

Sustainable Fuels for High School Students: Synthesis of Biodiesel from an Amazon Region Oil (Babassu)

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Abstract We describe here a four-stage project on the synthesis of a sustainable fuel (biodiesel, BD) from an Amazon region oil, namely, babassu oil (BO). The project was carried out both at Humboldt high school (project discussion and evaluation) and at the Institute of Chemistry, the University of São Paulo, ChemUSP (properties of BO; synthesis of BD). The students were asked to discuss aspects (origin, economic and environmental) of (petroleum-based) diesel oil and BD; suggest a candidate oil/fat from the Amazon region and an alcohol for the synthesis of BD, and discuss experimental variables that bear on biofuel yield. The students discussed this assignment in class, as well as what they are going to do during the third (experimental) stage of the project, carried out at ChemUSP. The students determined the acid- and saponification number of BO, synthesized BD using an optimized experimental protocol, prepared mixtures of (BO + authentic BD) and were introduced to gas chromatography. The students evaluated the project positively because of the socioeconomic- and environmental relevance of BD, its production from an Amazon region oil, and their contact with ChemUSP. We recommend this project for high school students because of its simplicity, safety, low cost, and because the school/university contact is beneficial for both sides.

Keywords Babassu oil, Biodiesel synthesis, Acid-base titration, Transesterification, Catalysis, Chemical equilibria

1. Introduction

Biodiesel, BD, denotes the fuel composed of mixtures of fatty acid esters (usually methyl or ethyl) obtained by the transesterification of oils and fats. We developed a four-stage project for twelfth-grade high school students on the synthesis of BD, with emphasis on connecting theory to practice. In the first stage of the project, conducted at Humboldt school, we introduced some aspects of BD, including its economic importance (in 2021, Brazil consumed 5.57 billion liters of biodiesel) [1], some relevant properties of oils and fats (acid- and saponification number), and aspects of the reactions used to produce BD (esterification; transesterification; catalysis) [2]. We divided the seventeen participating students into 4 groups, each of 4 or 5, and gave them the following assignment for the next class, based on literature that we provided [3–6]: (i) discuss aspects (origin, economic and environmental) of (petroleum-based) diesel oil and BD [7]; (ii) suggest candidate oil/fat from the Amazon region [8]; and an alcohol (methanol, ethanol, and 1-propanol) to synthesize BD; (iii) discuss some experimental variables that affect the

yield of BD [9]. Regarding this assignment, we explained that the transesterification reaction will be base-catalyzed; we asked the students to consider the availability/price of the starting materials.

In the following class (project stage 2 in the school), the students discussed differences in origin and combustion products of diesel oil and BD. Because of CO₂ fixation during plant growth, the contribution of BD combustion to the green-house effect is much smaller than that of diesel oil, in addition to being Sulphur-free, and emits less CO, CO₂, nitrogen oxides and particulate matter [10,11]. The students also showed how BD is synthesized, chose an Amazon region oil (babassu oil, BO), and discussed briefly what they are expected to do during their visit to the Institute of Chemistry, the University of São Paulo (ChemUSP).

In project stage 3, the four student groups visited ChemUSP, where they did the following experiments in the undergraduate laboratory, under our supervision: determined the acid- and saponification number of BO; prepared six mixtures of (BO + authentic BD) for viscosity measurement, and synthesized BO-based BD. The students visited the Central Analytical Laboratory (CAL) of ChemUSP where they were introduced to gas chromatography (CG), the technique most employed for the qualitative and quantitative analysis of BD [12]. Later, the instructor determined the viscosity of the above-mentioned (BO + authentic BD) samples, the four BD samples that the

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students prepared, and then sent the results to the students who calculated the BD yields of their experiments. The last stage of the project was carried out at the school, where the four groups presented their data and evaluated the project. We attribute the students' highly positive evaluation to: the socioeconomic relevance of BD and the lower environmental impact of its combustion, relative to diesel oil; participation of the students in choosing the starting materials; their visit to ChemUSP, and the connection between the experimental part of the project and the theory they study (esterification, acid-base titration, and chemical equilibrium). We recommend this project for high school students because of its safety, low cost, and the simplicity of the equipment required. The project offers an opportunity to link experiment to theory, and enhances school/university interaction.

