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Properties of Soybean Biodiesel Blended with Palm or Tallow During Long Term Storage Under Different Conditions

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Abstract – *Soybean biodiesel has been commercially produced mainly due to its environmental benefits and availability. Despite of being economically feasible, most of its esters molecules have at least one unsaturated double bond, which can be oxidized and reducing biofuel shelf life. In order to improve qualities of soybean biodiesel without using chemical performance compounds, this research investigated the behavior of different types and quantities of biodiesel blends stored at 25 °C along 60 days. Palm and tallow biodiesel (10 %, 20 % and 30 %) were separately blended with soybean biodiesel. Their properties were analyzed freshly, after 30 and 60 days comparing them with pure soybean, palm and tallow biodiesel. Relationship between percentage of saturated biodiesel and time of storage was also established by using response surface methodology. Results from this procedure showed that percentage of saturated biodiesel needed to be effective against oxidation depends on the temperature of storage. However, induction period was extremely affected by time of storage at high temperatures. Long term storage test reveals that only when 30 % of tallow was used, the soybean blend reaches the adopted standard limits in Brazil.*

Keywords*: Biodiesel, Oxidation Stability, Palm, Storage, Surface Response, Tallow*

I. Introduction

Soybean biodiesel is largely used around the word, especially in Brazil where this energy matrix is supplied by almost 80% of the internal consumption [1]. Such as largely reported in literature [2]-[3]-[4], soybean biodiesel has low oxidation stability due to its fatty acid profile, which is composed by more than 80% of unsaturated double bonds. Biodiesel properties are intrinsically correlated with their fatty acids features such as carbon chain size and its bonds, quantity and quality of fatty acid bonds, presence of non-glycerides compounds [5]-[6].

Oxidation process can be mitigated through chemical additives, such as phenolic substances, which have been used as antioxidant, despite of their high costs.

Besides being an alternative to chemicals of performance, the employment of multiple feedstocks allows varying type and quality and, hence, cost. Price remains a serious threat to the economic viability of biodiesel industry and mixing feedstock could also be one of the solutions to feedstock cost, which currently accounts for over 85% of biodiesel production expenses [7]-[8]. Most of biodiesel studies reports properties from a single feedstock source. Those few that investigates the influence of mixed feedstocks on biodiesel properties generally applies unusual oils [9]-[10].

This study aims to analyze the influence on fuel properties of biodiesel mixtures from the most used oils and fat in the word.

For that, it was studied soybean blends with palm and tallow in several proportions along 8 weeks in 3 different temperatures of storage.

II. Material and Methods

Soybean biodiesel was obtained from an edible refined commercial local brand. It was transesterified with 35 % (w / w) of methanol 99.9 % (Tedia) and 0.75 % (w / w) of potassium hydroxide (KOH) from Proquimios by 1 hour under constant stirrer. For palm oil and tallow, the following proportions were used: 40 % (w / w) of methanol and 1 % (w / w) of KOH. This adjustment was necessary due to the high content of saturated fatty acid in those feedstocks in order to avoid losses. Both were washed with 3 % of citric acid solution and, after that, dried.

II.1. Surface Response Methodology

It is known that time of storage and fatty acid profile directly interfere on oxidation stability [4] and because of that they were selected as independent variables. To evaluate how these factors influence oxidation stability of samples stored at 5 °C, 25 °C and 45 °C, it was carried out a statistical test. For that, an experimental design was employed to the 22 soybean biodiesel samples blended with tallow or palm (11 samples for each saturated feedstock). A central composite was applied by using

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axial points with alpha for rotatability equal to 1,4142. Dependent variable (in this case oxidation stability) was studied in three different temperatures for each type of biodiesel blend (soybean with tallow and soybean with palm), providing empirical models to predict induction period in the range of temperature studied. Analyses of results were obtained by Statistica 7.0 software.

