

Short-term Ageing Study on the Palm Oil and Mineral Oil in the Presence of Insulation Paper, Moisture, Low Molecular Weight Acid, and Oxygen

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ABSTRACT

This study presents the short-term ageing study on refined, bleached and deodorised palm oil (RBDPO) and mineral oil (MO) in the presence of insulation paper, moisture, low molecular weight acid (LMA) and oxygen. The ageing experiment was performed for 7 days at

140°C. The oil was maintained dried while the paper's moisture was varied between 0.5% and 3.5%. In total, 0.2 g of LMA and 20 mbar of oxygen pressure were initially introduced in the oil before the ageing started. Several analyses were conducted after the ageing experiment, which include the AC breakdown voltage (BDV) oil/paper, tensile strength, degree of polymerization (DP) and thermogravimetric analysis and differential scanning calorimetry (TGA-DSC). After being subjected to ageing in the

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presence of LMA and oxygen, the reduction of AC BDV of RBDPO is lower than MO at all moisture levels. At the same condition, the AC BDV of RBDPO-impregnated paper also maintains higher than MO-impregnated paper. The RBDPO-impregnated paper, in the presence of LMA and oxygen, has higher resistance toward ageing than MO-impregnated paper based on DP and tensile index, even in high moisture. All RBDPO are more resistant to ageing than MO in the presence of LMA and oxygen based on the high onset temperatures of the TGA-DSC analysis.

Keywords: Breakdown voltage, degree of polymerization, insulation paper, tensile strength

INTRODUCTION

Recently, vegetable oil (VO) has been identified as a possible substitute for mineral oil (MO) for application in transformers due to its biodegradability, environmental friendliness and fire safety (Maharana et al., 2018; Martin et al., 2006; Matharage et al., 2016; Maharana et al., 2018; Raj et al., 2020; Raymon et al., 2013; Suwarno & Pasaribu, 2017;). Palm oil (PO) is among the most widely accessible VOs in Asian countries (RSPO, 2015; Suryani et al., 2020). Refined, bleached and deodorised palm oil (RBDPO) is one of the common varieties of PO originating from the oil palm fruit (Azis et al., 2014). Different studies are conducted on RBDPO to explore its application as a dielectric insulating fluid (Makmud et al., 2018; Makmud et al., 2019).

The ageing characteristics of MOs are previously examined, which cover the physiochemical and electrical aspects (Abdelmalik, 2015; Carcedo et al., 2015; Coulibaly et al., 2012; Matharage et al., 2016; Munajad et al., 2017; N'cho et al., 2016). Similar studies are carried out for VOs such as coconut, palm, soya, sunflower, rapeseed and corn oils, whereby among the important finding is that the ageing of cellulose insulation can be retarded as a result of its water scavenging and hydrolytic protection mechanisms (Gomna et al., 2019; Rapp et al., 2005; Vihacencu et al., 2013). The ageing can affect the electrical properties of VOs, such as the electrical dissipation factor and resistivity (Ciuriuc et al., 2014; Wilhelm et al., 2011). In addition, the AC breakdown voltage (BDV) of the natural ester can increase up to 30.96 % after being subjected to ageing (Maharana et al., 2019). A previous study shows that most of the acid generated in MO is low molecular weight acid (LMA). The LMA generated in VO is lower than MO (Azis & Wang, 2011). The presence of LMA can further enhance the degradation of paper (Kouassi et al., 2018).

The ageing characteristics of RBDPO are also examined in recent years (Ismail et al., 2013; Kiasatina et al., 2011; Mohamad et al., 2016; Sinan et al., 2014; Suleiman et al., 2014). A previous study revealed that the AC breakdown voltage of aged RBDPO can decrease between 9.8% and 28.2% after being subjected to ageing (Mohamad et al., 2016). Similarly, it is found that the ageing rate of paper aged in MO can be up to 1.6 higher than

RBDPO and coconut oil samples based on previous ageing models (Mohamad et al., 2015). The percentage of reduction of the tensile index (TI) of RBDPO-impregnated paper is lower than that of MO-impregnated paper at the end of the ageing period (Mohamad et al., 2016). The thermogravimetric analysis and differential scanning calorimetry (TGA-DSC) for aged RBDPO reveals that the onset temperature is 408°C while MO is 297°C (Raof et al., 2019). Currently, the study on the effect of ageing accelerators such as moisture, oxygen and acid on the ageing performance of RBDPO is still lacking.

This paper discusses the impact of short-term ageing on the RBDPO and MO in the presence of insulation paper, moisture, acid and oxygen. The type of acid used in the study is LMA. The properties such as AC BDV of the oil/paper, tensile strength, degree of polymerization (DP) and TGA-DSC are measured and analysed. The ageing factor is derived from the influence of different ageing accelerators. The current study provides a fundamental understanding of the RBDPO and MO aged in the presence of ageing accelerators for possible future applications in transformers.

