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Quality Assessment of Aged Mineral Oil-Synthetic Ester Retrofilled Transformer Oil through Total Acid Number and Fourier Transform Infrared Spectroscopy

Nur Amirah binti Othman[1], Hidayat bin Zainuddin[1,*], Muhammad Sharil bin Yahaya[1], Norhafiz bin Azis[2], Zulkifli bin Ibrahim[1], and Mohd Affendi bin Che Musni[3]

[1]Faculty of Electrical Technology and Engineering, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100, Durian Tunggal, Melaka., Faculty of Engineering, [2]Universiti Putra Malaysia UPM, 43400, Serdang, Selangor Darul Ehsan., [3]Power Transcoteq & Services, Senawang Commercial Park, 70450, Seremban, Negeri Sembilan
nuramirahothman@yahoo.com, *hidayat@utem.edu.my, sharil@utem.edu.my, norhafiz@upm.edu.my, zulkifliibrahim@utem.edu.my, affendi.musni@transcoteq.com.

Abstract: The retrofilling process involves the substitution of mineral oil in an existing transformer with ester oil serves as an alternate method to prolong the transformer lifetime. This pertains to the excellent characteristics of ester oil, recognized for its moisture resilience which in turns imparts a unique advantage by extending the lifespan of insulation paper. Nevertheless, the retrofilling procedure involves mixing the new ester oil with the existing aged mineral oil (AMO) in the transformer oil, which ultimately impacts the performance of the new ester oil. Hence, it is crucial to evaluate the performance of mixed oil to identify any indications of oil deterioration due to retrofilling before energising the transformer. In evaluating the performance of aged mineral-synthetic ester (AMO/SE) retrofilled oil, this work presents the analysis of the total acid number (TAN) and Fourier Transform Infrared (FTIR) spectra. Prior to the retrofilling process using fresh-treated synthetic ester oil, four distinct amounts of aged mineral oil (AMO) were prepared, comprising 3 %, 5 %, 7 % and 9 %. This option provides the opportunity to simulate varying levels of remaining mineral oil present in the transformer. The AMO samples were prepared by subjecting the fresh mineral oil to thermal ageing in a vacuum oven at a temperature of 130 °C for two different durations, i.e. 75 and 150 hours. Throughout the collected data, the amount of acids in AMO/SE oil has exhibited a rising trend with a decreased in proportions of AMO in the fresh-treated synthetic ester oil for both AMO conditions. Meanwhile, the severity of AMO conditions give a significant effect to the acid growth in AMO/SE retrofilled oil. In conjunction with FTIR spectra, the rise in acid concentration in retrofilled AMO/SE oils was clearly observable in the C=O stretch in the region of 1710-1770 cm^{-1}, which exhibited a significant association with the TAN value.

Keywords: Power transformer, retrofilling, synthetic ester oil, mineral oil, total acid number (TAN), water content, hydrolysis, fourier transform infrared (FTIR) spectra.

1. Introduction

Power transformer is a crucial component in power systems. It requires the use of high-quality insulating material for optimal performance. An oil-filled transformer is a type of transformer that consists of insulating oil and cellulose-based paper as widely used insulating material subject to compliance to certain standards and requirements. However, these insulation materials are prone to the impacts of ageing. This situation keeps on great interest to researchers and engineers, driving their focus towards assessing the performance of insulation system in power transformers.

Mineral oil (MO) is widely utilized in power transformers, serving for various purposes. It serves as a dielectric, facilitates heat transfer as a coolant, acts as a protective layer for the core-coil assembly, and helps reduce oxygen exposure to cellulose-based paper [1]. In addition, MO exhibits a cost-effective advantage and possesses a low kinematic viscosity, contributing this insulating oil to endure for several centuries [2]. Nonetheless, the environmental sustainability

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of MO is compromised by its weak decomposability and significant dependence on non-renewable energy sources. In addition, the power transformer will faces a challenge due to its low flash point when need to operate at elevated temperatures [3]. As of this point, many researchers in the modern day are actively seeking alternative other types of insulating liquids that could potentially replace the mineral oil.

