


## Article

# Physiochemical and Electrical Properties of Refined, Bleached and Deodorized Palm Oil under High Temperature Ageing for Application in Transformers

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**Abstract:** This paper presents a high temperature ageing study of refined, bleached, and deodorized palm oil (RBDPO) olein at 170 °C in the presence of air. In total, two types of RBDPO were examined. The physiochemical and dielectric properties of RBDPO were measured and analysed. We found that the moisture and viscosities for both RBDPO increased as the ageing progressed, whereas the acidities fluctuated at very low levels at less than 0.005 mg KOH/g. The ageing on the AC breakdown voltages of both RBDPO were not affected throughout the ageing processes. The dielectric dissipation factors and relative permittivities for both RBDPO increased as the resistivities decreased with the ageing time.

**Keywords:** high temperature thermal ageing; open condition; refined, bleached and deodorized palm oil; physiochemical and electrical properties

## 1. Introduction

Palm oil (PO) has been identified as possible insulating fluid for transformer applications. PO is included in the natural ester group and has almost the same characteristics as other types of vegetable oils, such as having high flash and fire points, and being biodegradable and environmentally friendly [1,2]. Previous studies revealed that the electrical and physiochemical properties of new PO are close to other types of natural esters and mineral oil (MO) [3–6]. The majority of these properties are comparable to other types of vegetable oils under ageing at temperatures ranging from 85 to 150 °C, which are maintained at acceptable levels according to standards [7–11]. Currently, the high temperature ageing performance of PO has not been well established and requires further investigation.

One of the main purposes of high temperature ageing is to examine the performance of vegetable oils under extreme conditions where the influences of oxygen and moisture are also considered [12,13]. Previous high temperature ageing studies on vegetable oils examined acidity, viscosity, AC breakdown voltage, and dielectric properties [12–18]. Several ageing studies at high temperatures ranging from 140 to 170 °C showed that acidity and viscosity of natural ester can be affected by oxygen [14,17]. Natural ester showed a large increase in acidity and viscosity when aged under open conditions

at temperatures between 140 and 160 °C [14,19]. In addition, natural ester can start to gel after subjected to high temperature ageing at 170 °C [15]. The AC breakdown voltage of natural ester is maintained at an acceptable level regardless of the ageing conditions [16,18]. However, the dielectric dissipation factor of natural ester can be affected by ageing at high temperatures ranging from 140 to 170 °C [14,18]. A previous ageing study at 170 °C showed that the dielectric dissipation factor of natural ester increased by five- and eight-fold than the initial value when tested at 25 and 100 °C, respectively [18]. The ageing of natural ester at temperatures between 140 and 160 °C has been reported to cause a three- to six-fold increase in the power factor [14]. The impacts of high temperature ageing on moisture and dissolved gases generation of natural ester vegetable oils are not clear since the trends fluctuate as ageing progresses [12,18].

Oxidation is one of the main degradation mechanisms for natural ester. The rate of oxidation is governed by temperature, which can be accelerated by oxygen and metal catalysts [20]. The process begins with the initiation stage where free radicals are created as a result of the dissociation of the hydrogen atom from the C-H bond in the fatty acid triglyceride [21,22]. Interaction between oxygen and the free radicals initiates the propagation stage, which leads to the formation of peroxides. These peroxides interact with the C-H bond to create hydroperoxides [22]. With the initiation of the chain scission stage, the hydroperoxides dissociate and the interaction with oxygen promotes further generation of radicals and peroxides [22]. The final products of oxidation, as a result of the decomposition of hydroperoxides, are ketones, alcohols, aldehydes, and high molecular weight polymers [20,22–24]. The secondary non-volatile compounds formed via oxidation are subjected to cyclisation and polymerization processes. As a result, high molecular weight compounds, such as gel and lacquer, are formed at high temperatures and pressures [22]. Apart from oxidation, natural ester can also be subjected to the hydrolysis degradation mechanism. The process has three stages that begin with the formation of diglyceride as result of the interaction between triglyceride and water [25]. Further interactions between diglyceride and water lead to the formation of monoglyceride [25]. Glycerol is produced as a final product from the interaction between monoglyceride and water [25]. All stages in the hydrolysis of natural ester are reversible and fatty acids are produced at each of the stages [25].

In this paper, a high temperature ageing study, in the presence of air at 170 °C for 28 days, was completed on refined, bleached, and deodorized palm oil (RBDPO). Multiple physiochemical and electrical properties measurements were recorded and analysed.

## 2. Materials and Methods

### 2.1. Thermal Ageing Procedure

Two different RBDPO were tested as shown in Table 1. Both RBDPO were olein types from cooking products on the market. The fatty acid composition for both RBDPO were determined via gas chromatography analysis according to the Malaysian palm oil board (MPOB) test methods p3.4:2004 and p3.5:2204, as shown in Table 1 [26]. Both RBDPO had almost balanced compositions of fatty acids dominated by palmitic and oleic acids.

The oil was first filtered through a membrane filter and was dried in the oven for 48 h at 85 °C. The final moisture contents for both RBDPO were less than 120 ppm. Next, both RBDPO were sealed and left at room temperature for 24 h before being placed inside an air circulating oven at 170 °C. Each RBDPO was aged individually in a separate borosilicate glass bottle. The oil samples were aged under open conditions for 7, 14, 21 and 28 days.