METHODOLOGY

Thermal Ageing Procedure

The MO and RBDPO were initially filtered 3 times using a membrane filter with a pore size of 0.2 μm . The properties and appearances of oils are shown in Figure 1 and Table 1. These oils were dried for 2 days at 85°C in a vacuum oven. All samples were filled with nitrogen to reduce the interaction with oxygen. The final moisture contents of RBDPO and MO were 109 ppm and 12 ppm, respectively. Next, the paper was dried at 90°C or 105°C in a vacuum oven to produce the sample with different moisture contents known as base, low, medium and high moistures. The paper was dried at 105°C for 48 hours, 105°C for 96 hours, 90°C for 48 hours and 90°C for 24 hours to produce base, low, medium and high moisture samples. The final moisture for the base, low, medium and high moisture paper samples are 0.87%, 0.55%, 1.66% and 2.65%. The paper was then impregnated with oils in a vacuum oven for 24 hours at 85°C. These oils were introduced with 0.2 g of formic acid

Table 1
Oil properties

Properties (Unit)	MO	RBDPO
Viscosity, 40°C (mm ² /s)	7.6	21.2
Density, 150°C (kg/m ³)	890	915.5
Flashpoint (°C)	154	320
Water content (mg/kg)	<20	60
Breakdown voltage (kV)	40-60	60-70
Acidity (mg KOH/g)	<0.2	<0.06
Dielectric dissipation factor, 90°C	<0.001	0.03



Figure 1. MO and RBDPO

as LMA and 20 mbar of oxygen pressure above the bottle's oil surface. The oil-to-paper ratio was set to 20:1, with 450 g of oil and 22.5 g of paper, and it was aged in borosilicate glass. The bottle cap reinforced with polytetrafluoroethylene tape was used to seal the borosilicate glass containing the oil and paper, aged for 7 days at 140°C to minimise the environmental interaction.

AC Breakdown Voltage of Oil

An automatic BAUR DPA 75C was used to obtain the AC BDV of oil as per ASTM D1816-12, as shown in Figure 2. The test was conducted at room temperature between 28.4 and 32.9°C (ASTM D1816-12, 2019). The gap spacing between the 36 mm diameter VDE electrodes was fixed to 2.5 mm. In total, 400 ml of oil was carefully poured into the test cell to prevent the formation of any bubbles. The oil was given 15 minutes to rest prior to the test. The voltage was then gradually increased at 0.5 kV/s until the breakdown occurred. Next, the oil was stirred continually using a magnetic stirrer while the interval between breakdowns was set to 5 minutes. In total, 50 measurements of AC BDV were obtained for MO, and the average value was used for the analysis.

AC Breakdown Voltage of Oil Impregnated Paper

The AC BDV of paper was measured using BAUR DPA 75 C as per IEC 60156, as seen in Figure 3. First, the gap distance of the sphere electrodes with a 12.5 mm diameter was set based on the thickness of the oil-impregnated paper. In total, 2 layers of oil-impregnated paper were used due to the measurement limit caused by the very small gap distance based on the thickness of 1 layer of oil-impregnated paper. Next, 400 ml of pre-processed oil was carefully poured into the test cell, whereby the voltage ramping rate was set to 2 kV/s. The oil-impregnated paper was moved to other positions after each of the breakdowns. In total, 20 measurements of AC BDV were recorded for the oil-impregnated paper, whereby the average value was used for the analysis.

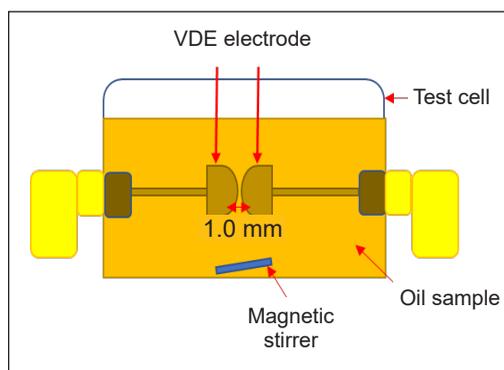


Figure 2. AC breakdown voltage test of oil

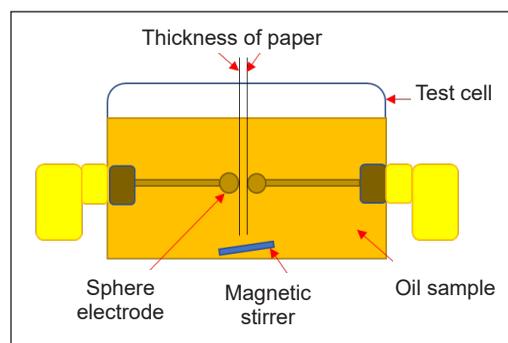


Figure 3. AC breakdown voltage test of oil-impregnated paper

Tensile Strength

Tensile strength was performed using an Instron 5566 model universal testing machine as per BS EN ISO 1924-2. A 10 kN universal testing machine load cell was used, as shown in Figure 4. The crosshead speed and full-scale load range were adjusted to 20 mm/min and 0.5 kN. The distance between the two clamps was set to 100 mm. The paper width and gap distance of the paper were set to $16 \text{ mm} \pm 0.1 \text{ mm}$ and $180 \text{ mm} \pm 0.1 \text{ mm}$, respectively. In total, 5 samples were tested for each type of paper and the average value was used for the analysis. Next, the maximum load of TI was calculated based on Equation 1.

