ASSESSING ELECTRICAL AND PHYSICOCHEMICAL PERFORMANCE OF CHEMICALLY MODIFIED PALM OIL AS AN ALTERNATIVE TRANSFORMER LIQUID

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ABSTRACT
Numerous studies have been conducted on enhancing the dielectric properties of palm oil (PO) with the addition of nanoparticles (NPs). However, insufficient emphasis was given to its physicochemical properties, which are also critical for the function of an insulating liquid. The high viscosity of PO is one of the key factors which deteriorates the usage of natural ester in general, and PO in our case. This study was intended to investigate the performance of chemically modified PO by adopting a sequential transesterification process – NPs addition. The present work explored the transesterified PO blended with insulative (SiO2) and semiconductive (ZnO) NPs at various concentrations (0.1-0.6 g L–1). The modified oils’ dielectric strength and physicochemical properties (density, kinematic viscosity, and water content) were measured using standard testing methods. The results showed that the transesterification process effectively reduced the viscosity of PO, and adding ZnO NPs substantially impacted the PO in terms of AC breakdown voltage, with an optimum performance at 0.2 g L–1 concentration of 85% improvement. In contrast, while using SiO2, its breakdown voltage decreased irrespective of the concentration.

Keywords: chemically modification, nanoparticles, physicochemical properties, transesterification.

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INTRODUCTION
For decades, petroleum-based mineral oil (MO) has been utilised conventionally as a transformer insulating liquid. However, the usage of MO derived from non-renewable energy resources is affecting the environment due to its non-biodegradable property. As a result, researchers shifted their interest to biodegradable and renewable alternatives. Most research in biodegradable oils for transformer applications has focused on natural ester oils (NEOs) or vegetable oils (VOs). In the late 1990s, ABB developed the first commercial electrical insulating oil, BIOTEMP®, a sunflower oil-based (Boss and Oommen, 1999). In 2000, McShane et al. (2000) of Cooper Industries Inc. introduced Envirotemp FR3™ derived from soybean oil, and later, in 2008, Lion Corporation of Japan successfully developed Palm Fatty Acid Ester (PFAE) with the brand name Pastell Neo®, as potential natural ester (NE) insulating liquids for the transformers. VOs are renewable, cost savings, environmentally friendly, biodegradable, and safer alternative insulating oil (Amin et al., 2019). Due to the advantages mentioned above, VO is considered a substitute.
The wealth of literature conducted with the above NEOs has motivated researchers from other countries to begin researching their plant-based insulating liquids that utilise available and accessible renewable and biodegradable energy resources. Several plant oils, such as palm oil, pongamia pinnata oil, olive oil, marula oil, coconut oil, etc., have been used as the raw materials. Their potential suitability as transformer oils has been investigated and examined thoroughly by many researchers (Ariffin et al., 2017; Baruah et al., 2019; Farade et al., 2020; Genga Devi et al., 2016; Koutras et al., 2020a; Oparanti et al., 2020; Samikannu et al., 2021).

Researchers eventually realised that VO-based liquid insulation needed various modifications to overcome its shortcomings and be employed as the insulating liquid in transformers (Moosasait and Maria Siluvairaj, 2021). One of the issues with VOs is their high viscosity, which undoubtedly reduces their efficacy as liquid insulation, making them unsuitable for cooling purposes (Raeisian et al., 2019; Yusof et al., 2017).

Over the years, researchers have successfully modified VOs for use as transformer liquid (Menkiti et al., 2017). Many studies have recognised the chemical modification of NEOs to lower their viscosities and make them comparable to that of MO for transformer application (Baruah et al., 2019). These studies have used various methods and base fluids, as summarised in Table 1. These methods are direct purification, esterification, transesterification, and epoxidation-esterification methods. Among all, transesterification is one of the options that has gained popularity, similar to the production of biodiesel as performed by various researchers (Ishola et al., 2020; Venugopal et al., 2021).