2. Experimental

2.1. Material

Babassu oil (CAS 91078-92-1) was from Coppali, Lago do Junco, Maranhão, Brazil. Based on literature data [8], the molar mass of this BO was taken as $654.37 \text{ g mol}^{-1}$. Commercial "anhydrous" bioethanol (CAS 64-17-5; hereafter designated as "ethanol"; 99.8 wt%), standardized HCl- (CAS 7647-01-0) and standardized KOH solutions (CAS 1310-58-3), diethylene glycol (CAS 111-46-6), NaCl (CAS 7647-14-5) and anhydrous MgSO_4 (CAS 7487-88-9) were from Synth, São Paulo, all were used as received. An authentic sample of BD was synthesized by the instructor, by reacting BO with EtOH in the presence of H_2SO_4 catalyst as given elsewhere [13]. Based on CG analysis carried out by CAL, the purity of the authentic BD sample was 99.8%.

2.2. Equipment

The students used Corning 6798-420D PC-420D digital stirring hotplate; Sorvall Legend X1R centrifuge (Thermo Scientific). The instructor dried the mixtures prepared in section 2.3.3 (BO + authentic BD), and the students' BD samples (section 2.3.4) under reduced pressure using Fisher model 281A vacuum oven, and determined their viscosities using Brookfield model R/S-CPS cone-plate rheometer. The density was measured at 25°C using Anton Paar DMA-4500 digital densimeter. The purity of the authentic BD sample was determined at CAL using Shimadzu model CG-msQP2010 gas chromatograph, equipped with 30m capillary column (BPX5, 5% phenyl methylpolysiloxane).

2.3. Procedures

2.3.1. Determination of the Acid Number (AN) of Babassu Oil

Dissolve accurately weighed oil (ca. 10 g) in 75 mL of ethanol (EtOH) and add 0.5 mL of phenolphthalein indicator. Using a magnetic stirrer, titrate the solution using the

furnished standardized ethanolic KOH solution (0.05 mol L^{-1}). Additionally, perform a blank titration (using EtOH only). Calculate the value of AN of BO by the Eq. 1.

$$\text{AN} = \frac{56.1(Vb - Va)C}{m} \quad (1)$$

Where: Vb is the volume used for titrating the blank solution; Va is the volume used for titrating the BO solution; C is the molar concentration of the KOH solution; (m) is the mass of oil used in g [14]. (Caution: before using this base, read the corresponding safety data sheet (<http://www.labchem.com/tools/msds/msds/75365.pdf>).

2.3.2. Determination of the Saponification Number (SN) of Babassu Oil

Dissolve accurately weighted BO (ca. 1.5 to 2 g) in 25 mL of ethanolic KOH solution (0.5 mol L^{-1}). Reflux the mixture for 1h to ensure saponification, characterized by a visually clear solution. Wash the condenser with ca. 10 mL of hot EtOH, add the washing to the saponification solution. Add 0.5 ml of phenolphthalein indicator solution, stir the solution magnetically, titrate the excess KOH present in the solution using the provided, standardized HCl solution (0.5 mol L^{-1}). Additionally, perform a blank titration (using KOH solution only).

Calculate the SN by Eq. 2 below:

$$\text{SN} = \frac{56.1(Vb - Va)C}{m} \quad (2)$$

Where Vb is the volume required to titrate the blank sample, in mL; Va is the volume required to titrate the ethanolic BO solution, in mL; C is the concentration of HCl, (m) is the mass of oil used, in g [15].

2.3.3. Preparation of Mixtures of BO + Authentic BD for BD Yield Determination

Using 5 mL burettes, prepare 2 mL mixtures of (BO + authentic BD) containing 15, 30, 45, 60, 75, and 90 volume % of the provided authentic BD. For example, pipette into the provided tubes 0.3, 0.6, 0.9 mL, etc. of authentic BD and 1.7, 1.4, and 1.1 mL, etc. of BO. Mark the tubes and give them to the instructor. Calculate the mass % of authentic BD in these mixtures from the densities at 25°C of both components, 0.9176 and 0.8857 g mL^{-1} , for OB and BD, respectively.

Caution: BO is a viscous liquid that flows slowly. Consequently, it takes time for the meniscus to stabilize in the burette.

2.3.4. Synthesis of Babassu Oil-Based Biodiesel

Place the stirring hotplate on a laboratory jack; place the provided diethylene glycol (DEG) heating bath on the hotplate; introduce a thermometer (range $0\text{-}230^\circ\text{C}$) into the DEG, and start stirring/heating the liquid to $100 \pm 5^\circ\text{C}$ (set the heating power dial of the hotplate at 4). Parallely, introduce a magnetic stirring bar into 125 mL 3-necked

round bottom flask; use 25 mL and 10 mL burettes to introduce into the flask the volumes shown in **Table 1** of BO and ethanol, respectively; close all necks with stoppers. Assemble the transesterification reactor above (*not in contact with*) the DEG bath, by inserting a reflux condenser provided with CaCl_2 drying tube into the central neck of the flask. Using a 10 mL burette, introduce the provided catalyst solution (ethanolic sodium ethoxide) into a 10 mL addition funnel with a pressure equalizing arm (caution: the stopcock of the funnel should be closed); insert the funnel into one neck of the reaction flask.

