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FULL PAPER

# Ascorbic Acid as Antioxidant for Soybean Biodiesel

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## Abstract:

The intensive use of fossil fuels has had consequences coming from the instability of the reserves, due to the finitude of the resource, as well as the environmental impacts that the use of these fuels entails, such as the increasement of greenhouse gases. In this way, a promising alternative found to attenuate such problems is the production of biofuels, such as biodiesel, in which are used raw materials from renewable sources. They contribute to decrease dependence on oil and reduce the greenhouse effect. However, after biodiesel is produced, it is subject to degradation reaction when in contact with air, light, and temperature, which promotes the reduction of its quality. To slow down the degradation reaction, antioxidants are added to the biodiesel, providing greater oxidation stability. Antioxidants are substances that retard oxidation and can be natural, such as ascorbic acids or synthetic substances. Thus, this work aimed to evaluate the efficiency of ascorbic acid as an antioxidant in soybean biodiesel. Oxidative stability tests were performed and it was observed that the best concentration of ascorbic acid in soybean biodiesel, as antioxidant, is 27.5 g $L^{-1}$.

**Keywords:** antioxidant; bioenergy; biofuel; oxidation

## 1. Introduction

The intensive use of fossil fuels in transport has had consequences that come from the instability of the reserves, due to the finitude of the resources, as well as the environmental impacts that the use of these fuels entails, such as the increasement of greenhouse gases [1, 2]. In this way, a promising alternative found to attenuate such problems is the production of biofuels, such as biodiesel [3, 4].

Biodiesel can be produced from vegetable oils and animal fat and it can be used pure or mixed with mineral diesel [2, 5-6]. An advantage that the use of this biofuel provides, is the production made from raw materials from renewable sources, thus reducing dependence on oil and reducing the greenhouse effect. Biodiesel from vegetable sources (soybean, castor oil, palm oil, jatropha and other oil plants) has advantages because it presents a partially closed cycle of carbon, so when the biodiesel is consumed, the combustion process releases carbon dioxide, which is used by plants in photosynthesis, even though the amount absorbed by plants is not equal to that released in the combustion [4, 7-8]. In addition to the aforementioned characteristics, biodiesel has been prominent in Brazil due to the great diversity of raw materials, climatic factors, subsidy policies and territorial expansion [9].

Regardless of the raw material, biodiesel undergoes degradation when in contact with air, light, temperature, among other factors [10]. Thus, oxidation stability is an important characteristic to

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be evaluated in the quality control of this biofuel [11].

This oxidation reaction may be due to the storage condition of the biodiesel, thus influencing the potentiality of the fuel, influencing the homogeneity in the burning that is related to the ignition delay for the compression engines [2,8,12]. As a consequence, the quality of the biofuel is reduced. In order to minimize the oxidation reaction, antioxidants are added to biodiesel [4, 13].

Antioxidants are substances that slow down the initial stage of self-oxidation that form free radicals. These may be natural as ascorbic acid, α-tocopherol, phenolic compounds among others or may be synthetic as butyl hydroxytoluene (BHT), butyl hydroxyanisole (BHA), propyl gallate (PG) and tert-butyl hydroquinone (TBHQ) [2,14-15]. Ascorbic acid ($C_6H_8O_6$), known as vitamin C, has the characteristic of being a great natural antioxidant, being found mainly in citrus fruits. The use of natural antioxidants has been widely studied to promote the increase of biodiesel stability, besides being derived from the oil and obtained from biomass [4, 8, 15].

Ascorbic acid (AAS) is considered a natural antioxidant, which is obtained from different plants, through extracts and does not present toxicity to humans [15]. According to Pereira [16] ascorbic acid acts as an antioxidant, available for an energetically favorable oxidation, that is, with air easily oxidizes ascorbic acid, oxidizing before the final product, preserving and reducing oxidation [2].

The added amount of antioxidants varies according to their origin and the origin of biodiesel. Each raw material used in biodiesel production provides higher or lower concentrations of unsaturated fatty acids in the oil. Thus, the higher the concentration of these fatty acids, the greater the oxidation tendency, requiring a higher antioxidant addition to achieve oxidation stability [4,11].

When an antioxidant is added to biodiesel, it has the function of reacting with the radicals formed in the initiation and propagation steps to minimize or extinguish a number of free radicals. In the period of initiation occurs a low consumption of oxygen and of peroxide formation and in the period of propagation these amounts of peroxide and the products in decomposition increase. Thus, the product of the antioxidant reaction is the formation of a neutral and a radical product (with the structure of the antioxidant), but both do not have the ability to propagate the radical reaction, since the radical formed is stabilized by the structure of the antioxidant, which is usually phenolic [6,14,17,18].