**Table 1.** Fatty acid compositions of all samples.

Types of Fats	Types of Fatty Acids	Sample (%)	
		RBDPO A	RBDPO B
<b>Saturated</b>	C6: Caproic	-	-
	C8: Caprylic	-	-
	C10: Capric	-	-
	C12: Lauric	0.3	0.3
	C14: Myristic	1.1	0.9
	C16: Palmitic	37.7	39.0
	C18: Stearic	3.9	4.2
<b>Mono-unsaturated</b>	C18: Oleic	42.3	43.0
<b>Poly-unsaturated</b>	C18: Linoleic	12.4	10.4
	C18: Linolenic	0.3	0.2
	Others	0.1	0.1

## 2.2. Measurement of Properties

### 2.2.1. Moisture Content

A Metrohm 831 Karl Fischer (KF) Coulometer was used to measure moisture based on ASTM D 6304 [27]. In total, 1 g of RBDPO was used for each of the measurements. For each of the samples, two measurements were carried out in order to obtain the mean value.

### 2.2.2. Acidity

The measurement of acidity was performed according to ASTM D 974 with a Metrohm SM Titrino 702 [28]. For each of the measurements, 10 g of RBDPO were used where only one measurement was performed for each of the samples.

### 2.2.3. Viscosity

The viscosity was determined based on ASTM D 445 using a SVM 3000 Stabinger viscometer [29]. In total, 5 mL of RBDPO were used for the measurement. In this study, only one measurement at 40 °C was recorded for each of the RBDPO.

### 2.2.4. AC breakdown Voltage

A BAUR DPA 75C was used to determine AC breakdown voltage according to ASTM D 1816 [30]. The measurement was obtained based on two VDE electrodes (diameter = 36 mm) where the distance between these electrodes was set to 1 mm. These electrodes were fully immersed in 400 mL of RBDPO used for the test. The voltage increased at rate of 0.5 kV/s with a 5 min interval between each breakdown. To obtain the mean value, 50 measurements of AC breakdown voltages were recorded for each of the samples.

### 2.2.5. Dielectric Dissipation Factor, Relative Permittivity and Resistivity

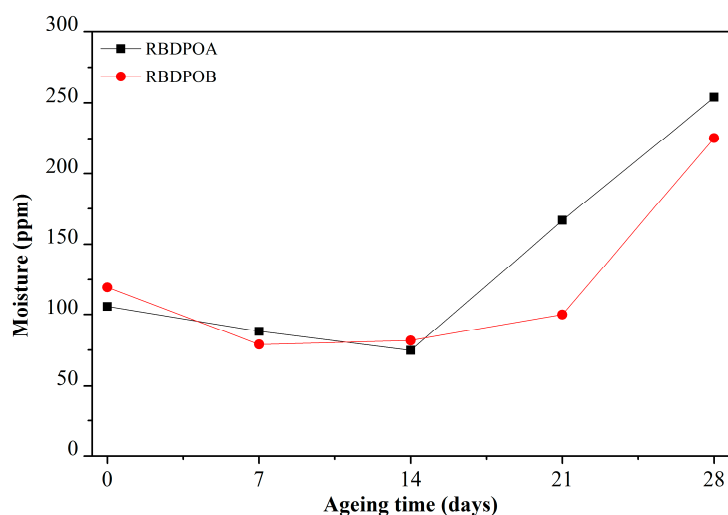
Dielectric properties of RBDPO was measured using a BAUR DTL C oil tester according to IEC 60247 [31]. In total, 45 mL of RBDPO were used for the measurement and only one measurement at 90 °C and 50 Hz was recorded for each of the RBDPO. The applied voltages for dielectric dissipation factor, relative permittivity, and resistivity measurement were 1999 V (AC) and 500 V (DC), respectively.

### 3. Results

#### 3.1. Physicochemical Properties

##### 3.1.1. Moisture

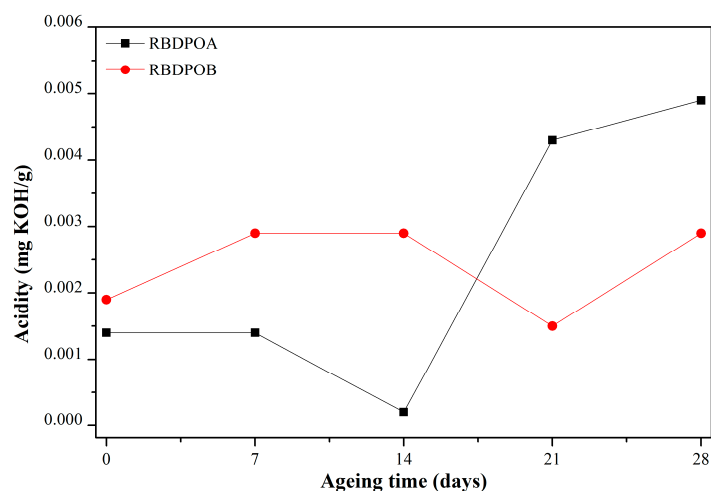
Overall, both RBDPO A and RBDPO B had almost the same increasing trend in moisture as shown in Figure 1. The moisture for both RBDPO slightly decreased with ageing up to 14 days and subsequently increased as the ageing progressed. After 28 days of ageing, the moisture for RBDPO A and RBDPO B were 254 ppm and 225 ppm, respectively.