When the DEG temperature is reached, circulate water into the reflux condenser and use the laboratory jack to carefully raise the hot plate/DEG bath until the liquid in the 3-necked flask is below the DEG level. When ethanol starts refluxing gently, open the stopcock of the addition funnel to introduce the base catalyst solution at once, and start recording the reaction time, as listed in **Table 1**.

At the end of the specified time, turn off the heating, lower the hot plate/DEG bath, put the reaction flask into a cold-water bath, wait for the solution to cool and then disassemble the equipment by removing the condenser and the addition funnel. To the reaction mixture add 20 mL of cold NaCl solution (10%, w/v) and transfer the mixture to a 50 mL Falcon tube, close the latter and centrifuge the mixture for 10 minutes at 4000 rpm, at room temperature. Introduce the two-layer liquids carefully (avoid mixing of the layers) into a 125 mL separation funnel (caution: the stopcock of the funnel should be closed). Wait for phase separation and remove the aqueous (lower) layer *slowly*. To the organic layer, add 40 mL of the NaCl solution, agitate the mixture (caution: open the stopcock of the funnel occasionally to release pressure), wait for 5 minutes for

phase separation and remove the aqueous (lower) layer *slowly*. Repeat the washing two more times, each with 40 mL of cold, distilled water. After the fourth washing, use a pH test strip (pH range 0-14) to measure the pH of the aqueous phase. Repeat the washing if the pH is not equal to that of distilled water. Remove the remaining water from the organic (turbid) phase as follows: cover the surface of a Büchner funnel (48 mm diameter) with a filter paper (qualitative, fast speed filtration), cover the latter with a thin layer of anhydrous MgSO_4 (a drying agent) and filter the oil phase *slowly* through the MgSO_4 layer into a 50 mL Erlenmeyer flask. Take care to moisten the entire surface of the drying agent. Transfer the product (a clear oil) to a marked 10 mL Falcon tube, close the tube and give it to the instructor. These oils will be further dried by the instructor, by heating at 60°C for 6 hours, under reduced pressure, over P_4O_{10} .

2.3.5. Calculation of the Biodiesel Yield from Viscosity Measurement

This was carried out by the instructor and the students. The former determined the viscosities of the (BO + authentic BD) mixtures prepared by the students, as well as the viscosities of the BD samples furnished by the students after drying, using the Brookfield viscosimeter. Based on these data, the students calculated BD yields, *vide infra*.

2.4. Hazards

There are no significant hazards in this project that require precautions other than wearing personal protective equipment. The aqueous waste was discarded by the safety division of ChemUSP.

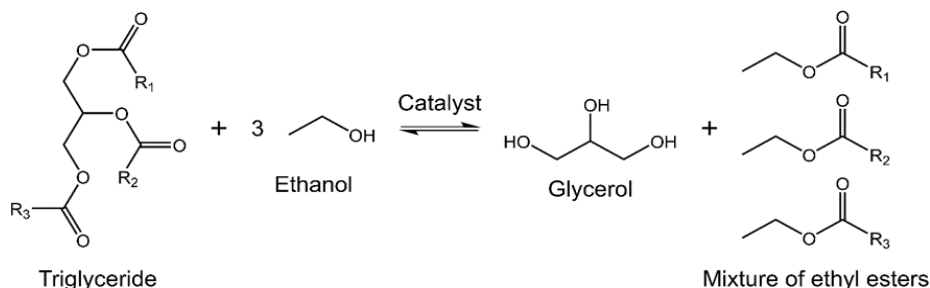
Table 1. Reaction conditions employed for the synthesis of biodiesel by student groups G1 to G4, and reaction yield

Group	Babassu oil volume, mL	Ethanol volume, mL	Molar ratio ethanol/BOa	Catalyst solution, mLb	Reaction time, min	Reaction BD yield, m%c
G1	12	5.9	6:1	2.4	30	88.8
G2	12	5.9	6:1	2.4	60	93.6
G3	12	11.8	12:1	3.2	30	90.4
G4	12	11.8	12:1	3.2	60	95.8

