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New near-infrared absorbance peak for inhibitor content detection in transformer insulating oil

Yang Sing Leong[a], Pin Jern Ker[a,*], M.Z. Jamaludin[a], Saifuddin M. Nomanbhay[a], Aiman Ismail[a], Fairuz Abdullah[a], Hui Mun Looe[b], C. Nuru Saniyyati M. Shukri[b]

[a] Electronics Research Group, Institute of Power Engineering, Universiti Tenaga Nasional, Kajang, Selangor, Malaysia
[b] TNB Research Sdn. Bhd., Bandar Baru Bangi, Selangor, Malaysia

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ABSTRACT

Monitoring the condition of transformer insulating oil has been considered as a crucial and effective measure for preventive maintenance of power transformers. Various properties of the oil can be monitored such as the dissolved gases, furan content, and inhibitor content. This paper focuses on the inhibitor content in insulating oil. Currently, Fourier transform infrared (FTIR) spectroscopy in accordance with the IEC 60666 standard is used for the measurement of inhibitor concentration in insulating oil. However, this technique involves site sampling, transportation to a laboratory and an expensive instrument. This work proposes the characterization of inhibitor content in insulating oil in the near-infrared (NIR) waveband, which would lead to the design of a faster and cheaper detection system for inhibitor content. It was found that the inhibitor content exhibits an optical absorbance peak at 1403 nm, which was not reported in any previous work. A mathematical model was then created to describe the relationship between the concentration of inhibitor, the area under the absorbance spectrum and the peak optical absorbance. The model was verified, and the results showed a root mean square error (RMSE) of 0.0458.

1. Introduction

Preventive maintenance of power transformers is crucial to ensure that the power transformer is operating under optimal conditions. Any early signs of fault detected in the transformer will be addressed so that catastrophic failures can be avoided. Typical preventive maintenance programs consists of three tasks, including routine equipment inspections, maintenance tasks and repairs [1]. One of the maintenance tasks is the monitoring of the condition of the insulating oil in the power transformers [2–4].

Insulating oil, also known as transformer oil, is one of the main components in power transformers. It serves as a coolant to regulate the temperature of the power transformer so that overheating does not occur during operation. Insulating oil also electrically insulates the windings and core due to its good electrical insulation properties. Furthermore, noise due to the vibration of the transformer can be reduced. More importantly, insulating oil acts as an information carrier in that it holds information on the physical and chemical condition of the transformer. After years of service, the insulating oil is subjected to continuous thermal, electrical and mechanical stresses [5–7]. These stresses will change the properties of the oil, thus degrading it. Therefore, monitoring of the insulating oil condition is important to ensure reliable and safe operation of power transformers.

Various properties of insulating oil, such as acidity, color, water content, furan content, interfacial tension (IFT), inhibitor content and dissolved gases [8,9], are commonly monitored during routine maintenance. This paper focuses on the concentration of inhibitor content (*%IC*) in the insulating oil. Inhibitors are organic chemical compounds used to decrease the oxidation process in insulating oil [10]. They are also known as antioxidants or oxidation inhibitors. There are various types of inhibitors for transformer oil such as 2, 6-di-tertiarybutyl phenol (DBP), 2,6-ditertiarybutyl-*para*-cresol (DBPC), 1,2,3-Benzotriazol (BTA), dibenzyl disulfide (DBDS), 2-*tert*-butyl-*p*-cresol (2-t-BPC), *N*-phenyl-1-naphthylamine and methylated-BTA [11]. However, DBPC is the most commonly used inhibitor and is approved universally as a highly desirable inhibitor material with excellent properties [10] that minimize any oxidation process in transformer oil during operation.

It is commonly known that insulating oil is subjected to degradation during operation and that the inhibitors deplete with time. According to the IEC 60422 standard (Mineral insulating oils in electrical equipment – supervision and maintenance guidance), if the inhibitor content drops below 40% of its initial level, either close

* Corresponding author.
E-mail address: pinjern@uniten.edu.my (P.J. Ker).

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Table 1
Summary of the techniques for the measurement of inhibitor concentration based on IEC60666 [13].