$$TI = ((\bar{F}/W) / G) \times 10^3 \quad [1]$$

Whereby TI is the paper's tensile index in Newton's metres per gram, \bar{F} is the maximum load in Newton, W is the paper's width in millimetres, and G is the paper's grammage in grams per square metre. The paper's grammage under study was around 51.8 g/m^2 .

Acidity

The acidity of the oils was determined using a Metrohm 877 oil Titration plus as per ASTM D974 (2023). For each type of oil, 10 g was utilised for the measurement, as seen in Figure 5. In total, 1 measurement was obtained for each type of oil.

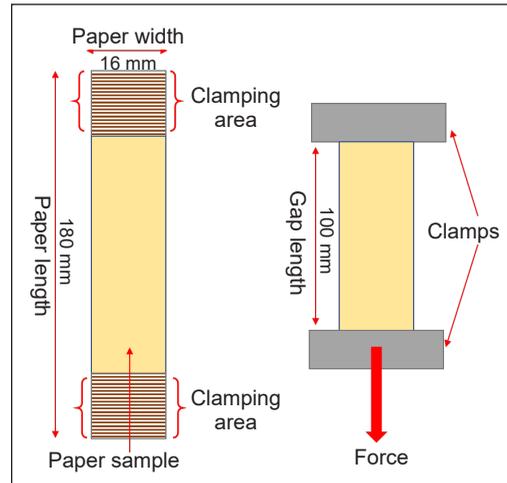


Figure 4. Tensile strength test of paper

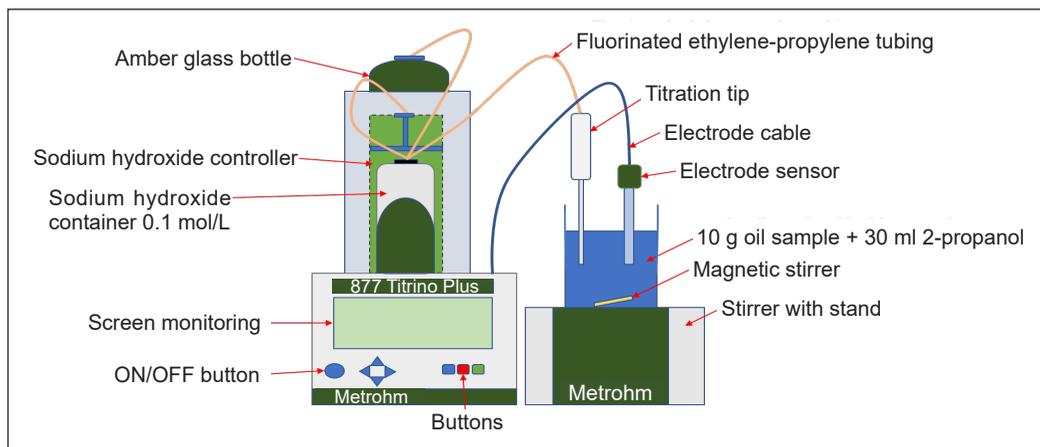


Figure 5. Acidity test of oil

Moisture in Oil and Paper

A Metrohm 831 Karl Fischer (KF) Coulometer was used to measure the moisture in oil based on ASTM D6304 (2021). For each type of oil, 1 ml of oil was used for the moisture measurement, as shown in Figure 6. A Metrohm 774 Karl Fischer Coulometer measured the moisture content of the insulation paper according to IEC 60814. The moisture in the paper was extracted via an oven technique. The total weight of paper used for the moisture measurement is 0.5 g, as seen in Figure 7. In total, 2 moisture measurements were taken for RBDPO and MO for the insulation paper, where the average value was used for the analysis.

