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RESEARCH ARTICLE

EXTRACTION AND CHARACTERIZATION OF JACKFRUIT SEED OIL FOR BIODIESEL PRODUCTION: A GREEN MANAGEMENT OF AGRO-INDUSTRIAL RESIDUES TO BIOFUEL

Licianne Pimentel Santa Rosa^{1*}, Roberta Flávia Romero², Amanda Keyla Oliveira Candido²,
Janaina Carvalho Melo Argôlo², João Victor Santos Pinto², Rick Brener Queiroz Alves²
and Itallo Oliveira Macedo Sampaio²

¹Federal University of Sergipe, Department of Chemical Engineering, Av. Marcelo Déda Chagas, s/n, Rosa Elze, São Cristóvão - SE, 49107-230; ²Jorge Amado University Center, Av. Luís Viana Filho, 6775, 41.745-130, Paralela, Salvador-BA

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ABSTRACT

In recent years, global energy systems have undergone a transition, with their energy matrices gradually shifting towards more renewable and cleaner sources. Among the various alternative energy sources, biofuels have become the subject of extensive academic research and scientific studies. The aim of this paper is to utilize jackfruit seeds, an agro-industrial residue, to obtain biofuel using the transesterification method. The oil was extracted from the seeds using the Soxhlet Extraction method, with hexane as the solvent. The jackfruit seeds exhibited an extractive content of 42.21 wt.%, which represents a significant portion of the biomass composition. Biodiesel production was achieved through transesterification with basic catalysis in ethanol, resulting in a conversion rate of 80.4%. The biofuel obtained was characterized by evaluating its physical-chemical parameters, including pH, electrical conductivity, acidity index, and density. The obtained results were satisfactory, and they met the specifications set by the ANP (Brazilian National Petroleum Agency) for biodiesel. Furthermore, they align with specifications found in the literature for other types of seeds. Therefore, it can be concluded that jackfruit seeds are a promising source for biofuel production. Additionally, the findings of this study provide motivation for further research in this field, particularly when synthesizing other types of seeds for biofuel extraction and comparing the results with those obtained from jackfruit seeds.

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INTRODUCTION

In the context of global climate changes caused by factors such as global warming and ecological unbalance, it leads to the necessity of searching for renewable sources of energy. It is essential to look after the planet earth, as habitable planet as well as essential and secure economic viability to foment balanced means of productions in the social/economic and sustainable development. There is a worldwide effort to substitute the sources of non-renewable energy (petroleum-based), polluting, expensive and limited for alternative sources, less pollutant, renewable and diverse. Therefore, one of the main sources of energy to replace the petroleum-based energy is biodiesel, which is obtained from vegetable or animal fat. Due to the diversity of biomass existent in Brazil, biodiesel becomes a very promising source to reduce the use of non-renewable energy. Among many other raw materials used to produce biodiesel, vegetable oils, rich in lipids, are the most used. The lipids are most commonly found in the pods, fruits and leaves. According to Esaú (1986), seeds are very rich in lipids. As they are important in obtaining biodiesel and are found in seeds, it is feasible to study seeds as raw material for biodiesel production.

According to Silva (2006) several crops are indicated for the extraction of biodiesel, such as: cotton, peanut, canola, coconut, palm oil, sunflower, castor bean, soybean, etc. Among these species, oil palm and coconut have the greatest productive potential in addition to the advantage of being perennial crops with continuous harvesting throughout the year. As noted, biodiesel has a variety when it comes to environmental advantages compared to diesel oil. According to Silva (2006) the energy balance varies according to the system used in the cultivation of oil-producing species. Therefore, rationality, research and innovation will be needed in the handling of agricultural practices from planting to the final product, so that there is no reversal of the purpose, that is, the use of clean and sustainable energy. Still, according to Silva (2006), the cultivation of these species will require the agricultural system to be adapted and consequently, it will favor crop rotation, in addition to activating the manipulation in areas already used. According to Knothe (2005), biodiesel is determined to be monalkyl ester of long-chain fatty acids derived from vegetable oils or animal fats and alcohol, obtained with or without the use of a catalyst. Fazal (2011) affirms that it contains less sulphur, a net carbon balance close to zero, lower emissions of pollutants such as