Techniques	FTIR Spectroscopy	HPLC	GC–MS
Test Method	• Does not require trained personnel to conduct experiment • Need to prepare reference sample by solid phase extraction using silica gel • Non-destructive test	• Require trained personnel to conduct experiment • Complex sample preparation required. • Destructive test	• Require trained personnel to conduct experiment • Sample preparation required • Complex experimental setup • Destructive Test
Precision	• Unable to detect trace levels of inhibitor in insulating oil • Results can be affected for used oil samples due to oxidation by-products in oil	• Capable of detecting trace levels of inhibitors in insulating oil • Results can be affected during sample preparation.	• Capable of detecting trace levels of inhibitors in insulating oil • Results can be affected during sample preparation

observation of the transformer is needed or additional inhibitor must be added [8]. These actions are necessary because once the inhibitor amount is below the minimum level, the oil starts to degrade at a higher rate. To increase the useful life of the insulating oil to an acceptable period of time, monitoring and replenishment of inhibitor content in insulating oil must be carried out [10].

The ASTM D2668 (Standard Test Method for DBPC and DBP in Electrical Insulating Oil by Infrared Absorption) and IEC60666 (Detection and determination of specified additives in mineral insulating oils) standards are commonly used for the detection and measurement of the concentration of inhibitor content in insulating oil [9,12]. The ASTM D2668 standard covers the determination of inhibitor content using Fourier transform infrared (FTIR) spectroscopy, while the IEC60666 standard covers several techniques to determine the concentration of inhibitor content such as FTIR spectrophotometry, high-performance liquid chromatography (HPLC) and gas chromatography-mass spectrometry (GC–MS) [12,21]. Table 1 shows the comparison of each technique described in the IEC 60666 standard.

Among the three techniques, FTIR spectroscopy is most commonly used for measuring the *%IC* in insulating oil for routine maintenance. Although the technique lacks the precision to measure inhibitor content at the trace level, it does not require trained personnel to conduct complex sample preparation and measurement, which saves time and cost. Furthermore, it is a non-destructive test, as the technique involves optical spectroscopy.

Optical spectroscopy studies the interaction between matter and electromagnetic (EM) radiation. When an EM wave interacts with matter, the matter absorbs, reflects, refracts, diffuses or even emits EM waves of different wavelengths. Depending on the objectives of a research study, different properties are investigated. Recently, optical spectroscopy has been popular among researchers in exploring new techniques for monitoring the condition of transformer oil [14–20]. In this paper, optical absorption by the inhibitor content is investigated. Theoretically, each substance will have unique spectral properties that are discernible from the properties of all others [21]. The region of detection used to determine the concentration of DBPC inhibitor content is the phenolic OH stretch at a wavenumber of 3650 cm^{-1} (equivalent to a wavelength of 2739.73 nm) [13].

However, this FTIR spectroscopy technique still requires relatively expensive equipment, and oil sampling with transportation to a laboratory is still necessary, which incurs additional running cost. Therefore, a more convenient and affordable method is needed. Thus, this paper reports on the optical characterization of inhibitor content in the NIR region, which enables the possibility of designing a cost-effective and portable detection system. A mathematical model that describes the relationship between the optical response of the inhibitor in the NIR region and the *%IC* was formulated and verified.

2. Experiment details

In this research work, a set of transformer oil samples was taken from 40 different operating power transformers. Each sample was collected and transported properly in accordance with the IEC 60475 standard [22]. This procedure requires careful handling of the samples to ensure that there is no contamination or modification of the samples that will jeopardize the analysis. The oil samples were collected in two amber glass bottles, of which one was sent to the accredited laboratory for inhibitor content analysis using FTIR in accordance to the IEC 60666 standard, while the other bottle was analyzed using a laboratory-based NIR spectrophotometer. This method was carried out to ensure that there was no large time gap between the accredited laboratory analysis and the experiment conducted in this study. In the NIR spectrophotometer study, oil samples were divided into two sets: 70% of the oil samples tested with inhibitor content were used for the mathematical modeling, while the remaining 30% of the oil samples were used for verification of the model.

The laboratory-based NIR spectrophotometer used was the Agilent Cary5000 UV–vis-IR spectrophotometer. The Cary5000 is a high-performance double-beam spectrophotometer with good photometric performance in the 175–3300 nm range. Fig. 1 shows the basic working principles of the double-beam spectroscopy.

