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STUDY OF ETHANOL – DIESEL (E-DIESEL) BLENDS STABILITY USING A RENEWABLE TERNARY ADDITIVE

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Abstract. *With the shortage of conventional energy and the increasingly stringent emission standards, it is necessary to develop new internal combustion engines with low emissions, high fuel efficiency and high specific power. In this context, the use of biofuels (mainly biodiesel and ethanol) presents an attractive potential as it constitutes a renewable energy source. However, there are many technical barriers to the direct use of ethanol in diesel fuel: indeed, Diesel engines cannot operate normally on ethanol–diesel blend without special additives. Based on a literature review, ternary additives composed of biodiesel and vegetal oil from two renewable sources and n-butanol were synthesized through a Doehlert experimental design. This work studied the impact of four additives to stabilize Brazilian diesel fuel (with 7 vol% of biodiesel, B7) blended with aqueous ethanol, both commercial. Preliminary results showed that high content of biodiesel was required and that high n-butanol content had negative effect on stabilization. Nevertheless, the additives only delayed the phase separation for less than one hour. Consequently, the effect of ethanol concentration and temperature of these four additives were investigated using anhydrous ethanol. In presence of the additive, the range of instability was narrower and, even if phase separation was observed, organic compounds were dragged into ethanol phase.*

Keywords: *Compression ignition flex fuel motor, Biofuels, Ethanol-diesel blends (E-diesel), Miscibility*

1. INTRODUCTION

Most scenarios developed by economists are based on a regular growth of global energy demand over the next twenty years. In this balance, nuclear and renewable energy (wind, hydro, solar, etc.), although in full expansion, will remain marginal compared to fossil fuels. Oil demand for transport and petrochemical is expected to grow by over 15% until 2040 (IEA, 2014). With the shortage of conventional energy and the increasingly stringent emission standards, it is important to develop new internal combustion engines with low emissions, high fuel efficiency and high specific power (Lei, *et al*., 2012). In this context, the use of biofuels presents an attractive potential. Biofuels are produced from biomass and, therefore, as long as the growing cycle is respected, constitute renewable energy source. In addition, the development of biofuel production chains can participate in the energy independence in many countries (Ballerini, 2007). Biodiesel-diesel blends can be used on existing engines to achieve both environmental and energy benefits. Biodiesel has properties similar to those of traditional fossil diesel and it can substitute diesel with little or no engine modification. Substantial reduction in particulate and CO emissions can be obtained through the addition of biodiesel to diesel (Shi, *et al*., 2006). As biodiesel cannot entirely replace fossil diesel demand, ethanol is considered as an alternative. In order to introduce ethanol in Diesel engines, three main techniques have emerged during recent years: alcohol fumigation where alcohol is added to the intake air charge; dual injection where each fuel is injected separately;

and ethanol-diesel blends (E-diesel) where the fuels are mixed prior to injection (Abu-Qudais, *et al*., 2000; Boretti, 2012). The last method is the easiest as ethanol could be used in the form of solutions, without technical modifications. Many investigations showed an increase in brake thermal efficiency and in specific fuel consumption, a slight decrease in engine power and a more significant decrease in exhaust emissions in E-diesel blends compared to the use of diesel.

However, there are many technical barriers to the direct use of ethanol in diesel due to its properties, including low cetane number and lubricity and poor miscibility of ethanol in diesel in cold weather (Hansen, *et al*., 2001). E-diesel blends are not accurately described as either miscible or immiscible. Miscibility of ethanol in diesel depends on the hydrocarbon composition and wax content of the base diesel, ethanol content, and the temperature of the diesel fuel (Ribeiro, *et al*., 2007). Aromatic contents and intermediate distillate temperatures has a significant impact on the miscibility limits. It was verified that, for blends of ethanol with paraffinic and olefinic hydrocarbons, the temperature of phase separation decreases gradually with the increase of the molecular mass and the number of unsaturations. Naphthenic and aromatic hydrocarbons show low temperatures of phase transition. It has been found that the presence of ethanol in any proportion in a mixture with biodiesel does not cause phase separation under the conditions of use in Brazil (Caetano, 2003; Can, *et al*., 2004). Anhydrous ethanol is highly soluble in diesel fuel at contents of approximately 0-30 vol% and 70-100 vol% for temperatures higher than 20°C. However, at temperatures below 10°C, ethanol is almost immiscible in diesel and this affects the fluidity and good filterability of the E-diesel in cold climatic conditions. Miscibility also depends on the water content of ethanol, as ethanol is hygroscopic and easily picks water up from ambient air and from the distribution system. The miscibility and the cloudiness in the mixture followed by phase separation have been observed when the water content of the ethanol exceeded 1 vol% (Satgé de Caro, *et al*., 2001; Fernando and Hanna, 2004; Weber de Menezes, *et al*., 2006; Ribeiro, *et al*., 2007).