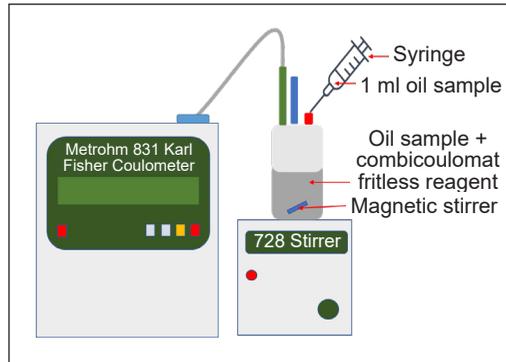


Figure 6. Moisture content test of oil

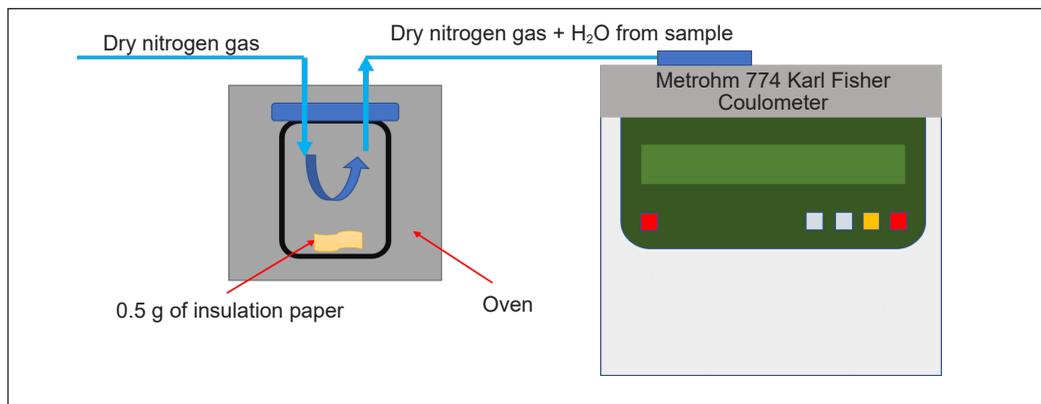


Figure 7. Moisture content test of paper

Thermogravimetric and Differential Scanning Calorimetry Analysis

The measurements of the oils were performed under non-isothermal conditions based on the standard TGA from Mettler Toledo, TGA-DSC HT 3, as shown in Figure 8. The weight of the oil used was 5 mg. The system was first purged with nitrogen gas at 50 ml/min for around 20 minutes at 25°C to release the trapped gases. The sample was then heated from 25°C to 600°C at a steady rate of 10°C/min, and the temperature was maintained for 10 minutes.

Degree of Polymerization

The DP of the paper was obtained based on the average intrinsic viscosity according to ASTM D4243 (2023), as seen in Figure 9. The residual oil was removed from the paper through soxhlet extraction using hexane for up to 8 hours to obtain the dry weight value.

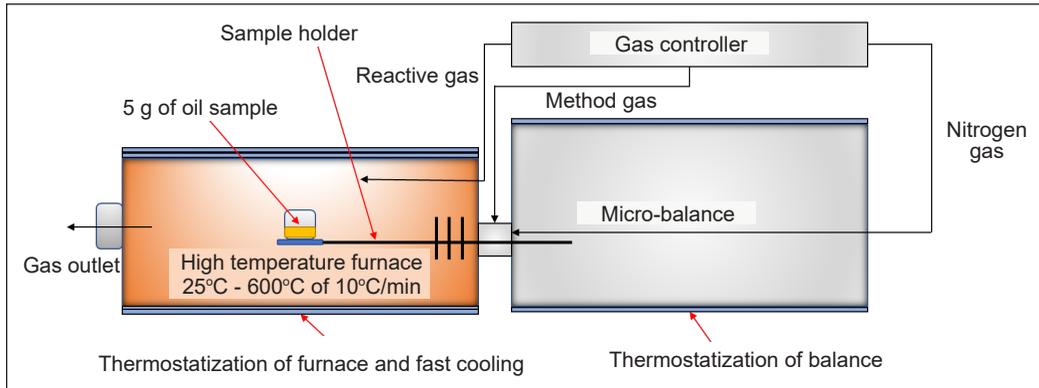


Figure 8. Thermogravimetric analysis and differential scanning calorimetry test of oil

Next, the paper was cut into confetti with dimensions 2 mm before the moisture content was determined using the oven method. In total, 0.1 g of the sample was weighed and mixed with 22.5 ml of distilled water in a beaker. Next, the solution was left for 30 minutes before fragmented in a wet kitchen blender for up to 15 minutes. Next, 22.5 ml Copper (II) ethylenediamine (CED) solution was added and stirred for 2 hours. The mixture was added to the Cannon-Ubbelohde capillary tube viscometer. The viscometer was inserted into a constant water bath at 20°C. Once the tube’s temperature was stabilised, the viscosity measurement was performed. The measurement was carried out for 4 times, whereby the average was used for the analysis. The calibration was performed based on the viscosity determination of the blank solution with a CED-to-water ratio of 50:50.