Radiation from the light source passes first through a monochromator and a slit so that only a narrow band of light passes through. The light beam is then focused on a switching disk. The role of the switching disk is to switch among 3 distinct positions, for which light will either pass through, be reflected or blocked. At the first position, the light beam passes through the disk, directly shines onto a 1-cm path-length quartz cuvette containing the oil samples (Sample cell), and then reaches the detector for measurement. The disk subsequently switches to the second position, for which the light beam shines on the mirror surface to be reflected at 90°. The light then shines through another 1-cm path-length quartz cuvette containing the reference sample (reference cell) and then reaches the detector for measurement. In this experiment, clean, new uninhibited transformer oil was used as the reference sample. Finally, the disk switches to the third position, for which the light beam strikes the black surface, thus ensuring that no light passes through the disk to reach the detector. This part of the cycle is essential for the instrument to measure the dark current so that it can be subtracted from the overall light measurement made by the system. For every measured wavelength, there will be three measurements taken at the three different positions of the switching disk. The process is repeated until it covers the entire range of wavelengths.

The measured results were recorded as transmittance and then converted to absorbance values by applying the Beer-Lambert's Law [24] as in Eq. (1).

$$Abs = -\log_{10}(S_i - B_i/R_i - B_i) = \varepsilon_\lambda \cdot c \cdot l \quad (1)$$

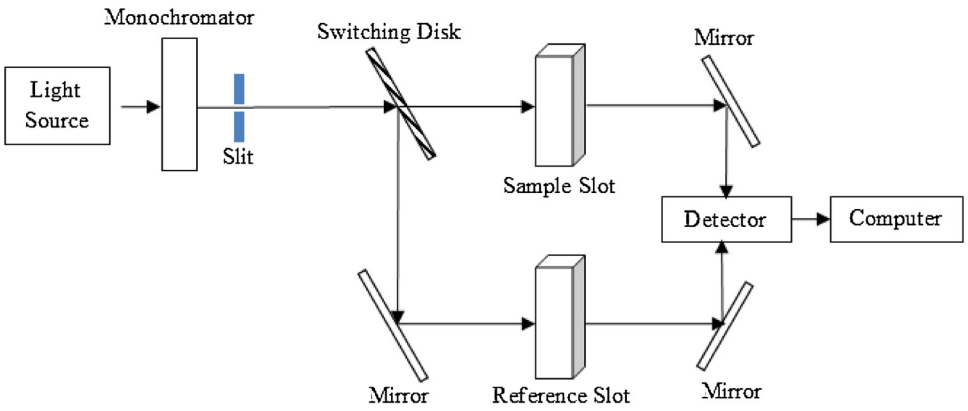

Fig. 1. Simple block diagram of a double-beam spectrophotometer [23].

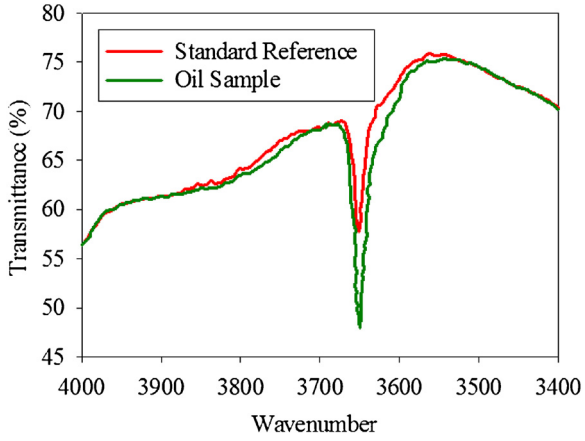

Fig. 2. An example optical spectrum of a standard reference and of an oil sample from FTIR measurements.

where *Abs* is the optical absorbance, S_i is the transmittance of light passing through the sample in the sample slot, R_i is the transmittance of light passing through the sample in the reference slot, B_i is the baseline, ε_λ is the absorbance coefficient of the absorbing sample at a certain wavelength, c is the concentration of the absorbing sample, and l is the path-length traversed by the light.

In this study, the collected oil samples were measured in the range of 200 nm–3300 nm to obtain their respective absorbance spectra. Then, both the IR absorbance spectra and the respective data from laboratory analysis using FTIR were compared for data analysis.