Indeed, diesel engines cannot operate normally on ethanol–diesel blend without special additives. There are two additive-based approaches to maintaining stable blends: adding surfactants (or emulsifiers) that produce stable (micro)emulsions or adding co-solvents that produce stable homogeneous solutions (Ribeiro, *et al*., 2007). Among the applicable co-solvents, biodiesel are primarily used because of their similarity to diesel, which allows the use of ester - diesel blends in any proportion. Moreover, biodiesel is miscible with alcohols. Its addition prevented phase separation acting as an amphiphile, which improves the tolerance to water, the lubricating properties and the cetane number of the blends. Moreover, this addition increases the oxygen level in the blend. Numerous studies have been done since 1980 using diesel – ethanol – biodiesel blends with anhydrous ethanol (up to 30 vol%) or aqueous ethanol (up to 10 vol%) and using different biodiesel sources. The results indicated that methyl ester was better than ethyl ester in improving the miscibility of the E–diesel blend. Overall, investigations showed that diesel – ethanol – biodiesel blends reduce NOx emissions while increasing CO and HC emissions, perhaps due to the cooling effects of alcohols (Kwanchareon, *et al*., 2007; Lapuerta, *et al*., 2007; Lapuerta, *et al*., 2009; Guareiro, *et al*., 2009; Chotwichien, *et al*., 2009; Cheenkachorn, *et al*., 2010; Torres-Jimenez, *et al*., 2011; Yilmaz, *et al*., 2014).

This work studied the impact of a new additive to stabilize Brazilian diesel S10 fuel (with 7 vol% of biodiesel, B7) blended with aqueous ethanol, both commercial. Based on literature review and Lei's work (Lei *et al*., 2012), ternary additives composed of biodiesel, vegetal oil and *n*-butanol were synthesized through a Doehlert experimental design considering all the possible combinations with sources for biodiesel-vegetal oil (A-A, A-B, B-A and B-B). All compounds of the additive can be obtained from renewable sources. The impact of 2 vol% of such additive to stabilize E-diesel blends (ratio 51: 49), as the influence of additive concentration, is studied. Based on these results, the best additive for each couple was defined. Moreover, the effect of ethanol concentration and different temperatures (10, 20 and 30°C) on these four additives were investigated using anhydrous ethanol.

2. METHODOLOGY

2.1 Material

The phase behaviour of the E-diesel blends was studied using commercial additivated S10 diesel B7 and aqueous ethanol fuel samples bought from a gas station in Rio de Janeiro, Brazil, and anhydrous ethanol 99.9° INPM. Some physical–chemical properties of the commercial fuels are given in Tab. 1 and Tab. 2, respectively.

Table 1. Physico-chemical properties of the Brazilian additivated S10 diesel B7 studied.

Property	Unity	Method	Limit	Value
Aspect	-	Visual	Limpid and exempt of impurities	Limpid and exempt of impurities
Colour	-	Visual	Colourless to yellow	Yellow
Colour ASTM	-	ASTM D1500	max 3.0	1.5
Water by Karl Fischer	wt%	ASTM D6304	max 0.02	0.01
Total sulfur	mg/kg	ASTM D5453	max 10.0	9.6
Fatty acid methyl esters	vol%	EN 14078	7.0 ± 0.5	7.2
Flash point	°C	ASTM D93	min 38.0	48.7
Cold Filter Plugging Point	°C	ASTM D6371	max 5.0	-4.0

Table 2. Physico-chemical properties of the aqueous ethanol studied.