In addition, modifications are attempted on the base fluids to achieve superior thermal and dielectric properties by introducing nanoparticles (NPs), resulting in a nanofluid (NF) mixture. The physical, chemical, and thermal characteristics of dispersed NPs, along with the base fluid, as well as their weight or volume concentrations (Pyrgioti et al., 2020), have impacted the improvement and performance of NFs (Hussain et al., 2020). The influence of conductive (Fe$_3$O$_4$, Fe$_2$O$_3$), semiconductive (TiO$_2$, ZnO, CuO) and insulative (SiO$_2$, Al$_2$O$_3$) NPs on the dielectric and physicochemical properties of biodegradable insulating liquids have been extensively studied in the literature. Koutras et al. (2020b) reported that the addition of TiO$_2$ NPs into FR3 enhanced alternating current breakdown voltage (AC BDV) by 22.40% at 0.02 vol% concentration. Oparanti et al. (2021) examined the effects of dispersing TiO$_2$ and Al$_2$O$_3$ NPs into palm kernel oil (PKO) on the physicochemical properties, dielectric response, and BDV. The results showed that the dispersion of these two types of NPs enhanced the physicochemical properties of the

### Table 1. Summary of Literature Related to Methyl Ester Oils as Alternative Transformer Liquid

| Modification method                  | References                          | Methyl ester oils                               | Performance                                      |
|--------------------------------------|-------------------------------------|-------------------------------------------------|-------------------------------------------------|
|                                      |                                     | Performance                                     | Dielectric strength [kV] | Kinematic viscosity (40°C) [mm$^2$ s$^{-1}$ or cSt] |
| Direct purification                   | Menkiti et al. (2017)               | Modified *Terminalia catappa* kernel oil (MTCKO)|                       |                                                       |
|                                       |                                     |                                                 | 32.88                                           | 15.22                                                  |
| Esterification                       | Sitorus et al. (2016)               | *Jatropha* methyl ester oil (JMEO)              | 87.00                                           | 10.45                                                  |
| Transesterification                  | Maharana et al. (2018)              | *Karanji* oil methyl ester (KOME)               | 85.40                                           | 12.00                                                  |
|                                      | Agu et al. (2019)                   | Modified *T. catappa* kernel oil (MTCKO)       | 48.55                                           | 10.29                                                  |
|                                       | Nkoutetcha et al. (2019)            | *Castor* oil methyl ester (COME)                | 74.67                                           | 18.42                                                  |
|                                       | Ravulapalli et al. (2019)           | Methyl ester *sesbania* seeds oil (MESSO)      | n/a                                             | 0.98 × 10$^{-11}$                                      |
|                                       | Asse et al. (2022)                  | *Palm* kernel oil methyl ester (PKOME)          | 32.22                                           | n/a                                                    |
|                                       | Oparanti et al. (2022)              | Neem oil ester                                  | n/a                                             | 5.17                                                   |
| Epoxidation-esterification           | Abdelmalik et al. (2011)            | *Palm* kernel oil epoxy methyl ester (PKOEME)  | 45.58                                           | 6.14                                                   |
|                                       | Agu et al. (2019)                   | Modified *T. catappa* kernel oil (MTCKO)       | 50.05                                           | 8.56                                                   |
| Blending with fatty acid or fatty esters | Mohd et al. (2021)                | Blended RBDPO olein                             | 76.80                                           | 2.48 - 5.12 (depends on types of fatty acid or fatty esters) |

Note: Recommended specification for natural ester (IEEE C57.147:2018) – Dielectric strength ≥35 kV, and kinematic viscosity ≤50 cSt. n/a – not available.
PKO. In other studies, for instance, Raj et al. (2021) reported the excellent performance of marula oil blended with Al2O3 by showing up to 64.00% on its breakdown voltage (BDV) enhancement, and Duzkaya and Beroual (2021) reported the increment of AC BDV of FR3 blended with ZnO at 0.1 to 0.3 g L⁻¹ concentrations. On the other hand, some types of NPs do not constantly improve the dielectric strength of NEOs, which others have also reported. For example, dispersion of SiO2 into commercial NEOs, MIDEL 1204 (Khaled and Beroual, 2019) and FR3 (Charalampakos et al., 2017) demonstrated a reduction of their dielectric strengths.