3. Results

3.1. FTIR spectroscopy using the conventional FTIR method

Conventionally, the inhibitor content in transformer oil is measured using FTIR spectroscopy based on the IEC 60666 standard. Before measuring the oil sample, a reference sample is scanned for calibration purposes. The FTIR spectra of both the reference sample and oil sample are recorded in terms of transmittance, as shown in Fig. 2.

Different transmittance values correspond to different values of *%IC* in transformer oil depending on the calibration of the instrument. Based on the results from FTIR spectroscopy, 11 samples out of 40 collected samples were found to contain inhibitor, while the other samples had zero or non-traceable inhibitor content. Table 2 shows the transmittance, derived absorbance values based on Eq. (1) and their corresponding inhibitor concentrations for 11 samples with inhibitor content based on the results of FTIR. The same sample labeling was used in the following sections.

Table 2
Summary of the transmittance, absorbance and inhibitor concentrations for 11 samples.

Sample	Inhibitor Content Concentration	Transmittance	Absorbance
1	0.23	61.3%	0.2125
2	0.60	47.8%	0.3206
3	0.33	57.1%	0.2434
4	0.22	61.5%	0.2111
5	0.26	59.5%	0.2255
6	0.42	54.1%	0.2668
7	0.12	64.8%	0.1884
8	0.28	59.1%	0.2284
9	0.59	48.1%	0.3179
10	0.65	45.8%	0.3391
11	0.29	58.8%	0.2306

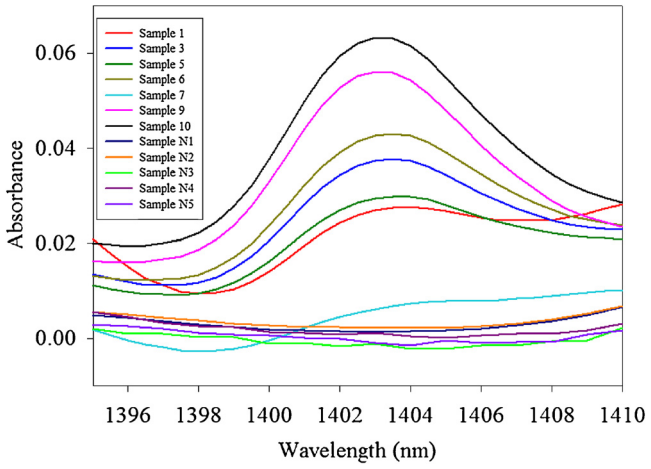

Fig. 3. Optical absorbance spectra of several oil samples with different concentrations of inhibitor content.

3.2. NIR resonant wavelength

After carrying out intensive data analysis comparing the obtained absorbance spectra and data from the laboratory analysis using FTIR, it was found that the samples with inhibitor content also exhibit a peak absorbance at the wavelength of 1403 nm, as shown in Fig. 3.

Samples 1, 3, 5–7, 9 and 10 contain different amount of inhibitor content, while samples N1–N5 have zero or non-traceable inhibitor content by the conventional method. To verify that the optical absorbance at the wavelength of 1403 nm is due to the inhibitor

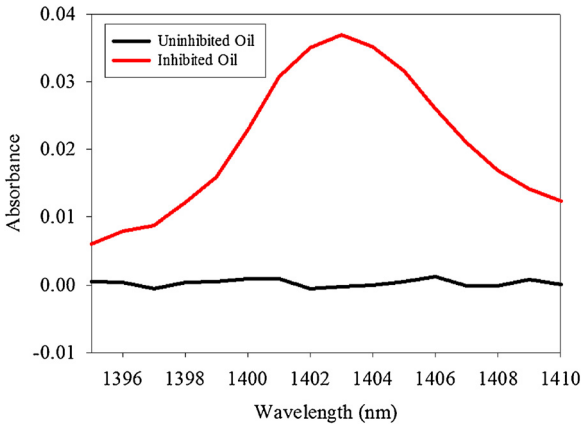

Fig. 4. Optical absorbance spectra of uninhibited and inhibited transformer oil.

content of the oil, two new oil samples of the same brand were tested as a control. One of these is IEC 60296-accredited uninhibited oil, which contains no inhibitor, while the other is IEC 60296-accredited inhibited oil, which contains inhibitor at weight percentage of 0.25. Fig. 4 shows the absorption spectra of both uninhibited and inhibited insulating oil measured using UV–vis-IR spectrophotometer. Fig. 4 clearly shows the same peak absorption response as observed for the collected samples. This result proves that 1403 nm is the resonant absorption peak of the inhibitor.