In order to determine the efficiency of the antioxidants, tests of accelerated oxidation, such as the oxidation stability regulated by the ANP (national petroleum agency), are carried out through resolution Nº. 45, 25.8.2014 (EN 14112), period of induction of the biodiesel sample, which evaluates the biodiesel quality in relation to the oxidation of the biofuel [11]. According to the ANP standard, the induction period should be at least 8 hours [18]. Therefore, this work has as main objective to evaluate the efficiency of the ascorbic acid solutions as antioxidant for the soybean biodiesel added in the washing step

## 2. Material and Methods

### 2.1 Soybean biodiesel production

For the production of biodiesel, the transesterification of soybean oil with methanol and alkaline catalysis in the ratio of 100:30: 2 (v/v/m) was used. In the first step the oil was heated to 80 ºC, simultaneously in another vessel the catalyst (KOH) was dissolved in methanol, and then this solution was warmed to 30 °C. When the stipulated temperatures are reached, the contents of the methanol + KOH solution are transferred into the vessel with soybean oil at 80 °C. Subsequently, the temperature of the reaction mixture was maintained at 80 °C for 1 hour under agitation to ensure high efficiency in the transesterification process. After this period the sample was added to a settling funnel, to separate the biodiesel from the glycerol, for a period of 24 h. Figure 1 shows the flowchart for biodiesel production and wash.

### 2.2 Biodiesel washing

After biodiesel is produced it is not exempt of catalytic and glycerol residues, which influence some physicochemical properties, for this reason, the biodiesel must be washed, so that by dragging the waste is transferred from the oil phase to the aqueous phase (Figure 1).

Two different ways were used, conventional and with the presence of ascorbic acid solution. In the first step, about 30% by volume of distilled water was added to the biodiesel, which after stirring was conditioned for 24 hours in a separating funnel to separate the aqueous phase (water + residues) from the biodiesel.

As far as the second type of washing is concerned, the steps performed were identical to those described, the only difference being that the water used for washing was replaced by a solution of ascorbic acid of 10, 15 and 20 g/L.

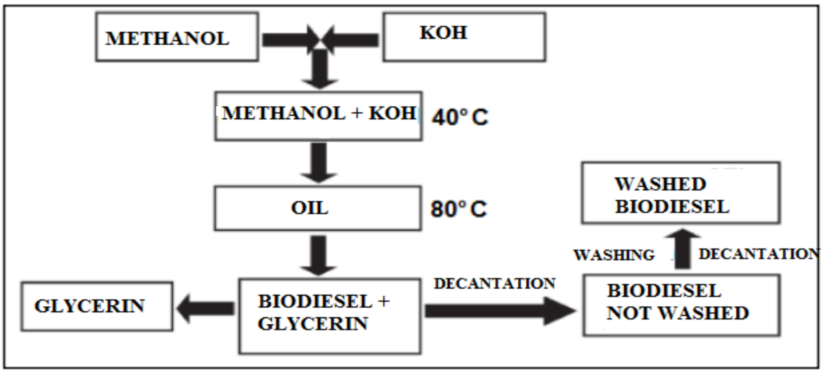


**Figure 1**. Flowchart for the production and washing of soybean biodiesel [6].

### 2.3 Evaluation of ascorbic acid as antioxidant for Biodiesel

#### 2.3.1 Biodiesel + antioxidant samples

To evaluate the effect of AAS as an antioxidant, the biodiesel samples were divided into three lots. In the first batch samples were washed in a conventional manner, in the second batch samples were washed with ascorbic acid solutions and in the third batch, samples were washed in a conventional manner with the subsequent addition of ascorbic acid powder at the concentration of 15, 20 and 25 g/L.

#### 2.3.2 Oxidation stability tests

In order to measure the effect of AAS as an antioxidant for biodiesel, the conductibility technique was used with the Rancimat 873 equipment of the Metrohm®, following the EN14112 standard, according to ANP Resolution Nº. 45, DE 25.8.2014 [18].

#### 2.3.3 Statistical analyzes

The analysis of variance and the Scott-Knott's test were used to compare the efficiency of AAS as an antioxidant for biodiesel, to compare means at 5% probability. These tests were performed using R software and the ExpDes package.