**Figure 1.** Moisture of refined, bleached, and deodorized palm oil (RBDPO) aged at 170 °C under open conditions.

##### 3.1.2. Acidity

No clear trend was observed in the acidities for either RBDPO as shown in Figure 2. The acidity of RBDPO A slightly decreased after 7 days of ageing and increased after 14 days. However, the acidity of RBDPO B fluctuated between 0.0015 mg KOH/g and 0.0029 mg KOH/g. Both RBDPO had very low acidities even after 28 days of ageing.



**Figure 2.** Acidity of RBDPO aged at 170 °C under open condition.

### 3.1.3. Viscosity

The viscosity of RBDPO A remained almost unchanged during the first 14 days of ageing as shown in Figure 3. After 14 days of ageing, the viscosity of RBDPO A increased and remains almost unchanged from 21 to 28 days. The viscosity of RBDPO B slightly decreased during the first 7 days of ageing and then increased exponentially as the ageing progresses. After 28 days of ageing, the highest viscosities for RBDPO A and RBDPO B were 50.8 cSt and 70.9 cSt, respectively.

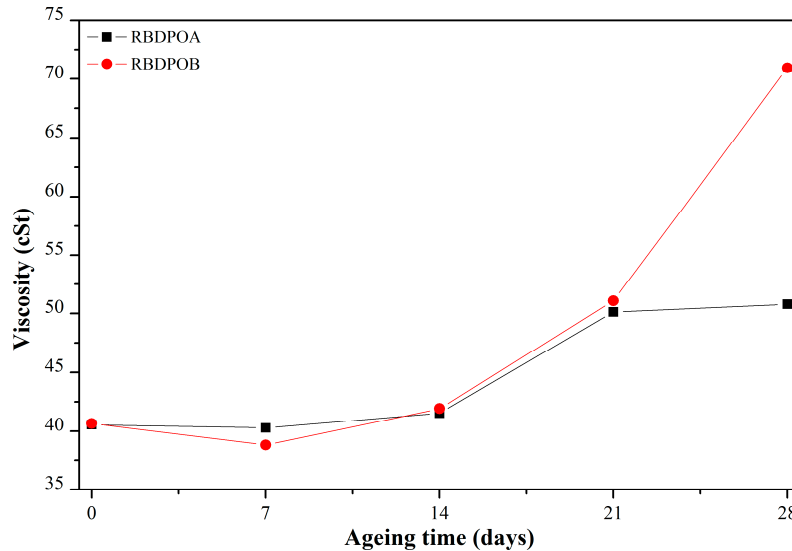


Figure 3. Viscosity of RBDPO aged at 170 °C under open conditions.

## 3.2. Electrical Properties

### 3.2.1. AC Breakdown Voltage

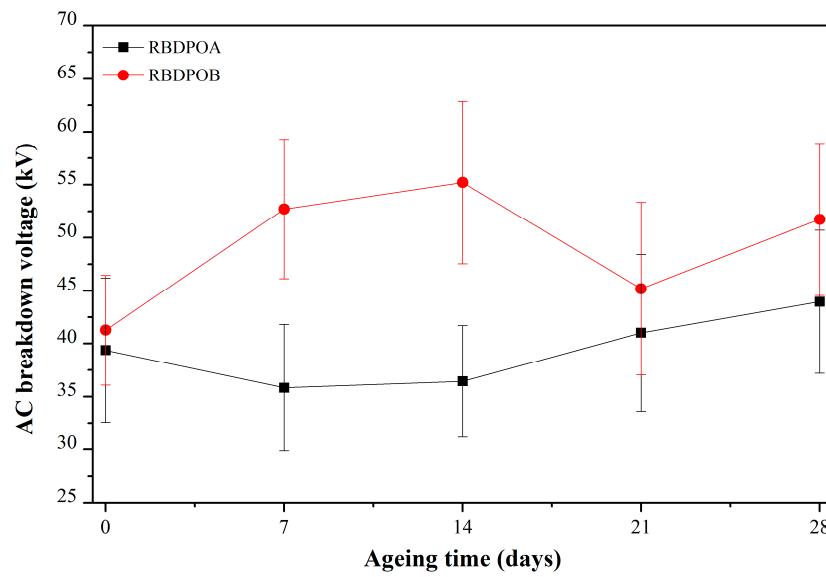
RBDPO B had a higher AC breakdown voltage than RBDPO A throughout the ageing period with differences in percentages between 9.3% and 40.9%, as shown in Figure 4. The AC breakdown voltage of RBDPO A slightly decreased during the first 7 days of ageing and increased after 14 days. For RBDPO B, the AC breakdown voltage fluctuated between 41.3 kV and 55.2 kV throughout the ageing period.

The AC breakdown voltages for both RBDPO were analysed based on cumulative normal and Weibull distributions. Equation (1) shows the cumulative normal distribution where  $\mu$  is the mean of the breakdown voltage data and  $\sigma$  is the standard deviation. Equation (2) shows the cumulative Weibull, where  $\alpha$ ,  $\beta$  and  $x$  are the scale and shape parameters and breakdown voltages for each of the samples, respectively.