Property	Unity	Method	Limit	Value
Colour	-	Visual	Orange	Yellow
Aspect	-	ASTM D4176	Limpid and exempt of impurities	Limpid and exempt of impurities
Specific mass at 20°C	kg/m^3	ASTM D4052	810.1	809.2
Alcohol content	wt%	ASTM D4052	92.5 to 93.8	93.21
pH at 25°C	-	NBR 10891	6.0 to 8.0	7.2
Total acidity	mg/L	NBR 9866	max 30	8.2
Electric conductivity	µS/m	NBR 10547	max 389	372.0
Evaporation residue	mg/100mL	NBR 8644	max 5.0	3.1

Biodiesel and refined vegetal oil from two sources (named A and B in this study) and *n*-butanol, purity PA, have been used for additive synthesis. To evaluate the impact of temperature, samples were stored in a BOD incubator (model 101M/3, ELETROlab) which allows a control of temperature in the range of -6 to 60°C with a precision of 0.3°C.

2.2 Synthesis of the additives through a Doehlert Experimental Design

A two variables Doehlert matrix, a second order design with interaction of order two, was set up to study the stability of E-diesel blends and gives a model as in Eq. (1):

$$Y = b_0 + b_1X_1 + b_2X_2 + b_{11}X_1^2 + b_{22}X_2^2 + b_{12}X_1X_2 \quad (1)$$

where Y is the experimental response, X_1 and X_2 represent the variables to be optimized, b_0 is an independent term, b_1 and b_2 are coefficients of the linear terms, b_{11} and b_{22} are the coefficients of the quadratic terms and b_{12} is the coefficient of the interaction term (or rectangular term).

With anhydrous ethanol, higher biodiesel content reduced the range of ethanol in which phase separation is observed. Consequently, the two factors were the concentrations of biodiesel and vegetal oil: the concentration of biodiesel was varied from 0-95 vol% whereas the concentration of vegetal oil was maintained in the range of 0-10 vol%. 3 levels were assigned to vegetal oil content (0, 5 and 10 vol%) and 5 levels were given to biodiesel concentration (5, 27.5, 50, 72.5 and 95 vol%). One sample was repeated in order to evaluate the repeatability of the experiments (samples 8 and 9). Tab. 3 sums up all the experimental conditions investigated for each couple of biomass for biodiesel and vegetal oil (A-A, A-B, B-A and B-B).

Table 3. Doehlert matrix in term of reduced and real coordinates for each biomass source.

Sample reference	x_1	x_2	X_1 (Biodiesel content, vol%)	X_2 (Vegetal oil content, vol%)	*n*-butanol content, % vol
1	0	0	50.0	5.0	45.0
2	1	0	95.0	5.0	0.0
3	0.5	0.866	72.5	10.0	17.5
4	-0.5	0.866	27.5	10.0	62.5
5	-1	0	5.0	5.0	90.0
6	-0.5	-0.866	27.5	0.0	72.5
7	0.5	-0.866	72.5	0.0	27.5
8	0	0	50.0	5.0	45.0
9	0	0	50.0	5.0	45.0

2.3 Study of the additives in aqueous ethanol – diesel blends at ambient temperature (T=20±2°C)

All additives were tested at the concentration of 2 vol% in pure diesel and aqueous ethanol to confirm the miscibility of the additive in these pure fuels. 200 mL of diesel-ethanol-additive blends (proportion 50:48:2) were also investigated. For all unstable samples, a non-dimensional parameter, which is defined as the separation ratio (SR), were calculated, after 1, 2 and 3 hours of decantation, as the ratio between the final volume of separated ethanol (V_{sep}) with respect to the initial ethanol content in the blend (V_e), as wrote in Eq (2). A sample with the same proportion of diesel and ethanol without additive was used as a reference to evaluate the stability effect.

$$SR = V_{sep} / V_e \quad (2)$$

Moreover, the maximum addition of aqueous ethanol in diesel initially additivated with 4 vol% of co-solvent was determined. For this purpose, aqueous ethanol was added by step of 2 mL in 100 mL of diesel until a phase separation

could be observed after 15 min of decantation. After the observation of phase separation, an additional addition was done to confirm the previous observation. At the equilibrium (after one day of decantation), the separation ratio SR were calculated. An E-diesel sample without additive was used for comparison.

