

Article

# Physicochemical Properties of Biodiesel Synthesised from Grape Seed, Philippine Tung, Kesambi, and Palm Oils

Hwai Chyuan Ong <sup>1,\*</sup>, M. Mofijur <sup>1,\*</sup>, A.S. Silitonga <sup>2</sup>, D. Gumilang <sup>2</sup>, Fitranto Kusumo <sup>1</sup> and T.M.I. Mahlia <sup>1</sup>

<sup>1</sup> School of Information, Systems and Modelling, Faculty of Engineering and Information Technology, University of Technology Sydney, 15 Broadway, Ultimo 2007, Australia; fitrantokusumo@yahoo.co.id (F.K.); TMIndra.Mahlia@uts.edu.au (T.M.I.M.)

<sup>2</sup> Department of Mechanical Engineering, Politeknik Negeri Medan, Medan 20155, Indonesia; ardinsu@yahoo.co.id (A.S.S.); gumilang.dewananta@gmail.com (D.G.)

\* Correspondence: hwaichyuan.ong@uts.edu.au (H.C.O.); mdmofijur.rahman@uts.edu.au (M.M.)

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**Abstract:** The production of biodiesel using vegetable oil is an effective way to meet growing energy demands, which could potentially reduce the dependency on fossil fuels. The aim of this study was to evaluate grape seed (*Vitis vinifera*), Philippine tung (*Reutealis trisperma*), and kesambi (*Schleichera oleosa*) oils as potential feedstocks for biodiesel production to meet this demand. Firstly, biodiesels from these oils were produced and then their fatty acid methyl ester profiles and physicochemical properties were evaluated and compared with palm biodiesel. The results showed that the biodiesel produced from grape seed oil possessed the highest oxidation stability of 4.62 h. On the other hand, poor oxidation stability was observed for Philippine tung biodiesel at 2.47 h. The poor properties of Philippine tung biodiesel can be attributed to the presence of  $\alpha$ -elaeostearic fatty acid. Furthermore, synthetic antioxidants (pyrogallol) and diesel were used to improve the oxidation stability. The 0.2 wt.% concentration of pyrogallol antioxidant could increase the oxidation stability of grape seed biodiesel to 6.24 h, while for kesambi and Philippine tung, biodiesels at higher concentrations of 0.3% and 0.4 wt.%, respectively, were needed to meet the minimum limit of 8 h. The blending of biodiesel with fossil diesel at different ratios can also increase the oxidation stability.

**Keywords:** biodiesel; alternative energy; antioxidant; blending ratio; biofuel; viscosity

## 1. Introduction

Fossil fuels are non-renewable energy sources used to support economic growth and are intricately woven into our daily lives. However, the excessive exploitation of fossil fuel has caused the depletion of reserves and negatively affected the environment, especially in relation to transportation [1]. At this moment, the most crucial issue is global warming due to fossil fuel combustion, which creates carbon dioxide [2,3]. Because of this negative environmental impact, researchers have tried to find alternative energy sources. Some of the most promising sources are renewable energy sources, including wind, wave, solar, and bioenergy [4–6]. Solar and wind energy are very promising; however, they are only available for a certain period and required storage devices. The only storage devices available commercially at the moment are batteries. Battery storage is very limited; hence, researchers have attempted to find other types of materials for energy storage [7–9]. Therefore, the industry is still very interested in liquid types of renewable energy that have no storage issues. There are two types of biofuel currently used for vehicles, namely bioethanol, which replaces gasoline, and biodiesel.

Biodiesel has appeared as an alternative energy source to meet the global energy demand, which at the same time could minimize environmental concerns [10–12].

Currently, 95% of global biodiesel is produced from edible oils, which causes competition between food resources and energy applications [13]. Biodiesel is more expensive than fossil fuel due to cost of procurement of raw materials, which accounts for about 80% of the total production cost [14,15]. In order to promote the commercialization of biodiesel, non-edible feedstock sources are a possible solution [16]. Recently, a study showed microalgae-based biodiesel fuels to be promising energy sources due to their high yield [17–19]. The benefits of utilizing biodiesels are that they are (a) renewable, (b) biodegradable, and most importantly, (c) that they can be used neat or blended with diesel fuel, without the need for modification of existing diesel engines [20–23]. Many researchers have proven that the carbon monoxide (CO) and hydrocarbon (HC) emissions and the smoke opacity are lower for all types of biodiesel blends [24–26].