For each temperature, it was developed a second-order model to represent how the induction period (IP) of those samples behavior according to variation of time of storage (TS) and percentage of saturated biodiesel (PS) into soybean biodiesel, which formula is shown in Equation (1):

$$IP = a + b \cdot PS + c \cdot PS^2 + d \cdot TS + e \cdot TS^2 + f \cdot PS \cdot TS \quad (1)$$

where a, b, c, d, e and f are the numeric parameters of the second-order model.

II.2. Biodiesel Properties During Storage

Six blends were prepared with soybean biodiesel adding 10 %, 20 % and 30 % of tallow biodiesel (called T 10, T 20, T 30) and 10 %, 20 % and 30 % of palm biodiesel (P 10, P 20, and P 30). Besides that, three blank samples of each biodiesel (S 100 - soybean, T 100 - tallow and P 100 - palm) were also stored at the same way, in a closed transparent glass flask at room temperature (± 25 °C) during 60 days.

Analysis of content of ester - CE (EN 14103) [11], acid value - AV (ASTM D664) [12], iodine value - IV (EN 14111) [13], kinematic viscosity at 45°C - KV (ASTM D445) [14], oxidation stability - OS (EN 14112) [15], flash point - FP (ASTM D93) [16], content of water - CW (ASTM D6304) [17] and cold flow plugging point - CFPP (ASTM D6371) [18] were carried out with fresh samples and after 30 and 60 days to monitor the influence of time in these features.

III. Results and Discussion

III.1. Surface Response Analysis

Sixty six experiments were carried out and the results from central composite design are shown in Table I. It is clear that, to the same level of percentage of saturated biodiesel, both blends presented higher levels of induction period the lower the time of storage, showing the negative influence of the time in the stabilization of biodiesel.

Samples of the same blend presented higher oxidation stability to those with higher concentration of saturated biodiesel, considering the same time of storage, what implies that increasing saturated esters also increases the shelf life of biodiesel. Not only the quantity of saturated fatty acid methyl ester is important to the oxidation stability, but also the quality of them, such as carbon chain size and the presence, quality and quantity of non-glyceride compounds. Therefore, there is a difference in the behavior of the blend with tallow and with palm. Rate of oxidation to tallow is higher than to palm when at different temperatures. This fact may be occurred due to the high level of natural antioxidant present in palm biodiesel, such as beta-carotene. This class of non-glyceride tends to capture the free radical, i.e., it is oxidized first preventing the ester from the attack of the oxidizing agent.

TABLE I
CENTRAL COMPOSITE DESIGN OF EXPERIMENTS AND THEIR EXPERIMENTAL RESULTS

Exp [a]	PS (w/w)	TS (days)	Induction period for blend with palm (h)			Induction period for blend with tallow (h)		
			5 °C	25 °C	45 °C	5 °C	25 °C	45 °C
1	10	10	5,82	4,83	2,59	6,96	5,56	1,23
2	10	50	5,45	3,72	0,00	6,62	3,65	0 [b]
3	30	10	6,27	6,02	3,14	7,65	6,71	1,76
4	30	50	6,06	4,84	0,00	7,92	5,18	0
5	5,86	30	5,44	4,26	0,56	6,78	4,97	0
6	34,14	30	6,36	5,44	0,69	8,17	5,85	0
7	20	2	5,84	5,84	5,50	6,83	6,59	5,1
8	20	58	5,60	3,76	0,00	6,18	4,91	0
9	20	30	5,96	5,28	0,84	7,46	5,69	0
10	20	30	5,95	5,20	0,87	7,39	5,67	0
11	20	30	5,91	5,25	0,89	7,46	5,62	0

[a]number of experiment; [b]zero value was considered when IP was very low, less than the sensitiveness of equipment.

Statistically, empirical data for palm blend showed that, at 5 °C, the percentage of palm biodiesel into soybean biodiesel is the variable that most influences the oxidation stability. Time of storage also is significant, in a less dimension and presents a negative influence though. Therefore, the higher the time of storage, the lower the induction period. At 25 °C, time of storage is still negative and becomes the variable that most influence the oxidation stability. Percentage of palm significantly contributes to increase induction period. Such as at room temperature, time of storage is also the most important variable that negatively influences biodiesel stability at 45 °C, when percentage of palm has almost no influence on IP.

