

Research Article

Effect of Selected Metal Contaminants on the Stability of Castor Oil Methyl Ester

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This study investigates the effect of iron (Fe) and aluminium (Al) contaminants on the stability of castor oil methyl ester (COME). COME was synthesized via transesterification reaction and the peroxide value of the COME was used to monitor its stability. Varied amounts of Fe and Al (0.2 g, 0.4 g, 0.6 g, 0.8 g, and 1.0 g) in powdered form were added to 100 mL of COME and their effect on the peroxide value was monitored for a day, for the first phase of the experiment, while for the second phase, 0.3 g and 0.8 g of these metals were added to 100 mL COME, and their effect was monitored for 9 days. At the end of the study, it was observed that as the amount of the metals added to the COME increases from 0.2 to 1.0 g/100 mL COME, the peroxide values of the COME decrease from 1.6 to 0.3 meq/kg and 1.2 to 0.3–0.2 meq/kg for Al and Fe contaminated COME after 24 h. Also in the second phase of the experiment, the peroxide value of the 0.3 g Fe metal contaminated COME increased from 1.4 to 11.6 meq/kg while that of 0.8 g Fe contaminated COME increased from 1.4 to 3.0 meq/kg after the nine-day period of investigation.

1. Introduction

Biodiesel also known as fatty acid alkyl esters (FAAE) is an alternative fuel, derived from vegetable oils, animal fats, or waste cooking oils through transesterification of triglycerides with an alcohol in the presence of a catalyst [1–5]. In recent years, researches on biodiesel have attracted a considerable amount of attention because of its renewability and its several advantages over the conventional petroleum diesel. These advantages include nontoxicity, biodegradability, and the reduction in vehicle emissions such as carbon monoxide and particulate matters [6]. The use of vegetable oil derivatives as direct replacements for diesel fuel is not only environmentally advantageous but is also rapidly becoming an economically viable strategy for sourcing part of the global fuel oil requirements. The use of edible vegetable oils for biodiesel production has not been successful in some parts of the world because of its unstable price [7]. As the demand for vegetable oils for food has increased substantially in recent years, it has become necessary to use nonedible oils for biodiesel production. Castor oil has been identified as an important nonedible source for the production of biodiesel [5, 8]. Castor

crops, from which castor oil is obtained, require relatively fewer inputs such as water, fertilizers, and pesticides. These crops can be grown on marginal land, thus providing an excellent opportunity for many regions of the world to utilize their land resources more effectively.

The properties of biodiesel depend on the composition of the feedstock for production. If the feedstock is composed of high amounts of unsaturated fatty acids, the oxidation stability of the resulting biodiesel is low [6–11]. Biodiesel instability results in the degradation of the fuel which alters the fuel properties and forms undesirable products. Such products include peroxides and gums; it also leads to an increase in the viscosity, increase in acid value of the fuel, a slight reduction in the heating value, and also an increase in the cetane number. Biodiesel, when exposed to air or oxygen, results in the formation of alcohols which are further oxidized to acids [11]. The presence of alcohol will lead to a reduction in the flash point, and also an increase in the total acid number results in the corrosion of the fuel line [3, 5, 10]. The sensitivity of biodiesel to oxidation is due to the unsaturated fatty acid content in the oil [12]. Other factors which also have an influence on the oxidation stability of biodiesel are

light, temperature, presence of metal contaminants, nature of storage containers, and the presence of antioxidants [6, 7, 11, 12].

The chemistry of oxidation is based on the primary and secondary oxidation processes [13, 14]. The primary oxidation processes involve the production of low molecular weight compounds such as hydroperoxides in the fuel. The secondary oxidation process on the other hand results in the formation of higher molecular weight species that leads to an increase in viscosity and solid deposits.

The mechanism for the primary oxidation processes of biodiesel can be divided into three stages, namely, initiation, propagation, and the termination stages [6].