Biodiesels consist of fatty acid methyl ester (FAME) compounds and are generally synthesized by base-catalyzed transesterification of vegetable oils or animal fats with alcohol at low temperatures and pressures [27]. Sodium hydroxide (NaOH) and potassium hydroxide (KOH) are usually used as base catalysts in the transesterification process. These catalysts have higher reaction rates, and are readily available and inexpensive. Depending on the amount of free fatty acid (FFA) present in the oil, its usage may lead to the formation of soap, causing lower biodiesel yield [28]. The usage of acid catalysts such as sulphuric acid ( $H_2SO_4$ ), hydrochloric acid (HCl), and phosphoric acid ( $H_3PO_4$ ) could overcome this issue. Additionally, utilization of a heterogeneous catalyst and a lipase-catalyzed transesterification process can also solve this issue, providing high sustainability, high surface area, and high porosity [29,30]. However, those heterogeneous catalysts and lipase-catalyzed transesterification processes are much more expensive compared with other homogeneous catalysts. As a pretreatment step, FFA and water are converted into ester before transesterification [31]. However, a higher ratio of alcohol to oil and longer reaction time are needed to complete the reaction. The resultant biodiesel contains a high amount of oxygen but almost zero sulphur content. This helps to reduce tail pipe emissions, especially particulate matter, sulphur dioxide, carbon dioxide, and unburned hydrocarbon, when compared to diesel [32].

Oxidation stability remains the major drawback pertaining to the quality and commercialization of biodiesel [33]. Due to the higher tendency for oxidation compared to diesel, extended storage is a problem. The instability is mainly caused by the fatty acid composition, water content, metal traces, and storage conditions, such as the temperature, light, and material used for storage [34]. The affected parameters would be higher kinematic viscosity, density, and acid values due to the formation of allylic hydroperoxides [35]. An antioxidant can be used to increase the oxidation stability in these cases [36]. The hydroxyl group contained in antioxidants allows the donation of hydrogen to free radicals to form a stable compound, interrupting the propagation of free radicals [37,38].

In this work, the potential use of *Vitis vinifera*, *Reutealis trisperma*, *Schleichera oleosa*, and palm oil in biodiesel production and the resultant biodiesel conformity to ASTM D6751 and EN 14214 biodiesel standards were studied. The impacts of the addition of the phenolic antioxidant pyrogallol (PY) and the blending ratio on grape seed, Philippine tung, and kesambi biodiesels were also investigated. For blending comparison, the ASTM D7467 standard was used for B5 to B20 blends.

## 2. Materials and Methods

### 2.1. Materials and Reagents

Palm oil methyl ester (POME) (or palm biodiesel), which is clear and transparent, was purchased from Forest Research Institute Malaysia (FRIM). Grape seed oil and crude kesambi oil were obtained from Basso Fedele and Figli S.R.L., Italy, distributed by the Sunland Volonte Agency in Malaysia and from local farmers in Probolinggo, East Java, Indonesia respectively. The Philippine tung seeds were collected from Majalengka, West Java, Indonesia. The crude Philippine tung oil was extracted manually

using a meal grinder and hydraulic press. All chemicals, such as methanol, ethanol, phosphoric acid ( $\text{H}_3\text{PO}_4$ ), sulfuric acid ( $\text{H}_2\text{SO}_4$ , 96%), potassium hydroxide (KOH), and sodium hydroxide (NaOH), were purchased from Sigma-Aldrich, Malaysia. The pyrogallol (PY) antioxidant, having a purity of 99%, was supplied by Axon scientific, Malaysia.

#### 2.1.1. Palm (*Elaeis guineensis*)

*Elaeis guineensis* belongs to the Arecaceae family and is generically known as the palm tree. It is an indigenous plant of West Africa, between Angola and Gambia. Palm trees have large pinnate leaves (3–5 m long) and can reach a height of 20–30 m. The annual palm oil yield is reported to be 10–35 tonnes/ha from the fresh fruit bunches, with each piece of fruit in a fruit bunch containing 50% oil [39].

#### 2.1.2. Grape Seed (*Vitis vinifera*)

*Vitis vinifera* commonly refers to the grape tree, which belongs to the Vitaceae family [40]. The fruit contains amino acids, micronutrients, water, minerals, and sugars [41]. Growing up to 34 m in height, the plant is a primary source of wine and generates 99% of the world's wine. The plant is primarily grown for wine grapes, but also includes table and raisin grapes. The grape seed produces 10–20% (w/w) oil, which contains a high percentage of unsaturated fatty acids, especially linoleic acid (67.15–69.58%) [42].

#### 2.1.3. Philippine Tung (*Reutealis trisperma*)

*Reutealis trisperma* is a fast-growing tree that belongs to the Euphorbiaceae family and the genus *Reutealis* Airy Shaw [43]. The plant is native to the Philippines, but is now widespread in Majalengka and Garut, West Java, Indonesia. The diameter of the trunk ranges from 75 to 200 cm, with a height of 10–15 m. The Philippine tung produces seeds once a year and reaches its maximum productivity by four years. The seed contains 45–50% (w/w) pale yellow oil [44].

