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***Research Paper***

# Studies on the Effect of Antioxidants on the Long-term Storage stability and Oxidation Stability of Jatropha Biodiesel

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***Abstract*** *- Biodiesel is defined as the mono alkyl esters of vegetable oils. Biodiesel is more susceptible to oxidation or autoxidation during long term storage than conventional petro diesel. This paper presents results of studies on the effectiveness of various individual antioxidants to improve the oxidative stability and storage stability of Jatropha biodiesel (JBD).The results of Rancimat experiments show that the induction point (IP) increased substantially on adding certain antioxidants to the Jatropha biodiesel. The study reveals pyrogallol (PY) to be the best antioxidant which showed the best improvement in the oxidative stability of JBD, the induction time being enhanced to 18.62 h at a PY concentration of 3000 ppm at 110$^0$C. The storage stability studies were carried out according to the ASTM standard procedures 1) ASTM 4625 @30$^0$C/50 weeks 2) ASTM 4625@43$^0$C/ 12 weeks by adding different antioxidants like Butylated hydroxytoluene(BHT), Butylated hydroxyanisole (BHA), Pyrogallol (PY), Gallic acid (GA) and tertiary Butylhydroquinone (TBHQ). The effectiveness of these five antioxidants on JBD was examined at varying loading level during the storage period.*

**Keywords:** Jatropha oil, Biodiesel, Oxidation stability, Storage stability, Antioxidant.

## Introduction

Biodiesel, defined as fatty acid mono-alkyl esters made from vegetable oil or animal fat, is an alternative fuel for combustion in compression–ignition (diesel) engines. Several recent reviews have reported on the technical characteristics of biodiesel. In short, biodiesel is made from domestically renewable feedstocks is environmentally innocuous, is relatively safe to handle (high flash points), and has an energy content, specific gravity, kinematic viscosity (KV) and cetane number (CN) comparable to those properties of petro diesel [1,2].

With a production of almost one million tons in Europe, fatty acid methyl ester (FAME) more generally called biodiesel has become a fast growing renewable liquid biofuel within the European Community. Not only vegetable oils or fats but also fatty acid methyl esters undergo degradation over time, mainly influenced by temperature and oxygen. Degradation products of biodiesel such as insoluble gums and sediments, or the formation of organic acids and aldehyde may cause engine and injection problems [2-4]. Fuel properties degrade during long-term storage as follows: (i) oxidation or autoxidation from contact with ambient air (ii) thermal or thermal-oxidative decomposition from excess heat (iii) hydrolysis from contact with water or moisture in tanks and fuel lines or (iv) microbial contamination from migration of dust particles or water droplets containing bacteria or fungi into the fuel [5]. Monitoring the effects of autoxidation on biodiesel fuel quality during long-term storage presents a significant concern for biodiesel producers, suppliers, and consumers[6].

Therefore, especially engine and injection pump producers insisted on the parameter of oxidation stability which was finally fixed at a minimum limit of a 6-hour induction period at 110$^0$C [6, 7]. The method adopted for determination of the oxidation stability is the so called Rancimat method which is commonly used in the vegetable oil sector. Especially high contents of unsaturated fatty acids, which are very sensitive to oxidative degradation, lead to very low values for the induction period. Thus, even the conditions of fuel storage directly affect the quality of the product. Several studies showed that the quality of biodiesel over a longer period of storage strongly depends on the tank material as well as on contact to air or light. Increase in viscosities and acid values versus decreases in induction periods could be observed [6–9] to retard oxidative degradation and to guarantee a specific stability, it will be necessary to find appropriate additives for biodiesel. Researchers have evaluated the beneficial effects of the antioxidant additive tert-butylhydroxyquinone (TBHQ) on sunflower oil methyl and ethyl ester stability [10]. Similar results on soybean oil methyl esters were observed by Dunn using TBHQ and a tocopherol, even under accelerated conditions [11].

Although there are numerous publications on the effect of natural and synthetic antioxidants on the stability of oils and fats used as food and feed, little is available on the effect of antioxidants on the behavior of FAME used as biodiesel. Simkovsky, et al. studied the effect of different antioxidants on the induction period of rapeseed oil methyl esters at different temperatures but did not find any significant improvements [12]. Canakci, et al. tested the influence of the antioxidant TBHQ on the PV of soybean oil methyl esters during storage and found good improvement of stability [13]. Most recently Das, et al. described the effect of commercial antioxidants used in kharanja biodiesel for storage stability [14]. In the previous studies, numerous methods for assessing the oxidation status of biodiesel have been investigated, including acid value, density and kinematic viscosity. The peroxide value may not be suitable because after an initial increase it decreases due to secondary oxidation reactions, although the decrease likely affects only samples oxidized beyond what may normally be expected. Thus there is the possibility of the fuel having undergone relatively extensive oxidation but displaying an acceptable peroxide value. The peroxide value is also not included in biodiesel standards. Acid value and kinematic viscosity however are two facile methods for rapid assessment of biodiesel fuel quality as they continuously increase with deteriorating fuel quality [15].

The aim of the present study is to investigate the oxidative stability and storage stability of Jatropha curcus oil methyl ester. The Rancimat procedure for oxidation stability and the ASTM procedure for storage stability have been used in this study. Using different antioxidants in different concentrations, the fuel properties such as Acid value (AV) and Kinematic viscosity (KV) of Jatropha Bio-Diesel (JBD) were determined at regular period of time.