This shows that, increasing temperature, the percentage of saturated esters ceases to be important and fails to exert a positive influence on the response. Likewise, the time of storage of the sample becomes decisive for the stability of the mixture of the biofuels.

All calculated models had a satisfactory coefficient of determination (R^2), above 0.90, except to tallow blend at 45 °C, which not presented enough variation of results to be able to provide a model, not being possible to do any statistical analyzes in this temperature. It means that, at this temperature, tallow blend is extremely sensitive to oxidation. Its decrease in induction period is more pronounced than to palm blend.

Low errors of the models were obtained by the triplicates of center points and expanded to whole model. Besides that, predicted values by modeled equations were very near to what were experimentally observed. Effects of the parameters obtained for tallow and palm blends are shown in Table II. These models can be used when dependent variables are coded (+1, -1).

TABLE II
EFFECT ESTIMATES FOR TALLOW AND PALM BIODIESEL IN EACH TEMPERATURE

T (°C)	Statistical parameters						R^2	Error
	a	*b*	*c*	*d*	*e*	*f*		
Palm Blend								
5	5,94	0,59	0,01	-0,23	-0,18	0,08	0,96	0,0007
25	5,24	0,99	-0,38	-1,31	-0,43	-0,04	0,98	0,0016
45	0,87	0,18	-0,37	-3,38	1,76	-0,28	0,98	0,0006
Tallow Blend								
5	6,44	0,99	0,19	-0,25	-0,78	0,30	0,92	0,0016
25	4,66	0,98	-0,40	-1,45	-0,06	0,19	0,92	0,0013
45[a]	-	-	-	-	-	-	0,59	-

[a]Data not gave enough information. Underlined parameters were considered insignificant (p value > 0,05).

Parameter *a* decreases drastically with the increase of temperature, as it was expected, because the higher the temperature, the lower will be the initial IP. Temperature variation did not promote any alteration on the influence of linear saturated percentage (*b*) in the case of tallow biodiesel.

Only to palm this variation was observed, meaning that the higher the temperature, the higher the percentage of palm necessary to stabilize the biodiesel blends at tallow levels, because palm has less saturated esters than tallow.

Increasing the temperature, the effect of the time of storage parameter (*d*) becomes higher, in both cases, i.e., the higher the temperature, the larger will be the negative influence of time of storage over IP because it will accelerate the oxidation process. Interaction terms were very low, being not considered.

Linear parameter of percentage of saturated esters was positive and higher to tallow (6.44) than to palm (5.94) at 5°C, which shows that blend with tallow has oxidation stability a slightly more sensitive to PS than palm. At the same temperature, level of significance for linear time of storage parameter was very near comparing palm and tallow blends, always being negative. It means that time of storage has almost the same negative impact in induction period to both blends.

Afterward the evaluation of effects, it was possible to obtain the surfaces responses for each blend in each temperature. At 5°C, palm blends basically not depend on the TS, unlike tallow blends that are slightly affected (Figs. 1 and 2, respectively). Natural antioxidants can be responsible to make palm blends more resistant at this temperature, in contrast to tallow blends, which presented a restricted time of storage.

At room temperature (Fig. 3 to palm and Fig. 4 to tallow), the IP starts to be restricted to a limited quantity of days of storage. Nonetheless, to both blends, the lower time of storage and the higher percentage of saturated ester, the higher will be the oxidation stability of the biodiesel blend.

Palm and tallow blends become quickly oxidized at 45°C, no matter PS, as shown in Fig. 5 for palm blend.