#### 2.1.4. Kesambi (*Schleichera oleosa*)

*Schleichera oleosa* is a member of the Sapindaceae family. It is generally known as the kusum tree in India and the kesambi tree in Indonesia and Malaysia [45]. It originated in South and Southeast Asia, however the plant is also grown in countries such as India, Bangladesh, Myanmar, and Sri Lanka, with similar cultivation cultures [46]. Kesambi plant grows well on light, well-drained gravelly or loamy soil and is drought-tolerant. Traditionally, the kesambi oil is used for culinary, lighting, medicinal, hairdressing, pain treatment, itch relief, and hair growth purposes in India [47]. Kesambi plant is often used to produce a high-quality lacquer gum, where this tree is commonly referred to as the lac tree [46]. The oil content of the seed is around 59–72%, with a yellowish green color.

### 2.2. Equipment and Apparatus Setup

The biodiesel production was conducted using three neck flasks together as a reactor. The three-neck flask was placed on a heating plate equipped with a magnetic stirrer. A Friedrichs condenser was fixed together with the reactor to avoid the evaporation of alcohol during the heating process. A thermometer was also fitted using a retort stand and immersed into the reactor to measure the reaction temperature during biodiesel production.

### 2.3. Biodiesel Production

#### 2.3.1. Grape Seed Biodiesel

The production of grape seed biodiesel (GSME) involves a double base catalyzed transesterification process, as it has a low acid value of 0.2 mg KOH/g. First, 500 mL oil with 32% (v/v) of methanol and 0.5 wt.% of KOH is transesterified at 60 °C for 1 h at 1000 rpm. Then, the same oil is mixed with 0.5% (v/v) of methanol and 0.1 wt.% of KOH at the same speed and temperature,

and for the same duration. The resultant crude biodiesel is washed with 50 °C distilled water and vacuum dried at 70 °C for 2 hours to evaporate the extra water and methanol.

### 2.3.2. Philippine Tung Biodiesel

To produce Philippine tung biodiesel (PTME), three steps were involved, due to its high acid value of 15 mg KOH/g. The first step was the degumming process to remove the gum from the crude oil. Then, 0.1 wt.% H<sub>3</sub>PO<sub>4</sub> (85% concentration) was added to preheated crude oil (90 °C) for 10 min and the mixture was stirred at 1000 rpm, then allowed to stand. Gum appeared at the lower layer and was removed from the oil before washing with 10% (w/w<sub>oil</sub>) distilled water.

The esterification process was performed by mixing the oil with methanol (10:1 molar ratio of methanol/oil) and H<sub>2</sub>SO<sub>4</sub> as acid catalyst (1% (v/v) of oil). The reaction was maintained at 65 °C with constant stirring at 1000 rpm for 3 h, and then it was allowed to cool to room temperature (28 °C). In the transesterification process, the NaOH (1% (w/w) of oil) as the base catalyst was mixed with 20% methanol by volume of oil into the esterified Philippine tung oil and left to react for 2 hours. The mixture was transferred into a separating funnel and allowed to settle for 24 h. The bottom layer containing methanol, glycerol, and catalyst was removed, and the upper layer was washed with warm distilled water (50 °C). The final product (biodiesel) was dried at 70 °C for 2 hours to evaporate the extra water and methanol.

### 2.3.3. Kesambi Biodiesel

Kesambi oil methyl ester (KOME) was prepared following the procedure described by Sudradjat et al. [48]. The degumming process was performed with 0.1 wt.% H<sub>3</sub>PO<sub>4</sub> (85% concentration) at 90 °C for 15 min. Then, the esterification process was conducted under the following conditions: reflux temperature of 60 °C, molar ratio of methanol/oil of 10:1, and with 1% (v/v) H<sub>2</sub>SO<sub>4</sub> as an acid catalyst. Next, the 8:1 molar ratio of methanol/oil and KOH (1% (v/v) of oil) were transesterified at 1000 rpm at 60 °C for 1 h. The mixture was allowed to separate in a separating funnel. The resultant methyl esters were washed with warm distilled water (50 °C) until the pH of the washed water became neutral. Finally, the produced KOME was dried at 70 °C for 2 hours to evaporate the extra water and methanol.

## 2.4. Biodiesel Characterization

Table 1 shows the important biodiesel fuel properties and equipment used in this study, according to ASTM D6751 and EN14214 standards [49–51].

**Table 1.** Equipment and test methods for biodiesel properties.

Properties	Test Method	Equipment
Flashpoint	ASTM D93	NPM 440 automatic (Normalab, France)
Cloud point	ASTM D2500	NTE 450 automatic (Normalab, France)
Pour point	ASTM D97	NTE 450 automatic (Normalab, France)
Cold filter plugging point (CFPP)	ASTM D6371	NTE 450 automatic (Normalab, France)
Kinematic viscosity at 40 °C	ASTM D445	SVM 3000 (Anton Paar, UK)
Density at 15 °C	ASTM D1298	DM 40 (Mettler Toledo)
Oxidation stability	EN 14112	873 Rancimat (Metrohm, Switzerland)
Carbon residue (100% sample)	ASTM D4530	NMC 440 automatic (Normalab, France)
Copper corrosion	ASTM D130	Seta copper corrosion bath 11300-0 (Stanhope-Seta, UK)
Sulfur content (S500 grade)	ASTM D5453	Multi EA 5000 elemental analyzer (analytikjena, UK)
Water content	EN ISO 12937	831 KF coulometer (Metrohm ion analysis)
Moisture	EN ISO 12937	831 KF coulometer (Metrohm ion analysis)
Calorific value (Gross)	-	Parr 6200 calorimeter (Parr instrument, US)

## 2.5. Antioxidants

The addition of antioxidants can significantly improve the induction period (IP) of biodiesels [52,53]. The effects of different concentrations of the phenolic antioxidant pyrogallol (PY) from 0.1 wt.% to

0.5 wt.% of biodiesel on the biodiesel's oxidation stability and kinematic viscosity were investigated. Table 2 shows the physicochemical properties of PY.