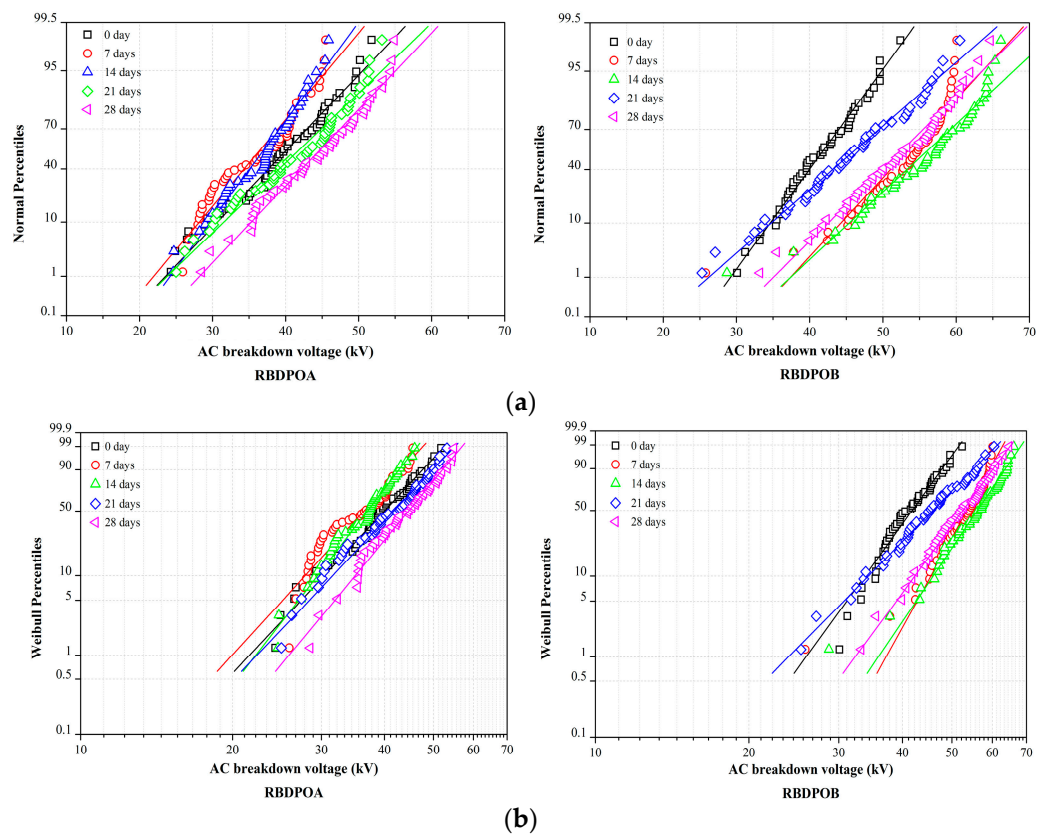
$$F(x|\mu, \sigma) = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^x e^{-\frac{(t-\mu)^2}{2\sigma^2}} dt \quad (1)$$

$$F(x|\alpha, \beta) = 1 - e^{-\left(\frac{x}{\alpha}\right)^\beta} \quad (2)$$

The breakdown voltage of RBDPO A is well-represented by normal distribution compared to the Weibull distribution, as shown in Figure 5. Conversely, Weibull distribution better represents the breakdown voltage of RBDPO B. For all ageing times, we found that the breakdown voltage of both RBDPO at 1% probability for normal distribution was higher than Weibull distribution, as shown in Table 2. At 50% probability, the majority of the breakdown voltage of both RBDPO for Weibull distribution was higher than normal distribution. For normal distribution and 1% probability, the lowest breakdown voltages for RBDPO A and B were 21.94 kV and 26.23 kV, whereas they were 19.95 kV and 19.8 kV for Weibull distribution, respectively.



**Figure 4.** AC breakdown voltage of RBDPO aged at 170 °C under open conditions.



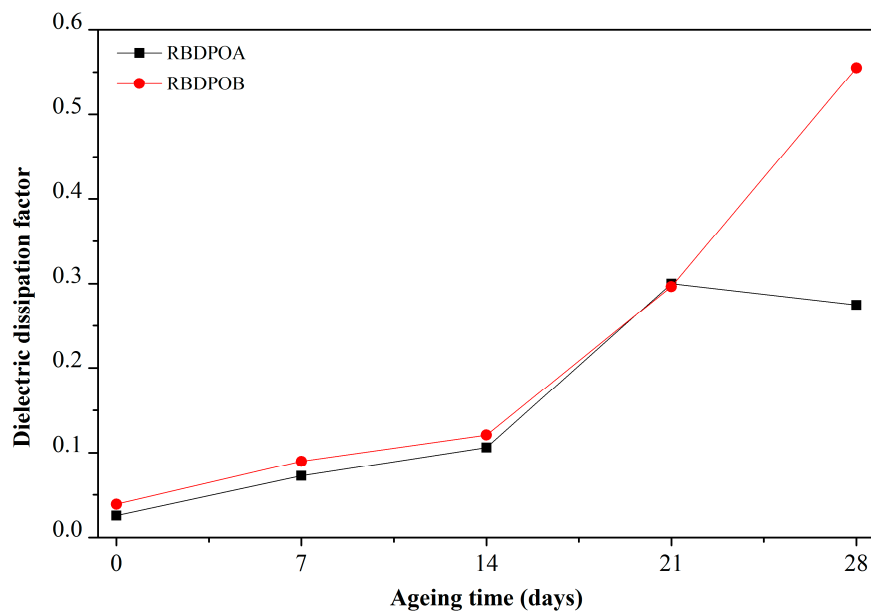
**Figure 5.** Probability plot of RBDPO aged at 170 °C under open conditions: (a) normal distribution and (b) Weibull distribution.