Further inspection of Fig. 3 reveals a good correlation between the absorbance spectra and the inhibitor concentration. As the concentration of inhibitor increases, the peak absorbance at 1403 nm (Abs_{1403nm}) and the area under the graph from 1398 nm to 1408 nm (*Area*) also increases, as shown in Fig. 5(a) and (b).

3.3. Mathematical modeling

A mathematical model based on the results can be used to describe the relationship between the spectral response and the concentration of inhibitor. The model was formed using linear regression on three parameters, *%IC*, Abs_{1403nm}, and *Area*. A 3-dimensional plot was generated to study the relationship between the Abs_{1403nm}, *%IC* and *Area* values, as shown in Fig. 6, along with the regression plane for 7 oil samples. The residual plot of the 3-dimensional plot contains data points randomly dispersed around the horizontal axis, indicating that linear regression is appropriate for the data.

Based on the results of the linear plane regression analysis, the mathematical model that describes the relationship between the *%IC*, Abs_{1403nm} and *Area* values was formed as shown in Eq. (2).

$$\%IC = 0.065 + 55.87Abs_{1403\,nm} - 6.313Area \quad (2)$$

3.4. Model verification

To validate the mathematical model of Eq. (2), the optical absorbance of the remaining 4 collected oil samples was measured. Fig. 7 shows the absorbance spectral response of the 4 oil samples.

For the verification process, the Abs_{1403nm} and *Area* of the 4 oil samples were determined and used to estimate the *%IC* of the oil samples using Eq. (2). The estimated *%IC* values were then compared with the actual *%IC* values obtained using FTIR spectroscopy as shown in Table 3.

Based on Table 3, it can be observed that the average percentage difference between the actual value and estimated value is 10.74%.

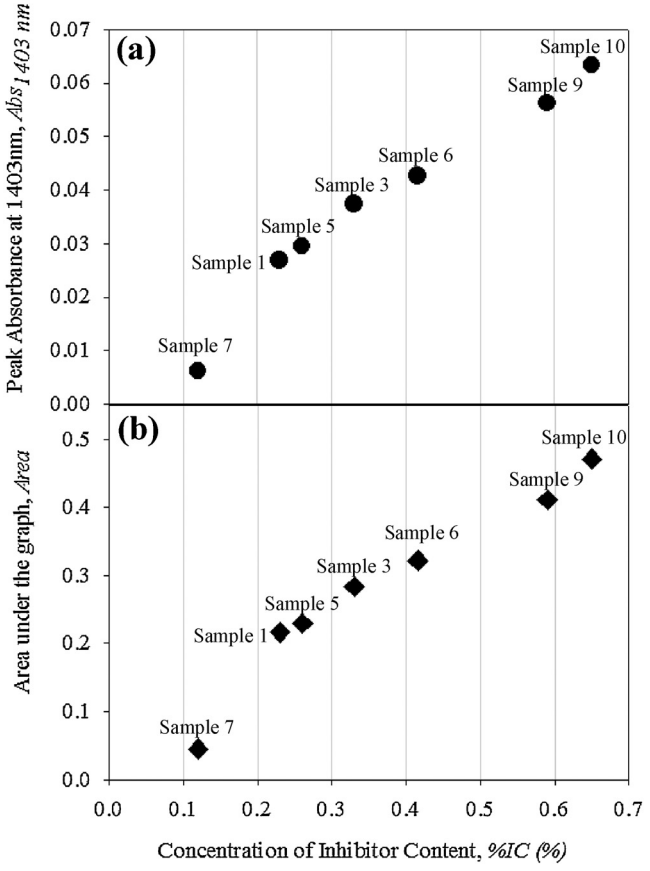

Fig. 5. (a) Peak absorbance at 1403 nm versus *%IC* in oil samples. (b) Area under the graph versus *%IC* in oil samples.