CO, particulate matter, unburned hydrocarbons, in addition to greater amounts of oxygen (which leads to complete combustion and provides a reduction in emissions). The first step to obtain biodiesel from seeds is to extract the oil. Usually, it is done by a solvent, hexane being the most used. According to Rotta (2015) this occurs because hexane meets the recommended requirements for extraction, due to some properties such as high oil solubilization, high extraction rate when in equilibrium, immiscibility in water and the fact that it is not oxidizable pipes and equipment. There are three types of processes to obtain biodiesel: transesterification, thermal cracking and esterification. A common factor among the mentioned methods is the formation of a chemical reaction of oil and alcohol caused by the catalyst. Transesterification is the most applied method, since it only occurs in a single step, where the processing of the organic compound takes place due to the action of a catalyst. In this procedure, an ester, a substance obtained from the chemical reaction between an alcohol and a carboxylic acid, is obtained. Thus, considering the Brazilian territorial extension, favourable climatic conditions and plant biodiversity, it can be said that the country has the potential to produce biodiesel on a large scale. Therefore, this paper aims to study the application of jackfruit seeds (Figure 1), to produce biodiesel, through the transesterification method of vegetable oils. Jackfruit (*Artocarpus Heterophyllus L.*) has a great ease of dissemination, which occurs due to its spontaneous proliferation in the hottest regions of the country. The fruit of the jackfruit consists of three parts, pulp, seeds and peel, with the pulp constituting, on average, 30% of the weight of the fruit and the seeds around 12%. According to John and Narasimham (1993) the number of fruits produced per jackfruit tree is an average 45 fruits, and the productivity is 475 kg/tree, after picking only the seeds, one fruit has up to 500 units.



Figure 1. Jackfruit seeds

Even though very predominant in Brazil, Jackfruit is little studied. Its consumption is basically restricted to production sites, mainly for food. Its normally consumable parts are the cooked or roasted seeds and the pulp. In addition, Fonseca (2010) stated that jackfruit peel can be used in animal feed. Currently, the jackfruit tree has low prospects for a sustainable economic use, thus making the need for investments in research in the most varied areas and applications. Due to the vast abundance in Brazilian territory, another characteristic of jackfruit that facilitates its exploitation on a commercial scale is the good prices on the market. Therefore, jackfruit seeds have great potential for biodiesel production, and as the authors of this article acknowledge, this seed has not been investigated for this purpose. In addition, investing in research and production of jackfruit on a commercial scale to produce biofuel is extremely promising for the poorest regions in the north and northeast of Brazil. It will directly and indirectly generate jobs, influence the development of family farming and cooperatives and improve the infrastructure and economy of these regions. So, this article aims to verify the viability of utilising jackfruit seeds to produce biodiesel through transesterification method, split into steps, such as: preparing the seeds, extracting oil, producing biodiesel and analysing its categorization. However, this article also follows presenting the methodology and material used throughout the whole process, pH analyses, electrical conductivity, acidity index (free fatty acids), volumetric mass and as a the findings will be presented and discussed, and the study is concluded.

METHODOLOGY AND EXPERIMENTAL PROCEDURES

The jackfruit seeds were collected from a tree in the metropolitan region of city of Salvador in the state of Bahia in Brazil. Figure 2 shows a simplified flowchart with the general steps for oil extraction and biofuel production, which will be shown later in this paper with further details. Firstly, the jackfruit seeds were ground to facilitate the oil extraction. Secondly it was used the Soxhlet bench of extraction to extract the oil from the jackfruit mince with hexane as the solvent. Afterwards, to extract the more volatile part the asset was submitted to a process of separation on a rotatory evaporator. After being concentrated, the oil obtained was used to produce biofuel by heating transesterification method. The whole heating process the biofuel went through the three steps of washing. In the end, the characterizations were carried out. The parameters for the characterization are: electric conductivity, chemical pH, acidity index and density.