## Material and Methods

Jatropha curcus oil used for the study was purchased from Sakthi sugars Ltd, Tamil nadu, India. Certified methanol of high purity grade and catalyst Sodium hydroxide of pure grade was obtained from Merck, India. Jatropha bio-diesel (JBD) was prepared in our laboratory according to the methodology described by Chitra, et al. [16] and Naik, et al. [17]. The characteristic of Jatropha oil methyl ester (JBD) are given in Table respectively (Table 1).

### Storage condition

All the biodiesel samples of volume 200 ml were stored in open Borosil glass bottles of 250 ml capacity and kept in doors, at a room temperature of $30^0$ C and $42^0$ C. The samples area that exposed to air as daylight condition. 50 ml space is unoccupied biodiesel. That container is fully opened for air contact. Room humitity is 41 % to 72% day time humidity is low but night time is high humidity. Every week take the samples for analyzed.

### Determination of oxidative stability

Oxidative stability (OS) of biodiesel sample was studied with a Rancimat 873 instrument (Metrohm, Switzerland). In the Rancimat procedure the sample was heated at a constant temperature with an excess airflow, which passed through a conductivity cell filled with distilled water. During this oxidation process volatile acids are formed and the conductivity increases at an end and the period up to this point is called ‘‘Induction period”. The induction period of JBD was determined without antioxidant and with different antioxidants (BHA, BHT, TBHQ, PY and GA) and under different concentrations of the antioxidants (500, 1000, 2000 & 3000ppm) at $110^0$C. This method of determination was followed by Das, et al. [14] and Dinkov, et al. [18], Karavalakis, et al. [19], Bouaid, et al. [20] and Knothe [21] in their earlier works.

### Evaluation of Storage stability

To evaluate the storage stability the ASTM procedures 4625 -50 weeks and 4625 – 12 weeks were carried out. In the ASTM 4625 -50 weeks procedure, the KV and AV values were determined at $30^0$C over a period of 50 weeks at regular intervals. In the ASTM 4625- 12 method, the KV and AV values were monitored at $43^0$C over a period of 12 weeks at regular intervals.

## Results and Discussion

### Determination of oxidation stability

The Rancimat test is the specified standard method for oxidative stability for biodiesel in accordance with EN 14112 (European standard) [22]. The absolute difference between two independent single test results did not exceed the repeatability limit of the EN 14112 method. The IP for 100% biodiesel (B100) specified in ASTM D6751 -05 is not less than 3 h [23]. The induction period of Jatropha biodiesel without addition of antioxidant is 2.02 h. So different antioxidants at four different concentration levels were used to improve the oxidation stability. Table 2 shows the induction period of Jatropha biodiesel using different antioxidants at different concentration. The study reveals Pyrogallol (PY) to be the best antioxidant which showed the best improvement in the OS of Jatropha biodiesel that is 18.62 h at a concentration of 3000 ppm at $110^0$C. The study also reveals that with TBHQ the Oxidation stability of biodiesel is enhanced to 11.83 h at a concentration of 3000 ppm at $110^0$C. All the other antioxidants used have small improvement in the induction period. The improvement in the induction time using different antioxidants in different concentrations is presented in table. (Table 2)

### Evaluation of Storage Stability

### Kinematic Viscosity (KV)

During storage, the viscosity of the methyl esters increases by the formation of more polar, oxygen containing molecules and also by the formation of oxidized polymeric compounds that can lead to the formation of gums and sediments that clog filters. In ASTM 4625 – 50 procedure, the KV and AV values were determined at $30^0$C over a period of 50 weeks at regular intervals. The kinematic viscosity of Jatropha biodiesel at the initial stage at $30^0$C was8.38 cP. When the biodiesel was left by itself for a duration of 50 weeks in an open to air condition, the oxidation process started and the KV value rose to a enormously high value of 18.41 cP which is an indication that storage stability of biodiesel is a serious problem. On employing antioxidants to retard the oxidation process during storage it is found from table 3 (ASTM 4625 50 weeks @ $30^0$C) that the antioxidants definitely improve the storage stability (Table 3). All the five antioxidants tested showed that at a concentration of 3000 ppm, they are able to substantially retard the oxidation process during a 50week storage period at $30^0$C and improve the storage stability of the biodiesel. Antioxidants BHA and TBHQ seem to have a better effect on the storage stability of JBD than the other antioxidants over a 50 week period at a concentration of 3000 ppm. In the other accelerated method namely ASTM 4625 @ 12 weeks at $43^0$C it is found again that in all the five antioxidants a concentration of 3000 ppm of the antioxidants suppresses the oxidation and improves the storage stability of the biodiesel. (Table 4) In fact it is found from table 4 that the KV values are maintained as that of the standard value of KV as prescribed by ASTM for biodiesel, when the concentration of the five antioxidants used are 3000 ppm. At a concentration of 3000 ppm BHT, TBHQ and PY all improve the storage stability of biodiesel and maintain the KV value around the standard value of 8.4 cP.