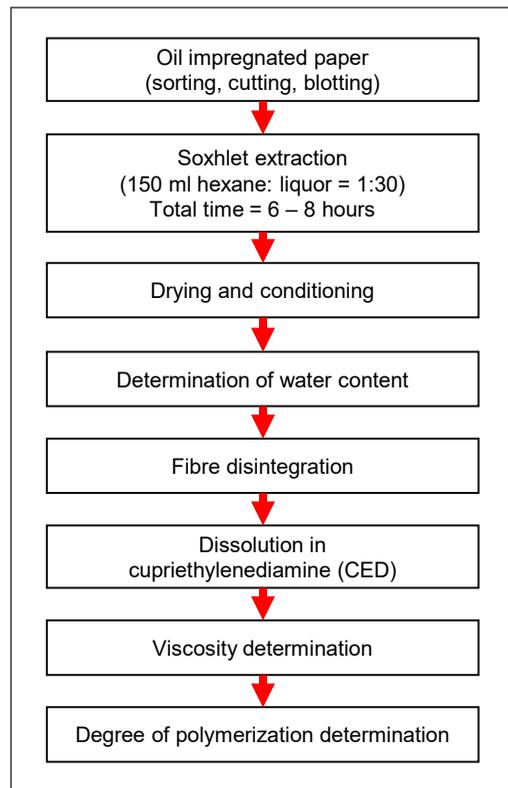


Figure 9. Measurement process of degree of polymerization

From the solution prepared, the concentration (c) was calculated based on Equation 2

$$c = \frac{100m (100 - \%MC)}{4500 + m(\%MC)} \quad [2]$$

Where m is the mass of paper, g, and MC is the moisture content of the paper, %.

DP was calculated based on Mark Houwink constants where α and K were defined as 1 and 7.5×10^{-3} , respectively (Equation 3).

$$DP^\alpha = \frac{[\eta]}{K} \quad [3]$$

Where $[\eta]$ is the intrinsic viscosity related to the specific viscosity, η_s and can be determined based on Equation 4.

$$\eta_s = \eta_{rel} - 1 = [\eta](10^{K[\eta]c}) \quad [4]$$

Where η_{rel} is the relative viscosity that can be defined based on Equation 5.

$$\eta_{rel} = \frac{\text{Kinematic viscosity of the solution}}{\text{Kinematic viscosity of CED}} \quad [5]$$

$[\eta].c$ can be obtained from Table 1 in ASTM D4243 (2023). The intrinsic viscosity $[\eta]$ can be determined using the c from Equation 2. DP can be estimated based on Equation 3.

RESULTS AND DISCUSSION

AC Breakdown Voltage of RBDPO and MO

The AC BDV of RBDPO is higher than MO at all moisture levels after 7 days of ageing, as shown in Figure 10. The decrement pattern of AC BDV for MO is steeper than RBDPO as the moisture increases. In the presence of high moisture, LMA and oxygen, the AC BDV of RBDPO decreases by 12%, while for MO, it decreases by 73%.

AC Breakdown Voltage of RBDPO and MO-Impregnated Paper

At all moisture levels, the AC BDV of RBDPO-impregnated paper is higher than MO-impregnated paper after 7 days of ageing, as seen in Figure 11. The AC BDV of RBDPO-impregnated paper slightly increases with the introduction of low moisture and decreases with the moisture increment. The same pattern is found for AC BDV of MO-impregnated paper. In the presence of high moisture, LMA and oxygen, the AC BDV of RBDPO and MO-

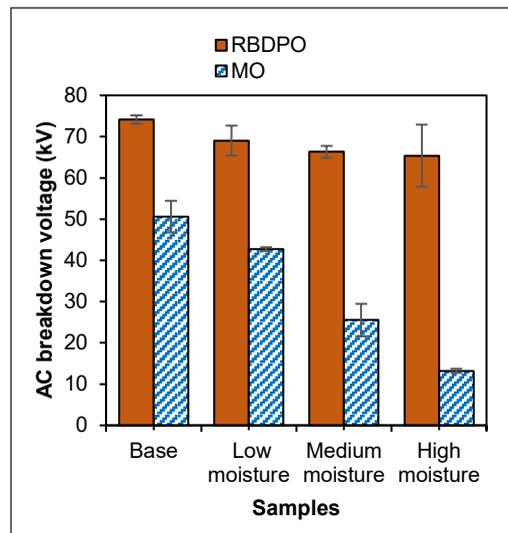


Figure 10. AC breakdown voltage of RBDPO and MO in the presence of moisture, LMA and oxygen

impregnated paper slightly increased by 16.88% and 18.02% compared to the base.

