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ORIGINAL PAPER

Comparison of a New Estolide Oxidative Stability Package

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Abstract The rotating pressurized vessel oxidation test (RPVOT) was used in the analysis and determination of a new oxidative stability package (OSP) for a series of estolide based materials. Three antioxidants (BHT, two different alkylated diphenyl amines) were used in either 0.5 or 1.0 wt/wt%, in different ratios, and in conjunction with one another (hindered phenol/alkylated diphenyl amines or hindered phenol/mixed alkylated diphenyl amines). The estolide-based samples analyzed for their resistance to oxidation were two pure (distilled) estolides (oleic estolide 2-EH esters and coco-oleic estolide 2-EH esters), an estolide mixture that was analyzed straight from the reaction (coco-oleic estolide 2-EH esters with coco 2-EH esters) and finally the ester fraction from the estolide mixture (coco 2-EH esters). The coco estolide mixture and coco 2-EH esters had the best overall RPVOT times with 1.0% of the alkylated diphenyl amine, coco estolide mixture, 326 min, and coco 2-EH esters, 310 min. Coco estolides were expected to have an advantage over the simple oleic estolides due to the increase in saturation in the estolide. Unexpectedly, the two distilled estolides (oleic and coco) had very similar RPVOT max times with all the antioxidants, and were much higher than the other oxidative packages tested to date. In general, the alkylated diphenyl amine outperformed mixed alkylated diphenyl amines in the majority of the individual samples tested specially the coco 2-EH esters and distilled coco-oleic estolide 2-EH esters material at 1% OSP. Overall, a series of new antioxidants were tested and compared to other commercial products. A variety of physical properties of the four estolide based material were collected and compared to commercially acceptable material. Coco-oleic estolide 2-EH esters were formulated to have excellent pour points (−36 °C), were both oxidatively and hydrolytically stable (RPVOT, 310 min), with expected good biodegradability which should help commercialization.

Keywords Alkylated diphenyl amines · Estolides · Hindered phenol · Oxidative stability · Physical properties · RPVOT

Introduction

Estolides have been used to help develop new products from industrial crops [1, 2]. Estolides are obtained by the formation of a carbocation that can undergo nucleophilic addition with or without carbocation migration along the length of the chain. The carboxylic acid functionality of one fatty acid links to the site of unsaturation of another fatty acid to form oligomeric esters. The extent of oligomerization is reported by estolide number (EN) which is defined as the average number of fatty acids added to the base fatty acid. The estolide carboxylic acid functionality

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Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by USDA implies no approval of the product to the exclusion of others that may also be suitable.

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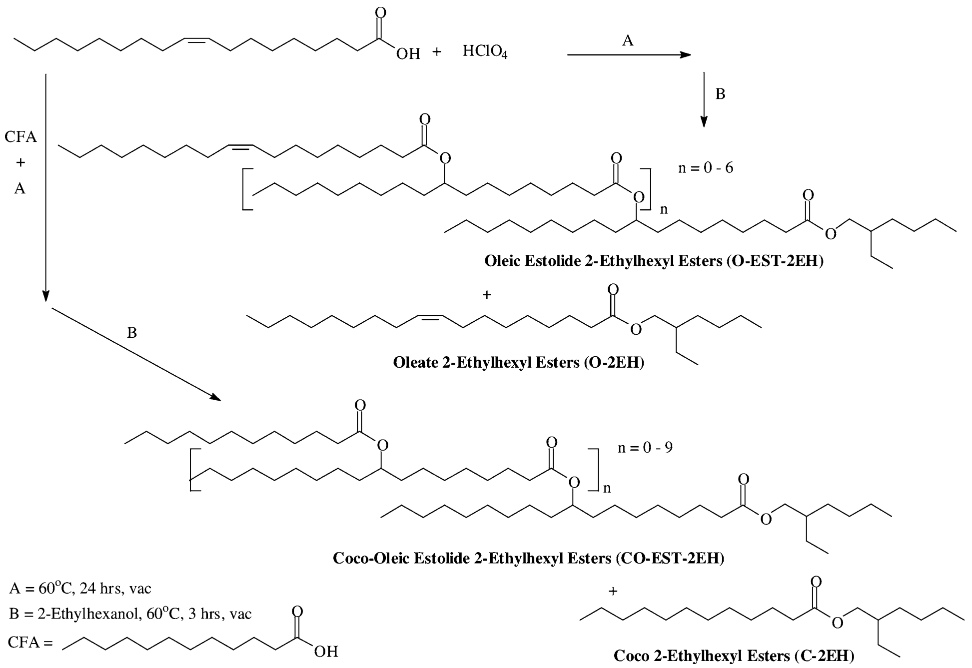
can be converted to the corresponding estolide ester under esterification conditions with the addition of an alcohol (Fig. 1).