The change of kinematic viscosity of the biodiesel was plotted for every week of storage with a particular antioxidant at four different concentration (Figure 1, a to e). The change of kinematic viscosity of the biodiesel was also plotted for every week of storage with a particular concentration of all the five antioxidants (Figure 1, f to i). The graph plotted brings out clearly that at a concentration of 3000 ppm, each of the five antioxidants (BHT,BHA, GA, TBHQ and PY) improves the storage stability of the biodiesel to a very large extent (Figure 1, a to e). In the graph plotted (Figure 1, f to i) it is distinctively seen that at a particular concentration, a particular antioxidant improves the storage stability to a maximum. At a concentration of 500 ppm, TBHQ is found to be the best antioxidant. Similarly at a concentration of 3000 ppm BHA is shown to be the best antioxidant. From the Figure 2 for the ASTM 4625 @ $43^0$C – 12 weeks procedure it is seen clearly, that pyrogallol is the best antioxidant at any concentration between 500 ppm to 3000 ppm (Figure 2, f to i). The ASTM 4625 @$43^0$C 12 weeks method, again proves distinctively that the antioxidant concentration of 3000 ppm is the optimum concentration between 500 ppm to 3000 ppm for the highest improvement in storage stability of the biodiesel sample (fig 2, a to e). (Figure 2)

### Acid Value (AV)

The acid value (AV) of biodiesel samples also increased with increasing storage time as a result of hydrolysis of fatty acids methyl esters (FAME) to fatty acids (FA). The specification limit of 0.5 mg KOH/g was exceeded when the methyl ester (ME) samples were not exposed to daylight but kept open to air over a storage time of 350 days (50 weeks). The acid value of Jatropha biodiesel initially was 0.44 mgKOH/g. when the biodiesel was stored in an open to air condition and kept for 50 weeks it was found to undergo oxidation and the AV rise up to a very high value of 7.2 mg KOH/g. From table 3, it is evident that the addition of antioxidants to retard the oxidation is found to be effective with all the five antioxidants when the antioxidants concentration is 3000 ppm. In the other accelerated procedure of ASTM 4625@ 12 weeks / $43^0$C, it is clearly established that a concentration of 3000 ppm of all the five antioxidants improves greatly the storage stability of the biodiesel sample. The AV is brought down substantially to the standard AV of biodiesel samples. From the table 4 it is also evident that the antioxidants BHT, TBHQ and PY have greater effect on the storage stability of Jatropha biodiesel over a storage period of 12 weeks at $43^0$C

The change of Acid value of the biodiesel was plotted for every week of storage with a particular antioxidant in four diferent concentration (Figure 3, a to e). The change of acid value of the biodiesel was also plotted for every week of storage with a particular concentration of all the five antioxidants (Figure 3, f to i). The graph plotted brings out clearly that a concentration of 3000 ppm of each of the five antioxidants (BHT,BHA, GA, TBHQ and PY) improves the storage stability of the biodiesel to a very large extent (Figure 3 a to e). In the graph plotted (Figure 3, f to i) it is distinctively seen that at a specific concentration, only, a particular antioxidant improves the storage stability to a large extent. At a concentration of 500 ppm, PY is found to be the best antioxidant. Similarly at a concentration of 3000 ppm BHA is shown to be the best antioxidant. In the accelerated test ASTM 4625 @ $43^0$C – 12 weeks, the graph plotted clearly proves that pyrogallol is the best antioxidant at any concentration between 500 ppm to 3000 ppm (Fig 4, f to i). Again it is evident that the antioxidant concentration of 3000 ppm is the optimum concentration between 500 ppm to 3000 ppm for the best improvement in storage stability of the biodiesel sample (Figure 4, a to e).

## Conclusion

Biodiesel, which consists of monoalkyl esters of long-chain fatty acids made from biolipids, generally suffers from inferior oxidative stability. In this study, we investigated the oxidative stability and storage stability in an open to air storage condition of Jatropha biodiesel. The oxidative stability of JBD decreased i.e the kinematic viscosity and acid value increased with increase in storage time of the biodiesel. The results of this study can be summarized as follows:

All the samples were stored under open to air and not exposed to day light condition. The effect of Pyrogallol as an antioxidant for JBD is found to be the best as it increased the induction time from 2.02 h to 18.62 h, when the concentration of pyrogallol was 3000 ppm. KV and AV values are good indicators of storage stability of biodiesel. They increase on increase of storage time. A concentration of 3000 ppm of antioxidants like BHT, TBHQ, PY, GA, and BHA has a beneficial effect on the storage stability of Jatropha biodiesel.