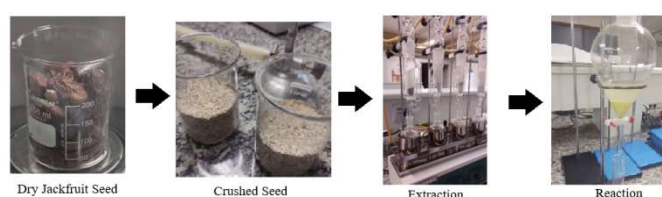


Figure 2. Biofuel Production Simplified Flowchart

Jackfruit Seeds Preparation: The seeds were extracted from the jackfruit by hand and thoroughly washed in running water to ensure that no residual fruit was left attached to them. At that point, these seeds went through a heat treatment 10 minutes in a microwave (Pmo26b, Philco) to optimize the oil extraction, as recommended by Azadmard-Damirchi (2011). Those seeds were then dried for 20h in a dehydrator (MOD-EL-1.1, Odontobrás), at 60°C. In order to maximise the interaction solute-solvent in the extraction the first layer of the seeds was removed to then grind the seeds in a processor (TA-2BIVMF60N5, Metvisa). After that, the grinded seeds were passed through a sift to reach maximum granulometry of 1mm, resulting in 236.9g of jackfruit seeds meal, which was stored for biofuel extraction.

Oil Extraction Process to Produce Biofuel: The jackfruit oil extraction from the meal was done through the method of continued extraction with solvent in a soxhlet (MA-487, Marconi) extractor as recommended by Darge (2019). First, a sample of 100 g of crushed jackfruit seeds was used which was placed in the flask with 250 mL of the solvent. The flask was attached to the Soxhlet extractor operated at 60°C for 2 hours. After extraction, the oil/solvent mixture was taken to the evaporator rotary to remove the solvent and the samples stored in a desiccator until mass constant, ensuring that only the oils were present. The remaining oil was quantified and stored at -10°C for the biodiesel production.

Biodiesel production by transesterification: In order to carry out the biodiesel production by the transesterification route the reagents were used: potassium methoxide (mixture of methanol with KOH potassium hydroxide), jackfruit oil, hydrochloric acid, distilled water, saturated NaCl solution. First, the potassium methoxide solution was prepared in order to achieve a better yield in the production of the ester. For that, 1.5 g of potassium hydroxide (basic catalyst) was dissolved in 35 mL of methanol with the aid of stirring and control of temperature at 45°C. Subsequently, 100 mL of jackfruit oil were added to a flat-bottomed flask, where it was heated with the aid of a magnetic stirrer (Fisatom, 752 A). It remained under stirring until reaching a temperature of 45 °C. Then, the recently prepared potassium methoxide solution was added to it, and the reaction mixture was stirred for 60 minutes at 45°C. The reaction mixture was transferred to a decantation funnel for phase separation: the upper

layer was biodiesel, and the lower layer was glycerin, formed by soap, excess alkali and alcohol. The total separation time was 15 minutes. The lower phase was collected in a 50 mL beaker. A 250 mL beaker was used to measure the volume of biodiesel, which was then returned to the separation funnel for the washing process. The washing was carried out, which had the purpose of purifying the biodiesel. This process was started with 50 mL of 0.5% (v/v) hydrochloric acid aqueous solution in order to neutralize the catalyst action. The wash step continues with 50 mL of saturated NaCl solution and finally 50 mL of distilled water in order to remove the remain impurities.

Oil and Biodiesel Parameters: The Brazilian legislation that regulates the sale of biodiesel in the country is ANP Resolution No. 42 of 24.11.2004. It establishes parameters regarding the quality and conformity of biodiesel produced in the national territory. Therefore, below the main physical and chemical properties of biodiesel are presented. They were used for the characterization of the biodiesel produced in this work.

Total Lipids Percentage of the Jackfruit Oil: In order to obtain a characterization of the jackfruit oil obtained, the lipid content was calculated. The percentage of total lipids present in the samples was calculated using Equation 03:

$$\% \text{ lipids} = (m_o/m_s) \cdot 100 \quad (01)$$

where m_o is oil mass and m_s is the mass of crushed seeds.

Acidity index of the Biodiesel: The acid index (AI) measures the presence of free fatty acids (FFA) and was determined through the quantitative volumetric method. Potassium hydroxide (KOH) was used as standard and phenolphthalein as indicator. In an Erlenmeyer flask, 0.70 g of biodiesel was weighed, 25 mL of ethyl alcohol and 1.0 mL of 1% phenolphthalein were added. The sample was titrated under agitation with a 0.15 mol/L KOH solution until the end point of the reaction. Then the index calculated according to Equation 02:

$$IA = \frac{V \cdot N \cdot f}{m} \quad (02)$$

where, V is the volume of hydroxide used in the titration, N is the normality of the KOH solution, f is the correction factor of the KOH solution and m is the mass of the sample used.