In 200 mL of diesel-ethanol blends (proportion 51:49), additive concentration was tested in the range of 2, 3, 4 and 5 vol%. The volume ratio between the inferior diesel phase and the superior ethanol phase were measured after 1 and 2 hours of decantation. A sample with the same proportion of diesel and ethanol without additive was used as a reference to evaluate the stability effect.

2.4 Impact of the best additives in anhydrous ethanol - diesel blends at 10, 20 and 30°C

Based on the precedent results, mathematical models allowed the determination of the best additive composition. Best additives for all combinations biodiesel – vegetal oil were tested at the concentration of 2 vol% in 200 mL E-diesel blends where concentration of anhydrous ethanol varies from 0 to 100 vol% by step of 10 vol%. Three temperatures were investigated to represent different climatic conditions: 10, 20 and 30°C. Again, separation ratios SR were measured after 1, 2 and 3 days of decantation.

3. RESULTS AND DISCUSSSION

3.1 Stability study of diesel-ethanol-additive blends (proportion 50:48:2) at ambient temperature

All tested additives were miscible at ambient temperature (20 ± 2°C) in pure diesel and pure aqueous ethanol at the concentration of 2 vol%. In term of stabilization, only the additive 7 with biodiesel and vegetal oil from source A delayed the separation from less than 2 hours. Nevertheless, it can be observed in this sample the formation of three phases: a limpid diesel phase (bottom), a cloudy intermediary phase (it can be supposed that there are crystals of biodiesel in suspension) and a limpid ethanol phase (superior). Other additives did not present such a behaviour and phase separation are observed after a few minutes. Samples 1, 8 and 9 had similar behaviour, showing a good repeatability of the experiments.

According to Tab. 4, all additivated and reference samples presented a separation ratio SR higher than 1. This indicated that, during the migration process of ethanol toward the superior phase, where diesel, biodiesel or additive compounds were dragged with the ethanol. The separation ratios of additivated samples were higher than the reference samples except for samples 1 to 4 and 6 to 9 with biodiesel and vegetal oil from source B. This indicates that materials from source A have higher affinity than materials from source B. This behaviour could be related to the chemical structure of the biodiesel: both biodiesel main compounds have the same number of carbon, but the biodiesel from source B has an additional double bounds while biodiesel from source A has an alcohol function, allowing hydrogen interaction with ethanol. Samples 5 were mainly composed of *n*-butanol which has higher affinity with alcohols and water through hydrogen bounds than with diesel.

Table 4. Separation ratio (SR) for each biodiesel – vegetal oil sources after 3 hours of decantation.

Biodiesel – Vegetal oil Sources	Samples	1	2	3	4	5	6	7	8	9	Reference
	A-A	1.167	1.188	1.167	1.181	1.188	1.188	1.198	1.181	1.194	1.120
	A-B	1.188	1.188	1.167	1.167	1.208	1.194	1.167	1.188	1.188	1.113
	B-A	1.167	1.160	1.146	1.188	1.201	1.188	1.167	1.167	1.146	1.127
	B-B	1.109	1.042	1.063	1.104	1.125	1.111	1.069	1.104	1.104	1.120

3.2 Influence of additive concentration (2, 3, 4 and 5 vol%) in diesel - ethanol (49:51) blends at ambient temperature

The increase of additive content in the studied range did not impact the miscibility of ethanol in the E-diesel blends as phase separation was observed. An apparent equilibrium was observed after 1 hour of decantation. Moreover, the additive generally went into the ethanol phase, as the volumetric ratios between diesel and ethanol phases decreased with the addition of additive (Fig. 1). The additives with high content biodiesel from source B have the opposite behaviour. Again, this indicates that materials from source A have higher affinity than materials from source B. For additives with biodiesel from source A, all volumetric ratios between diesel and ethanol phases were lower than for the reference sample confirming the affinity of these materials with ethanol. Samples 1, 8 and 9 presented the same tendency even if the values were different, due to the experimental error.