Initiation Stage. The initiation stage as shown in (1) involves the removal of hydrogen from the carbon atom of the biodiesel (RH) by an initiator radical (I°) to form a carbon-based fatty acid radical (R°):

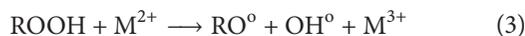


These initiator radicals are formed by different mechanisms including

- (i) thermal dissociation of hydroperoxides (ROOH) that are present as impurities:

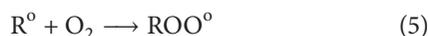


- (ii) metal (M) catalyzed decomposition of hydroperoxides:



- (iii) photooxidation: this is the increase rate of oxidation of biodiesel exposed to light.

Propagation Stage. During the propagation stage, the carbon free radical R° formed at the initiation stage is very reactive and it easily combines with a biradical oxygen in the air resulting in the formation of peroxy radical, ROO° . This peroxy radical abstracts a hydrogen atom from a biodiesel molecule to form another carbon-free radical and a hydroperoxide:



During this stage, up to 100 new radicals are created from one single radical, meaning that the decomposition occurs at an exponential rate and results in the formation of byproducts [15].

Termination Stage. The propagation process continues until two radicals combined together to form a stable product as shown in



The secondary oxidation process results in the formation of acids, esters, aldehydes, ketones, and so forth [16–19]. This leads to changes in the biodiesel properties, such as viscosity, acid number, and oxidation stability [9, 16, 20, 21]. The stability of biodiesel can be improved by adding an appropriate antioxidant [6, 19, 22]. Commercial antioxidants, such as pyrogallol (PY), propyl gallate (PG), catechol, nordihydroguaiaretic acid, butylhydroxyanisole (BHA), butylhydroxytoluene (BHT), and tert-butylhydroquinone (TBHQ), are usually added to biodiesel to enhance its stability [9, 19, 20, 22].

Biodiesel is stored in metallic and nonmetallic containers. These containers, especially the metallic containers, are prone to corrosion in the presence of biodiesel [21–23]. The adverse effects of the presence of metal contaminants on biodiesel stability have been reported in terms of the induction periods [23–25]. No attempt has been made to study the effect of these metal contaminants on the primary oxidation products of biodiesel.

The objective of this study, therefore, is to investigate the stability of castor oil methyl ester in the presence of aluminium and iron contaminants.

2. Materials and Methods

Castor oil was purchased from Stanvac Laboratory, Benin City, Edo State. All the reagents used, methanol, potassium hydroxide, glacial acetic acid, chloroform, potassium iodide, starch indicator, and 0.1 N sodium thiosulphate solution, were of analytical grade and were obtained from Onitsha Head Bridge Market, Anambra State, Nigeria. Iron and aluminium particles in powdered form ($5 \mu\text{m}$) were obtained from the Mechanical Engineering Laboratory of the University of Benin, Benin City.

2.1. Castor Oil Methyl Ester Synthesis. Castor oil methyl ester (COME) was synthesized by reaction of methanol (200 mL) with castor oil (1000 mL) in the presence of KOH (1 wt. % of oil) in a hot magnetic plate stirrer for one hour. After completion of reaction, the material was transferred to a separating funnel and both the ester (biodiesel) and glycerin phases were separated. The upper phase was biodiesel and lower part was glycerin. The upper biodiesel phase was washed with warm water to remove traces of glycerin, unreacted catalyst, and soap formed during the transesterification reaction. The pH of the washing water was initially high at 10.4 due to dissolved $KOCH_3$. After three successive washings, it reduces to about 7.8. The washing was continued until a pH of 7.1 was achieved. Finally, the biodiesel was heated to remove any traces of water that might still be present in it.

2.2. Physicochemical Analysis. Physicochemical analysis was carried out on the castor seed oil (CSO) and the COME according to the American (ASTM D 6751) standards. Analysis was done in triplicate and recorded as mean value \pm standard deviation.