Estolides have been developed in order to overcome some of the short falls associated with vegetable oils, which are known to have poor thermal oxidative stability [3], low hydrolytic stability [4] and poor low temperature properties [5]. Some of these deficiencies can be improved with the use of large volumes of additive packages, but usually at the sacrifice of biodegradability, toxicity, and cost.

There have been numerous ways that the oxidative stability of an oil have been measured; some of the common ways are: oxidative stability index (OSI) [6]; rotating pressurized vessel oxidation test (RPVOT) [3]; differential scanning calorimetry (DSC) [7]; Indiana stirring oxidation test (ISOT) [8]; and the thin-film microoxidation test [9]. The oleic based estolides were developed as an industrial base oil or as an industrial oil additive, so the material needs to be evaluated under conditions associated with industrial materials. These industrial oils will be replacing petroleum oils for which the recommended tests are generally the microoxidation or RPVOT for which the American Society for Testing Materials has developed a detailed test procedure for these materials. In RPVOT, the time is reported in minutes to failure which is when the pressure drops by 175 kPa from the maximum recorded pressure. The larger the RPVOT time the better the oxidative stability of the material, typical commercial engine oils have RPVOT times in the range 210–250 min. The RPVOT test method calls for the oil to be tested with materials that would be present in most applications i.e. water, copper, and oxygen. The test has been accepted by bio-based material producers as a suitable method to test the oxidative stability of these fluids. With vegetable based materials, the RPVOT tests both the thermal oxidative stability and hydrolytic stability. Pure vegetable-based materials are known to have low hydrolytic stability [4]. Thus, they do not perform well under the RPVOT ASTM method D 2272–98. One way of testing the oxidative stability of vegetable based materials without complication from their low hydrolytic stability is by modifying the published ASTM method. In one example [10] soybean oil, which has a poor hydrolytic stability, was tested with water removed from the RPVOT ASTM method. Thus resulted in increased RPVOT times. Because the oil was no longer able to suffer from the poor hydrolytic stability, the oil could then be evaluated for oxidative stability.

Estolides esters have previously been shown to have improved oxidative stability over vegetable oils. Cermak and Isbell [3] reported that the saturated-capped estolide esters synthesized from coco and oleic fatty acids provided a vary oxidatively stable product when mixed with a commercial oxidative stability package. These coco-oleic estolide esters had RPVOT times ~245 min with 1% of a Lubrizol oxidative stability package. The simple oleic estolide (RPVOT times ~132 min) did not perform as well as the coco-based material (RVPOT times ~264 min) when 1% of a Lubrizol oxidative stability package was used. The coco-estolide esters had lower iodine values (IV) because it contained more saturates in the molecule than the simple oleic estolide esters, which resulted in the

Fig. 1 Reaction scheme for the synthesis of oleic (O-EST-2EH) and coco oleic (CO-EST-2EH) estolide esters and coconut (C-2EH) and oleic esters (O-2EH)



increase in the oxidative stability of the coco-estolides. The increased oxidative stability for the coco-estolide esters were not unexpected but the simple oleic estolide ester would need to perform to a higher standard if it is to be used as an industrial fluid.

Thus far, estolide esters physical properties have compared favorably to commercially available industrial products such as petroleum-based hydraulic fluids, soy-based fluids, and petroleum oils. In this paper, we report the RPVOT times of four different estolide based samples formulated with either 0.5 or 1.0% of three different oxidative stability packages in varying ratios.