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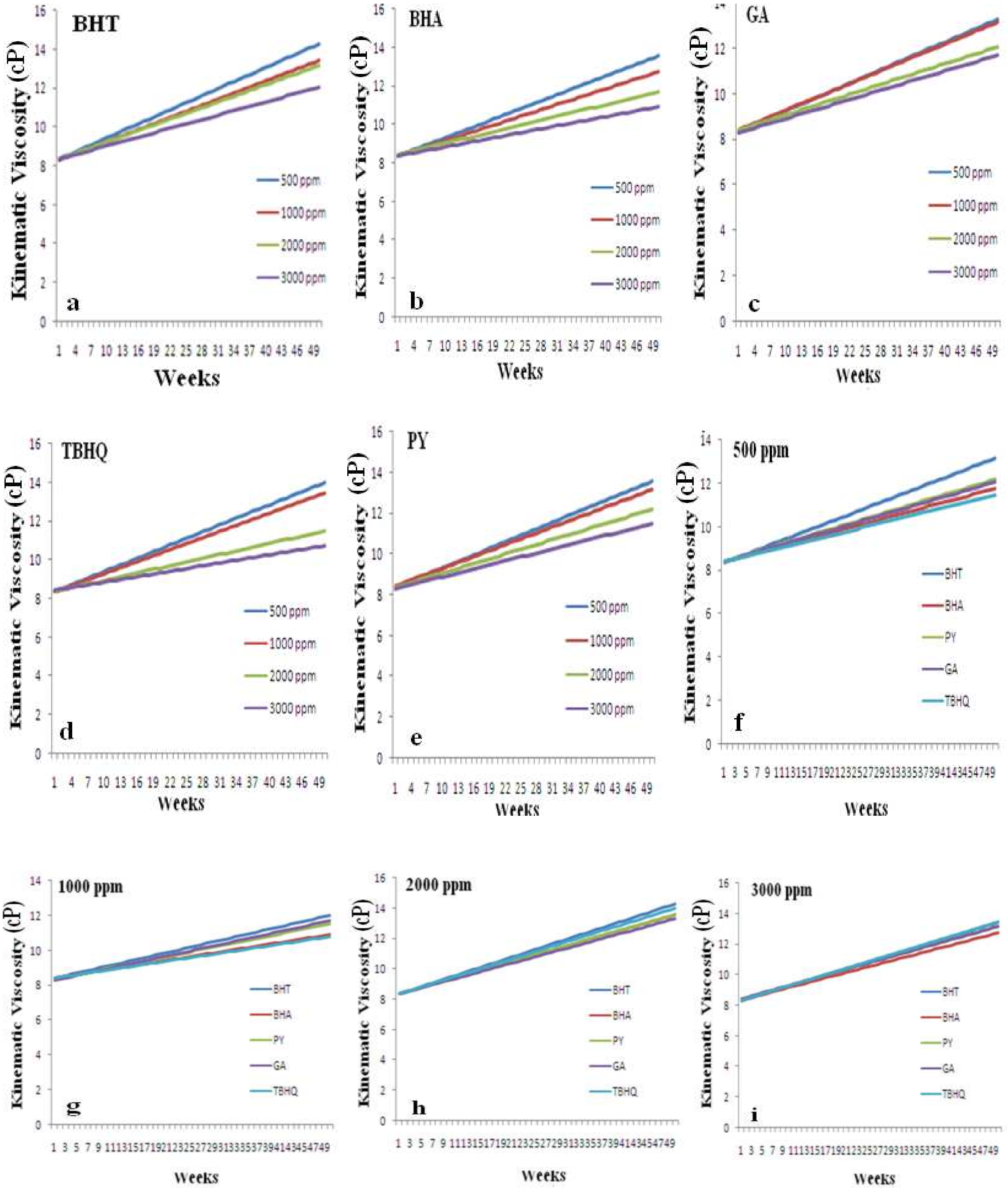

**Figure 1: Change of Kinematic viscosity of JBD with time. Figure ( a to e) indicate the variation of kinematic viscosity with different antioxidants at different concentrations. Fig (f to i) indicate the variation of kinematic viscosity with different antioxidants at a particular concentration of antioxidant (ASTM 4625 @ 30$^0$C at 50 weeks).**