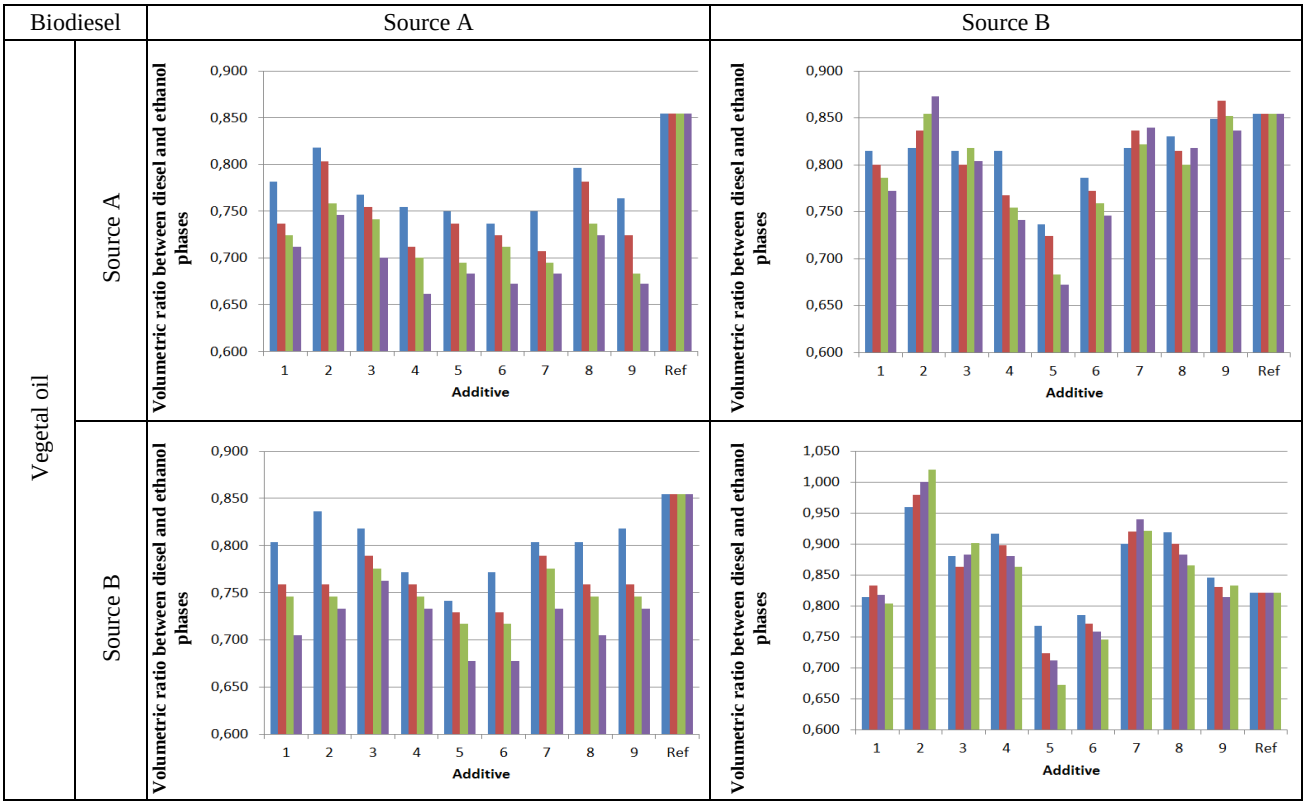

Figure 1. Volumetric ratio between the ethanol phase and diesel phase after decantation for different additive's content: ■ 2 vol% ■ 3 vol% ■ 4 vol% ■ 5 vol%

3.3 Maximum addition of aqueous ethanol in diesel with 4 vol% of additive until phase separation at ambient temperature

Fig. 2 represents the iso-lines of maximum addition of aqueous ethanol given by the mathematical models. In this figure, it could be observed that a maximum of approximately 20 mL can be added to the diesel until separation after 15 minutes of decantation with additive with high concentration of biodiesel and vegetal oil from source A. For all combination A-A, A-B, B-A and B-B, it could be seen that the additives with high *n*-butanol content did not contribute to stabilization. Furthermore, they had an opposite effect as samples with these additives had lower miscibility than E-diesel blends without additives: *n*-butanol acted as an extraction solvent, enhancing phase separation. In a general point of view, additives with high biodiesel and high vegetal oil content presented higher stabilizing ability. Biodiesel and vegetal oil presents a good miscibility with ethanol because of its polar character and their long lipophilic carbon chain contributes to the stability with diesel fuel. For additive with biodiesel and vegetal oil from source B, the vegetal oil content had lower effect than in the other additives.