Experimental Procedures

Materials

Oleic acid (90%) and 2,6-di-*tert*-butyl-4-methylphenol (BHT) were obtained from Aldrich Chemical Co. (Milwaukee, WI). Perchloric acid (70%) and 2-ethylhexyl alcohol were obtained from Fisher Scientific Co. (Fairlawn, NJ). Potassium hydroxide was obtained from J. T. Baker Chemical Co. (Phillipsburg, NJ). Filter paper was obtained from Whatman (Clifton, NJ). Ethanol was obtained from AAPER Alcohol and Chemical Company (Shelbyville, KY). Coco fatty acids was obtained from Acme-Hardesty (Blue Bell, PA). Petroleum oil (Valvoline® 5 W–30, 10 W–30, 10 W–40, 20 W–50, SAE–30) and synthetic oil: Castrol Synthetic® 10 W–30 were obtained from Wal-Mart Department Store® (Peoria, IL). Soy based oil (Soylink®) was obtained as free samples from University of Northern Iowa (Cedar Falls, IA). Meadowfoam oil was supplied by the Fanning Corp. (Chicago, IL). Biobased Oils (Environlogic 132, 146, and 168) obtained as free samples from Terresolve Technologies Ltd. (Eastlake, OH). Alkylated diphenyl amine (Vanlube NA) and a mixture of alkylated diphenyl amines (Vanlube SL) antioxidants were obtained from Vanderbilt Corporation (Norwalk, CT) and were used as supplied. Figure 1, synthesis of estolide esters and esters, gives the structures, names, and abbreviations of the various estolide esters and esters used in this study. The O-EST-2EH is a homo-oligomer estolide obtained by the self-condensation of oleic acid. The CO-EST-2EH is a co-oligomer estolide and was obtained by the reaction of the carboxylic group of coco fatty acids (~50% lauric acid) with the double bond of oleic acid. The reaction conditions for the synthesis of O-EST-2EH, CO-EST-2EH, C-2EH, and a mixture of CO-EST-2EH and C-2EH are shown in Fig. 1. Pure (distilled) estolide esters and esters were obtained by purifying the reaction mixture on a Myers Pilot 15 Molecular Distillation Unit (Kittanning, PA). The purity of the estolides was highly dependent on the distillation cut and temperature of the distillation process. Another interesting characteristic of this particular synthetic procedure was that the estolide produced was comprised of mixtures of oligomers with varying degrees of oligomerization ($n = 0$–6 for O-EST, $n = 0$–9 for CO-EST). Details on complete synthesis and purification of the estolides can be found elsewhere [1, 3, 11].

GC

Hewlett-Packard 6890 N Series gas chromatograph equipped with a flame-ionization detector and an autosampler/injector was used for GC analysis. Analyses were conducted on a SP-2380 30 m × 0.25 mm i.d. column (Supelco, Bellefonte, PA). Saturated C_8–C_{30} FAMEs provided standards for making fatty acid and by-product assignments.

Parameters for SP-2380 analysis were: column flow 1.4 ml/min with helium head pressure of 136 kPa; split ratio 50:1; programmed ramp 120–135 °C at 10 °C/min, 135–175 °C at 3 °C/min, 175–265 °C at 10 °C/min, hold 5 min at 265 °C; injector and detector temperatures were set at 250 °C. Retention times for eluted peaks were: methyl stearate 12.3 min, methyl oleate 13.3 min, hydroxy methyl stearate 22.1 min, hydroxy methyl oleate 22.3 min, 14-hydroxy eicosanate 23.3 min, 14-hydroxy-*cis*-11-eicosanate 23.5 min, 14-hydroxy eicosanate 23.8 min, and 14-hydroxy-*cis*-17-eicosanate 24.0 min.

Estolide Numbers (EN)

EN were determined by GC from the SP-2380 column analysis as described previously [1].

GC Analysis of Hydroxy Fatty Acids

Analytical estolide samples for GC were prepared using procedures described by Cermak and Isbell [1].

Viscosity and Viscosity Index

Calibrated Cannon-Fenske viscometer tubes obtained from Cannon Instrument Co. were used to measure viscosity. Measurements were run in a Temp-Trol viscometer bath set at 40.0 and 100.0 °C. Viscosity and viscosity index were calculated using ASTM methods D 445–97 and ASTM D 2270–93, respectively. Duplicate measurements were made and the average values are reported.

Pour Point

ASTM method D97–96a was used to measure pour points to an accuracy of ±3 °C. The pour points were determined by placing a test jar with 50 ml of the sample into a cylinder submerged in a cooling medium. The sample temperature was measured in 3 °C increments at the top of the sample until the material stopped pouring. This point was determined when the material in the test jar did not flow when held in a horizontal position for 5 s. The temperature of the cooling medium was chosen based on the expected pour point of the material. Samples with pour points that ranged from +9 to −6, −6 to −24, and −24 to −42 °C were placed in baths of temperature −18, −33, and −51 °C, respectively. The pour point was defined as the coldest temperature at which the sample still poured. All pour points were run in duplicate and average values were reported.