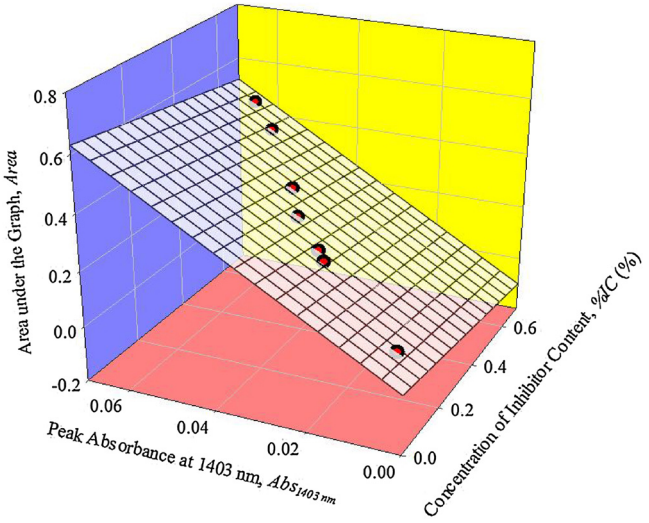

Fig. 6. Graph of *%IC* of transformer oil samples vs. Abs_{1403nm} vs *Area* (Red circles with black borders) with linear plane regression (White plane). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 3
Comparison between the measured and calculated *%IC* of 4 samples for verification.

Sample	Actual *%IC*	Estimated *%IC*	Difference in *%IC*[a]	Percentage Difference
2	0.60	0.681	0.081	13.33%
4	0.22	0.188	−0.032	12.14%
8	0.28	0.312	0.032	10.54%
11	0.29	0.268	−0.022	6.96%

[a] Difference in *%IC* = Estimated *%IC* − Actual *%IC*.

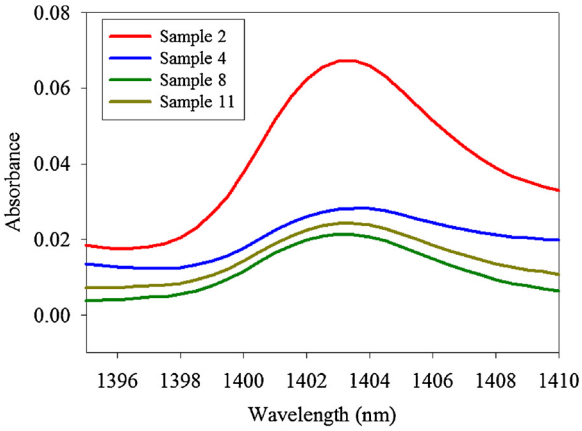

Fig. 7. Optical absorbance spectra of oil samples for verification.

The RMSE between the actual *%IC* value, $\hat{y}$, and the estimated *%IC* value, y, was calculated using Eq. (3).

$$\text{RMSE} = \sqrt{\frac{1}{n}\sum_{i=1}^{n}\left(\hat{y}_i - y_i\right)^2} \tag{3}$$

where n is the total number of samples.

Based on Table 3, the RMSE for the 4 oil samples is 0.0458. The considerably high average percentage difference is attributed to the fact that the model was produced using only 7 samples. It is expected that by increasing the data population, more accurate constants in Eq. (2) can be calculated, which will increase the accuracy of the model and reduce the RMSE.

4. Discussions

The focus of this study is to explore and discover new, unreported absorbance maxima in the NIR range due to inhibitor content. According to previously reported works [13,25–28], the inhibitor (specifically, DBPC) can be detected at a wavelength of 2739.73 nm, or 3650 cm^{-1} wavenumber, due to the phenolic OH stretch. These existing data lead researchers to carry out measurements using FTIR spectroscopy. However, based on the results obtained, a new absorption wavelength of the inhibitor was discovered to occur at 1403 nm. The preliminary results of a reported work also demonstrate the same optical properties of the inhibitor content at this NIR wavelength [29]. Since there is a good correlation between the spectral response and the *%IC*, a mathematical model was formulated. However, its accuracy can be further improved if a larger number of samples with inhibitor content are measured.