**Table 2.** Withstand voltage based on normal and Weibull distribution at 1% and 50% probability.

Samples	Ageing Time (Days)	Normal Distribution		Weibull Distribution	
		1%	50%	1%	50%
RBDPO A	0	23.56	39.37	21.6	39.97
	7	21.94	35.84	19.95	36.41
	14	24.18	36.43	22.22	36.98
	21	23.8	41.02	22.35	41.71
	28	28.23	43.96	25.86	44.63
RBDPO B	0	29.2	41.27	19.80	40.9
	7	37.38	52.71	37.14	53.51
	14	37.4	55.22	35.86	56.08
	21	26.23	45.19	23.90	45.86
	28	35.06	51.74	32.29	52.5

### 3.2.2. Dielectric Dissipation Factor

Figure 6 shows that the trends in the dielectric dissipation factors for both RBDPO are the same as for viscosity. The dielectric dissipation factor for RBDPO A increased steadily initially, and then slightly decreased after 21 days of ageing. The dielectric dissipation factor of RBDPO B increased exponentially throughout the ageing time.

**Figure 6.** Dielectric dissipation factor of RBDPO aged at 170 °C under open conditions.

### 3.2.3. Resistivity

The resistivities for both RBDPO showed clear decreasing trends as shown in Figure 7. RBDPO A experienced a steady reduction in resistivity until 21 days of ageing. The resistivity of RBDPO A remained almost unchanged between 21 and 28 days of ageing. The resistivity of RBDPO B decreased almost linearly as the ageing progressed. The resistivity of RBDPO A was slightly higher than RBDPO B at all ageing times. RBDPO A and RBDPO B suffered 86.6% and 93.7% reductions in resistivities at the end of the ageing time, respectively.

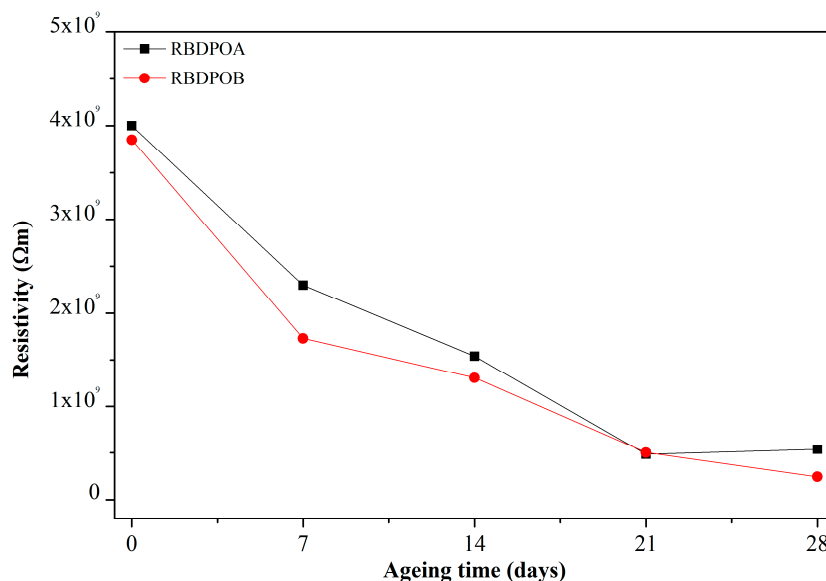


Figure 7. Resistivity of RBDPO aged at 170 °C under open conditions.

### 3.2.4. Relative Permittivity

Figure 8 shows the slightly increasing trend of the relative permittivity for both RBDPO. The relative permittivity of RBDPO A was almost unchanged after 21 days of ageing. The relative permittivity of RBDPO B increased almost linearly throughout the ageing period. After 28 of ageing, the relative permittivity of RBDPO A and RBDPO B increased by 7.8% and 12.9% as compared to the initial values, respectively.