Cloud Point

ASTM method D 2500–99 was used to measure cloud points to an accuracy of ±1 °C. The cloud points were determined by placing a test jar with 50 ml of the sample into a cylinder submerged into a cooling medium. The sample temperature was measured in 1 °C increment at the bottom of the sample until any cloudiness was observed at the bottom of the test jar. The temperature of the cooling medium was chosen based on the expected cloud point of the material. Samples with cloud points that ranged from room temperature to 10, 9 to −6, and −6 to −24, −24 to −42 °C were placed in baths of temperature 0, −18, −33, and −51 °C, respectively. The cloud point was defined as the coldest temperature at which the sample remained opaque. All cloud points run in duplicate and average values reported.

Rotating Pressurized Vessel Oxidation Test (RPVOT)

Test determinations were conducted on a (RPVOT) apparatus manufactured by Koehler (Bohemia, NY) using the ASTM Method D 2272–98. All oil samples were run at 150 °C. Samples were measured to 50.0 ± 0.5 g with 5.0 ml of reagent grade water added to the sample. The copper catalyst was measured to 3 m and sanded with 220 grit silicone carbide sand paper produced by Abrasive Leaders and Innovators (Fairborn, OH) and was used immediately. The wire was wound to have an outside diameter of 44–48 mm, weight of 55.6 ± 0.3 g and to a height of 40–42 mm. The pressure vessel was assembled with above reagents and slowly purged with reagent grade oxygen twice. The pressure vessel was charged with 90.0 ± 0.5 psi (620 kPa) of reagent grade oxygen then tested for leaks by immersing it in water. The test was stopped and the time recorded when the pressure in the vessel dropped by 175 kPa from the maximum pressure. All samples were run in duplicate and the average time in minutes was reported.

OSP Sample Preparation

The individual commercial antioxidant(s) were added to the estolide esters and esters samples on a % wt/wt basis. Formulated samples were stirred for 24 h before evaluation on the RPVOT.

Results and Discussion

The four different materials shown in Fig. 1 were evaluated for their oxidative stabilities. All the materials were synthesized under an acid catalyzed process that converted fatty acids to the free acid estolides under vacuum at 60 °C followed by an in situ esterification under similar conditions. The final products underwent simple vacuum distillation to remove excess 2-ethylhexyl (2-EH) alcohol. The simplest estolide, oleic estolide 2-EH esters (O-EST-2EH), underwent a further distillation at 180–200 °C to remove the fatty esters, oleate 2-EH esters (O-2EH), to give the distilled estolide (O-EST-2EH). A coco-oleic estolide 2-EH ester (CO-EST-2EH) was prepared under similar conditions to yield a distilled coco based estolide sample. Then a more complex mixture was examined; the complete coco-oleic estolide reaction was used as is (no distillation) so the sample contained the coco 2-EH esters (C-2EH) as well as the oleate 2-EH (O-2EH) esters in a ratio of 1:1:4 (C-2EH:O-2EH) as previously reported [12]. Finally the ester fraction which was obtained as the distillate during the purification step (distillation) of the synthesis of CO-EST-2EH contained the esters O-2EH and C-2EH in the same ratio as reported above.

Table 1 outlines the physical properties, pour and cloud points, viscosity, viscosity index, estolide numbers, and percent of estolide saturated-capped, from the estolide based reactions (Fig. 1) that were later mixed with the different oxidative stability packages (OSP). The pure (distilled) estolide materials had outstanding pour points (−33 and −36 °C for O-EST-2EH and CO-EST-2EH, respectively) while the esters, C-2EH and O-2EH, have higher pour points (−24 °C for C-2EH) than the estolides. The pour point of the pure (distilled) coco based estolides is one of the main physical properties that make estolides such an interesting industrial fluid. Generally, as saturation is added to a compound the pour points tend to increase. However, when estolides are used the pour points are decreased due to the branching within these molecules. The estolides all had excellent viscosity indexes, 170–185, and the viscosities were similar to other commercial oils.