Tensile Index of RBDPO and MO Impregnated Paper

The reduction pattern of TI is quite similar to DP at all moisture levels after 7 days of ageing, as shown in Figure 12. RBDPO-impregnated paper experiences a lower reduction of TI than MO-impregnated paper. Similar to DP, the TI of both RBDPO and MO-impregnated papers still suffer advanced degradation at low moisture. The lowest TI for RBDPO-impregnated paper is still higher than the 50% retention strength limit per IEEE standard C57.91-2011 (IEEE Standards Association, 2012). With high moisture, the TI of MO-impregnated paper exceeds the limit with a percentage reduction of 59.54%. The ageing factor based on TI for RBDPO-impregnated paper in the presence of low, medium and high moistures is 1.41, 1.40 and 1.45. For MO-impregnated paper, the ageing factors are 1.64, 1.80 and 2.47, respectively.

Degree of Polymerization of RBDPO and MO-Impregnated Paper

The DP reduction of RBDPO-impregnated paper is lower than MO-impregnated paper at all moisture levels after 7 days of ageing, as seen in Figure 13. Even with the introduction of low moisture, both RBDPO and MO-impregnated papers still experience significant degradation, possibly due to the presence of LMA. The lowest DP for RBDPO-impregnated paper is 319 in the presence of high moisture. On the other hand, the DP for MO-impregnated paper decreases lower than 200, reaching the end of its life (Emsley et al., 2000.). The ageing factor based on DP for RBDPO-impregnated paper in the

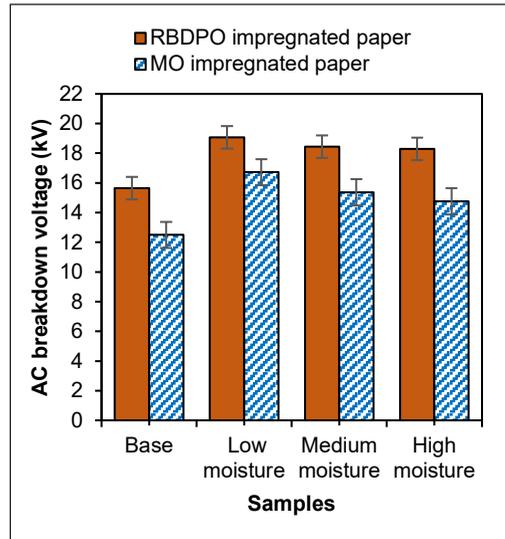


Figure 11. AC breakdown voltage of RBDPO and MO-impregnated paper in the presence of moisture, LMA and oxygen

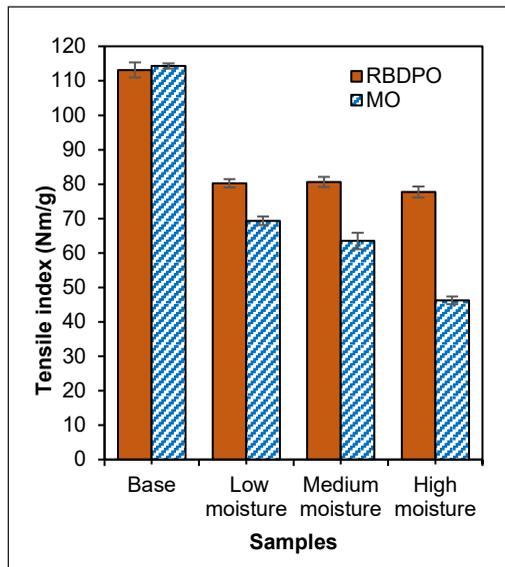


Figure 12. Tensile index of RBDPO and MO-impregnated papers in the presence of moisture, LMA and oxygen

presence of low, medium and high moistures is 2.04, 2.07 and 2.96. For MO-impregnated paper, the ageing factors are 2.47, 4.44 and 4.57, respectively.

Thermogravimetric and Differential Scanning Calorimetry Analysis of RBDPO and MO

The onset temperature, or the temperature at which degradation begins, is useful for the oil stability (Raof et al., 2019). All RBDPOs are more resistant against degradation as compared to MO since the onset temperatures are high at all moisture levels after 7 days of ageing, as shown in Table 2 and Figure 14. The base RBDPO is stable up to 396°C, whereas base MO is only stable up to 296°C. MO exhibits a high weight loss of 72% in high moisture at 315.5 °C.

The weight loss at the low-temperature range for MO is attributed to the evaporation of low molecular weight hydrocarbons and degradation of the base oil. According to Tripathi & Vinu (2015), the degradation of MO can occur at the temperature range between 150°C and 350°C. For example, the paraffinic chain in the MO molecules can decompose into ethane (C₂H₆), ethylene (C₂H₄), methane (CH₄), hydrogen (H₂) and graphite carbon. At a similar moisture level, the weight loss for RBDPO is only 61% at 431.5°C. RBDPO contains high triglyceride molecules such as palmitic and oleic acids, contributing to its higher thermal stability than MO. The increment of the hydrocarbon chain length and branching in the RBDPO's molecule decreases the weight loss and decomposes much slower (Raof et al.,