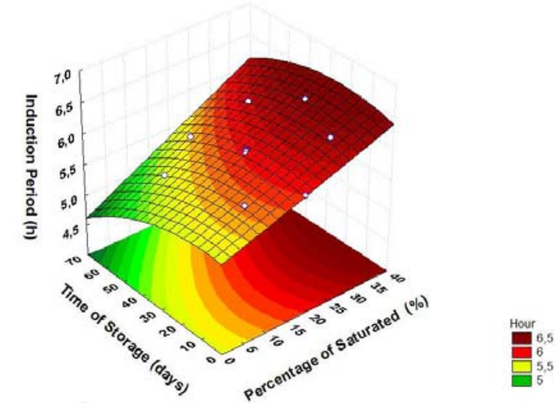

Fig. 1. Surface response to palm blend stored at 5°C

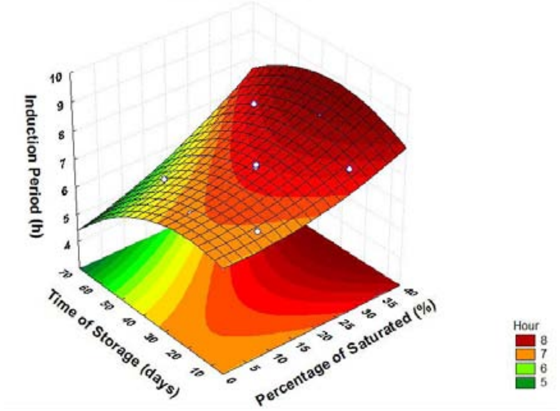

Fig. 2. Surface response to tallow blend stored at 5°C

III.2. Biodiesel Properties over Time of Storage

Since biodiesel must be stored and handled before it is dispensed into vehicle fuel tanks, its properties can varies not only relative to its free fatty acid profile, but also to the environment in what it is kept.

Likewise, free fatty acid compositions of biodiesel from soybean, tallow and palm were measured by European EC standard and showed in Table III.

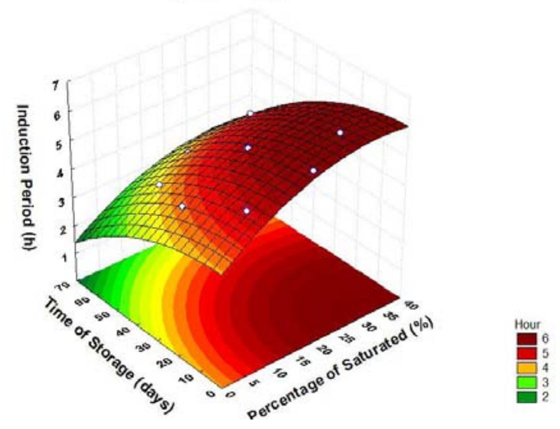

Fig. 3. Surface response to palm blend stored at 25°C

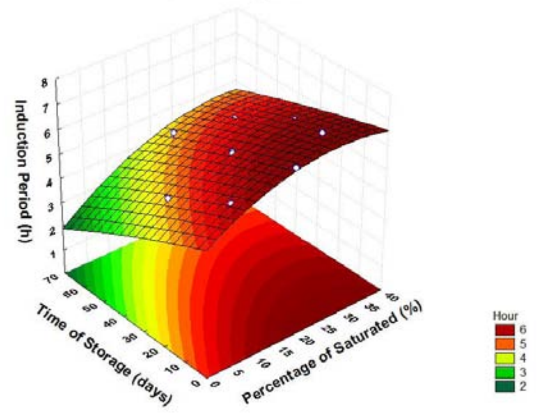

Fig. 4. Surface response to tallow blend stored at 25°C

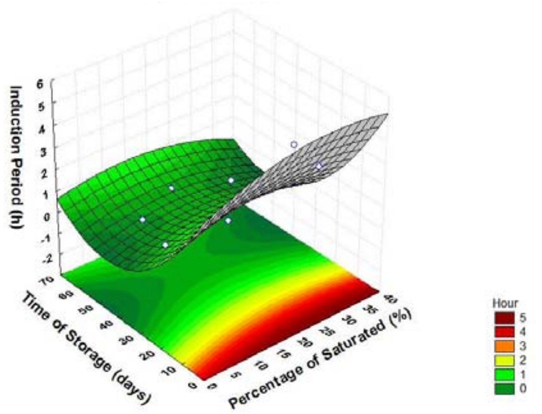

Fig. 5. Surface response to palm blend stored at 45°C

As a consequence to each different profile, physical and chemical biofuel properties will be different. It was observed through this research that mixing saturated esters can avoid or postpone some undesirable changes in soybean biodiesel, as shown in Table IV.