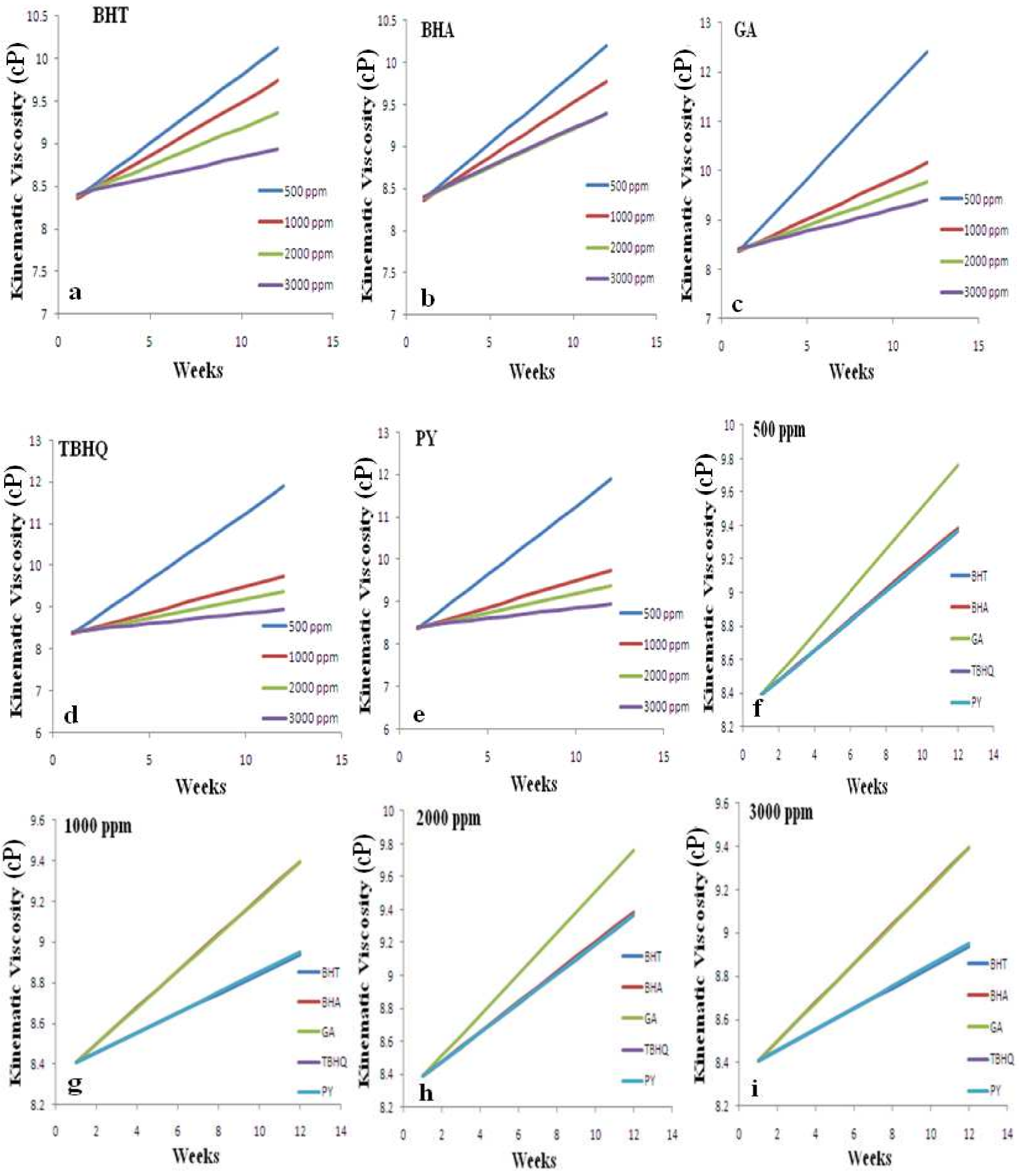

**Figure 2: Change of Kinematic viscosity of JBD with time. Fig (a to e) indicate the variation of kinematic viscosity with different antioxidants at different concentrations. Figure (f to i) indicate the variation of kinematic viscosity with different antioxidants at a particular concentration of antioxidant (ASTM 4625 @ 43$^0$C at 12 weeks).**