### 2.4 Biodiesel Quality Control

After all the oxidation stability tests and statistical analysis, physical-chemical tests were performed to ascertain the quality of the biodiesel samples with the best results of the induction time (IT). The tests for quality control were: color and appearance, hydrogenation potential (pH), conductivity, specific mass and flash point.

#### 2.4.1 Flash Point

The flash point analyzes were performed using the PENSKY-MARTENS Flash Point equipment, coupled with a thermometer with a scale of 0 to 200 ºC. The standard used was an ASTM D92 [18].

#### 2.4.2 Specific mass

The specific mass tests at 20 ºC were performed using the Incoterm brand densimeter, with a graduated rod ranging from 0.800 to 0.900 g/cm$^3$. The standard used was ASTMD1298 and NBR 7148 [18].

#### 2.4.3 Visual color and appearance

The visual color and appearance of the samples were determined in accordance with ANP Ordinance No. 310/2001 [18]. The appearance of the samples was classified according to the following classifications:

- Clean and free of impurities;
- Clear and with impurities;
- Turbid and free from impurities;
- Turbidity and impurities.

#### 2.4.4 Hydrogen ionic potential (pH)

The pH was measured using a digital Phmeter with bench meter and calibration check, of the brand HANNA model HI 2221 [18].

#### 2.4.5 Electrical Conductivity

The electrical conductivity was obtained using a Digimed device, model DM-3P-PE2, following the standard ASTM D2624 [18].

### 2.5 Oxidation Kinetics

The study of oxidation kinetics has the purpose of evaluating the mechanism and influence of the antioxidant on the oxidation reaction of biodiesel. In order to perform this study, the data of the oxidation stability test, at temperatures of 90 °C, 100 °C, 110 °C and 120 °C, were used for the samples that presented the highest IT (induction time) in the presence of AAS comparing them with the control (biodiesel without addition of AAS).

## 3. Results and Discussion

### 3.1 Analysis of AAS efficiency as antioxidant

The results for the first part of the experiments are shown in Table 1.

**Table 1**. Induction time for soybean biodiesel washed with AAS in solution (10, 15 and 20 g/L), AAS powder (15, 20 and 25 g/L) and the control.

| **Types of biodiesel washing** | Mean of induction time (h) | Standard deviation |
|---|---|---|
| Ascorbic Acid in solution (10 g/L) | 3.2$^a$ | 0.3252 |
| Ascorbic Acid in solution (15 g/L) | 3.8$^a$ | 0.3040 |
| Ascorbic Acid in solution (20 g/L) | 4.8$^a$ | 0.1484 |
| Dissolved powder ascorbic acid (15 g/L) | 4.2$^b$ | 0.0141 |
| Dissolved powder ascorbic acid (20 g/L) | 4.8$^b$ | 0.1838 |
| Dissolved powder ascorbic acid (25 g/L) | 5.1$^b$ | 0.1484 |
| Control | 5.6$^b$ | 0.2333 |

Considering the results presented in Table 1, a statistical test was performed comparing the types of biodiesel washing (in which the types of washing with the letter "a" differed from the control and those with letter "b" did not differ from the control), suggesting that the washing of biodiesel in a conventional manner with subsequent addition of the antioxidant powder did not differ from the control. In this way these tests were excluded, remaining the study only with the washing with antioxidant solutions.

Among the samples washed with the antioxidant in solution the concentration that obtained the highest induction time was 20 g/L, suggesting that in concentrations higher than 20 g/L there is the possibility of obtaining higher IT than those already found. Thus, new oxidation stability tests were performed using samples with concentrations of 25, 30 and 35 g/L. The IT results of the new tests are presented in table 2.

**Table 2**. Induction time for the soybean biodiesel samples washed with AAS in solution (25, 30 and 35 g/L) and the control.

| **Types of biodiesel Washing** | Mean of induction time (h) | Standard deviation |
|---|---|---|
| Ascorbic Acid in solution (25 g/L) | 4.2$^a$ | 0.1202 |
| Ascorbic Acid in solution (30 g/L) | 4.2$^a$ | 0.0777 |
| Ascorbic Acid in solution (35 g/L) | 0.4$^a$ | 0.2333 |
| Control | 0.6$^b$ | 0.3535 |

According to Table 2, the highest results were obtained at concentrations of 25 and 30 g/L, suggesting that concentrations between 25 and 30 g/L may present higher values than those found in these research Table 2. In this way, other tests were carried out at the concentration of 27.5 g/L in order to verify if the induction time had a higher value than was obtained. The data for this test are shown in Table 3.