Estolides are formed from the cationic homo-oligomerization of unsaturated fatty acids resulting from the addition of a fatty acid carboxyl adding across the olefin [13]. This condensation can continue, resulting in

Table 1 Physical properties of estolides 2-ethylhexyl esters and 2-ethylhexyl esters (see "Experimental Procedures" section, materials do not contain oxidative stability packages (OSP))

Sample no.	Description[a]	Pour point (°C)	Cloud point (°C)	Vis. @40 °C (cSt)	Vis. @100 °C (cSt)	Vis. index	Estolide number (EN)	Capped[b] (%)
1	O-EST-2EH	−33	<−33	95.9	15.4	170	1.44	18.0
2	CO-EST-2EH	−36	<−36	68.4	12.1	176	1.37	42.0
3	CO-EST-2EH & CO-2EH	−24	−20	19.5	4.8	185	0.35	NA
4	C-2EH	−24	−15	9.1	2.8	156	NA	NA

NA not applicable

[a] See Fig. 1 for structures

[b] Percent of estolide saturated-capped

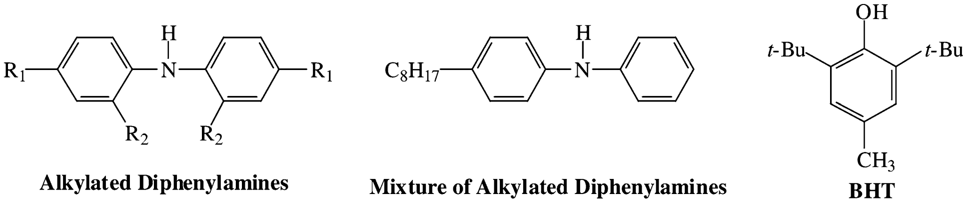


Fig. 2 Arylamines and hindered phenol inhibitors

oligomeric compounds where the average degree of oligomerization is defined as the estolide number (EN = $n + 1$, Fig. 1) [14]. When saturated fatty acids are added to the reaction mixture, the oligomerization terminates upon addition of the saturated fatty acid to the olefin since the saturate provides no additional reaction site to further the oligomerization and usually yields lower EN estolides. Consequently, the estolide at this point is prevented from further reaction, thus we term the estolide as being "capped" [1].

The two pure (distilled) estolides were analyzed for the amount of saturated capping material. The saturated capped % values in Table 1 were obtained from GC analysis of the estolide esters which were saponified and esterified. The components of the GC were classified as one of the following: unsaturated, saturated, and hydroxy fatty acids. The percent saturated-capped estolide was calculated as follows:

$$\%\text{sat. capped} = [(\text{sat. F.A.})/(100 - \text{hydroxy F.A.})] \times 100. \quad (1)$$

Analysis of GC data using Eq. (1) showed that the CO-EST-2EH contained 42% saturated capped estolides while the remaining material was capped with unsaturated fatty acids. Depending on the amount of the capped material, the physical properties of the estolides will vary, and the estolides interact differently i.e. the more saturation tends to lead to better oxidative stability.

A series of oxidative stability packages containing amines and hindered phenols were examined based on the fact that both class of compounds act as a radical scavengers through a hydrogen transfer mechanism [15]. Ten antioxidant packages were prepared by blending each amine (alkylated diphenyl amine or mixed alkylated diphenyl amines, Fig. 2) antioxidant with a hindered phenol (BHT, Fig. 2) antioxidant in the following ratios: 100:0, 75:25, 50:50, 25:75, and 0:100. Each antioxidant was blended into each of the estolide esters and esters to a concentration of 0.5 or 1.0% (wt/wt). Each sample was used in duplicate runs in the RPVOT. The RPVOT values reported were obtained following the RPVOT ASTM Method D 2272–98 exactly. Previously, soybean oil [10], which is known to have poor hydrolytic stability, was tested with water removed from the RPVOT ASTM method. The RVPOT times dramatically increased over RPVOT times when the ASTM method was followed as published with water, which demonstrates that materials that have good RPVOT time must be both thermally and hydrolytically stable. In our case, water was used in the RPVOT method thus the samples could potentially undergo hydrolysis that would yield unacceptable RPVOT times as most vegetable based materials, the oil samples tested here did not suffer from this condition. The RPVOT times that are reported for the estolides represent a combination of both the thermal oxidative stability and hydrolytic stability of the samples tested. The RPVOT results for the individual samples from Table 1 are displayed in Figs. 3, 4, 5 and 6. All samples without added oxidative stability packages gave RPVOT times of less than 20 min, which was similar to data on vegetable oils without antioxidants, Table 2. In general, as the amount of amine antioxidants increased the RPVOT times increased for all samples. With