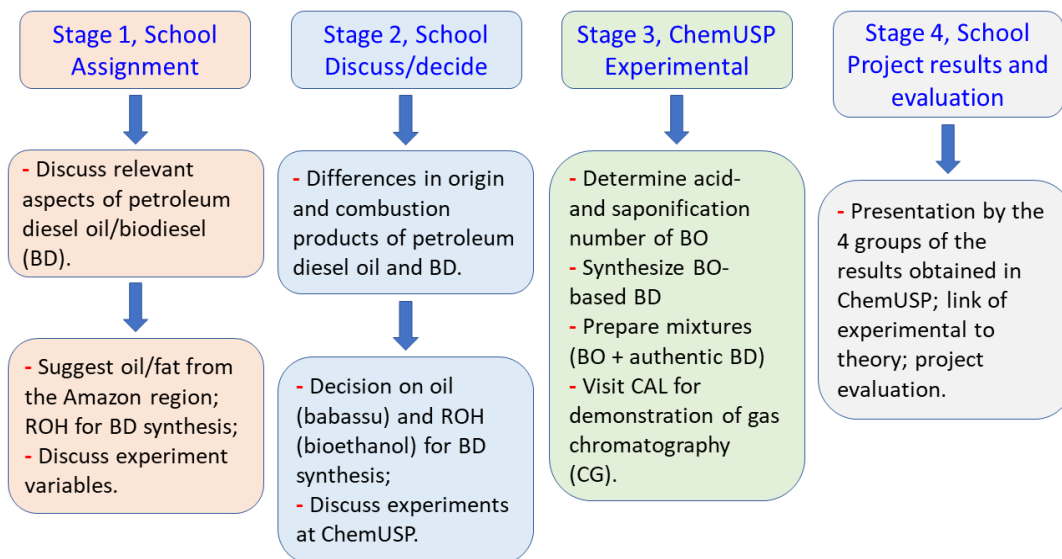
a- This molar ratio is based on a molar mass of $654.37 \text{ g mol}^{-1}$ for babassu oil [8].

b- The sodium ethoxide catalyst was prepared by the instructor at the beginning of the laboratory class by dissolving 1g sodium in 50 mL of commercial anhydrous ethanol; its concentration is 0.88 mol L^{-1} .

c- The BD yield in mass % was determined from viscosity measurement, as given in section 2.3.5.



Scheme 1. General scheme for transesterification of a vegetable oil (triglyceride) with an alcohol (usually methanol or ethanol) in the presence of acid- or base catalyst



Scheme 2. Activities carried out by the students at the school and ChemUSP during this project

3. Results and Discussion

Scheme 1 shows the transesterification reaction, under acid- or base catalyzed reaction, the students used the latter ($\text{C}_2\text{H}_5\text{ONa}/\text{C}_2\text{H}_5\text{OH}$); the activities carried out by the students are shown in **Scheme 2**.

As shown in **Scheme 2**, the activities during the four-hour visit to ChemUSP included determination of some properties of BO (acid- and saponification number), the synthesis of BD, preparation of (BO + authentic BD) mixtures, and demonstration of the CG technique at CAL. Although the students did not have enough time to finish the BD synthesis, they understood fully the experiments they are doing, as shown in stage 2 of **Scheme 2**.

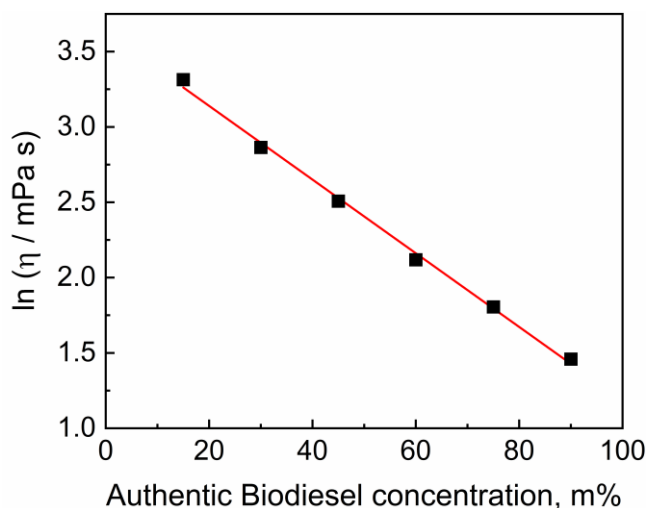


Figure 1. Dependence of the viscosity (η) of mixtures (BO + authentic BD) on the mass% of the latter component in the mixtures