In recent times, ester oil (EO) has emerged as an acceptable substitute for insulating liquids, offering an alternative to the existing issue. Exclusively, the oils that has been classified as readily biodegradable are natural ester (NE) and synthetic ester (SE) oils. This classification is based on two specific criteria. Firstly, these oils must undergo a minimum of 60 % biodegradation within 10 days following the surpassing of the 10 % degradation. Additionally, a minimum of 60 % degradation must be evident by the 28th day of the biodegradation test [3]. However, invest in completely new transformer oil, along with a new insulating oil can be considered as costliest option. This situation presents an opportunity to consider retrofilling approach, which involves substitution the conventional mineral oil with ester oil in an existing transformer. Nevertheless, this approach does not guarantee the complete elimination of all aged mineral oil. It is possible that a portion of the residual volume could remain confined within the strands of cellulose paper, core, winding, or any unreachable components of the transformer [4]. In a preceding study led by Fofana et al [4], it has revealed that a significant amount, reaching up to 8% of aged insulating liquid (specifically polychlorinated biphenyls, PCB oil) remains within the transformer unit. The residual insulating liquid has been thoroughly mixed with the new insulating liquid, which will consequently impact the performance of the new insulating liquid.

Considering the viewpoints presented above, this paper looks into the performance of fresh SE oil after being mixed with various amounts of aged mineral oil (AMO) during the retrofilling process. Hence, it is crucial to prioritise on accessing the retrofilled oil prior to its utilisation in industrial applications. This paper aims to assess the performance of retrofilled oil by using the Total Acid Number (TAN) and examining its relationship with Fourier Transform Infrared (FTIR) spectra.

2. Experimental Methodology

This section discusses the experimental methodology related to this work. It starts with a description of the insulating materials used including the procedure of oil pre-treatment for the purpose of baseline reference. Then, the preparation of aged mineral oil (AMO) through thermal ageing is discussed. Finally, the procedure for replacing the AMO with synthetic ester oil (SE) which is also called as retrofilling process is explained.

A. Insulation Materials

Mineral oil (MO) Nytro Libra and synthetic ester (SE) Midel 7131 were used. The MO refers to the fresh oil that will be aged through thermal ageing, and eventually will be regarded as aged mineral oil (AMO). Meanwhile, the SE refers to the fresh oil that will be used in the retrofilling of the AMO. Both the MO and SE oils have undergone initial treatment involving oil filtration and nitrogen purging, which will be further described in the subsequent sections.

In addition to the insulating oils, the study also included the Kraft paper. The Kraft paper, having a thickness of 0.018 mm, was supplied by Krempel GmbH & Co.

B. Oil Pre-treatment

Pre-treatment of oil is essential for enhancing the properties and overall performance of oil samples. This, in turn, allows for the standardization of the oil samples. During the pre-treatment procedure, the oil samples have been initially filtered, which is a crucial for removing particles or contaminants that may have accumulated in the oil samples over an extended duration [5]. In this study, both the MO and SE oil samples underwent filtration using a 47-mm vacuum filtration with filter paper having a pore size of 0.22 μm [6].

In this procedure, nitrogen gas was introduced into the oil samples to displace the air and moisture, enabling the water content of the oil to comply with the standard [7].

C. Thermal Aging Preparation

In this section, the thermal aging process was specifically applied to the treated MO oil. The Kraft insulating paper along with copper catalysts specifically copper rod [8] and copper powder [9] were used to speed up the aging process of the oil. The first dimension measured approximately 15 mm × 50 mm and was securely attached to an abraded and polished copper rod. The second dimension of 15 mm × 250 mm was not attached to the copper rod. Meanwhile, a quantity of 2.5 grams of copper powder was carefully wrapped in the filter paper.