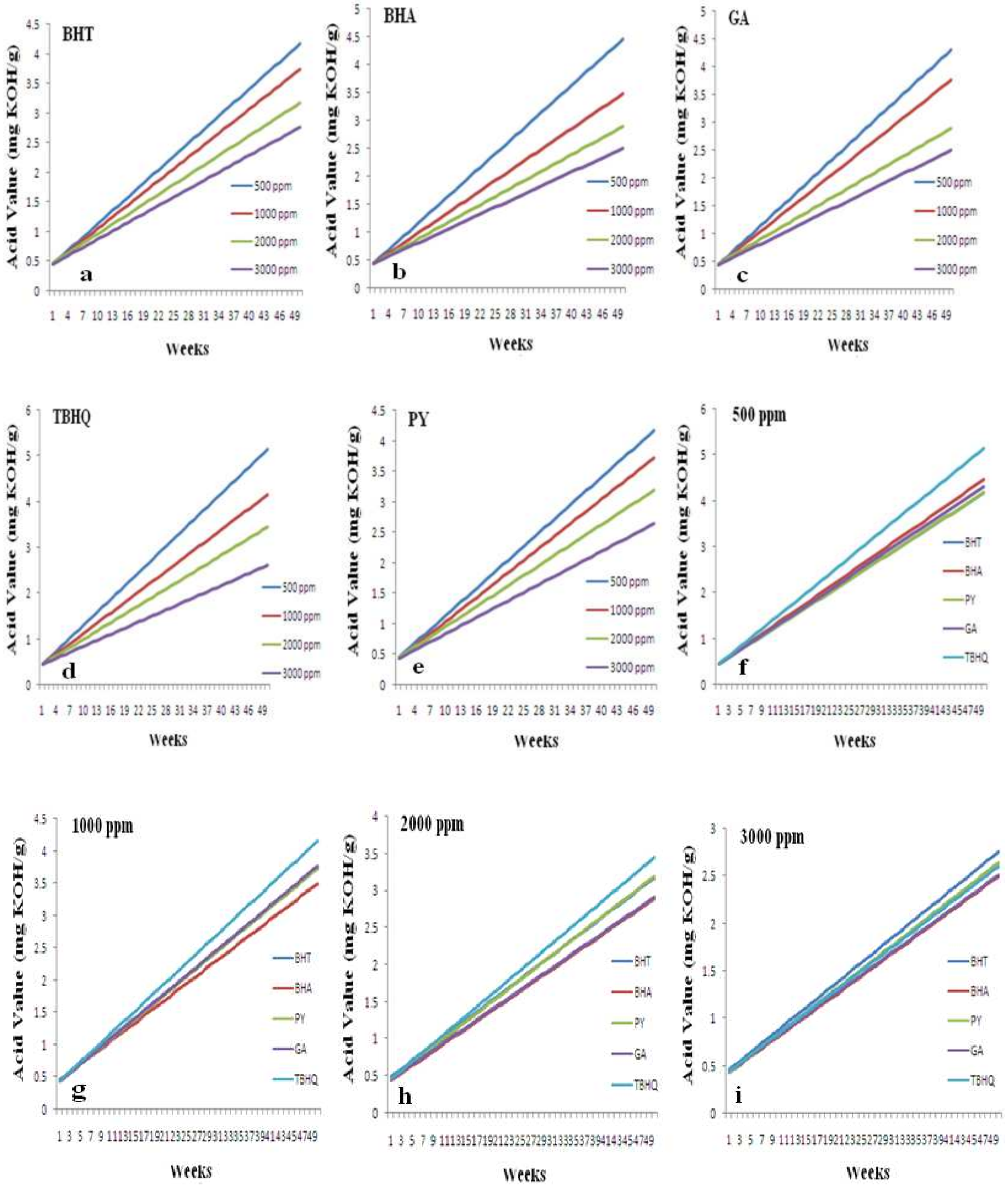

Figure 3 : Change of Acid value viscosity of JBD with time. Figure a to e indicate the variation of Acid value with different antioxidants at different concentrations. Figure f to i indicate the variation of Acid value with different antioxidants at a particular concentration of antioxidant (ASTM – $30^0$C at 50 weeks)

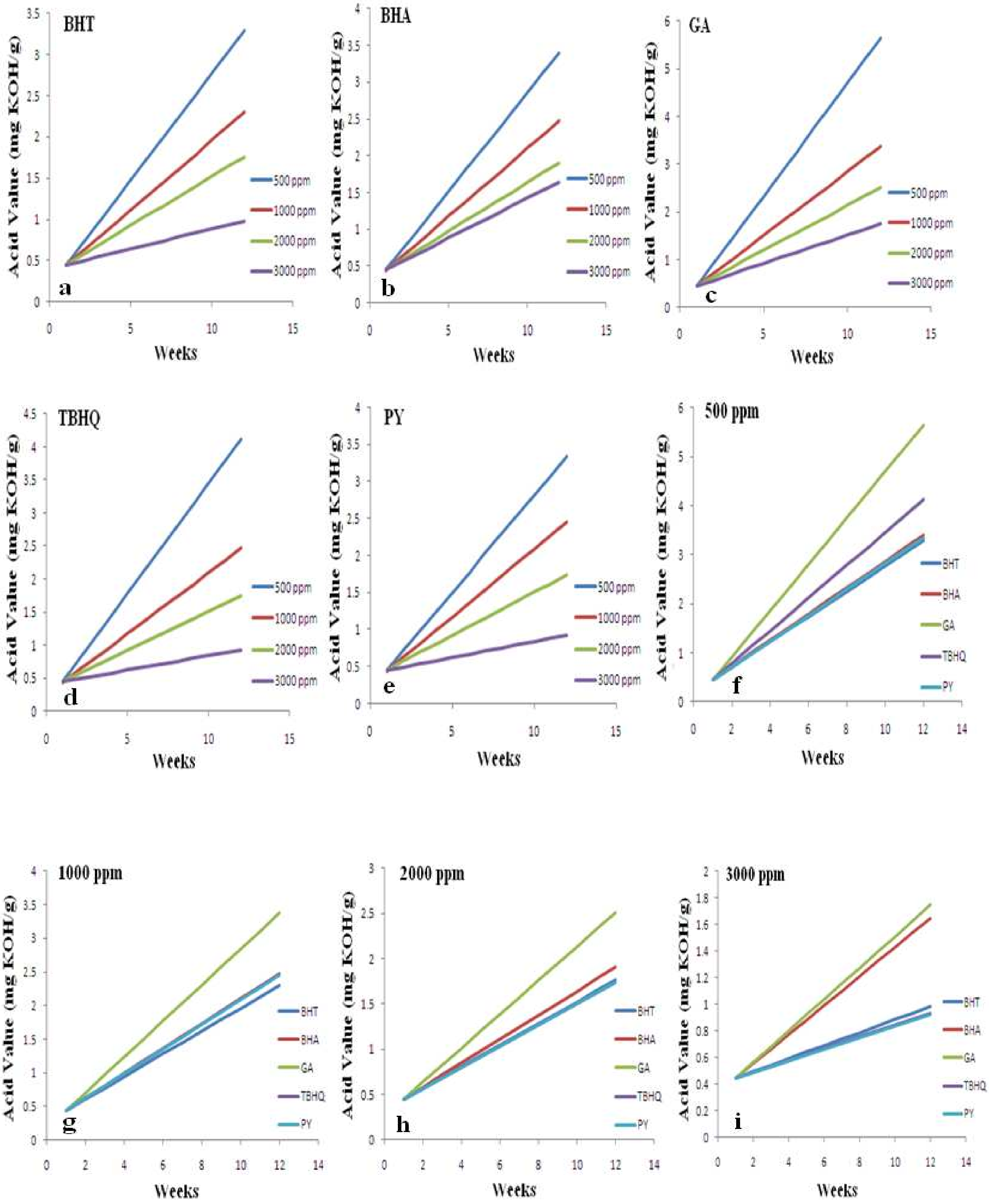

**Figure 4: Change of Acid value of JBD with time. Figure a to e indicate the variation of acid value with different antioxidants at different concentrations. Fig f to i indicate the variation of acid value with different antioxidants at a particular concentration of antioxidant (ASTM 4625 @ 43$^0$C at 12 weeks).**