Once the analyzes were carried out before and after 60 days of storage, the difference in each parameter in this period (final minus initial) was called delta. Firstly, delta of ester content (EC), which shows the biodiesel degradation after 60 days, is less when saturated biodiesel is added to soybean biodiesel, mainly with tallow, 15 % richer in saturated esters than palm. It is worth to note that beside this fact, tallow has the bigger saturated carbon chain size. Although the acid values (AV) are still according to the adopted standard limit, all mixtures presented higher acid values. This fact is probably related to the hydroperoxide decomposition, which breakage generates smaller carbon chain size of carboxylic acids [19]. To all mixtures, delta of kinematic viscosity at 40 °C (KV) values was lower than to the pure biodiesel. It can be associated to a diminution in polymeric oxidation because the increasing in the saturated compounds quantity presents in palm and tallow. Despite the fact that pure tallow biodiesel had the highest value to oxidation stability (OS) for fresh biodiesel, (13.42 h), followed by palm (10.31 h) and soybean (5.56 h), respectively, it had also the highest delta in induction period, reaching 5.97 h after 2 months.

Palm biodiesel presented a hazel-red color, what visually demonstrated the presence of natural antioxidants, such as its known beta-carotene. Although it has more polyunsaturated esters than tallow and therefore it could oxidize firstly, these specific non-glyceride compound slow the esters oxidation. On the other hand, the proportion of saturated esters in tallow biodiesel keeps it inside specification because its fresh induction period was highly superior.

Both P 100 and S 100 had almost the same delta decrease in OS (-1.27 h and -1.22 h, respectively), but their fresh oxidation stability values were different. Not even when fresh, the soybean biodiesel reaches the minimum of 6.0 h.

When only P 100 was evaluated, OS was kept at highest levels over the months. However, when it was mixed with soybean biodiesel, its mixtures not presented the same behavior. Perhaps it is necessary a larger quantity of saturated esters from palm oil to obtain this behavior with soy blends [20].

Although palm apparently seemed to be more resistant to oxidation process over the months, tallow blend T 30 was the only one that maintained the biodiesel according to the adopted specification.

TABLE III
ESTERS COMPOSITION (%) OF SOYBEAN, TALLOW AND PALM BIODIESELS

Methyl Ester	(C_{number}: $_{Unsaturation}$)	Soybean	Tallow	Palm
Laurate	C12:0	0,0035	0,1007	0,1358
Myristate	C14:0	0,0704	ND	0,4095
Palmitate	C16:0	10,7548	26,5373	30,9033
Palmitoleate	C16:1	0,0885	2,5371	0,1425
Stearate	C18:0	3,2738	23,1751	4,1927
Oleate	C18:1	30,4745	38,6652	40,9443
Linoleate	C18:2	49,5510	7,6153	21,1243
Linolenate	C18:3	4,7130	0,6643	1,4644
Arachidate	C20:0	0,3220	0,2130	0,3378
Gadoleate	C20:1	0,2625	0,3777	0,1739
Behenate	C22:0	0,4302	0,0600	0,1408
Erucate	C22:1	ND[a]	ND	0,0056
Lignocerate	C24:0	0,0558	0,0329	0,0035
Nervonate	C24:1	ND	0,0213	0,0216
∑*Saturated*	Cn:0	14,91	50,12	36,12
∑*Monounsaturated*	Cn:1	30,83	41,60	41,29
∑*Polyunsaturated*	Cn:>1	54,26	8,28	22,59