**Table 3**. Induction time results for soybean biodiesel washed with the ascorbic acid of 27.5 g/L and control.

| Types of Biodiesel Washing | Mean of induction time (h) | Standard deviation |
|---|---|---|
| Ascorbic Acid in solution (27.5 g/L) | 7.8 | 1.0465 |
| Control | 5.6 | 0.2333 |

According to table 3, the ascorbic acid concentration of 27.5 g / L presented a longer induction time and was statistically different from the control, suggesting that this would be the "best" concentration for AAS use as an antioxidant for the biodiesel.

Another relevant result was that when the AAS concentration is higher than 27.5 g/L, that is, 30 g/L, the induction time has decreased. According to the literature, the antioxidant effect of a compound only has an effect up to a certain concentration, since when the concentration is increased the TI also increases to a certain point, if the concentration is higher than this point, the opposite effect occurs, compromising the oxidation stability of biodiesel, this can occur due to an effect called mixed action, where the antioxidant used can act as an inhibitor to a certain concentration, after can have a catalytic action [4,19].

The results confirm the antioxidant action of AAS in biodiesel and according to the literature, the AAS molecules undergo oxidation before the biodiesel, like other antioxidants that absorb or free radicals from the initiation and propagation stages, minimizing the oxidation of biofuel [15].

### 3.2 Quality control of biodiesel

After obtaining an optimized concentration of AAS, physical-chemical tests were performed for the biodiesel sample, in order to investigate the influence of the addition of AAS on other biodiesel properties. The physical-chemical tests are presented in Table 4.

**Table 4**. Physico-chemical characterization of biodiesel washed with AAS 27.5g/L and control.

| Sample | Ascorbic Acid 27.5 g / L | Control | ANP Resolutions |
|---|---|---|---|
| Flash Point | 30 °C | 30 °C | 100 °C |
| Conductivity | 0 | 75.6 µS/m | 350 µS/m (máx.) |
| Especific mass | 876 kg/m³ | 875 kg/m³ | 850 – 900 kg/m³ |
| pH | 10.7 | 9.2 | 6.0 – 8.0 |
| Appearance | Clean and free of impurities | Clean and free of impurities | Clean and free of impurities |
| Visual color | Yellow | Yellow | Yellow |

According to Table 4, it is observed that the flash point test presented results with values lower than those recommended by the ANP, a phenomenon that can be associated with the biodiesel washing process, indicating that the agitation performed was not enough to remove methanol used in the production of biodiesel. For the same reason we can explain the fact that the pH results obtained for the samples did not conform to what is specified by the ANP, in other words, it is evident the presence of remnants of the catalyst used in the biodiesel production.

Regarding the results of the conductivity tests, the performed wash of the biodiesel proved to be efficient, since the values obtained were within the limits determined by the ANP. The specific mass results obtained for the samples conform to the range specified by the ANP. The physicochemical results, in general, suggest that the washing process can be more efficient, corroborating with the results of induction time, thus it would be possible to reach higher induction times with lower concentrations of AAS in the washing solutions.

### 3.3 Kinetic study

### 3.3.1 Order of oxidation reaction of biodiesel

From the knowledge of the optimized concentration of AAS in biodiesel, a kinetic study was carried out to verify the influence of AAS on the kinetics of the oxidation reaction of biodiesel. For this study, the integrated velocity laws were used, adapted for the present case. Since it is not possible to determine the biodiesel concentration directly, an indirect measure was used, which is the conductivity of the oxidation stability test.

In this way, it is known that the higher the oxidation of the biodiesel the greater the conductivity the laws of zero-order integrated velocities, one and two, are modified to those presented in equations 1,2 and 3.

$$\frac{1}{\Lambda} = \frac{1}{\Lambda_o} - kt$$

Equation 1: Zero order for integrated speed law modified.

$$\ln\left(\frac{1}{\Lambda}\right) = \ln\left(\frac{1}{\Lambda_o}\right) - kt$$

Equation 2: First order for modified integrated speed law.

$$\Lambda = \Lambda_o + kt$$

Equation 3: Second order for modified integrated speed law.

In equations 1,2 and 3 Λ is the concentration, and Λo is the initial concentration, k is the rate constant and t is the time. Table 5 shows the results of the kinetic study of the biodiesel with AAS and the control, obtained using the results of oxidation stability at 100 ºC.