The Kraft papers, both with and without copper rod, were subjected to a drying process in an air ventilated oven at a temperature of 105 °C for a duration of 12 hours. This procedure was carried out in accordance with the test method specified in the IEC 60641 standard, with the aim of eliminating any excessive moisture present in the Kraft paper [10]. Subsequently, all Kraft papers, both with and without copper rod along with wrapped copper powder were immersed in a 1000 ml volume of treated MO oil. Following that, accelerated thermal aging was conducted in a vacuum oven at a temperature of 130 °C under two distinct oil conditions, listed down in Table 1 [11], [12].

Table 1. Two Different Condition of Aged Mineral Oil

Oil Condition	Aging period
AMO-1	75 hours
AMO-2	150 hours

D. Retrofilling Preparation

During the preparation of retrofilling, each condition of the AMO was prepared using four distinct volume percentages, specifically 3 %, 5 %, 7 %, and 9 %. The balance portion was subsequently filled with fresh-treated SE oil. However, the fresh-treated SE oil was underwent an initial heating at 60 °C before retrofilling procedure [13] in order to guarantee thorough impregnation of the cellulose in the Kraft paper. Following that, the AMO and fresh-treated SE oil samples were mixed without the aid of a stirrer, in accordance with the respective percentages. Fig. 1 illustrates the experimental configuration for the AMO/SE retrofilled oil, while Table 2 provides the corresponding notation. The AMO/SE retrofilled oil was subsequently left for a period of 24 hours for the dispersion of air bubbles and ensure thorough impregnation of the insulation papers before the testing [13]. Table 3 depicts the specific information regarding the mixing percentages of AMO/SE retrofilled oil.

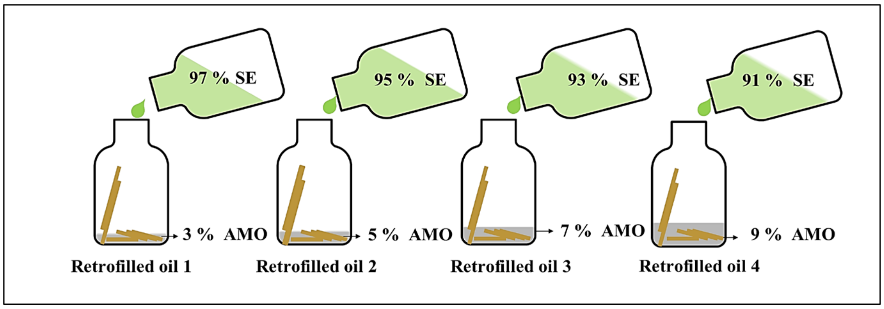


Figure 1. Experimental configuration for retrofilling of AMO/SE

Table 2. Visual Representation for Retrofilling Preparation

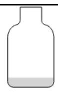
Graphic	Detail
	Aged Copper rod securely wrapped with Kraft paper (5 cm)
	Aged Kraft paper solely without copper (15 mm × 250 mm)
	Aged mineral oil specifically Nytro Libra
	Fresh-treated synthetic ester oil specifically Midel 7131

Table 3. Percentage of Mixed Oil for Retrofill

Types of Retrofilled Oil	Percentage of Oil Mixing		Ratio of Oil
	AMO	SE	
Retrofilled oil 1	3 %	97 %	3:97 AMO/SE
Retrofilled oil 2	5 %	95 %	5:95 AMO/SE
Retrofilled oil 3	7 %	93 %	7:93 AMO/SE
Retrofilled oil 4	9 %	91 %	9:91 AMO/SE

3. Result and Discussions

The condition of AMO/SE retrofilled oil samples was analyzed through two distinct measurements including the determination of total acid number (TAN) and the analysis of Fourier Transform Infrared (FTIR) spectra.