**Table 2.** Physicochemical properties of the antioxidant pyrogallol (PY).

Antioxidant	CAS Number	Molecular Formula	Molecular Weight (g/mol)	Melting Point (°C)	Boiling Point (°C)	Formula	Reference
Pyrogallol	87-66-1	C <sub>6</sub> H <sub>6</sub> O <sub>3</sub>	126.11	131–134	309	C <sub>6</sub> H <sub>6</sub> O <sub>3</sub>	[54]

## 2.6. Blending

In this research, POME, GSME, PTME, and KOME were blended with diesel at different ratios of B10, B20, B40, and B50 to examine the effect of blending on the oxidation stability, kinematic viscosity, and flashpoint. Their conformity against the ASTM D7467 specification for diesel blends of B6–B20 was checked.

## 3. Results and Discussion

### 3.1. Fatty Acid Composition of Biodiesels

Table 3 showed the fatty acid composition of all the produced biodiesels. For palm oil biodiesel, the percentage of saturated fatty acid was slightly higher than unsaturated fatty acid, while for Kesambi biodiesel, an inverse trend of total unsaturated fatty acids having a slightly higher percentage than saturated fatty acids was observed. On the other hand, grape seed biodiesel and Philippine tung biodiesel showed more extreme ratios where the unsaturated fatty acids accounted for more than 75% of the total fatty acid composition. The research in the past has indicated that the composition of fatty acids has a direct effect on some of the most important physicochemical properties of biodiesel, such as oxidation stability, kinematic viscosity, and cold flow properties. It is important to note that the highest percentage of total unsaturated fatty acid of grape seed biodiesel was mainly attributed to the high percentage of linoleic acid (C18:2) at 70.2%, in agreement with the results reported by Fernández et al. [55].

**Table 3.** Fatty acid composition of biodiesels.

Fatty Acid Name		Composition (wt. %)			
		Palm Oil	Grape Seed Oil	Philippine Tung Oil	Kesambi Oil
Lauric acid	C12:0	0.3	-	0.1	-
Myristic acid	C14:0	1.4	-	0.1	-
Palmitic acid	C16:0	47.2	6.8	14.7	11.7
Palmitoleic acid	C16:1	-	0.1	0.5	1.1
Stearic acid	C18:0	4.1	3.8	6.6	3.9
Oleic acid	C18:1	36.4	18.5	31.3	45.4
Linoleic acid	C18:2	10.5	70.2	38.2	7.2
Linolenic acid	C18:3	-	0.3	0.3	0.4
$\alpha$ -elaeostearic acid	C18:3	-	-	2.3	-
Arachidic acid	C20:0	-	0.2	0.2	30.2
Gondoic acid	C20:1	-	-	0.2	-
Behenic acid	C22:0	-	-	1.1	-
Erucic acid	C22:1	-	-	4.3	-
Lignoceric acid	C24:0	-	-	0.1	-
Total unsaturated fatty acids		46.9	89.1	77.1	54.1
Total Saturated fatty acids		53.0	10.8	22.9	45.8

Philippine tung biodiesel also contained high amounts of unsaturated fatty acids at 77.1% but was more evenly distributed between monounsaturated fatty acids (36.3%) and polyunsaturated

fatty acids (40.8%). The presence of fatty acid  $\alpha$ -elaeostearic at 2.3%, which was not found in the other three biodiesels tested, was also present in the Tung tree oil, *Vernicia Montana* (Lour.), and *Vernicia fordii* (Hemsl.) [56]. The total unsaturated fatty acid content of kesambi biodiesel was made up of polyunsaturated fatty acids (7.6%) and a higher amount of monounsaturated fatty acid (46.5%). Its saturated fatty acid content amounted to 45.8%, mainly due to the presence of palmitic acid (C16:0) at 11.7% and arachidic acid (C20:0) at 30.2%. A similar composition for kesambi oil was also reported by Sharma et al. [46].

### 3.2. Biodiesel Characteristics

Table 4 compares the fuel properties of non-edible biodiesel GSME, PTME, and KOME with that of edible biodiesel POME against the ASTM D6751 and EN 14214 biodiesel standards. Generally, all tested biodiesels conformed and fulfilled the biodiesel standard ASTM D6751, except PTME for oxidation stability, which was only 2.47 hours. However, the POME did not comply with CFPP. All the fuel properties are individually discussed in the following section.