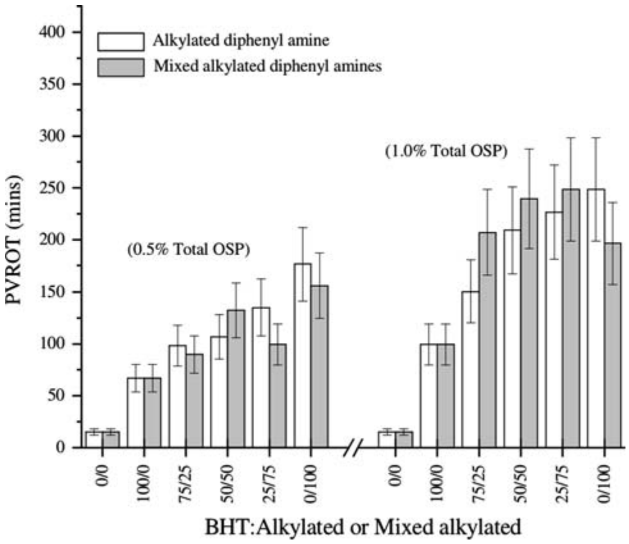


Fig. 3 RPVOT times for Oleic Estolide 2-EH Esters (O-EST-2EH, Sample 1) with 0.5 and 1.0% oxidative stability packages (OSP)

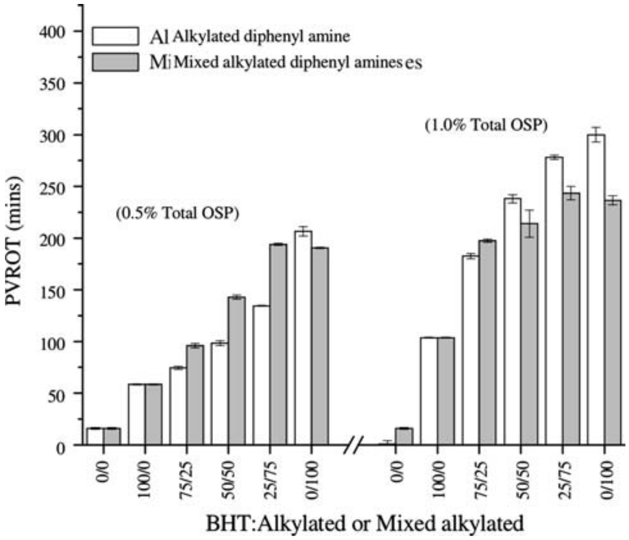


Fig. 4 RPVOT times for coco-oleic estolide 2-EH esters (CO-EST-2EH, Sample 2) with 0.5 and 1.0% oxidative stability packages (OSP)

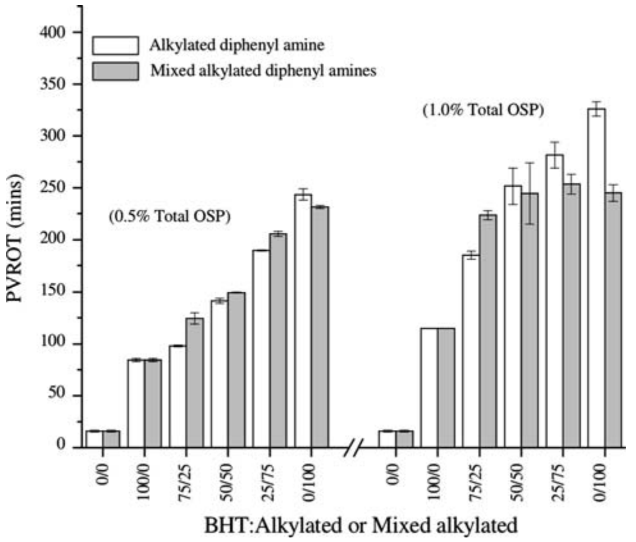


Fig. 5 RPVOT times for coco-oleic estolide 2-EH esters containing coco 2-EH esters (CO-EST-2EH and C-2EH, Sample 3) with 0.5 and 1.0% oxidative stability packages (OSP)