A. Total Acid Number of Retrofilled oil

TAN was used to determine the total acidic content of insulating oil. This includes the free organic and inorganic acids. In this work, the TAN was measured through coulometric titration technique according to ASTM D664 by using Metrohm 848 Titrino plus [14], as depicted in Fig. 2. In the measurement, the total acid number was determined by neutralizing the acid contained in the 1g of oil samples with potassium hydroxide (KOH).

Figure 2. Metrohm 848 Titrino Plus

Table 4 presents the TAN value for fresh-treated SE and MO as well as AMO oil conditions, namely AMO-1 and AMO-2. The TAN value for fresh-treated SE oils was observed to fall within specified range established by the oil standard IEC 61099 for new ester oil. In addition, it should be noted that both the fresh-treated MO and AMO-1 also comply with the mineral oil requirement standard, IEC 60296. In contrast, AMO-2 condition was not complied with the oil requirement standard, as it exceeded the maximum limit of 0.01 mgKOH/g.

Table 4. Initial TAN Value before Retrofilled

Type of Oil sample	TAN value (mgKOH/g)
Fresh-treated SE	0.017
Fresh-treated MO	0.009
AMO-1	0.010
AMO-2	0.035
Note: 1. IEC 61099: Acidity requirement of new ester oil is less than 0.03 mgKOH/g. 2. IEC 60296: Acidity requirement of new mineral oil is less than 0.01 mgKOH/g.	

Fig. 3 shows the line plot for TAN value for retrofilled AMO/SE at different oil ratios and AMO oil conditions. It can be observed that the TAN value for fresh-treated SE oil has increased after mixed with AMO oil under all oil ratios and AMO conditions. It is believed that the thermal aging of MO leads to an oil oxidation reaction, which can develop to the formation of degradation products, like carboxylic acid, in AMO oil [15], [16]. Consequently, the dissolving of carboxylic acid in aged mineral oil (AMO) will lead to increased acidity in fresh-treated synthetic ester (SE) oil when mixed with AMO oil at various mixing ratios.

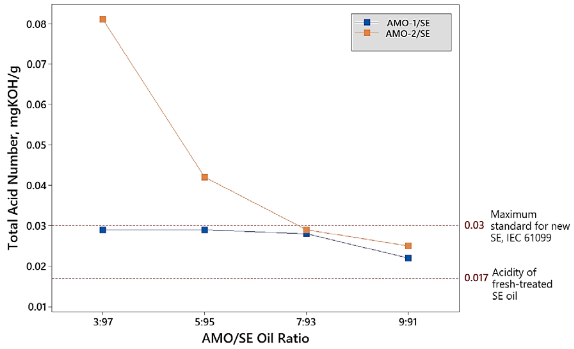


Figure 3. The TAN value for AMO/SE retrofilled oil at different oil ratios and AMO oil conditions.

When comparing the TAN value based on different retrofilled oil ratios, it is intriguing to observed that reducing the AMO oil to fresh-treated SE oil to lower oil ratios boosted the TAN value across all AMO conditions. The TAN value of the retrofilled AMO/SE oil exhibited a significant increased even though there is only small amount of AMO oil was mixed with fresh-treated SE oil. For instance, the TAN value of 3:97 AMO/SE retrofilled oil has increased more than 50 % from the TAN value of fresh-treated SE oil. The discussion of this particular observation initiates with an explanation that the significant increase in TAN value for 3:97 AMO/SE retrofilled oils is intricately associated with the hydrolysis reaction of ester oil. The increase in TAN value can be correlated well with the higher moisture content measured in the 3:97 AMO/SE retrofilled oil as opposed to the 5:95, 7:93 and 9:91 AMO/SE retrofilled oil. The differentiation is clearly illustrated in Fig. 4. Notably, higher water content in 3:97 AMO/SE retrofilled oil compared to 5:95, 7:93, and 9:91 AMO/SE oil ratios is believed can be attributed to the hygroscopic nature of ester, which allows the ester oil to hold more water from the AMO oil and draw in moisture from the cellulose insulation [17]. In this situation, it leads to a higher water consumption during the hydrolysis reaction. However, the inclusion of cellulose paper in the retrofilled oil could potentially lead to the equilibrium partition of water between the paper and oil and subsequently resulting in more water dissolved in the oil. Over time, large amount of free fatty acids was generated due to the hydrolysis reaction, resulting to an increase in the acidity of the oil [18].