## Table 1: Properties of JBD (Jatropha biodiesel)

| Properties | Jatropha biodiesel | Standard method | Standard Value |
|---|---|---|---|
| Iodine Value | 91.25 g iodine/100 g oil | EN 14214 | 120 max. |
| Peroxide Value | 5.83(meq/kg) | - | - |
| Kinematic Viscosity (@ 40$^{0}$C) | 5.94 cSt or 8.38 Cp | ASTM –D 445 | 1.9 – 6 cSt |
| Acid value | 0.467(mg KOH/g) | ASTM –D 664 | 0.5 max. |
| Saponification Value | 180.33 mg KOH/g Oil | - | - |
| Water content | 0.014 % | ASTM –D 2709 | Max. 0.05 % |
| Carbon Residue Value | 0.047 % | ASTM –D 4530 | Max. 0.05% |
| Cloud Point | 0 to 3 | ASTM –D 2500 | |
| Pour Point | -6 to -5 | ASTM –D 2500 | |
| Calorific Value | 33.596 MJ/kg | - | - |

## Table 2: Induction time of JBD with different antioxidants at different concentration.

| S. No. | AntiOxidants | Without Antioxidants Induction time (h) | Induction Time (h) | | | |
|---|---|---|---|---|---|---|
| | | | 500 ppm | 1000 ppm | 2000 ppm | 3000 ppm |
| 1 | BHT | 2.02 | 3.08 | 4.06 | 5.48 | 7.08 |
| 2 | BHA | 2.02 | 2.98 | 3.62 | 4.72 | 6.82 |
| 3 | TBHQ | 2.02 | 2.20 | 2.85 | 6.28 | 11.83 |
| 4 | GA | 2.02 | 2.19 | 2.96 | 3.09 | 3.27 |
| 5 | PY | 2.02 | 3.59 | 6.59 | 15.72 | 18.62 |

## Table 3: KV and AV data obtained from ASTM 4625 @ 30$^{0}$C – 50 weeks method

| Without Antioxidants Initial (0 weeks) | | Without Antioxidants (After 50 weeks) | | | 10th Week | | 20th Week | | 30th Week | | 40th Week | | 50th Week | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| KV (cP) | AV (mg KOH/g) | KV (cP) | AV (mg KOH/g) | With Antioxidants | KV (cP) | AV (mg KOH/g) | KV (cP) | AV (mg KOH/g) | KV (cP) | AV (mg KOH/g) | KV (cP) | AV (mg KOH/g) | KV (cP) | AV (mg KOH/g) |
| 8.39 | 0.44 | 18.41 | 7.2 | **BHT** | | | | | | | | | | |
| | | | | 500 ppm | 9.46 | 1.12 | 10.66 | 1.88 | 11.86 | 2.64 | 13.06 | 3.40 | 14.26 | 4.16 |
| | | | | 1000 ppm | 9.27 | 1.05 | 10.32 | 1.72 | 11.37 | 2.39 | 12.42 | 3.06 | 13.47 | 3.73 |
| | | | | 2000 ppm | 9.26 | 0.97 | 10.24 | 1.52 | 11.22 | 2.07 | 12.20 | 2.62 | 13.18 | 3.17 |
| | | | | 3000 ppm | 9.08 | 0.88 | 9.80 | 1.35 | 10.54 | 1.82 | 11.28 | 2.29 | 12.02 | 2.76 |
| 8.36 | 0.44 | 18.41 | 7.2 | **BHA** | | | | | | | | | | |
| | | | | 500 ppm | 9.3 | 1.18 | 10.41 | 2.00 | 11.47 | 2.82 | 12.54 | 3.64 | 13.61 | 4.46 |
| | | | | 1000 ppm | 9.12 | 1.00 | 10.07 | 1.62 | 10.96 | 2.24 | 11.85 | 2.86 | 12.74 | 3.48 |
| | | | | 2000 ppm | 9.02 | 0.89 | 9.69 | 1.39 | 10.37 | 1.89 | 11.05 | 2.39 | 11.73 | 2.89 |
| | | | | 3000 ppm | 8.85 | 0.81 | 9.32 | 1.23 | 9.89 | 1.65 | 10.41 | 2.07 | 10.93 | 2.49 |
| 8.36 | 0.45 | 18.41 | 7.2 | **GA** | | | | | | | | | | |
| | | | | 500 ppm | 9.27 | 1.16 | 10.28 | 1.95 | 11.29 | 2.74 | 12.30 | 3.53 | 13.31 | 4.32 |
| | | | | 1000 ppm | 9.29 | 1.05 | 10.26 | 1.73 | 11.23 | 2.41 | 12.26 | 3.09 | 13.17 | 3.77 |
| | | | | 2000 ppm | 9.06 | 0.90 | 9.81 | 1.40 | 11.56 | 1.90 | 11.31 | 2.40 | 12.06 | 2.90 |
| | | | | 3000 ppm | 8.91 | 0.82 | 9.6 | 1.24 | 10.29 | 1.66 | 10.98 | 2.08 | 11.67 | 2.50 |
| 8.37 | 0.44 | 18.41 | 7.2 | **TBHQ** | | | | | | | | | | |
| | | | | 500 ppm | 9.41 | 1.31 | 10.55 | 2.27 | 11.71 | 3.23 | 12.85 | 4.19 | 14.00 | 5.15 |
| | | | | 1000 ppm | 9.26 | 1.11 | 10.33 | 1.88 | 11.37 | 2.64 | 12.41 | 3.40 | 13.45 | 4.16 |
| | | | | 2000 ppm | 8.98 | 1.00 | 9.58 | 1.61 | 10.21 | 2.22 | 10.84 | 2.8 | 11.47 | 3.44 |
| | | | | 3000 ppm | 8.81 | 0.844 | 9.32 | 1.28 | 9.81 | 1.72 | 10.28 | 2.16 | 10.76 | 2.60 |
| 8.36 | 0.44 | 18.41 | 7.2 | **PY** | | | | | | | | | | |
| | | | | 500 ppm | 9.3 | 1.13 | 10.39 | 1.89 | 11.46 | 2.65 | 12.53 | 3.41 | 13.60 | 4.17 |
| | | | | 1000 ppm | 9.21 | 1.04 | 10.24 | 1.71 | 11.21 | 2.38 | 12.18 | 3.05 | 13.15 | 3.72 |
| | | | | 2000 ppm | 9.00 | 0.95 | 9.86 | 1.51 | 10.64 | 2.07 | 11.42 | 2.63 | 12.20 | 3.15 |
| | | | | 3000 ppm | 8.82 | 0.84 | 9.56 | 1.29 | 10.21 | 1.74 | 10.86 | 2.19 | 11.51 | 2.64 |