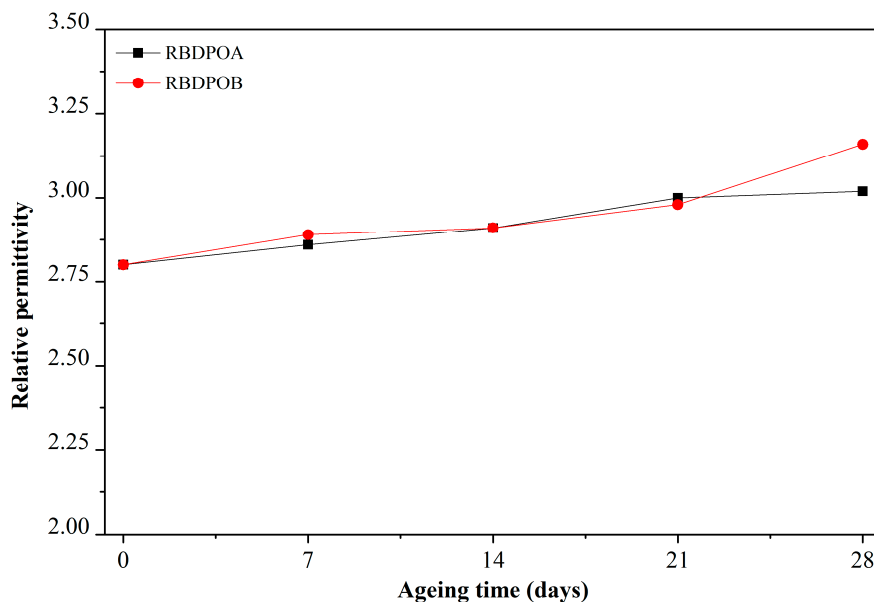


Figure 8. Relative permittivity of RBDPO aged at 170 °C under open conditions.

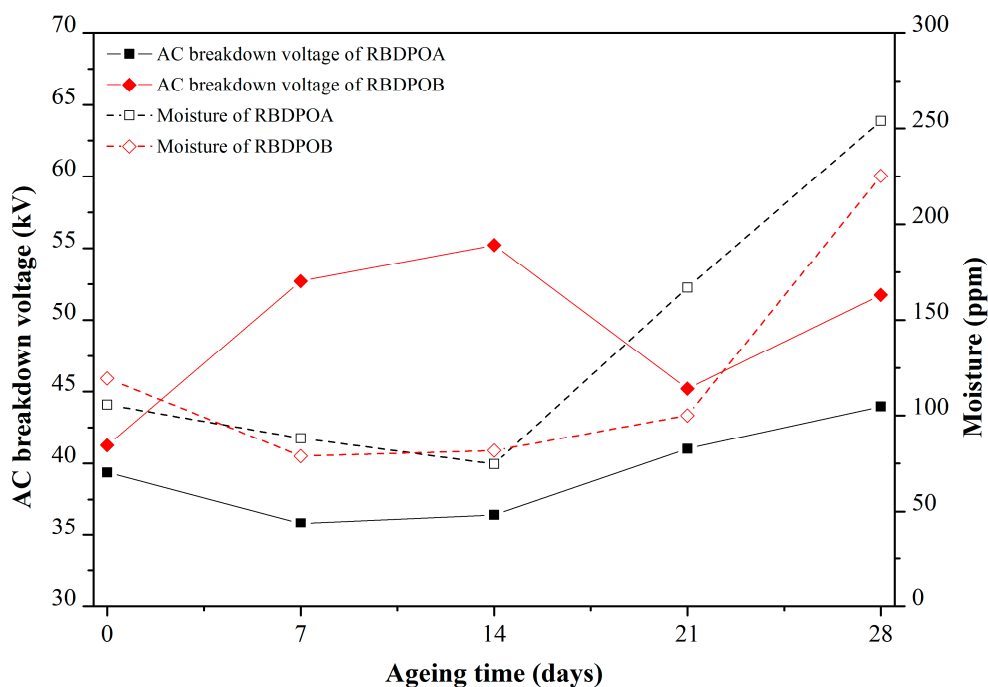
## 4. Discussion

A previous study [8] revealed that there is no apparent effect of the ageing on the physiochemical and electrical properties RBDPO at 115 °C under open conditions. However, the current study shows that several physicochemical and electrical properties of both RBDPO were affected by an ageing temperature of 170 °C under open conditions.