The activities that the instructor carried out were before-, and after the students' visit to ChemUSP. The former included providing ethanolic solution of an already saponified BO, so that the students can titrate the excess base,

and calculate the SN of BO. After the visit to ChemUSP, the instructor dried all oil samples, determined the dynamic viscosity (hereafter called "viscosity"; η) of six (BO + authentic BD) samples. and the four (transesterification) products of the students' experiments. Figure 1 shows the dependence of η on mass% authentic BD, from which Eq. 3 is calculated by regression analysis. For simplicity, we used a linear fit; including the data of the two pure components requires a 2nd degree polynomial fit.

$$\ln(\eta / \text{mPa s}) = -0.02445 (\text{authentic BD, mass\%}) + 3.62877 \quad (R^2 = 0.997) \quad (3)$$

Considering the limited time available to do the activities shown in stage 3 of **Scheme 2**, we developed an efficient, simplified experimental protocol, where: (i) the sequence of the steps was given in details; (ii) the students measured the volumes of the reactants using precision burettes, instead of pipettes; (iii) to shift the transesterification reaction to products, the students used excess ethanol and a relatively high catalyst concentration (final $[\text{C}_2\text{H}_5\text{ONa}] = 0.104 \text{ mol L}^{-1}$); (iv) the first (slow) aqueous/oil phase separation was accelerated by centrifugation. As shown by **Table 1**, excellent yields were obtained in relatively short times, 30, 60 minutes.

The experimental part of this project highlights some aspects of the chemistry course of these students. For example, the AN is an important quality control property of an oil. Rancid oils develop an unpleasant odor and flavor; the formed free radicals can cause cellular damage and have been linked to diabetes [16]. According to the Brazilian legislation, the maximum allowed AN is between 0.6-4.0 mg KOH/g for most edible oils [17]. The average AN calculated by the students was 1 mg KOH/g. On the other hand, the SN of oils and fats is of prime importance for making soft- and hard soaps [18]. Soaps that contain excess alkali may cause dryness and irritation of the skin and induce lesions of atopic dermatitis (eczema) in susceptible individuals [19,20]. Thus,

careful determination of SN is an essential initial step in soap manufacturing. The average SN that the students calculated was 246 mg KOH/g BO; both AN and SN agree with the range found in literature [21].

The synthesis of BD served to discuss aspects of the esterification and transesterification reactions; how to shift a reaction equilibrium to product, e.g., by using one reactant (ethanol) in excess, and the concept of catalysis. They also appreciated the power of CG in qualitative analysis, e.g., by comparing the obtained chromatogram of authentic BD with one from a data base.

Assessment of the project was the subject of the fourth stage of the project. The students appreciated the discussions, both intergroup, with their teacher and with the team from ChemUSP, as shown by the following excerpt: "during this project, I felt being a part of everything! The clarity of the preparatory classes helped me to understand the experiment". The seminars presented by the four groups were essential to conclude the project, as shown by the excerpts: "sharing the results with the rest of the class shows our interesting work and concludes the project"; "today, sharing experiences is one of the most important things; this seminar can increase interest of our colleagues". Other comments showed that the result of the visit to ChemUSP goes beyond doing the experiments, which many students reported as interesting: "I enjoyed getting to know the laboratory at ChemUSP and carrying out the experiments"; "I really liked the experience and the contact with the university". When we mentioned active-learning, the students linked it to doing the experiment. We called their attention to the fact that they were actively involved in all stages of the project, e.g., by choosing the starting materials. The students' overall evaluation was positive, with 58.8, 17.6, 17.6 and 5.9% evaluating the project as very interesting, innovative, motivating, and less interesting, respectively.

4. Conclusions

It is important to introduce the subject of sustainable fuels in high school because of their socio-economic importance, lesser environmental impact, and because it will take years before diesel engines are partially, or totally phased-out [22]. Using oils and fats that are not intended for foods for making BD is critical because of the "food versus fuel" debate [23], which led to the increased use of waste cooking oils to make BD [24-26]. In the present project the students chose an Amazon region oil that is not used in foods because of its high saturated fatty-acid content, ca. 89.5% [8]. The students assembled a reactor, carried out a transesterification experiment, and separated the product in one four-hour period, thanks to our supervision/support and the experimental simplifications that we introduced. Additionally, the students were introduced to a "novel, sophisticated" analytical technique (CG) during their visit to CAL. We recommend this project to high-school students because of its simplicity, safety, low cost, the connection

between experimental and theory, and because it represents an everyday situation, namely, manipulating a renewable material to produce BD, a fuel with less environmental impact than diesel oil.

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