TABLE 1: Physicochemical properties of castor oil and castor oil methyl ester.

Properties	CSO	COME	Biodiesel standard ASTM D6751	Test method
Specific gravity	1.562 ± 0.34	0.893 ± 0.18	0.87–0.90	ASTM D4502
Acid value (mg KOH/g oil)	1.60 ± 0.25	0.34 ± 0.21	0.80 max.	ASTM D664
Iodine value (mg I ₂ /100 g oil)	121.40 ± 8.3	95.0 ± 6.2	120 max.	D-664-11
Peroxide value (meq/kg)	8.80 ± 0.56	1.4 ± 0.28	—	EN 14111
Cloud point (°C)	n/d	−5 ± 1	—	ASTM D2500
Flash point (°C)	230 ± 4	190 ± 5	130.0 min.	ASTM D93
Viscosity (40°C) (mm ² /s)	214 ± 10.8	6.0 ± 2.3	1.9–6.0	ASTM D445
Cetane number	n/d	51.6	47 min.	ASTM D613

* n/d: not determined.

2.3. Sample Preparation. The effect of the two metal contaminants on the peroxide values of COME was carried out by adding varied amounts of Al and Fe particles to 100 mL of COME. In the first phase of the study, varied amounts (0.2, 0.4, 0.6, 0.8, and 1.0 g) of Al and Fe as contaminants were added to 100 mL COME and their effect on the peroxide value was monitored after 24 h. In the second phase of the study, 0.3 g and 0.8 g of both Al and Fe were added to 100 mL COME and their effect on the peroxide values of the contaminated COME was monitored for nine days.

2.4. Peroxide Value Determination. The peroxide value of the biodiesel samples was measured by titrating with sodium thiosulfate according to AOAC standards [26]. Briefly, 2 g of the biodiesel sample was weighed and 10 mL chloroform was added in a stoppered conical flask. 15 mL acetic acid and 1 mL potassium iodide 10% were added. The flask was shaken for 2 min and left in the dark for 5 min. Then, 75 mL of deionized water was added and titration was done with solution of sodium thiosulfate 0.01 N and 1% starch solution as indicator. A blank run was also carried out. The peroxide value expressed as meq/kg of biodiesel sample was calculated by the following formula:

$$\text{peroxide value (meq/kg)} = \frac{[(V - V_0) \times C \times 1000]}{W}, \quad (9)$$

where V is the volume (mL) of sodium thiosulfate solution for the biodiesel sample, V_0 is the volume (mL) of sodium thiosulfate solution for the blank, C is the molar concentration of sodium thiosulfate solution, and W is the biodiesel sample weight (g).

3. Results and Discussion

3.1. Physicochemical Properties of Castor Oil and Biodiesel Samples. The results in Table 1 show a comparison of the physicochemical properties of the castor seed oil (CSO) and the castor oil methyl ester (COME). The CSO has a specific gravity of 1.56 which reduced to 0.89 after transesterification reaction to produce the COME. The value for COME, however, falls within the range of values as specified by the ASTM D6751 standard. The iodine value (IV) of the castor oil was 121.40 mg I₂/g oil. This could be attributed to its high

content of unsaturated fatty acids. This value was higher than values obtained in previous studies carried out on castor oil [27, 28]. Akpan et al. [27] in their work got an iodine value of 87.72 mg I₂/g oil for crude castor oil which was later reduced to 84.8 mg I₂/g oil on modification by means of sulphation process. However, after the transesterification reaction, the iodine value for the resulting COME was found to be 95.0 mg I₂/g oil.

The cloud point of the COME was −5°C. This is the temperature at which wax first becomes visible as the temperature was lowered. This value was much higher than that obtained by Berman et al. [29] and Ramezani et al. [30], but the value obtained was within the ASTM D6751 standard. The viscosity obtained for the CSO was 230 mm²/s. As reported by Scholz and da Silva [31], the viscosities for castor seed oils range from 240 to 300 mm²/s. It was however observed that there was a sharp decrease in the viscosity after the transesterification reaction to COME. This viscosity for the COME was 6.0 mm²/s, which is within the biodiesel range of 1.9–6.0 mm²/s.