Electrical Conductivity of the Biodiesel: The electrical conductivity was performed with a conductivity meter (Omega, CDB 387), where the biodiesel was placed in a 50 mL beaker and stirred slightly, then introduced the electrode into the biodiesel sample and waited for the value to stabilize.

pH of the Biodiesel: The pH measurements were performed using the potentiometric method, with a digital pHmeter 0.00 - 14.00 pH/mv/°C (Bel Engineering, W3B). The biodiesel was placed in a 50 mL beaker under a slight agitation, then the electrode was introduced into the sample. After stabilization, temperature and pH were measurements.

Density of the Biodiesel: The density, d, was determined with a 25 mL pycnometer. The empty pycnometer with the lid was initially weighed. Then it was filled with biodiesel until it overflowed, covered and weighed. So, from the mass obtained and the pycnometer volume, the absolute density of biodiesel was determined, according to Equation 03.

$$d = (M2 - M1)/V \quad (03)$$

where, M2 is the mass of the full pycnometer, M1 is the mass of the empty pycnometer and V is the volume declared on the pycnometer calibration certificate.

RESULTS AND DISCUSSIONS

To adapt the raw material to acceptable humidity and acidity parameters for the process, there is a preparation step prior to the reaction. At this stage, the raw material is neutralized by washing with an alkaline hydroxide solution, followed by drying. The first sample of the jackfruit seeds weighed 78g, which was placed in a kiln at 60°C for 10h in a kiln. However, a colony of fungus grew in it, as shown Figure 3. This indicates that more time is needed to dry the sample. So, the first contribution of paper is about the time to dry the jackfruit seeds since 10h was not enough to fully dry out the sample.



Figure 3. Seeds show proliferation of fungi

The procedure was redone and the new sample (237.6 g of jackfruit seed) was submitted for 20 hours in the oven. From the total sample, 236.9 g of flour was obtained. Next step is to extract the oil from the seeds. The oil was extracted in the Soxhlet extractor. It was fed with 209.8 g of crushed and sieved seed flour and 1370 ml of solvent (Hexane). After passing by the rotary evaporator the volume of concentrated oil obtained was 130 ml. In order to measure the amount of lipids obtained in the oil extraction, the percentage of total lipids was determined, performing the analyses in triplicate. The result being expressed by the average of the three analyses. The percentage of total lipids present in the samples is $42.21 \pm 0.19\%$ per 100 g of crushed seed that is a high content of extract. This value agrees with the Ojwang et al. (2015) where the lipid content found was $0.45 \pm 0.24\%$ per 100 g of crushed seeds. According to Guimarães et al. (2022) compared to other agro-industrial residues, this value has great potential to be recovered as raw material for the production of biofuels, especially for biodiesel. Once the oil has been obtained, the next step is to carry out the transesterification reaction and obtaining biodiesel as shown the reaction in Figure 4. For that, methanol as a catalyst was used, instead of using ethanol, as methanol has a shorter chain and greater polarity, favouring the speed of the reaction and the removal of by-products. In addition, Barros (2020) states that ethanol promotes greater dispersion of glycerine in biodiesel, making it difficult to separate. When the transesterification procedure was finished, a volume of 102 mL was obtained. After decantation and phase separation, 82 mL of biodiesel and 20 mL of glycerine were obtained. So, the conversion achieved was 80.4%.

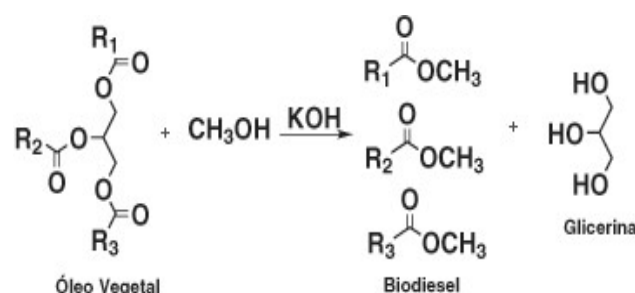


Figure 4. Chemical reaction of biofuel production

The results of the characterization in the biodiesel samples obtained by transesterification reaction via heating are presented below (Table 1) in comparison with the specifications of the norms of the ANP (Brazilian National Agency of Petroleum, Natural Gas and Biofuels) and results from literature.