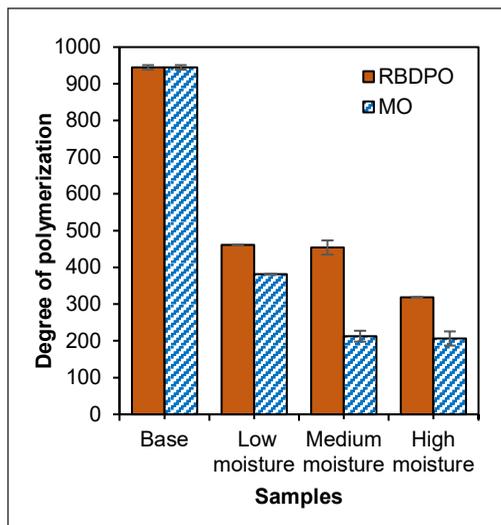


Figure 13. Degree of polymerization of RBDPO and MO-impregnated papers in the presence of moisture, LMA and oxygen

Table 2
The decomposition temperatures of the RBDPO and MO

Sample	Conditions	Onset temperature (°C)	Peak temperature (°C)	Weight loss (%)
MO	Base	295.6	330.6	57.6
	Low	293.4	329.8	56.1
	Medium	283.3	330.2	62.4
	High	238.5	315.5	72.1
RBDPO	Base	395.7	431.0	59.3
	Low	393.3	429.0	58.4
	Medium	394.4	431.7	61.0
	High	394.6	431.5	61.4

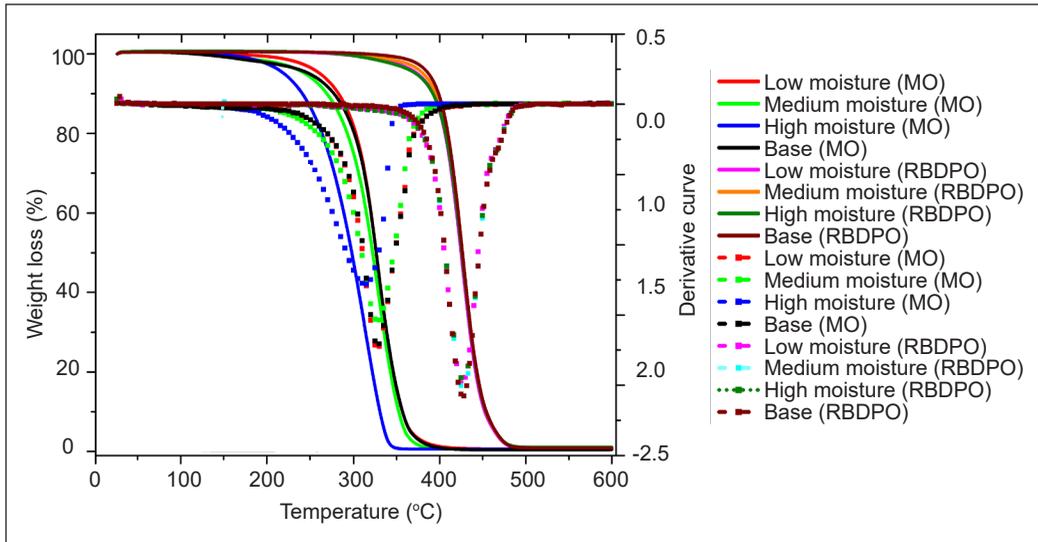


Figure 14. Thermogravimetric analysis and differential scanning calorimetry of the RBDPO and MO for weight loss and derivative curves in the presence of moisture, LMA and oxygen

2019). The relatively low decomposition temperature for RBDPO and MO with high moisture content can be attributed to the high volatility of water at a low temperature.

Correlation Between Degree of Polymerization and AC Breakdown Voltage of RBDPO and MO Impregnated Paper

The correlation between DP and AC BDV of RBDPO and MO-impregnated paper at all moisture levels after 7 days of ageing can be seen in Figure 15. The result shows that the correlation coefficient, R^2 , for RBDPO and MO-impregnated papers are 0.761 and 0.71, respectively. The correlation coefficient number, which ranges from -1 to 1, describes the strength and direction of the linear link between two quantitative variables. The R^2 from the linear relationship near 1 indicates a strong relationship whereby the range is between 0.7 and 0.99 (Ghoneim, 2021). Meanwhile, positive notation indicates the positive direction, showing that the 2 variables move in the same direction and vice versa. Moreover, it is found that there is a relationship between DP and AC BDV of RBDPO and MO-impregnated papers, which indicates that the reduction of mechanical strength leads to the reduction of electrical strength.

