEE/PES Transmission and Distribution Conference and Exposition (T&D)*, Dallas, TX, USA: IEEE, May 2016, pp. 1–5.

[27] T. Piotrowski, P. Rozga, and R. Kozak, "Comparative Analysis of the Results of Diagnostic Measurements with an Internal Inspection of Oil-Filled Power Transformers," *Energies*, vol. 12, no. 11, Jun. 2019.

[28] M. Duval & L. Lamarre, "The duval pentagon-a new complementary tool for the interpretation of dissolved gas analysis in transformers," *IEEE Electr. Insul. Mag.*, vol. 30, no. 6, pp. 9–12, Nov. 2014.

[29] Y. Yue, D. Yang, and D. Han, "Application of Duval Pentagon in State Diagnosis of On Load Tap Changer," *J. Phys.: Conf. Ser.*, vol. 2247, no. 1, Apr. 2022.

[30] L. Cheim, M. Duval, and S. Haider, "Combined Duval Pentagons: A Simplified Approach," *Energies*, vol. 13, no. 11, Jun. 2020.

[31] IEEE Std C57.155-2014, *IEEE Std C57.155. IEEE Guide for Interpretation of Gases Generated in Natural Ester and Synthetic Ester-Immersed Transformers IEEE Power and Energy Society*. 2014.

[32] N. A. Muhamad, B. T. Phung, and T. R. Blackburn, "Dissolved gas analysis for common transformer faults in soy seed-based oil," *IET Electr. Power Appl.*, vol. 5, no. 1, pp. 133–142, Jan. 2011.

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