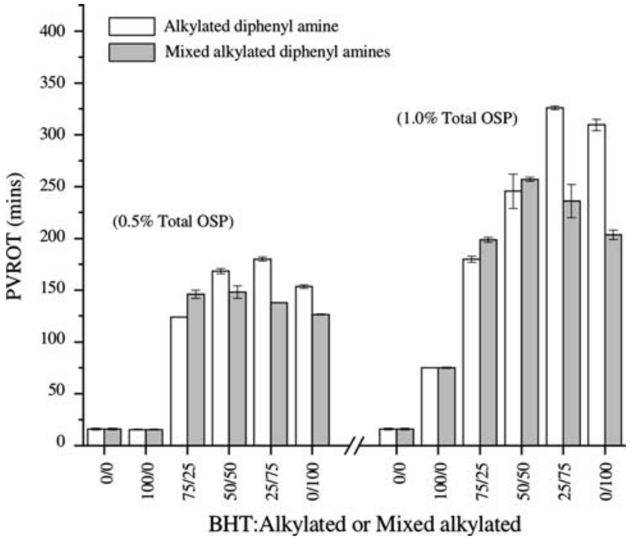


Fig. 6 RPVOT times for coco 2-EH esters (C-2EH, Sample 4) with 0.5 and 1.0% oxidative stability packages (OSP)

100% of a commercial hindered phenol (1% wt/wt) the RPVOT times in general increased from 16 min to about 100 min; which was not outstanding but in most cases an improvement was visible and comparable with current commercial bio-based materials (Table 2). In all cases, the samples with the highest RPVOT times had higher amounts of the amine packages added to the estolide based materials.

In the cases where the amines were the only antioxidant used, the alkylated diphenyl amine preformed better than the mixed alkylated diphenyl amine. The simple oleic estolide, O-EST-2EH (Fig. 3), with 1.0% OSP, the 100% alkylated diphenyl amine had a RPVOT time of 248 min whereas the mixed alkylated diphenyl amine was just 197 min. This trend held with all four oil samples as well as at both the 0.5 and 1.0% OSP. The most dramatic improvements were observed with two of the coco oil samples (samples 3 and 4, Figs. 5 and 6). The mixture of coco esters and coco estolide sample, CO-EST-2EH and C-2EH, (Fig. 5) showed an improvement of more than 80 min or 33% in the RPVOT times at the 1.0% OSP. While the coco esters, C-2EH, sample (Fig. 6) showed an improvement of more than 105 min or 52% in the RPVOT times at the 1.0% OSP.

Table 2 RPVOT values of common functional fluids

Sample	Average time (min)
Crambe oil[a]	13
Soybean oil[a]	13
Meadowfoam oil[a]-crude	20
Biobased oil 146	51
Biobased oil 132	67
Biobased oil 168	71
Soy based oil	83
Petroleum oil 20 W-50	214
Petroleum oil 10 W-30	223
Petroleum oil 10 W-40	224
Petroleum oil SAE-30	224
Petroleum oil 5 W-30	228
Synthetic oil 10 W-30	246

[a] Unformulated

The simple oleic estolide (O-EST-2EH), Fig. 3, and the coco based estolide (CO-EST-2EH), Fig. 4; both gave RPVOT times that were within experimental error of one another. Cermak and Isbell previously showed that complex estolides had the best RPVOT times with the addition of a different formulated commercial antioxidant additive when capped with a very short chain, saturated fatty acids such as those present in cuphea [16] and coconut [12]. The coco-estolide had the better RPVOT times due to the saturation in the molecule. With the amine antioxidants, the estolides unexpectedly performed about the same with longer RPVOT times than under previous conditions. The similar RPVOT times between the two estolides samples were contributed to the interactions of the estolides with the antioxidants.

The last two samples, 3 and 4, had significantly longer RPVOT times than the pure (distilled) estolides with the amine additive packages. The estolide and coco ester (CO-EST-2EH and C-2EH, Table 1, Sample 3) had longer RPVOT times than the simple coco estolide (CO-EST-2EH), Table 1, Sample 2. The 77 min increase was contributed to the increased amounts of saturated esters present in the sample. Table 1, Sample 4 (C-2EH) proved to yield the longer RPVOT times of 326 min which was expected as the sample contained the highest amounts of saturation of all the samples analyzed.

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