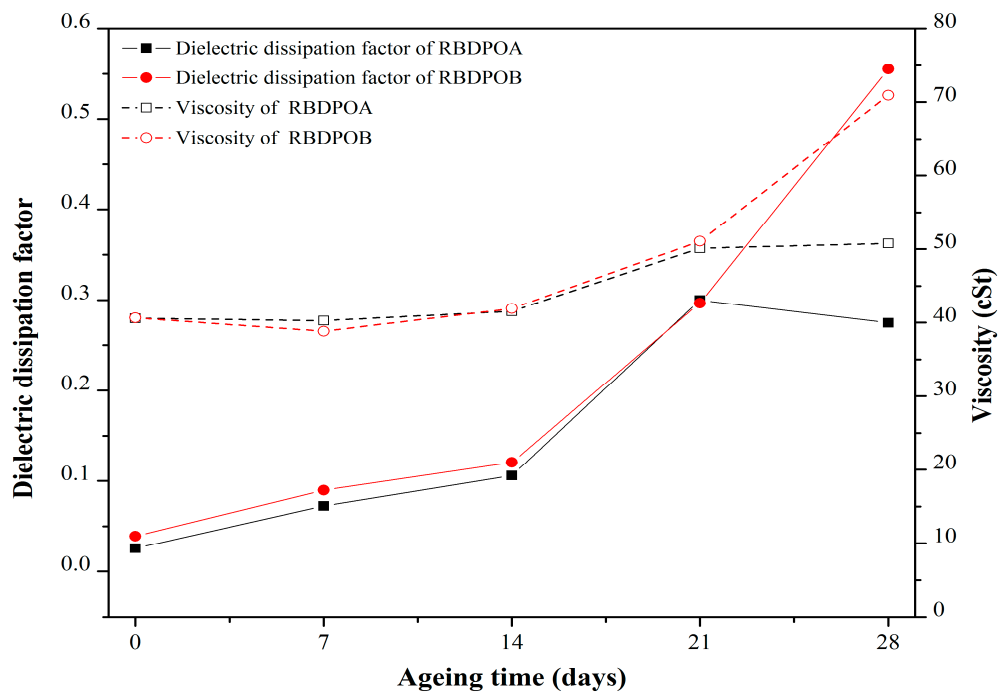


Apparent moisture increases were observed for both RBDPO (Figure 1). Apart from ageing, possible moisture ingress may have occurred from the exterior, since the experiment was performed under open conditions. In natural ester, the triglycerides will interact with moisture during hydrolysis to generate fatty acids [32]. However, the presence of moisture had no apparent effect on the hydrolysis of RBDPO, since the acidity remained low (Figure 2). The acidities of both RBDPO in this study remained lower than 0.005 mg KOH/g at all ageing times. Meanwhile, the increase in the viscosities of both RBDPO (Figure 3) were expected and in line with previous studies [14,20]. The triglycerides molecules were exposed to air, which in turn initiated the chain scission and cross linking [33]. Large molecules were generated that change the molecular weight and lead to changes in the viscosity of natural ester [33].

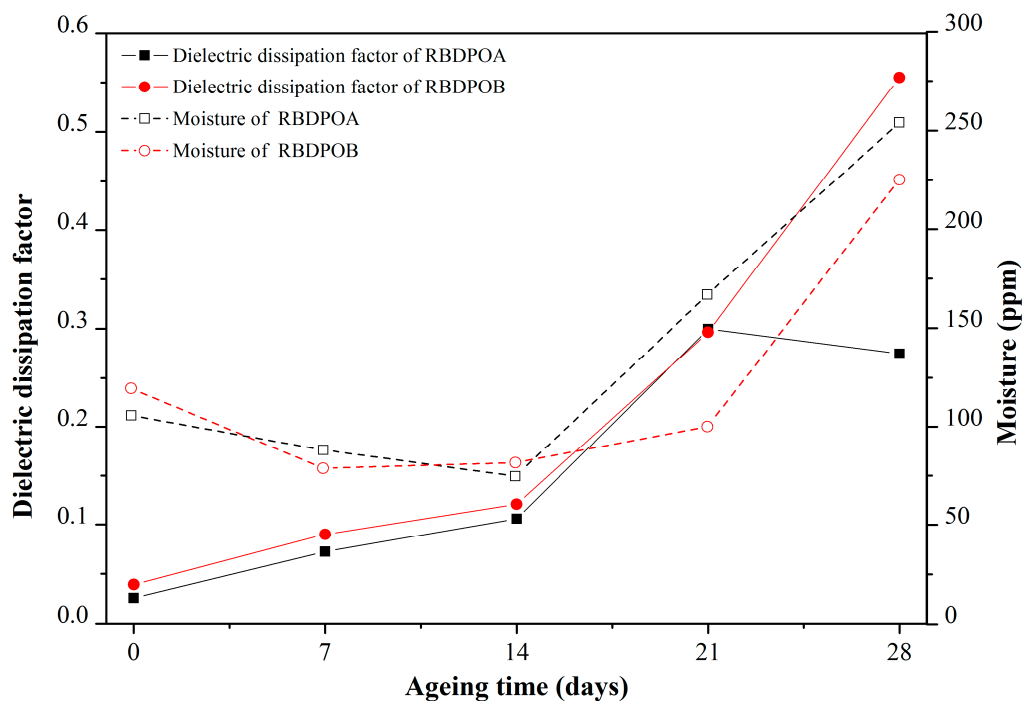
The effect of the current ageing study on the AC breakdown voltages of both RBDPO was not apparent, as shown in Figure 4. Furthermore, Figure 9 shows that no clear relationship exists between moisture and AC breakdown voltages of both RBDPO. Since the moisture saturation limit of RBDPO was not available, the effect of moisture on the AC breakdown voltage could not be examined extensively. The increases in the dielectric dissipation factors of both RBDPO were similar to viscosities (Figures 6 and 10). The dielectric dissipation factor is known as an indicator of the degree of contamination and moisture in dielectric insulating fluids [34]. Apart from moisture, large molecules generated as a result of thermal polymerization are suspected to also affect the dielectric dissipation factors and viscosities of both RBDPO, as shown in Figures 10 and 11 [35]. For both RBDPO, the decrease in the resistivities in Figures 7 and 12 are in line with the increase in moisture. The slight increase in the relative permittivity in Figures 8 and 13 are consistent with the increases in viscosities, which indicate changes in the chemical structures of both RBDPO.