The discovery of the new NIR absorbance wavelength for detecting the inhibitor content in insulating oil provides new possibilities for its detection. Since 1403 nm is within the telecommunication waveband, obtaining a light source and a photodetector operating at 1403 nm at a reasonably low cost is more easily achievable. Furthermore, this finding also opens up the possibility of producing a cheap and portable handheld detection device that can be used for on-site maintenance. The portable optical sensing device would also be able to reduce the transformer maintenance cost.

DBPC inhibitor is also commonly added to mineral oil-based lubricants such as turbine oils, hydraulic oils, gear oils, compressor oils and crankcase oils [27]. Thus, the discovery of the new NIR absorbance wavelength could also be very useful in determining the concentration of inhibitor content in other mineral oil-based lubricants besides transformer oil.

In addition to its role in lubricants, DBPC, which is also known as Butylated Hydroxytoluene (BHT) [30,31], is very commonly used in plastics applications and production [32,33]. An online optical monitoring system using the NIR absorbance wavelength can also be developed to monitor the BHT level in plastic products, such as plastic water bottles, during the production process.

Finally, BHT is also used in food [34] and cosmetic [35] products as an antioxidant. Although BHT is considered to be safe to use in food and cosmetic products and does not cause significant damage to the human body [34,35], its concentration in these products is still relatively low. For example, the allowable range of BHT concentration in cosmetic products is from 0.0002% to 0.5% [35]. The BHT concentration must be monitored carefully so that it does not exceed the acceptable limit. Thus, the discovery of the new NIR absorbance wavelength could also be very useful in monitoring the concentration of BHT in food and cosmetic products. However, the detection system needs to be designed with very high sensitivity to detect the very low concentration of BHT.

5. Conclusion

This work was carried out to explore the characteristics of insulating oil content using optical detection focusing specifically on the detection and measurement of inhibitor content in the insulating oil using optical spectroscopy. The existing method of measuring the concentration of inhibitor content in insulating oil was first investigated. This conventional technique, which involves oil sampling and transportation, incurs additional operating cost and is time consuming. In addition, an expert is needed to operate the expensive laboratory-based instrumentation, which also requires periodic maintenance. Therefore, this work explored a new NIR wavelength that is suitable for the design of a portable handheld device for the determination of the inhibitor content in insulating oil.

The results have identified a new wavelength corresponding to absorption by the inhibitor, which was discovered to be 1403 nm. The new wavelength was verified. A model that describes the relationship between the concentration of inhibitor, the area under the absorbance spectrum and the peak optical absorbance was derived. Despite the limited number of samples used in the mathematical modeling, the modeled equation produced reasonably good results with a RMSE of 0.0458.

This proposed technique has opened up new opportunities in the detection of inhibitor content using optical techniques. It demonstrates the possibility of a cost-effective and portable device that can be used for on-site measurement, resulting in lower maintenance cost.

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Biographies

Yang Sing Leong received the B.Eng. (Hons) degree in electrical and electronic engineering from the Universiti Tenaga Nasional, Malaysia in 2016. Currently, he is research assistance and a Master student at Universiti Tenaga Nasional. His research interest is optical sensing and monitoring.

Pin Jern Ker received the B.Eng. (Hons.) degree in electrical and electronic engineering from the Universiti Tenaga Nasional (UNITEN), Malaysia in 2009, and the Ph.D. degree in electronic and electrical engineering at The University of Sheffield, U.K. Currently, he is a senior lecturer at the Department of Electrical Power Engineering, UNITEN. He is also the Head of Unit (Electronics & IT) at the Institute of Power Engineering, a research institute of UNITEN. His research interests are the simulation and characterization of photodetectors, optical sensing, design of monitoring and control system for energy-related applications.