The cetane number, which is a measure of the ignition quality of diesel fuels and one of the prime indicators of the quality of diesel, was calculated to be 51.60. Although the value fell within the limits of ASTM D6751 standard, this value was a little higher than values obtained by Berman et al. [29] and Sreenivas et al. [28] which were 48.9 and 42, respectively. The flash point of the CSO was measured to be 230°C which reduced to 190°C after the transesterification reaction to COME. The flash point is the lowest temperature at which the fuel ignites when exposed to a heat source. The flash point value obtained for the COME falls within that specified by the ASTM D6751, which should be a minimum flash point value of 130°C for biodiesel.

3.2. Effect of Metal Contaminants on the Peroxide Value (PV) of Castor Oil Methyl Ester. Figure 1 shows the amount of peroxides formed in the COME containing varied amounts of metal contaminants after a 24 h period. The result shows that as the amount of metal contaminants increases, the peroxide values of the COME decrease. The formation of peroxides in the Fe contaminated COME was more pronounced compared to that contaminated with Al. In previous studies carried out, the presence of metal contaminants has been found to

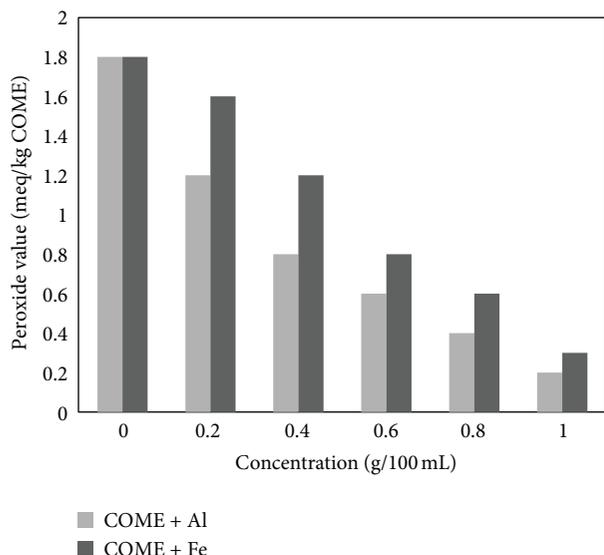


FIGURE 1: Effect of metal contaminants on the oxidation stability of castor oil methyl ester after a 24 h period.

reduce the induction period of biodiesel [32, 33]. For the Fe contaminated COME, the peroxide values reduced from 1.6 to 0.3 meq/kg COME as the amount of contaminants increases from 0.2 to 1 g/100 mL COME. Similarly, the peroxide values for the Al contaminated biodiesels reduce from 1.2 to 0.2 meq/kg COME as the amount of Al contaminants increases from 0.2 to 1.0 g/100 mL. This indicates that the more the metal contaminants, the more the decrease in peroxide values of the COME. This result confirmed that the presence of metal contaminants leads to decomposition of hydroperoxides [6].

The effect of varying concentrations of Fe and Al contaminants on the peroxide values of COME as monitored for nine days is shown in Figure 2. The peroxide values for both the contaminated and uncontaminated COME steadily increase with time during the first four days of our investigation. COME without metal contaminants experiences the greatest increase from 1.4 to 5.8 meq/kg COME, followed by 0.3 g Fe/100 mL COME, 0.3 g Al/100 mL COME, 0.8 g Al/100 mL COME, and 0.8 g Fe/100 mL COME in that order. The peroxide value of the uncontaminated COME is observed to be higher than the contaminated COME samples containing varying amounts of contaminants for the first four days. This was in line with the first phase of the experiment where it was discovered that the presence of metal contaminants reduces the amount of peroxides formation in the COME.