**Table 4.** Characteristics of biodiesels.

Properties	Unit	POME	GSME	PTME	KOME	ASTM D6751	EN 14214
Flashpoint	°C	178	188	168	173	130 minimum	120 minimum
Cloud point	°C	10	−5	2	5	−3 to 12	-
Pour point	°C	3	−5	1	4	−15 to 16	-
Cold filter plugging point (CFPP)	°C	11	0	0	4	-	+5 maximum
Kinematic viscosity at 40 °C	mm <sup>2</sup> /s	4.65	4.22	4.95	4.71	1.9 to 6.0	3.5 to 5.0
Density at 15 °C	kg/m <sup>3</sup>	876.9	886.7	889.6	875.6	-	860–900
Oxidation stability	h	3.57	4.62	2.47	3.21	3 minimum	8 minimum
Carbon residue (100% sample)	% m/m	0	0	0	0	0.050 maximum	0.3 maximum
Copper corrosion	-	1a	1a	1a	1a	3 maximum	class 1
Sulfur content (S500 grade)	ppm	3.74	2.64	5.02	3.83	500 maximum	-
Water content	ppm	472.4	246.7	356.2	Nil	500 maximum	500 maximum
Moisture	wt.%	0.0475	0.025	0.035	Nil	-	0.05 maximum
Caloric value (Gross)	MJ/kg	44.80	38.89	40.24	42.27	-	-

POME: Palm Oil Methyl Ester; GSME: Grape Seed Methyl Ester; PTME: Philippine Tung Methyl Ester; KOME: Kesambi oil Methyl Ester.

#### 3.2.1. Oxidation Stability

Meeting the oxidation stability limit is crucial for biodiesel storage and transportation purposes, as reported by Bouaid et al. [57]. In Table 4, GSME shows the highest stability at 4.62 h, followed by POME, KOME, and PTME at 3.57 h, 3.21 h, and 2.47 h, respectively. These values were far below that of petrol diesel at 59 h [58]. The low oxidation stability of biodiesel is caused by the presence of double bonds in unsaturated fatty acids found in biodiesels. However, the high level of water content may also contribute to lower oxidation stability in the final produced biodiesel. Although all biodiesels except for PTME generally meet the recommended oxidation stability limit of 3 h stipulated by ASTM D6751, nevertheless, for storage and transportation security, higher stability would still be preferred. The low oxidation stability of PTME might be attributed to the presence of erucic acid, which is a very long chain monounsaturated fatty acid that is more readily biodegradable than others. The addition of antioxidants can increase the oxidation stability of biodiesels.

#### 3.2.2. Kinematic Viscosity

Viscosity is a measurement of the resistance of a liquid to flow. A fuel with high viscosity will affect the operation during combustion due to poor spray and atomization, especially at low temperatures [59].



Oil and fat have very high viscosity and can be used directly in an engine. The transesterification of oil into fatty acid methyl esters (biodiesel) solves this issue. All biodiesels produced satisfy the limit set by both ASTM D6751 (1.9–6.0 mm<sup>2</sup>/s) and EN 14214 (3.5–5.0 mm<sup>2</sup>/s). The lowest kinematic viscosity recorded was for GSME at 4.22 mm<sup>2</sup>/s, while the highest was for PTME at 4.95 mm<sup>2</sup>/s. The low kinematic viscosity of GSME may be caused by the presence of the high linoleic acid (C18:2) content of 70.2%. Rodrigues et al. [60] mentioned that linoleic acid, the primary fatty acid of grape seed oil, showed significantly lower kinematic viscosity than the stearic, oleic, and palmitic acids. These are the primary fatty acids found in palm, Philippine tung, and kesambi oil. Additionally, the high linoleic acid content of GSME will lower the viscosity, as the presence of two or three double bonds will reduce the viscosity [61]. Thus, PTME and KOME have higher viscosity compared with GSME.

### 3.2.3. Flashpoint

Flashpoint denotes the temperature at which a fuel will ignite when exposed to a flame or spark. This parameter also indicates the volatility and contamination of leftover alcohol (methanol or ethanol) during the production of biodiesel [62]. The flashpoints of POME, GSME, PTME, and KOME measured in this study were 178 °C, 182 °C, 168 °C, and 173 °C respectively. They fulfilled the ASTM D6751 requirement of 130 °C min and EN 14214 of 101 °C minimum standards.

### 3.2.4. Cloud Point (CP), Pour Point (PP), and Cold Filter Plugging Point (CFPP)

The cloud point denotes the temperature at which a cloudy appearance appears in biodiesels, while the pour point marks the lowest temperature at which a fuel becomes semi-solid and loses its flow characteristics [63]. CFPP is the temperature at which the filter starts to plug and begins to crystallize. The limit set for each temperate country depends on the country's longitude.