At the equilibrium, the separation ratios of all samples are calculated (Tab. 5). It can be observed a good repeatability of the experiments. Only samples additivated with biodiesel from source B and vegetal oil from source A had SR higher than the reference samples. This indicates that these additives dragged more diesel, biodiesel or additive compounds than the others. Considering this aspect, additives from source B allowed an higher miscibility of ethanol in diesel.

Table 5. Separation ratio (SR) for each biodiesel – vegetal oil sources.

Biodiesel – Vegetal oil Sources	Samples	1	2	3	4	5	6	7	8	9	Reference
	A-A	1.000	1.000	0.929	0.909	1.000	1.000	0.923	0.917	0.917	1.120
	A-B	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.113
	B-A	1.400	1.250	1.231	1.500	1.500	1.500	1.400	1.400	1.400	1.127
	B-B	0.800	0.750	0.750	0.778	0.750	0.750	0.833	0.800	0.800	1.120

Based on these results, the determined mathematical models allowed the determination of the optimum composition of the best additives for each combination biodiesel – vegetal (Tab. 6). It could be observed that biodiesel was the main contributor to the additive, as ethanol presents a good miscibility with biodiesel because of its polar character.

Moreover, vegetal oil content was higher in the additive formulation when biodiesel and vegetal oil came from different sources, showing a synergy between them offered by the variety of carbon chain length.