Md Zaini Jamaludin received his Diploma in Electrical and Electronic Engineering from Institute Technology Mara (know University Technology Mara, UiTM) in 1983, the B.Sc. degree in electrical engineering from University of Miami, Florida, USA, in 1986, and M.Sc. in Electronic (Medical System) from University of Hertfordshire, UK, in 1994 and Ph.D. in Network Communication Engineering from Universiti Putra Malaysia 2007. He worked in Motorola Malaysia Sdn. Bhd in 1984 and 1986 as Assistant Engineer and Product Engineer respectively. He then joined UiTM in 1990, as Lecturer in the Faculty of Electrical Engineering. In 1997 he joined GITN Sdn. Bhd, the Malaysian e-government network service provider as a senior executive in the network security department. Joint Digicert Sdn. Bhd to set-up the first Certification Authority, company that issue the Digital Certificate for secured on-line transaction and appointed the Chief Operating Officer in 2000. He then joined the Department of Electronics and Communication Engineering, College of Engineering, Universiti Tenaga Nasional in 2001 as a Senior Lecturer. He was seconded to UNITEN R&D Sdn Bhd to setup the Research Company from 2008 until 2107 and hold the post of Managing Director since 2013 until 2017. He is currently a Professor, research focus on Photonics devices, Optical network, secured remote data acquisition systems, RF radiation, and Ethernet Passive Optical Networks. He is an active researcher with more than RM4.0 million worth of research grants secured from various Research funding and agencies such as eScience Fund, IRPA, TNBR and MCMC. He has authored and coauthored more than 130 research papers in journals and conference proceedings. Professor Md Zaini Jamaludin is a Senior Member of IEEE, has been an active executive committee member for of IEEE Photonics Society, including as Chairman in 2007–2008, and member of IEEE Malaysia, for the past 13 years.

Saifuddin M. Nomanbhay has been actively involved in the research field of microwave assisted reactions, biocatalyst, microwave pyrolysis, and advanced biodiesel/bioethanol production in the last 18 years at Universiti Tenaga Nasional (UNITEN). He is currently the principal researcher and professor at the Institute of Sustainable Energy (ISE) of UNITEN. His research now is more focus on renewable resources and enabling technologies using microwave technology and microwave driven plasma catalytic and non-catalytic reactions.

Aiman Ismail received his Bachelor in Electrical and Electronics Engineering and Masters in Electrical Engineering from Universiti Tenaga Nasional, Malaysia. He is now a lecturer at the Department of Electronics and Communication Engineering, Universiti Tenaga Nasional. His research interests are in Optical Sensors, Fiber Optic Sensors and Fiber Lasers.

Fairuz Abdullah received the B.Eng. degree in electronics from Universiti Tenaga Nasional, Malaysia, in 2001. He then completed M.Sc. in network and communication engineering from Universiti Putra Malaysia in 2004 and his doctoral degree in fiber laser sensor from Universiti Tenaga Nasional in 2012. He is currently an Associate Professor in Department of Electronics & Communication Engineering, College of Engineering, Universiti Tenaga Nasional and seconded to Innovation & Research Management Centre as Head of Unit, Intellectual Properties & Commercialisation Unit. He is also the head of Photonic Technologies Research Group focusing on optical communications, fiber lasers, amplifiers and sensors mainly for application in power enginering. To date, he has published more than 70 journal and conference papers. His research interests include fibre amplifiers, fiber lasers and optical sensors. He is also a senior member of IEEE Photonic Society and has been actively involve since 2003.

Looe Hui Mun graduated with a B.Eng (Hons) degree in 1997 and a PhD degree in 2004 from the Department of Electrical Engineering and Electronics, University of Liverpool, UK. He worked as a post-doctoral research associate at the University of Liverpool where he was with the Centre for Intelligent Monitoring System. Dr. Looe returned to Malaysia in 2013 under Returning Expert Programme offered by the government to contribute in the electrical supply industry. Currently, he is a principal researcher at the Smart Grid Unit of TNB Research Sdn. Bhd., which is a wholly subsidiary and in-house solution provider for Tenaga Nasional Berhad (TNB). He also received the Chartered Engineer (CEng) from the Institution of Engineering and Technology (IET), UK in 2014. His research interests include the development of data-driven diagnostic methods for industrial and electrical engineering applications, smart substation technology, sensor management and analytics.

Che Nuru Saniyyati received B.Eng (Hons) degree in Electrical Engineering from Universiti Teknologi Malaysia (UTM), Skudai Malaysia in 2012, and Master Degree in Electrical Engineering from UTM SKudai Malaysia in 2014. Currently, she is a researcher at TNB Research Sdn Bhd. Her research interests are Energy Efficiency which includes Volt-VAR Optimization, Energy Storage, Demand response and Energy efficiency applications.