GSME had the best cold flow properties for CP and a PP of −5 °C, the lowest among the four tested biodiesels (Table 4). On the other hand, POME was the worst among the four, where at 11 °C it started to plug the filters. Past studies have found that the higher the percentage of unsaturated fats in the fatty acid profile of a feedstock, the better its winter usability. Both GSME and PTME have a high amount of total unsaturated fatty acids compared with POME and KOME. Nonetheless, only GSME fulfilled the ASTM D6751 limit ranges for CP, PP, and CFPP.

### 3.2.5. Calorific Value

The calorific value of a fuel is defined as the heat that is released during its combustion [64]. As shown in Table 4, the highest caloric value of 44.8 MJ/kg was attained for POME, followed by KOME at 42.27 MJ/kg, PTME at 40.24 MJ/kg, and GSME at 38.89 MJ/kg. However, these values were slightly lower than diesel fuel at 45.76 MJ/kg, due to the oxygen content in the biodiesel fuels [65]. The presence of oxygen will assist the incomplete combustion of a fuel, leading to a better emissions profile.

### 3.2.6. Other Properties

Other properties, such as water and moisture contents, density, carbon residue, sulphur content, and copper corrosion, were within the specifications set by ASTM D6751 and EN 14214 standards.

## 3.3. Effects of Antioxidants on Oxidation Stability and Kinematic Viscosity of Biodiesels

### 3.3.1. Oxidation Stability

The results obtained in this study show that PY can enhance the oxidative stability of the biodiesels (Figure 1). A substantial improvement was visible for POME with every 0.1 wt.% increase of PY. The oxidative stability extended to at least 30% from the initial reading. At 0.3 wt.%, a two-fold increase of 8.09 h was observed, and the same increase occurred again from 7.58 h to 15.67 h at 0.2 wt.%. All the other biodiesel also showed a gradual increase in oxidation stability with every 0.1 wt.% interval. The minimum oxidation stability for the EN 14214 standard is 8 h; POME and GSME managed to

meet the requirement at 0.2 wt.% PY addition, while PTME was unable to meet the minimum 8 h requirement set by EN 14214.

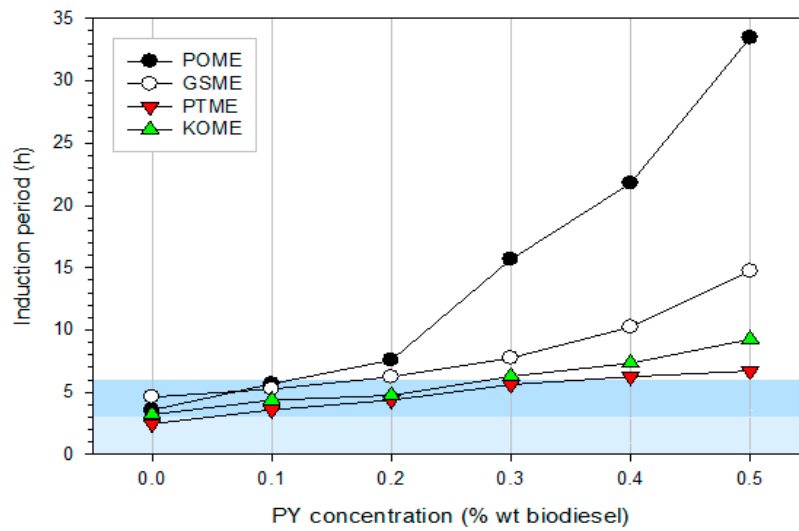


Figure 1. Effects of different pyrogallol (PY) concentrations on the oxidation stability of biodiesel.

### 3.3.2. Kinematic Viscosity

All of the biodiesels showed a gradual increase (from 0.2 to 1.1% of initial reading) in kinematic viscosity when subjected to increasing antioxidant concentration from 0.1 wt.% to 0.5 wt.% (Figure 2). Among all, KOME showed the highest increase with viscosity (1.1%) from 4.71 mm<sup>2</sup>/s to 4.76 mm<sup>2</sup>/s with the addition of 0.1 wt.% PY, followed by GSME, PTME and POME. All biodiesels still satisfied the ASTM D 6751 limit, even with the addition of PY up to 0.5 wt.%; however, if subjected to the EN14214 limit (3.5–5.0 mm<sup>2</sup>/s), PTME values were out of the stipulated range at 0.3 wt.% addition of PY and above.