**Table 5**. Order of the oxidation reaction for the samples of Biodiesel washed with AAS and the control.

| Sample of biodiesel | Order of reaction | Value of R | Linear coefficient | Angular coefficient |
|---|---|---|---|---|
| Ascorbic Acid 27.5 g/ L | 0 | 0.10469 | 1.40709 | $-3.1832\times10^{-5}$ |
| | 1ª | 0.80798 | -0.03847 | $-7.5652\times10^{-5}$ |
| | **2ª** | **0.97027** | **-1.13639** | **$3.94188\times10^{-4}$** |
| Control | 0 | 0.82433 | 14.36839 | -0.00274 |
| | **1ª** | **0.98068** | **4.66947** | **$-9.12942\times10^{-4}$** |
| | 2ª | 0.97027 | -1.13639 | $3.94188\times10^{-4}$ |

According to table 5, it can be observed that the order that best represents the oxidation reaction of the biodiesel washed with AAS is of the second order, whereas the control presented a kinetics that follows the first order.

According to the literature, the kinetics of oxidation of biodiesel usually follows the first order, with or without the addition of an antioxidant, however in the case of AAS it is possible to suggest that because this substance oxidizes before biodiesel it is determinant in kinetics of the reaction because without its presence there is only the dependence of biodiesel.

### 3.4 Activation Energy

Using the Arrhenius equation (Equation 4), the activation energy can be determined by constructing the graphs of lnk versus 1/T, with the values of the rate constants calculated for temperatures of 90 °C, 100 °C, 110 °C and 120 °C.

$$\ln k = \ln A - \frac{Ea}{R}\frac{1}{T}$$

Equation 4: Arrhenius equation.

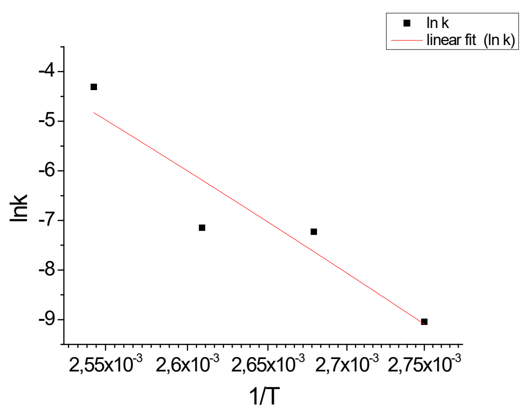


**Figure 2**. AAS activation energy.

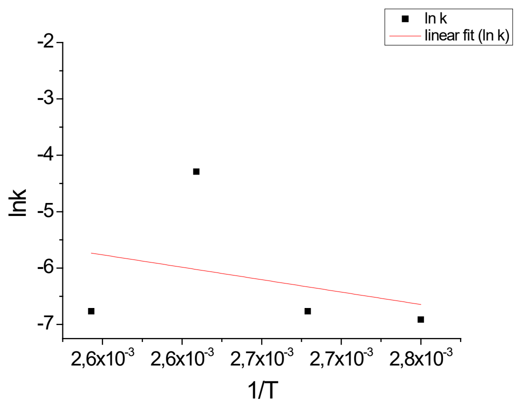


**Figure 3**. Activation energy of the control.

With the value of the coefficient that is equal to -Ea/R, the value of the activation energy is found by multiplying the slope value by the gas constant, see Table 6.

**Table 6**. Activation energy for samples of biodiesel washed with AAS and control.

| Sample of biodiesel | Activation energy ($kJ.mol^{-1}.s^{-1}$) |
|---|---|
| Biodiesel washed with ascorbic acid | 171.311 |
| Control | 19.452 |

As can be seen in Table 6, the activation energy of the control was lower, this indicates that with the addition of AAS the activation energy tends to increase, so the higher this energy the longer the biodiesel starts the oxidation process, proving the antioxidant effect of AAS

## 4. Conclusions

Ascorbic acid can be used as an antioxidant for biodiesel, the concentration at which the AAS has the highest efficiency is 27.5 g/L. The physical-chemical tests showed that the washing process was not enough to remove the catalyst and methanol residues. Kinetics of the oxidation reaction of the biodiesel washed with the ascorbic acid was of second order, this is because the AAS has the characteristic of oxidizing before biodiesel, and the control one was first order. The activation energy of the oxidation reaction washed with AAS and the control was 171.311 and 19.452 kJ/mol.s, respectively, thus it can be concluded that with the addition of AAS the biodiesel takes more time to start the process of oxidation.

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