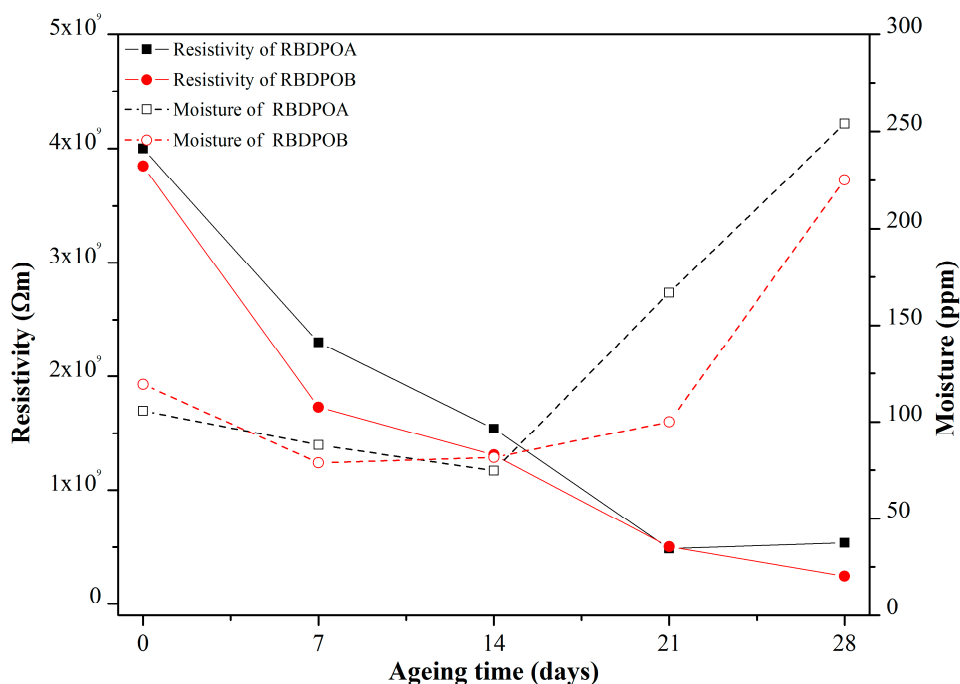
**Figure 9.** Relationship between AC breakdown voltage and moisture of RBDPO aged at 170 °C under open conditions.



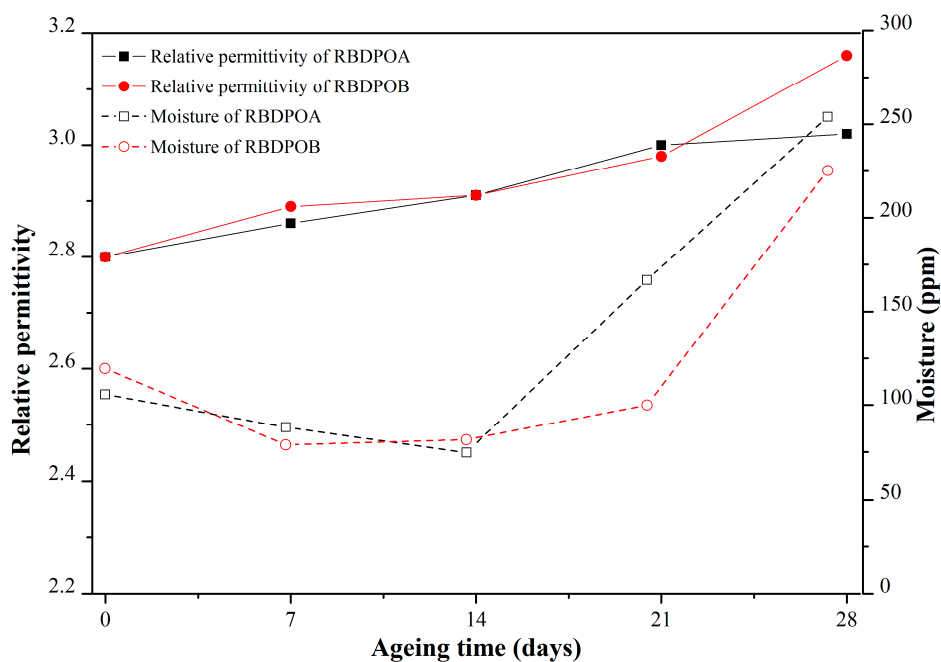
**Figure 10.** Relationship between dielectric dissipation factor and viscosity of RBDPO aged at 170 °C under open conditions.



**Figure 11.** Relationship between dielectric dissipation factor and moisture of RBDPO aged at 170 °C under open conditions.



**Figure 12.** Relationship between resistivity and moisture of RBDPO aged at 170 °C under open conditions.



**Figure 13.** Relationship between relative permittivity and moisture of RBDPO aged at 170 °C under open conditions.

## 5. Conclusions

In this study, the physiochemical and electrical properties of RBDPO were affected by the ageing scheme at 170 °C and in the presence of air. The moisture and viscosities of both RBDPO increased as the ageing time increased. The acidities of both RBDPO were not affected by the ageing and remained low throughout the ageing process.

The AC breakdown voltages of both RBDPO were not significantly affected as the ageing progressed. The dielectric dissipation factors, resistivities, and relative permittivities of RBDPO were affected by the ageing, where correlations between these parameters and moisture and viscosity were observed. Further study is required in the future to comprehensively examine the effect of high temperature ageing on RBDPO in the presence of insulation paper, pressboard, and metal catalyst.

**Author Contributions:** The research study was successfully completed with contribution from all authors. The main research idea, experimental works, and manuscript preparation were contributed by N.A.M. N.A. contributed to the manuscript preparation and research idea. J.J., M.Z.A.A.K. And R.Y. assisted with finalizing the research work and manuscript. Z.Y. provided several suggestions from an industrial perspective. All authors revised and approved the publication of the paper.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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