Similarly, some research studies reported the effects of NPs on the dielectric properties of PO, as demonstrated by Makmud et al. (2018) and Nor et al. (2017). The authors dispersed TiO2 NPs at various concentrations and reported an apparent enhancement in the dielectric breakdown strength of the NFs. Also, Hussin et al. (2021) and Saenkhumwong and Sukrri (2017) demonstrated an improvement in PO’s BDV using TiO2 and ZnO. Furthermore, Mohamad et al. (2019) compared the effects of Fe3O4, CuO, and Al2O3 with the presence of surfactant on the dielectric strength of PO. They also reported the enhancement of the BDV at a specific volume concentration. Meanwhile, SiO2 (Yahya and Amirrazzi, 2018), SiC (Yahya and Maliki, 2018), and carbon nanotubes (CNTs) (SUHAIMI, 2018) NPs caused the decrement of BDV of PO. From the literature, emphasis has been focused primarily on the dielectric characteristics while ignoring the physicochemical properties of PO. Consequently, in this present study, the authors investigate the chemically modified PO through the sequential transesterification process—NPs addition, for possible use as an alternative transformer liquid. The primary goal of this work is to examine the impact of sequentially optimising the physicochemical properties and addition of NPs on the transesterified refined, bleached, and deodorised palm oil (RBDPO) olein, known as palm oil methyl ester (POME). The characterisation of dielectric strength and physicochemical properties such as density, kinematic viscosity, and water content has been performed to determine the performance of the POME-based NFs.

MATERIALS AND METHODS

Materials

The RBDPO olein used was manufactured by Lam Soon Edible Oils Sdn. Bhd. and purchased from a local grocery store. It was used as received without filtration and purification, and methyl ester was synthesised from it. The SiO2 and ZnO NPs manufactured by Sigma-Aldrich were used to prepare the NFs. In addition, pure methanol (99.9%) was purchased from Merck. Pure potassium hydroxide (KOH) in the pellet form from the J. T. Baker was used as a catalyst, readily available in the laboratory.

Synthesis of POME by Transesterification Process

The RBDPO olein was transformed into POME via the transesterification process, as shown in Figure 1. The RBDPO olein (1000.0 g, 1.182 mol) and methanol (CH3OH) (227.3 g, 7.094 mol) were placed in separate beakers. In this work, methanol was particularly preferred to produce methyl ester because of its availability, cost, and physical and chemical advantages for quick reaction with triglycerides (Encinar et al., 2010; Musa, 2016). Then, KOH as a catalyst was added to the methanol beaker in the pre-determined amounts for the experiment and stirred with the magnetic stirrer until KOH pellets were fully dissolved. The second beaker containing RBDPO olein was heated to 60°C. The methanol and KOH mixture was poured into the RBDPO olein, and both mixtures were heated to 60°C for 60 min. The reaction was conducted using the recommended methanol; oil molar ratio of 6:1 (Baroutian et al., 2009; Musa, 2016). The ratio is universally accepted and often used to drive the reaction to the product side, as an incomplete reaction was observed for molar ratios below 6:1, and a higher molar ratios increase the cost to recover the excess alcohol after the completion of the reaction (Encinar et al., 2010).

The mixture was allowed to cool to ambient temperature before being transferred to a separatory funnel for separation. Glycerol and methyl ester were split into two layers when the transesterification process was completed. The bottom layer, which is the mixture of crude glycerol and KOH, was drained off. The upper layer, the fatty acid methyl ester (FAME), was then washed with warm distilled water several times to remove excess KOH until it achieved a neutral pH level. It was then heated and stirred at a temperature of 60°C for 30 min to eliminate the remaining water molecules and methanol. POME was used as the base fluid for preparing the NFs.

Preparation of POME-based NF Samples

In this study, the NFs were prepared using the two-step method. The NPs were heated in an oven to remove any moisture from the powder particles. Then, the pre-treated NPs were dispersed into the POME base fluid at different concentrations ranging from 0.1-0.6 g L⁻¹ and stirred using a magnetic stirrer for 30 min at 800 rpm. The concentration span provides a range to allow investigation of the optimal dielectric strength and physicochemical properties of the NFs. Figure 2 is the flow chart of the POME-based NFs preparation in the laboratory.
Then, the NF samples underwent an ultrasonicication for a 2 hr procedure to break up any agglomeration of NPs. This ultrasonic procedure using an ultrasonic cleaner with a heating power of 100 W and a frequency of 40 kHz was applied for 2 hr in intervals of 30 min of agitation with a 10 min waiting period between to avoid overheating of the NFs. The two-step method helped minimise the possibility of agglomeration resulting from the covalent bond between NPs, which induced NPs to be settled. After the drying procedure, a total of six NF oil samples having concentrations of: 0.1 g L⁻¹ SiO₂, 0.3 g L⁻¹ SiO₂, 0.5 g L⁻¹ SiO₂, 0.2 g L⁻¹ ZnO, 0.4 g L⁻¹ ZnO, and 0.6 g L⁻¹ ZnO were prepared for the subsequent experimental tests and analysis.

Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS) and Fourier Transform Infrared-Attenuated Total Reflection (FTIR-ATR) Characterisation

The characterisation of NPs was studied using SEM integrated with EDS analysis, taken with a JEOL JSM - IT500HR Electron Microscope operated
at 20 kV. On the other hand, the characterisation of pure POME and POME-based NFs samples was studied using FTIR-ATR analysis. The observed spectra were the absorbance of the differently prepared oil samples versus the wavenumber range of 4000-400 cm⁻¹.

Electrical and Physicochemical Characterisation

This study investigated the influence of SiO₂ and ZnO NPs, both in various sizes and at varying concentrations on the dielectric strength and physicochemical properties of POME. The physicochemical parameters studied were density, kinematic viscosity, and water content. AC BDV was measured using an insulating oil dielectric strength tester in accordance with IEC 156:1995 (En, 1996). The density at 20°C was measured using DMA 35 basic handheld density meter according to ASTM D7777 (ASTM Standard D7777-15, 2018); kinematic viscosity at 40°C was measured using HK kinematic viscosity apparatus in accordance with ASTM D445 (ASTM D445-06, 2008); and water content by Karl Fischer method in accordance to ASTM D6304 (ASTM Standard D6304-20, 2016). The dielectric strength and physicochemical characterisation of POME were compared to the requirement of IEEE Std C57.147:2018 – IEEE guide for acceptance and maintenance of natural ester insulating liquid in transformers (IEEE 2018). The effect of both NPs used in this study was investigated to understand better the behaviour of POME in the presence of different types of NPs as additives in transformer liquid dielectric.

RESULTS AND DISCUSSION

POME Production

Figure 3 shows the yield of POME from four samples (S1-S4) of RBDPO olein that underwent the transesterification process. From the graph, it can be seen that all the samples achieved more than 90.00% yield. The reaction was the same for all samples; however, a small yield percentage variability could be due to practical losses throughout the procedure. The average POME production yield was 98.43%.

SEM-EDS Analysis

Figures 4a and 4b show the SEM images of SiO₂ and ZnO NPs with three different magnifications, respectively. Both NPs had a variety of shapes and sizes and a homogeneous distribution with a wide range of dispersion. SiO₂ powder consists of more or less spherical and regular particles (Khaled and Beroual, 2019) with poor agglomeration. The particle size data of both NPs were obtained from measuring 50 particles using ImageJ software. SiO₂ and ZnO have an average size of 28.95 ± 14.9 nm and 40.64 ± 17.4 nm, respectively.

Figure 3. The percentage yield of POME.
Figures 4(a) and 4(b) show the SEM images of SiO$_2$ and ZnO NPs showing their morphology at three different resolutions at (i) ×20,000, (ii) ×50,000, and (iii) ×100,000 magnification.

Figures 5a and 5b show the EDS results of SiO$_2$ and ZnO particles. The spectrum indicates the presence of silicon, zinc particularly, and oxygen, indicating the formation of silica as silicon dioxide and zinc as zinc oxide, respectively.

**FTIR-ATR Spectra Analysis**

**Characterisation of Pure POME, POME-based SiO$_2$ NFs and POME-based ZnO NFs.** Pure POME and all NF samples were characterised using FTIR-ATR spectroscopy studies, as shown in Figures 6a and 6b. In these figures, the x-axis represents the wavenumber (cm$^{-1}$) and the absorbance in the y-axis. The absorption peaks at 2922.16 cm$^{-1}$ and 2852.72 cm$^{-1}$ represent the asymmetric stretching vibration of C-H of -CH$_2$ groups and the symmetric stretching vibration of CH$_2$ groups (Jacob et al., 2020; Jamo et al., 2019; Ramli et al., 2021), respectively.