| without Antioxidants Initial (0 weeks) | | Without Antioxidants (After 50 weeks) | | With Antioxidants | 3rd week | | 6th Week | | 9th Week | | 12th Week | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| KV (cP) | AV (mg KOH/g) | KV (cP) | AV (mg KOH/g) | | KV (cP) | AV (mg KOH/g) | KV (cP) | AV (mg KOH/g) | KV (cP) | AV (mg KOH/g) | KV (cP) | AV (mg KOH/g) |
| 8.39 | 0.44 | 14.5 | 7.2 | **BHT** | | | | | | | | |
| | | | | 500 ppm | 8.68 | 0.96 | 9.16 | 1.73 | 9.64 | 2.51 | 10.12 | 3.28 |
| | | | | 1000 ppm | 8.61 | 0.77 | 8.98 | 1.28 | 9.34 | 1.79 | 9.73 | 2.29 |
| | | | | 2000 ppm | 8.56 | 0.68 | 8.83 | 1.04 | 9.09 | 1.40 | 9.36 | 1.76 |
| | | | | 3000 ppm | 8.50 | 0.54 | 8.65 | 0.69 | 8.78 | 0.83 | 8.93 | 0.98 |
| 8.36 | 0.44 | 14.5 | 7.2 | **BHA** | | | | | | | | |
| | | | | 500 ppm | 8.70 | 0.98 | 9.20 | 1.78 | 9.87 | 2.59 | 10.20 | 3.39 |
| | | | | 1000 ppm | 8.62 | 0.80 | 9.00 | 1.36 | 9.39 | 1.91 | 9.78 | 2.47 |
| | | | | 2000 ppm | 8.56 | 0.71 | 8.84 | 1.11 | 9.205 | 1.50 | 9.38 | 1.90 |
| | | | | 3000 ppm | 8.58 | 0.66 | 8.85 | 0.99 | 9.21 | 1.32 | 9.39 | 1.64 |
| 8.36 | 0.45 | 14.5 | 7.2 | **GA** | | | | | | | | |
| | | | | 500 ppm | 9.10 | 1.39 | 9.83 | 2.80 | 11.30 | 4.22 | 12.40 | 5.64 |
| | | | | 1000 ppm | 8.68 | 0.97 | 9.17 | 1.77 | 9.66 | 2.57 | 10.15 | 3.37 |
| | | | | 2000 ppm | 8.63 | 0.82 | 9.01 | 1.38 | 9.38 | 1.94 | 9.76 | 2.50 |
| | | | | 3000 ppm | 8.58 | 0.68 | 8.85 | 1.03 | 9.15 | 1.39 | 9.39 | 1.74 |
| 8.37 | 0.44 | 14.5 | 7.2 | **TBHQ** | | | | | | | | |
| | | | | 500 ppm | 9.00 | 1.11 | 9.96 | 2.11 | 10.92 | 3.12 | 11.88 | 4.12 |
| | | | | 1000 ppm | 8.61 | 0.80 | 8.99 | 1.35 | 9.37 | 1.91 | 9.74 | 2.46 |
| | | | | 2000 ppm | 8.56 | 0.68 | 8.83 | 1.03 | 9.10 | 1.38 | 9.37 | 1.73 |
| | | | | 3000 ppm | 8.50 | 0.53 | 8.65 | 0.66 | 8.80 | 0.80 | 8.94 | 0.93 |
| 8.36 | 0.44 | 14.5 | 7.2 | **PY** | | | | | | | | |
| | | | | 500 ppm | 8.70 | 0.97 | 9.20 | 1.76 | 9.70 | 2.55 | 10.20 | 3.34 |
| | | | | 1000 ppm | 8.61 | 0.80 | 8.99 | 1.35 | 9.37 | 1.90 | 9.74 | 2.45 |
| | | | | 2000 ppm | 8.56 | 0.68 | 8.83 | 1.03 | 9.10 | 1.38 | 9.36 | 1.73 |
| | | | | 3000 ppm | 8.50 | 0.53 | 8.65 | 0.66 | 8.80 | 0.79 | 8.94 | 0.92 |

**Table 4: KV and AV data obtained from ASTM 4625 @ $43^0$C – 12 weeks**