Correlation Between Degree of Polymerization and Tensile Index

The correlation between DP and TI after being subjected to ageing at all moisture levels after 7 days of ageing can be seen in Figure 16. The result shows that the correlation coefficient, R^2 , for RBDPO and MO-impregnated papers are 0.953 and 0.841, respectively. The positive notation between the decreases and increases of DP and TI can be observed

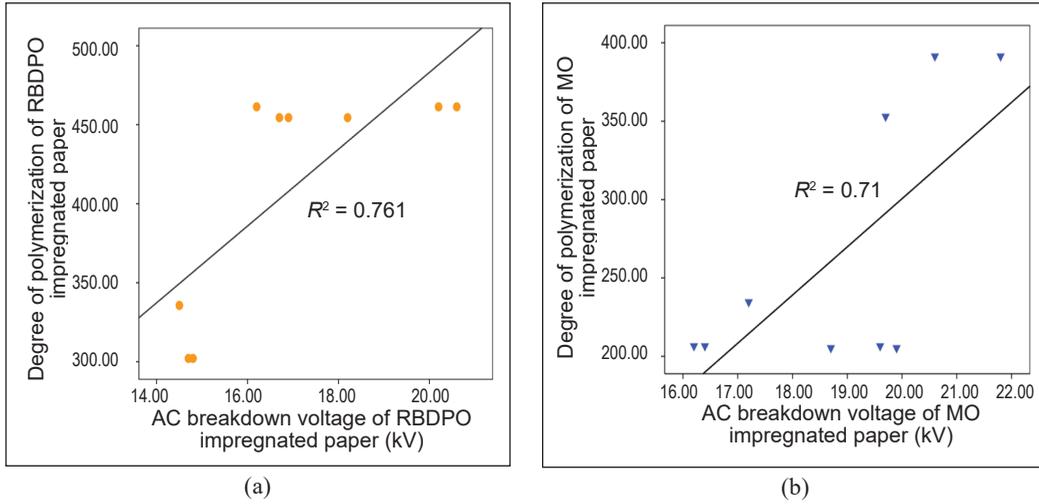


Figure 15. Positive correlation between DP and AC BDV of: (a) RBDPO; and (b) MO-impregnated papers

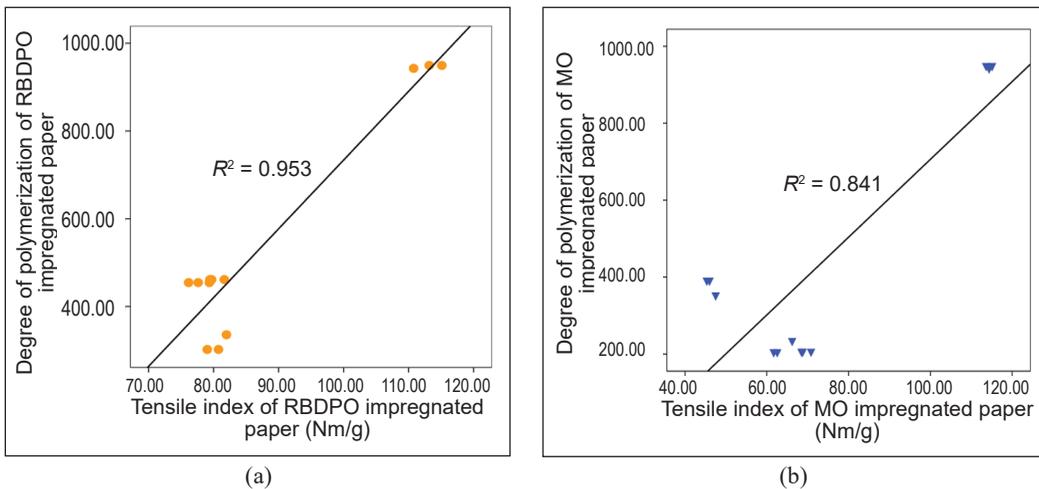


Figure 16. Positive correlation between DP and TI of: (a) RBDPO; and (b) MO-impregnated papers

through the linear regression. The results are in line with the study by (Arroyo et al., 2017), which suggests that the paper strength directly depends on the depolymerization of the cellulose as well as on factors of the inter-fibre bond strength among the cellulose fibres, individual fibre strength and the hierarchical structure of the paper.

CONCLUSION

This work examines the effects of initial ageing conditions in the presence of LMA, oxygen, and various degrees of moisture content of the paper at 140°C for 7 days are examined. The AC BDV for RBDPO experiences lower reduction than MO with the moisture

increment. The reduction of AC BDV of RBDPO-impregnated paper is still lower than MO-impregnated papers as the moisture level increases from low to high. The reduction trends of both DP and TI of RBDPO-impregnated papers show that the ageing performance is slightly better than MO-impregnated paper, even at high moisture content. RBDPO is more stable and resistant against ageing than the MO with high onset initial temperature. Overall, even with the presence of LMA, oxygen and high moisture, RBDPO is able to perform better than MO based on the condition under study.

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