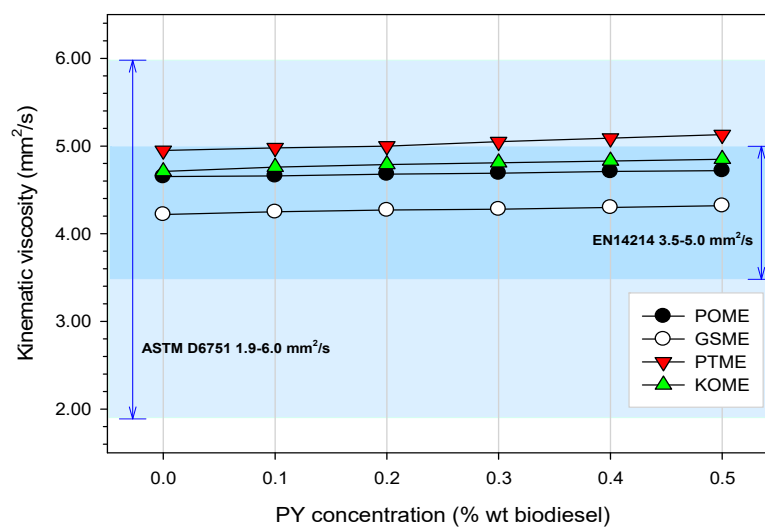


Figure 2. Effects of different PY concentrations on the kinematic viscosity of biodiesel.



### 3.4. Effect of Blending Ratio on Some Physicochemical Properties of Biodiesels

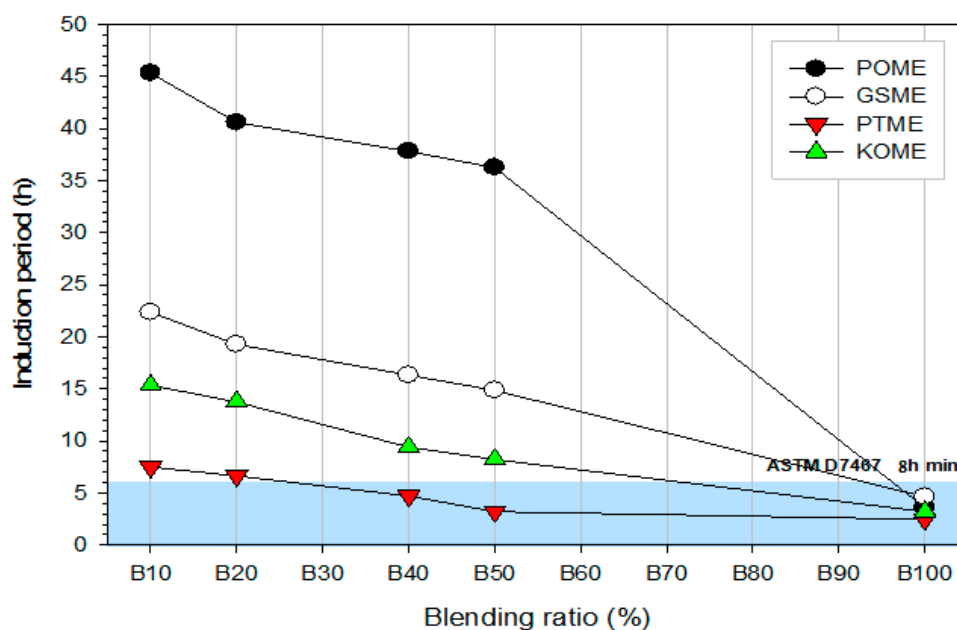
The influence of blending on oxidation stability, kinematic viscosity, and the flashpoint is discussed in the following section. It is important to note that for blending B6–B20, ASTM D7467 (Table 5) should be applied.

**Table 5.** ASTM D7467 requirements for diesel blends B6–B20.

Properties	Unit	Test Method	ASTM D7467 [66]
Oxidation stability	h	EN 15751	6 min
Kinematic viscosity at 40 °C	mm <sup>2</sup> /s	ASTM D445	1.9–4.1
Flashpoint	°C	ASTM D93	52 min

#### 3.4.1. Oxidation stability

Figure 3 shows the influence of blending on biodiesel oxidation stability. The minimum oxidative stability for B6–B20 was reached at 8 h [67,68]. All biodiesel blends fulfilled the minimum requirement of 8 h, except for PTME for B40 and higher; however, this limit only applies to a specific blending ratio. Blending of biodiesel significantly improved the oxidative stability of biodiesels. For example, POME was improved at B10 grade, for which the oxidative stability was enhanced 12-fold to 45.32 h compared with neat POME (B100) at 3.57 h. The same trend was also observed for other biodiesels, although the degree of improvement differed.



**Figure 3.** Effects of biodiesel–diesel blends on oxidation stability.

#### 3.4.2. Kinematic Viscosity

Figure 4 depicts the effects of the blending ratios on the kinematic viscosity of POME, GSME, PTME, and KOME. For lower percentage blending of B10 and B20, all biodiesels fulfilled the limit range set by ASTM D7467 of 1.9–4.1 mm<sup>2</sup>/s. Hasan and Rahman [69] also observed the same trend in their review. At B40 and above, PTME and KOME failed to meet the range limit. This limit range is for blends of B6–B20 only.