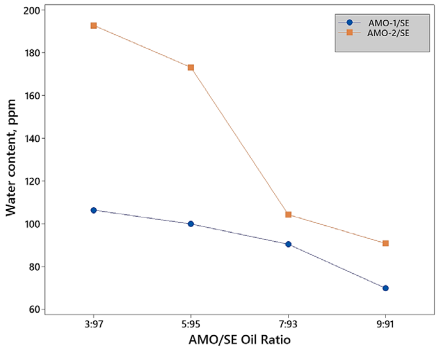


Figure 4. Water content for AMO/SE retrofilled oil at different oil ratios and AMO conditions.

In contrast to the various AMO conditions in affecting the TAN value for AMO/SE retrofilled oils as shown in Fig. 3, it is observed that the TAN value is higher for AMO-2/SE in comparison to AMO-1/SE retrofilled oils. The TAN value for AMO-2/SE has shown up to fivefold increase and it has grown to half the extent for AMO-1/SE, depending on the oil ratio, in comparison to the fresh-treated SE oil. It distinctly shows that increase in the severity of the AMO condition results in a more perceptible change in the acidity of retrofilled oil samples. Looking from an another angle, it is worth noting that the TAN value for AMO-2/SE retrofilled oil has beyond the maximum value specified in the oil requirement standard for new SE oil, with the exception of the 7:93 and 9:91 AMO-2/SE conditions. As discussed in the preceding paragraph, it is believed due to the higher water content in the ratios of 3:97 and 5:95 of AMO-2/SE in comparison to the 7:93 and 9:91 of AMO-2/SE, along with the ongoing absorption of water between the cellulose paper and retrofilled oil, leads to an elevated water consumption during the hydrolysis reaction. Therefore, it can be inferred that both AMO condition and the retrofilled oil ratio plays a significant role to the severity of retrofilled oil samples.

B. Fourier Transform Infrared Spectra of Retrofilled Oil

As elucidated in the preceding section (Section 3 (A)), the degradation of mineral and ester oil caused by oxidation and hydrolysis reactions will generate the formation of acid in the oil sample, specifically carboxylic acid [16], [19], [20]. The correlation between the acid number (TAN) in the oil samples and FTIR spectra is determined by identifying characteristic absorption bands that indicate the existence of acidic functional groups in the sample [15]. A Jasco FTIR-6100 was used for analyzing the FTIR spectra, as shown in Fig. 5.


Figure 5. Jasco FTIR-6100

The carboxylic acid content in the retrofilled oil samples of AMO/SE can be assessed by analyzing the carbonyl stretch region (C=O) within FTIR spectra [21], [22], [23]. The growth in intensity of the peak related to C=O bonds often occurs at the wavelength range of 1700 – 1800 cm^{-1} in FTIR spectroscopy [23], [24], [25].

Fig. 6 provides the area of interest within the wavelength range of 1710-1770 cm^{-1} providing clear evidence of the development of peak absorbance at 1739 cm^{-1} , corresponding to the C=O stretch. This reflects the increasing acid concentration in each AMO/SE retrofilled oil sample. At AMO-1/SE, the retrofilled oil samples with oil ratios of 3:97, 5:95, and 7:93 exhibited close peak absorbance, whereas the peak absorbance decreased when the retrofilled oil ratio was increased to 9:91. In contrast to AMO-2/SE, a distinct variation in the increase of peak absorbance is observed across the varied oil ratios. In general, it is evident that the AMO-2/SE retrofilled oil samples exhibit a more rapid increase in acid concentration in comparison to the AMO-1/SE retrofilled oil samples. Fig. 7 distinctly illustrates the correlation between the development of peak absorbance in the C=O stretch at a wavelength of 1735 cm^{-1} and the acid concentration for each retrofilled oil sample of AMO-1/SE and AMO-2/SE.