Table 1. Characterization of the biodiesel sample

| Parameter | Results | References from Brazilian legislation |
|---------------------------------|---------|---------------------------------------|
| Density (kg/m ³) | 792 | ~ 860 (Norma EN 14214) |
| Acidity index (mg KOH/g) | 0.13 | < 0.5 (ANP) |
| Electrical Conductivity (µS/cm) | 5.37 | 4.81 – 6.76 (Barros, 2020) |
| pH | 4.93 | 6 (ANP) |

As shown in Table 1, a value of 792 kg/m³ was obtained for the density. That is close (7,9% deviation) to that established by the EN 14214 standard, which requires that biodiesel have a density of around 860 kg/m³. It also confirms the fact that biodiesel is a less dense liquid than water (which has a density of 1000 kg/m³). The justification for obtaining a minimum value for specific mass is to obtain a maximum power for the engine, which uses the fuel with flow control in the injection pump. Therefore, the biodiesel complies with this specification. Density assessment is an important criterion because it measures the purity of biodiesel, considering that the presence of mono, di and triglycerides and remaining catalyst from the transesterification reaction incomplete can alter its density. So, the biodiesel obtained here is in agreement with the specification shown in Table 1.

The analysis of the acid index, that is, the measurement of free fatty acids, was carried out by titration. A value of 0.132 mg KOH/g was obtained, and the volume used for the turning point was 0.6 mL. As established by the ANP ordinance, aiming at the quality of the biodiesel, the acidity index cannot be greater than 0.5 mg KOH/g. Thus, the result obtained in this paper is in accordance with the value established by the ordinance. This demonstrates that the seed is an adequate raw material to the production of biodiesel, because if the acidity index is high, a strong presence of free fatty acids is indicated. Important mention that such raw material cannot be used in a basic transesterification, as these characteristic favours the formation of soaps in the reaction. Furthermore, high values of acidity reveal a failure in the reaction process and can affect fuel efficiency and cause damage to the engine. The analysis of the electrical conductivity reveals a value of 5.37 µS/cm. According to Delgado et al. (2007) conductivity measures the facility of the biofuel to dissipate the charge generated during oil transfer. It varies with the total amount of ionized substances dissolved in the fuel, the mobility of the ions with their valence and with the relative concentrations of each ion. A reference value was not found in the ANP, but the result is compatible with Barros et al. (2020) who, when performing two measurements, found respective values of 4.81 µS/cm and 6.76 µS/cm. Therefore, the result obtained in this work is within parameters established in the literature. The result obtained by the potentiometric method demonstrated a pH of 4.93 at 20°C of temperature. Through the resulting values, there is a divergence from the parameter in force in the ANP. It establishes that the pH of biodiesel must have a neutral profile, as acidic or basic pH values can cause damage to parts and corrosion of equipment and engines. This value found may be due to the washing step, that is, the neutralization of biodiesel occurred in two days, due to external interference. However, other authors also found a pH below the ordinance value, such as Silva et al (2019) who obtained a pH value of 3.16. It is important to mention that for the final application of biodiesel a certain amount of diesel from petroleum must be added. This addition can be a determining factor in obtaining a neutral pH.

CONCLUSIONS

This paper aimed to synthesize the vegetable oil from the jackfruit seed, an agro-industrial residue, to obtain biofuel by the transesterification method. Conversions of 80.4% were achieved. In

order to obtain quality biodiesel, it is necessary to meet the parameters established by the current ANP ordinance. The parameters evaluated in this article were pH, electrical conductivity, acidity index and density (density). The results indicate that the biodiesel sample obtained from the jackfruit seed is similar to those established by Brazilian norms and the results of other authors. However, it will be necessary to carry out other physical-chemical tests on this biodiesel to better characterize it, as well as redo the pH analysis after a new biodiesel neutralization step, since it was the parameter that most diverged from the established norms. It is necessary to emphasize that the use of jackfruit seed will provide several benefits, direct and indirect jobs, considering that the entire surrounding region will benefit, an increase in investment in family farming and, finally, the growth of cooperatives. Besides that, the studied seed shows potential for industrial application, since the equipment and materials for its production are already used in great-sized industries, such as soy oil which corroborates in the applicability and cost reduction for its implementation.

Availability Statement: The data that supports the findings of this study are available within the article.

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