At the absorbance wave number 1741.72 cm$^{-1}$, a long and narrow, strong peak can be seen for all samples; it represents C=O stretching indicating the formation of ester (Baruah et al., 2020; Jacob et al., 2020; Maharana et al., 2019). The absorbance peaks at a wavenumber of 1168 cm$^{-1}$ and 721.36 cm$^{-1}$ indicated the presence of the C-O band stretching, establishing the existence of an ester (Ravulapalli et al., 2019).

The resemblance in the FTIR-ATR spectra of both POME-based NFs at 2922.16, 2852.72, 1741.72, 1460.11, 1168.86 and 721.38 cm$^{-1}$ is an indication of the presence of a similar chemical group in their compositions (Awogbemi et al., 2019). The introduction of both NPs did not cause any changes in the spectra of investigated POME-based liquids; this infers that varying NPs concentration does not affect the sample’s compound structure.

**Dielectric Strength Characterisation**

**AC BDV.** The dielectric strength of the pure POME and NFs was studied using the AC BDV test. In this section, the AC BDV of POME-based SiO$_2$, and ZnO NFs were presented and discussed. Table 2 tabulates the AC BDV for both investigated NFs and shows the incremental percentage of their breakdown strength with different concentrations of NPs. A significant enhancement of AC BDV for POME-based ZnO NFs was observed compared to POME-based SiO$_2$ NFs. The results demonstrated that all NFs samples with ZnO addition have higher AC BDV than the pure sample of POME. The optimal concentration for the highest AC BDV was 0.2 g L$^{-1}$. The BDV increased from 20 kV to 37 kV, equivalent to an 85% increase in magnitude. However, the BDV started to decrease as the concentration of ZnO NPs increased. Similar results were reported by Saenkhumwong and Sukri (2017), where the authors tested the ZnO in palm ester, and the optimal concentration was found to be at 0.2 g L$^{-1}$. 
In contrast, while using SiO₂, the AC BDV decreased irrespective of the concentration, as shown by the negative incremental percentage in Table 2. This observation was similar to Yahya and Amirrazli (2018), who found that SiO₂ NPs did not improve the BDV of PO. Similarly, in other works where the addition of SiO₂ into different types of natural ester, i.e., MIDEL 1204 (Khaled and Beroual, 2019) and FR3 (Charalampakos et al., 2017), they demonstrated a similar reduction of dielectric strength of respective NEOs.

### Physicochemical Characterisation

**The density of oil samples.** In general, the density of the oils varies with each type and temperature, ranging from 0.81 to 0.91 g cm⁻³ (Rotimi, 2016). From the experimental results, compared to non-treated RBDPO olein, whose density was 0.91 g cm⁻³, POME's density was 0.87 g cm⁻³, met the IEEE C57.147 standard (≤0.96 g cm⁻³). An almost similar value was obtained by Jayakar et al. (2018), which was 0.873 g cm⁻³. The value is comparable to MO, which has a density of 0.88 g cm⁻³ (Kanoh et al., 2008).
**Kinematic viscosity.** The viscosity of PO is 37.300 cSt (Suwarno et al., 2003). After transesterification, the viscosity of the oil was reduced to 4.272 cSt, which implies that the POME complied with the required limit of ≤50 cSt. This study also looked at the effect of insulative NPs on the viscosity of POME. The measured viscosity of POME-based NFs at a temperature of 40°C varied between 4.233 to 4.326 cSt, which also complied with the required limit. This implies that the presence of NPs did not influence the viscosity of POME-based NFs at 40°C. The results demonstrated that the transesterification process had changed the viscosity of POME considerably. The conversion of triglycerides to methyl ester appeared to be the primary cause of the viscosity reduction. Low viscosity transformer oils are preferred because they promote heat transfer and circulation (Mohd et al., 2021; Walvekar et al., 2022).

**Water content.** According to Toudja et al. (2014), water content dissolved in oils is practically inevitable and adversely influences the oil’s properties. Consequently, the dielectric strength of the oil degrades. The water content of pure POME, POME-based SiO$_2$, and POME-based ZnO NFs can be seen in Figure 7. It was observed that the pattern in the graph demonstrated that as the amount of SiO$_2$ NPs employed increased, the water content of their NF decreased. However, when the concentration of ZnO NPs increased, so did the water content in the samples.