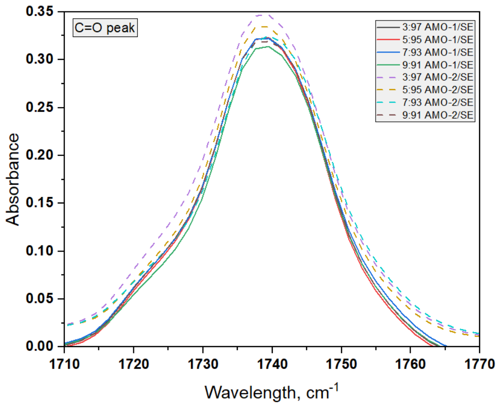
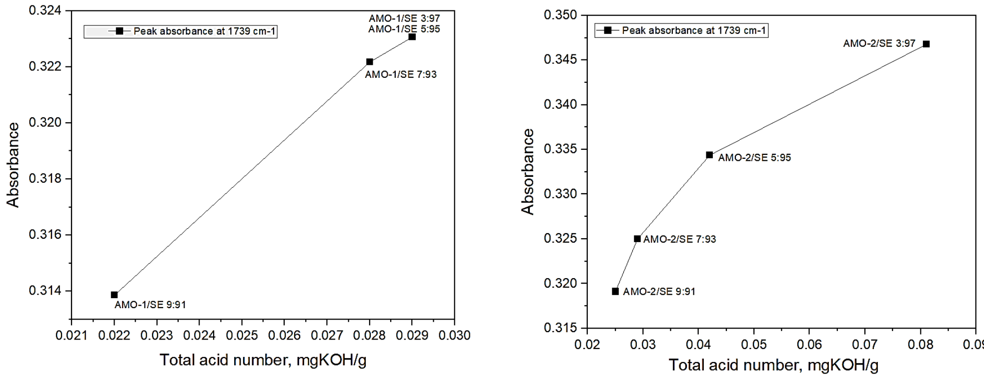


Figure 6. FTIR spectra at wavelength 1710-1770 cm^{-1} of AMO/SE retrofilled oil



(a) Peak absorbance for AMO-1/SE retrofilled oil

(b) Peak absorbance for AMO-2/SE retrofilled oil

Figure 7. Peak absorbance at wavelength 1739 cm^{-1} of AMO/SE retrofilled oil

4. Conclusion

Measuring the TAN value allowed the assessment of the performance of freshly-treated synthetic ester (SE) oil after its mixing with AMO oil. Additionally, further analysis of the chemical composition of the retrofilled oil sample is carried out by correlating it with FTIR spectra.

Prior to retrofilling, the initial steps include oil filtration and nitrogen purging, ensuring the standardization of oil samples and enhancing their performance upon extraction from the oil barrel. The treated mineral oil was then subjected to thermal aging at a temperature of 130 ℃ for durations of 75h and 150h. The AMO oil was prepared in four varying proportions particularly in 3%, 5%, 7% and 9% for retrofill with fresh-treated SE oil.

Various conclusions can be inferred from the obtained findings. A significant finding is that a decrease in the ratio of AMO in the AMO/SE retrofilled oil leads to an increase in the TAN value. This pattern is consistently observed in both AMO conditions. The hydrolysis reaction is believed to be the primary source of the large water consumption observed in 3:97 AMO/SE retrofilled oil. Meanwhile, there is a persistent process of water absorption from the cellulose paper into the oil, supported by the large water in 3:97 AMO/SE retrofilled oil leading to a significant increase in acid concentration. Besides that, it is evident that the severity of AMO conditions give paramount important to the TAN value, whereby the TAN value for AMO-2/SE condition is observed higher than AMO-1/SE.