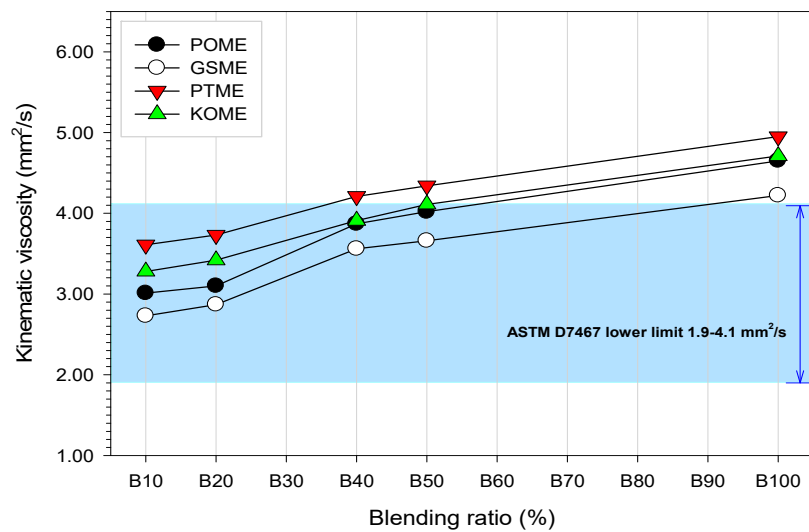


Figure 4. Effects of biodiesel–diesel blends on kinematic viscosity.

### 3.4.3. Flashpoint

Figure 5 illustrates the results of a flashpoint for different biodiesel blends. In this study, the measured flashpoints of different biodiesel blends (B10, B20, B40, and B50) were higher than diesel fuel (78 °C). This is in line with the average flashpoint reported by [69] of about 107.75 °C for all biodiesel–diesel blends, which is a significant increase of 68.67% in comparison with diesel. All tested blends fulfilled the limit set for B6–B20 at 52 °C min, as stipulated in ASTM D7467.

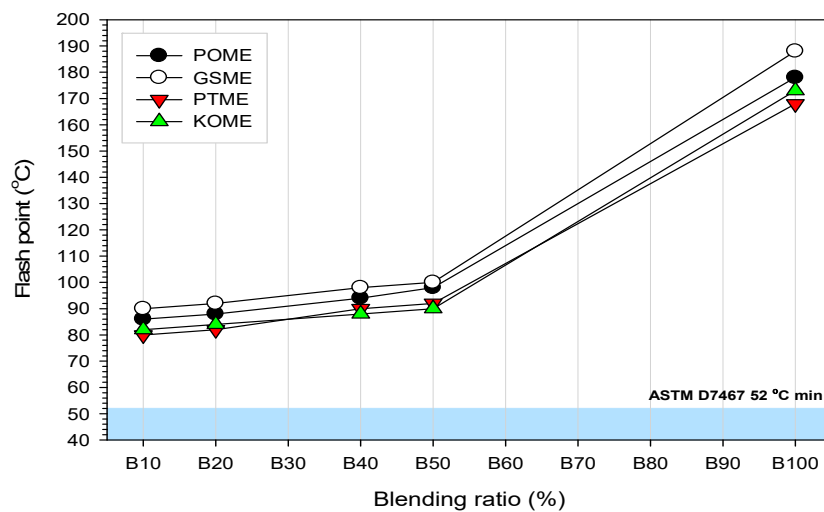


Figure 5. Effects of biodiesel–diesel blends on flashpoint.

## 4. Conclusions

In this study, biodiesel was produced from palm oil, grape seed, Philippine tung, and kesambi. Following this, the fatty acid methyl ester profiles were evaluated to identify the composition of each biodiesel. Finally, various physicochemical properties were evaluated, both by blending at different ratios with diesel and through adding an antioxidant. The following results were obtained:

- From the FAME analysis, palm oil and kesambi oil biodiesels had similar profiles, containing a 1:1 mixture of unsaturated and saturated fatty acids, while grape seed oil and Philippine tung oil biodiesels mostly contained unsaturated fatty acids.
- All tested pure biodiesels fulfilled the biodiesel standards for ASTM D6751, except for oxidation stability for PTME. However, POME did not fulfil the cold flow properties.

- (c) The addition of the phenolic antioxidant PY significantly increased the oxidative stability of the tested biodiesels.
- (d) All the biodiesel blends met the ASTM D7467 standards for blends of up to 20%. However, GSME and POME blends met the ASTM D7467 requirements up to 50%.
- (e) All of the biodiesel blends exceeded the minimum flashpoint requirement for the ASTM standard, which means all of them are safe for use. Furthermore, GSME exhibited the highest flashpoint for all blend ratios.

Blending of biodiesel and diesel causes an increase in the oxidative stability, especially at low concentrations; however, this lowers the kinematic viscosity and flashpoint of biodiesels. The results of this study demonstrate that grape seed, Philippine tung, and kesambi are some promising feedstock substitutes to edible palm oil for biodiesel production. The addition of antioxidants can improve the oxidation stability of biodiesels. As all the produced biodiesels met the standards, the decision for commercialization should consider the price of feedstock oil, availability, other uses of the feedstock, and the amount of chemicals and conversion steps required for commercial production of biodiesel.

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