TABLE IV
DELTAS BETWEEN RESULTS FROM FRESH SAMPLES AND SAMPLES STORED BY 60 DAYS

Sample	EC (%)	AV (mgKOH/g)	IV (g/100g)	KV (mm2/s)	OS (h)	FP (ºC)	CW (mg/kg)	CFPP (°C)
S 100	-7,81	0,0264	-4,90	0,0810	-1,27	-5,0	40	6
P 100	-6,14	0,0288	1,85	-0,0071	-1,22	-1,5	70	0
P 10	-6,46	0,0356	-4,20	-0,0304	-0,74	-1,0	40	9
P 20	-4,27	0,0682	-3,25	-0,1319	-0,77	-0,5	0	7
P 30	-1,77	0,0348	-2,73	-0,1317	-0,71	0,0	10	0
T 100	-0,54	0,0398	-10,90	0,0420	-7,45	7,0	80	0
T 10	-1,28	0,0812	-6,12	-0,1067	-1,51	6,0	70	5
T 20	-0,79	0,0838	-4,82	-0,1051	-1,69	4,5	20	5
T 30	-0,69	0,0552	-3,98	0,1030	-1,05	4,5	40	2

In tallow biodiesel, 23.18 % of esters were methyl stearate (C18:0), while palm had only 4.19 %. Moreover, comparing palmitate (C16:0) to stearate, this last is more stable due to its higher carbon chain size, providing to biodiesel blended with tallow a less degradation.

P 100 also maintains the integrity of the unsaturated bonds, as can be seen by IV, once again not only because its quantity of palmitate but also due to its naturals antioxidants. Besides that, palm biodiesel has more polyunsaturated esters (22.59 %) than tallow (8.28 %), and when these types of esters oxidize through primary oxidation, they trend to firstly generate monounsaturated compounds, which IV is higher than to those compounds richer in saturated bound. In the case of T 100, IV decreasing was higher. It is richer in monounsaturated esters (41.60 %), which oxidation results in saturated compounds making IV decreases after the time of storage, meaning that the degradation occurred due to the breakage of unsaturated bonds. It also follows the trend to increase delta with the increase of tallow percentage, i.e., the higher the quantity of saturated esters, the lower will be the decreasing of IV.

Both soybean and palm pure biodiesels presented a decrease in flash point (FP), contrary to what was observed to their mixtures, where flash points increased with the increase of palm percentage. Once soybean and palm are richer in polyunsaturated esters (54.26 % and 22.59 %, respectively), they can degrade their esters into lower compounds through secondary oxidation process, decreasing the mean carbon chain size and, consequently, the temperature at which they can vaporize to form an ignitable mixture in air, being lower to those who had less percentage of palm. To tallow and its mixtures blends, the flash points were higher, but there was a decreasing with the increase of tallow percentage. As tallow had a higher delta of oxidation, it may have contributed to this change, by modifying the vaporization temperature.

Content of water (CW) absorbed increased to all samples, being more hygroscopic over the months, but not enough to take it out of the specification.

Cold flow plugging point (CFPP) seemed to be practically constant over the months to pure biodiesel of soybean, palm and tallow. However, when mixed, they presented an increase in CFPP, which was higher to lower concentrations of saturated biodiesel. The breakage of unsaturated bonds occurred due to the oxidation process did the plugging point increases. Therefore, the lower the content of saturated esters in the mixture, the higher the degradation and, consequently, higher the increasing in CFPP.

IV. Conclusion

Both palm and tallow biodiesels improved induction period of soybean biodiesel. The higher the quantity of saturated esters, higher was the shelf-life of blend. At 5°C, the effect of percentage of saturated esters was more important than time of storage. Natural antioxidant of palm seemed to be sensitive at high temperatures, but effective against oxidation at low temperature. At 45°C, no matter the amount of saturated esters, time of storage was decisive to the stability of the blend. Samples stored for 60 days at 25°C presented an increase in acid value, flash point, content of water and a decrease in viscosity, iodine value, but not enough to take the blend out of conformity.

Oxidation stability only is kept according to specification after the time of storage when 30% of tallow biodiesel is used. The effect in stability of methyl stearate from tallow was superior to the combined effect of methyl palmitate and beta-catorene from palm.

Acknowledgements

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She has been published some articles in biodiesel, concerning esterification, heterogeneous catalysis, molecular modeling, kinetics. Currently, she works on quality control of biodiesel, mainly with biodiesel stability. He has been published many articles in different areas of Chemistry, including bioenergy, fossil fuels, and has been developing many others researches.
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