In context of FTIR spectra, it was discovered that the FTIR spectra aligns with the TAN value. In both AMO conditions, the 3:97 AMO/SE retrofilled oil sample exhibited a more prominent peak absorbance in the carbonyl stretch (C=O) at 1739 cm^{-1} compared to the 5:95, 7:93, and 9:91 AMO/SE retrofilled oil samples. In addition, the severity of the AMO condition has a significant role in influencing the growth of peak absorbance in retrofilled oil. Specifically, it is observed that the retrofilled oil with AMO-2/SE exhibits a greater peak absorbance compared to the retrofilled oil with AMO-1/SE.

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Nur Amirah Othman received the Bachelor degree in Electrical Enginering major in Control, Instrumentation and Automation from Universiti Teknikal Malaysia Melaka (UTeM) in 2014. She obtained her MSc in Electrical Engineering at UTeM in 2017. She is currently pursuing her PhD in Electrical Engineering. Her research focuses on the condition assessment of retrofilling aging transformer with ester oil.

Hidayat Zainuddin received his Bachelor of Engineering in Electrical from the Universiti Teknologi Malaysia in 2003. He obtained his MSc in Electrical Power Engineering with Business from the University of Strathclyde, Glasgow in 2005. He received his Ph.D degree at the University of Southampton in 2013. He has been as academic staff of the University Teknikal Malaysia Melaka (UTeM) since 2003 and at present is a dean in Faculty of Electrical Technology and Engineering, UTeM. His research interests include condition monitoring of high voltage equipment, testing and failure/degradation analysis of insulation/dielectric materials and finite element analysis modelling.

Muhammad Sharil Yahaya is a Senior Lecturer at Faculty of Electrical Technology and Engineering, Universiti Teknikal Malaysia Melaka (UTeM), Malaysia. He received his Ph.D. in Electrical Power Engineering from Universiti Putra Malaysia (UPM), Malaysia. He also received his Master of Science in Power Distribution Engineering in 2009 from Newcastle University, UK and bachelor's degree in electrical engineering in 2001 from Universiti Teknologi Malaysia (UTM), Malaysia. He had 6 years industrial working experience as Application Engineer and Technical Service Engineer. His research interests are predictive modeling, condition monitoring, condition assessment and asset management.

Norhafiz Azis received the B.Eng. degree in electrical and electronic engineering from Universiti Putra Malaysia, Serdang, Malaysia, in 2007, and the Ph.D. degree in electrical power engineering from The University of Manchester, Manchester, U.K. He is currently an Associate Professor with the Department of Electrical and Electronic Engineering, Universiti Putra Malaysia. His research interests are in-service aging of transformer insulation, condition monitoring, asset management, and alternative insulation materials for transformers.

Zulkifli Ibrahim is a Senior Lecturer at Faculty of Electrical Technology and Engineering, Universiti Teknikal Malaysia Melaka (UTeM), Malaysia. He received his Ph.D. in Electrical Power Engineering from Universiti Putra Malaysia (UPM), Malaysia in 2020. He obtained his M.Sc. in Electrical Power Engineering from Technische Universität Darmstadt, Germany in 2011. In 2002, he received his Bachelor of Electrical Power Engineering (Hons.) from Universiti Tenaga Nasional. He has 5 years industrial working experience as power transformer Design Engineer at transformer manufacturing company. He has experienced designing power transformer range from 6.6 kV to 132 kV with power up to 90 MVA. His research interests are transformer thermal modeling, transformer life management (TLM), solid and liquid insulation study.

Mohd Affendi bin Che Musni is a Director, Operations and Technical Service in Power Transcoteq & Services Sdn. Bhd based in Negeri Sembilan, Malaysia. He received his Bachelor of Elecrical Power Engineering from Universiti Tenaga Nasional in 2004. He has 9 years experiences in power transformer business in Operations, Testing, Maintenance and After Sales and Services.Besides that, he has also an experience in Oil & Gas project such as in Petronas.