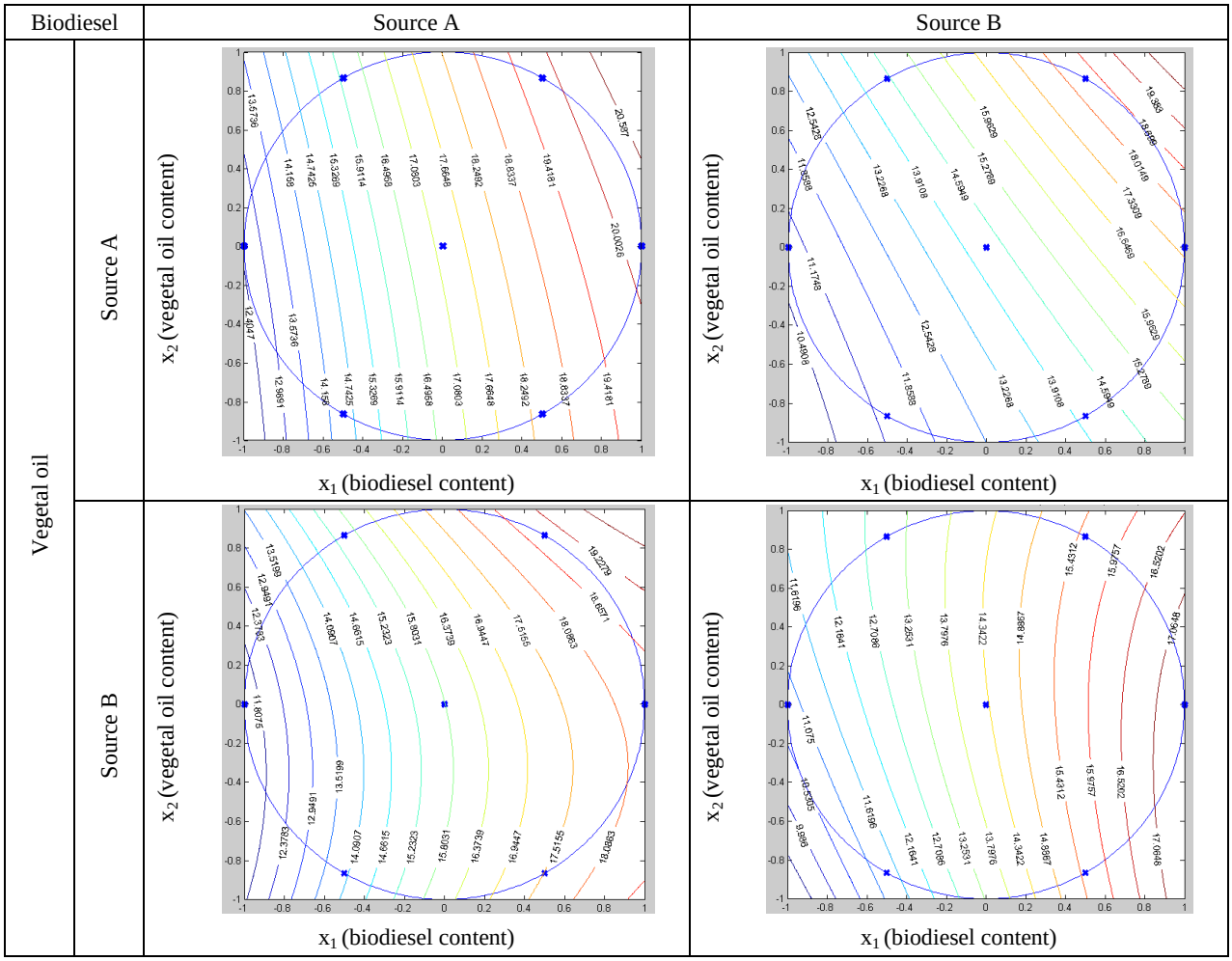

Figure 2. Maximum volume (mL) of ethanol in 100 mL of diesel with 4 vol% until phase separation after 15 min of decantation.

Table 6. Best additive's composition for each biodiesel – vegetal oil sources.

Biodiesel – Vegetal oil sources	Biodiesel content (vol%)	Vegetal oil content (vol%)	*n*-butanol content (vol%)
A-A	91.6	7.2	1.2
A-B	78.9	9.4	11.7
B-A	83.3	8.9	7.8
B-B	95.0	0.0	5.0

3.4 Influence of temperature for anhydrous ethanol – diesel blends with 2 vol% of additive

Fig. 3 shows that, without additive, when temperature decreased, the range of stability decreased and the separation ratios were quasi constant for two phases blends and tending to 1.0, as low temperatures were not favourable to drag organic compounds (diesel, biodiesel or additive) into ethanol phase. Moreover, when fuels were not miscible, it could be observed, at 10°C, the formation of a multiphasic solution (from 2 to 5 phases can be observed) and white crystals could be observed in E90 - Diesel blends.

At 30°C, all additivated samples were homogeneous while in absence of additive, diesel-ethanol blends were forming 2 phases with ethanol content from 30 to 60 vol%. At 20°C, samples with anhydrous ethanol content between 30 and 60 vol% presented phase separation while at 10°C, only blends with ethanol concentration in the ranges of 0 to 10 and 80 to 100 vol% were stable (Fig. 4). Nevertheless, at such low temperature and for high ethanol content (80 and 90 vol%), white deposits were observed at the bottom of the vessel, probably crystals of biodiesel, in additivated samples. Moreover, the presence of additive led to a better stability of ethanol-diesel blends as no separation were

observed at 30°C and, at 20 and 10°C, the range of instability was narrower and the separation ratio was higher with additive: even if blends were immiscible, the ethanol phase contained organic compounds. Based on this observation, additive with biodiesel from source A and vegetal oil from source B was the best additive, showing a synergy between the variety of carbon chain length and moieties.