Generally, as the water content of the NFs rises, their AC BDV is reduced. As demonstrated by other studies (Ab Ghani et al., 2016; Abdi et al., 2020), the water content is inversely proportional to the AC BDV. However, another factor influencing the mechanism of BDV characteristics of the NFs is the electrical conductivity of NPs (Duzkaya and Beroual, 2021). ZnO is a semiconductive NP that traps high mobility electrons by slowing down the electrons responsible for streamer development with trapping and de-trapping processes (Duzkaya and Beroual, 2021). Approximately 85% of the increment of AC BDV at a concentration of 0.2 g L$^{-1}$ is well explained by this mechanism. Meanwhile, the decreased BDV when NP exceeds a specific concentration may be attributed to the mechanism of bridging or tunnelling, as discussed in the previous study (Duzkaya and Beroual, 2021).

However, water content is still over the threshold under the required standard, which signals that more additional procedures or treatments are needed to lower the water content of POME NFs. These additional treatments include using adsorbents such as silica gel or drying the NFs in an oven at a specific temperature and a particular time to eliminate the existing moisture before conducting further tests.

**Comparative Analysis with Previous Studies**

Table 3 shows the comparative results obtained by various researchers in assessing the dielectric strength and viscosity properties of the PO. In the current study, the transesterification and addition of NPs methods used to modify the PO demonstrate comparable results to previous studies.

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![Figure 7. The water content of pure POME, POME-based SiO$_2$, and ZnO NFs.](image-url)
TABLE 3. COMPARATIVE ANALYSIS WITH PREVIOUS STUDIES

| Modification method | NPs addition / concentration | Breakdown voltage (kV) | Kinematic viscosity (40°C) (mm$^2$ s$^{-1}$ or cSt) | References |
|---------------------|-------------------------------|------------------------|-----------------------------------------------|------------|
| n/a                 | SiO$_2$ / 0.100%              | 25.46                  | 70.000                                        | Yahya and Amirrazli (2018) |
| n/a                 | SiC / 0.300%                  | 28.04                  | 140.000                                       | Yahya and Maliki (2018) |
| n/a                 | ZnO / 0.005 g L$^{-1}$        | 69.28                  | 46.660                                        | Hussin et al. (2021) |
| Transesterification | ZnO / 0.200 g L$^{-1}$        | 27.30                  | 0.934                                         | Saenkhumwong and Sukrit (2017) |
| Transesterification | TiO$_2$ / 0.200 g L$^{-1}$    | 29.00                  | 0.925                                         | Saenkhumwong and Sukrit (2017) |
| Transesterification | FeO$_3$ / 0.100 wt%           | 32.22                  | n/a                                           | Asse et al. (2022) |
| Transesterification | ZnO / 0.200 g L$^{-1}$        | 37.00                  | 4.326                                         | Current study |

Note: n/a - not available.

CONCLUSION

The transesterification process improved the physicochemical property of PO, in particular the viscosity of the oil, allowing it to be used as a potential insulating dielectric liquid for transformers. The addition of NPs to the POME appeared to have a minimal effect on the POME’s viscosity and can be considered negligible. POME-based SiO$_2$ and ZnO NFs complied with the viscosity requirement for their use in transformers. The effects of NPs on AC BDV of POME depend on their types and concentrations. The best increment achieved was 85%; at a concentration of 0.2 g L$^{-1}$ of ZnO NPs. Increasing ZnO concentration beyond this value suggests the implication of the conductivity of the NP, leading to the existence of an optimum concentration that has yet to be established because it is still in the research stage. On the other hand, adding insulative SiO$_2$ NPs reduced POME’s dielectric strength, with SiO$_2$ at a concentration of 0.3 g L$^{-1}$, causing a 20% reduction. This work revealed that the developed POME-based ZnO NFs were likely to serve better as an insulating liquid than POME-based SiO$_2$ NFs.

The study of the impact on chemical modification using the transesterification process and the addition of NPs in PO carried out here is a preliminary study. To ensure the reliability and possibility of POME-based NFs as an insulating liquid in transformers, other physicochemical parameters, including interfacial tension, acidity, pour point, and fire point will be included in future research work.

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