It was also observed that, as time in days elapses from the fifth day, the peroxide value of the contaminated COME containing 0.3 g Fe/100 mL COME was higher than that of the uncontaminated COME while that of the 0.3 g Al/100 mL was observed to be rising significantly but lower than that of the uncontaminated COME. However, there was a steady increase in the peroxide value for the 0.3 g Al/100 mL COME between the sixth and the ninth day as compared to that

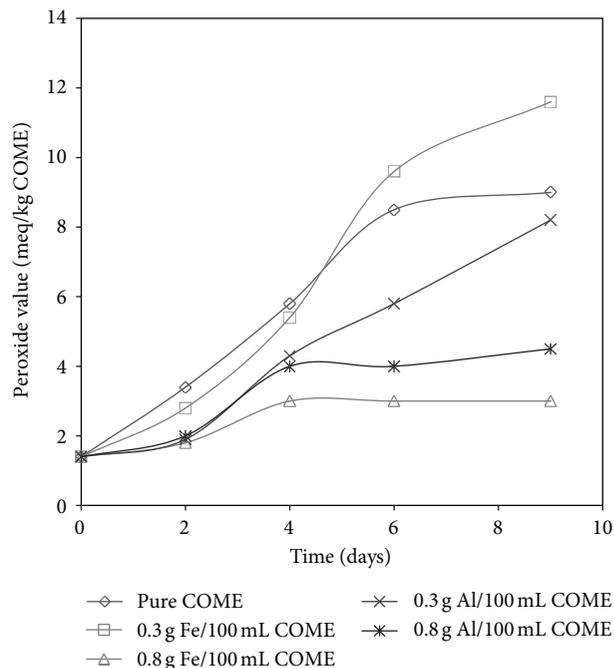


FIGURE 2: Effect of different concentrations of metal contaminants on the peroxide value of COME.

of the uncontaminated COME which had almost assumed a constant peroxide value during this period. Thus, the presence of these metal contaminants in small amounts (0.3 g/100 mL COME) can be said to enhance the formation of hydroperoxides. Although the COME contaminated with 0.3 g Fe had a stronger catalytic effect when compared to the COME contaminated with 0.3 g Al, both had strong positive influence on enhancing the formation of hydroperoxides as measured by their peroxide values.

The COME contaminated with 0.8 g Fe and Al was characterized by a slight increase in the peroxide values for the first four days, after which they both assume a constant value of 3.0 and 4.0 meq/kg COME for Fe and Al contaminated COME, respectively. In comparison with the COME in which 0.3 g Fe and Al were added, the peroxide values for COME contaminated with 0.8 g Fe and Al were lower.

The findings from this study reveal that, in the presence of small amount of metal contaminants, more hydroperoxides are produced as compared to when large amounts of metal contaminants are present. In this case, the presence of metals in the COME resulted in decomposition of hydroperoxides [6, 20]. This can be explained by the fact that the primary oxidation process is gradually tending towards the secondary oxidation process, where other oxidation products like aldehydes, alcohols, and shorter-chain carboxylic acids are being formed from the decomposition of the peroxides.

4. Conclusions

The stability of the COME is reduced in the presence of metals. Of the two metals investigated, Fe appears to have

a stronger detrimental and catalytic effect on stability than Al. Under short-term storage durations, for example, a day, the peroxide value of the COME was found to decrease as the concentration of metal contaminants increases. For longer period of days, it was observed that the increases in peroxide values in the COME contaminated with small amounts of metal contaminants are much higher than those in COME contaminated with large amounts of COME. From the study, it was observed that when metal contaminants are increased, the rate of hydroperoxide decomposition to other products is more than the rate of hydroperoxide formation. It can be concluded that, in the presence of large amounts of metal contaminants, the primary stage of oxidation is reduced leading to the formation of secondary oxidation products such as carboxylic acids and aldehydes.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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