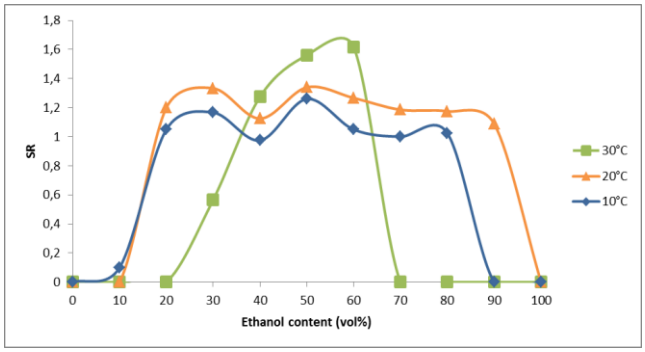

Figure 3. Separation ratio (SR) after 3 days of decantation in function of temperature of ethanol - diesel without additive at 10, 20 and 30°C.

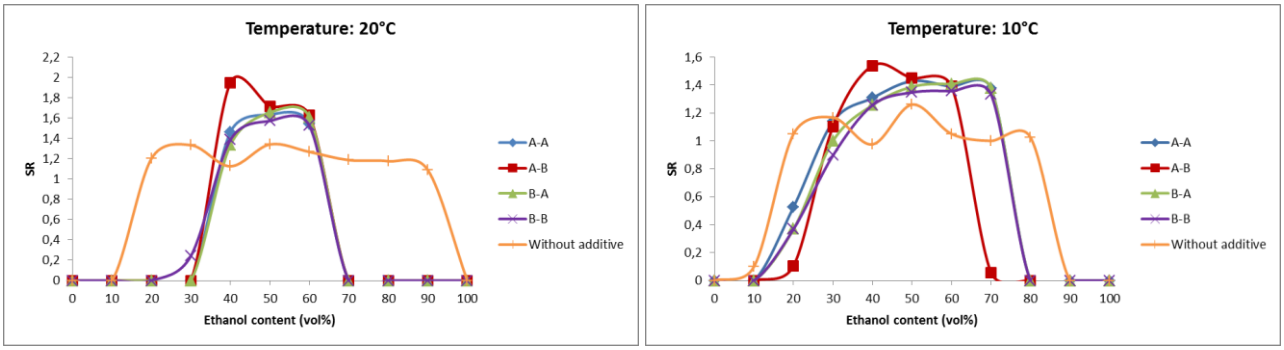

Figure 4. Separation ratio (SR) after 3 days of decantation in function of ethanol content for 20°C (left) and 10°C (right) in ethanol - diesel.

4. CONCLUSION

The additives synthesized from the experimental design showed that, for all combination of biodiesel – vegetal oil, the additives with high *n*-butanol content did not contribute to stabilization. It acted as an extraction solvent, due to the higher affinity of *n*-butanol with alcohols and water through hydrogen bounds than with diesel. It can be observed that biodiesel is the main contributor to the best additives for each combination biodiesel – vegetal oil. In a general point of view, additives with high biodiesel and high vegetal oil content presented higher stabilizing ability: ethanol presents a good miscibility with biodiesel and vegetal because of its polar character and their long lipophilic carbon chain contributes to the stability with diesel fuel. Moreover, vegetal oil content was higher in the additive formulation when biodiesel and vegetal oil came from different sources, showing a synergy between them offered by the variety of carbon chain length.

The increase of additive content in the studied range did not impact the miscibility of ethanol in the E-diesel blends as phase separation was observed. Moreover, the additive was generally going into the ethanol phase, but the additives with high content biodiesel from source B had the opposite behaviour. This indicates that materials from source A had higher affinity than materials from source B. This behaviour could be related to the chemical structure of the compounds: with the same number of carbon, materials from source B has an additional double bound while materials from source A has an alcohol function, allowing hydrogen bounds with ethanol.

Considering the ability of the best additives for each combination of biodiesel and vegetal oil to improve miscibility of blends with anhydrous ethanol, the presence of additive led to a better stability of diesel- anhydrous ethanol blends. No separation was observed at 30°C and, at 20 and 10°C, the range of instability was narrower and the separation ratio was higher with additive: even if blends were immiscible, the ethanol phase contained organic compounds. Among the tested additives, additive with biodiesel from source A and vegetal oil from source B presented the best stabilizing properties, showing a synergy between the variety of carbon chain length and moieties.

Based on the presented results, additives with higher refined vegetal oil fraction can be tested to increase the bridging power of the co-solvent and ethanol with low moisture content has to be used to guarantee the stability.

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