ChemPor 2023

14th International Chemical and Biological Engineering Conference

Book of Abstracts

Instituto Politécnico de Bragança | September 12-15

ORDEM DOS ENGENHEIROS

ipb INSTITUTO POLITÉCNICO DE BRAGANÇA

This volume contains the extended abstracts presented at the 14th International Chemical and Biological Engineering Conference (CHEMPOR 2023), held in Bragança - Portugal, from the 12th to the 15th of September, 2023.

Instituto Politécnico de Bragança & Ordem dos Engenheiros

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(CHEMPOR-2023)

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Edited by:

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Publisher

Instituto Politécnico de Bragança

September 2023

ISBN 978-972-745-327-6

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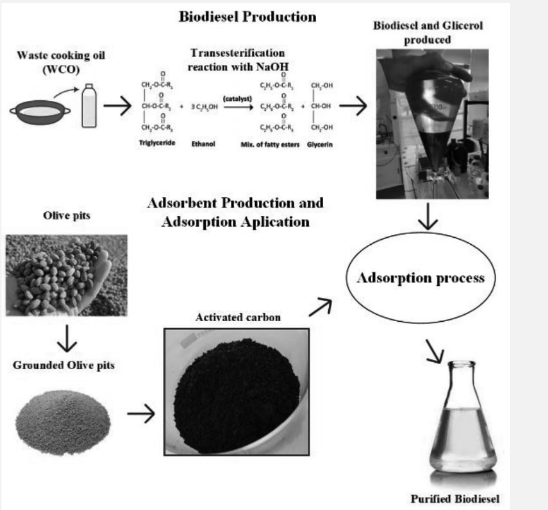
Study of biodiesel production from waste cooking oil by ethyl transesterification and its purification with the use of natural adsorbents

G.L. Camilo[1,2,3]*, M.C.S. Gomes[3], A. Queiroz[1,2], A. Ribeiro[1,2], P. Brito[1,2]

[1]Centro de Investigação de Montanha (CIMO), Instituto Politécnico de Bragança, Campus de Santa Apolónia, 5300-253 Bragança, Portugal; [2]Laboratório para a Sustentabilidade e Tecnologia em Regiões de Montanha (SusTEC), Instituto Politécnico de Bragança, Campus de Santa Apolónia, 5300-253 Bragança, Portugal; [3]Universidade Tecnológica Federal do Paraná, Jardim Paraíso, 86812-460, Apucarana, Brasil.

*glaminocamilo@gmail.com



Biodiesel production from waste cooking oil (WCO) has become an economic opportunity and an environmental strategy to help address the global challenges of renewable energy production. Considering classical industrial processes for biodiesel production, adsorption and ion exchange technologies are the most commonly used alternatives for crude biodiesel purification. These methods, also known as dry cleaning methods, use an appropriate adsorbent to selectively remove certain impurities from the liquid biodiesel phase through contact with the surface of the adsorbent. Dry cleaning offers several advantages, including simple integration into an existing industrial plant, shorter purification time, lower water consumption, and lower effluent generation. In this work, the main goals are the optimization of ethylic biodiesel production from WCO, followed by the study of its purification by adsorption, applying various types of previously characterized natural adsorbents, physically and chemically activated, which were obtained from residual biomass (olive pits), with a specific focus on glycerol removal.

Introduction

In recent years, a collective effort is being made in search of alternative forms of energy through renewable and friendlier to the environment sources. Currently, about 80% of the world consumption of energy comes from fossil fuels. The environmental problems associated with the use of these non-renewable fuels include air pollution and global warming [1]. In this scenario, biodiesel presents itself as a renewable fuel, environmentally friendly and with similar characteristics to common diesel. The cost of conventional biodiesel production is higher than the production of diesel from petroleum. Since it is produced mainly from high quality virgin oils, it is estimated that 70 to 80% of the total cost of biodiesel production is associated with the cost of their raw materials [2]. With this perspective, biodiesel production from waste cooking oil (WCO) has become an economic opportunity and an environmental strategy to help address global renewable energy challenges and contribute to a sustainable society [3].
Transesterification is the most used method to reduce the viscosity of vegetable oils, and the most accessible and available alcohols for this reaction include methanol and ethanol. The use of ethanol for production adds an even more sustainable character to the process, due to its world production being mainly through sugar cane plantations. Among the purification processes for crude biodiesel, the wet wash method, which uses water to purify the esters, is the most frequently used. On average, for each liter of purified biodiesel, the amount of water needed for purification varies from 0.2 to 10 liters. Wet washing is also energy and time consuming and economically inefficient. The literature indicates that effluent treatment costs account for 0.09 to 3.8% of the total cost of production [4]. Adsorption and ion exchange are the most commonly used affinity separation processes worldwide, which are also known as dry washing methods. Dry cleaning offers several advantages over wet cleaning, including ease of integration into an existing plant, shorter purification time, lower water consumption and lower effluent generation [5].
Activated Carbon (AC), activated fiber (carbon fiber) and activated alumina are among the most usual adsorbents in industrial applications. AC, which shows a large porous volume and high surface area, can be manufactured from any organic material rich in carbon, such as: sawdust, wood, coal, peat, fruit nuts, bituminous coal, lignite, coconut husks and olive pits [5].

Objectives

The main objectives of this work are the optimization of biodiesel production from waste cooking oil by alkaline transesterification using ethanol and the purification of the biofuel produced using activated carbon adsorbents produced from ground olive pits, focusing on the removal of free glycerol from biodiesel.

Methods

Initially, an optimization of the production of biodiesel from WCO by the ethylic transesterification route is sought. The optimization was carried out by a response surface methodology based on a Box-Behnken design for the study of 3 parameters: alcohol:oil molar ratio, reaction temperature and catalyst concentration. Subsequently, the synthesis of several adsorbent materials based on natural sources, such as olive pits, was implemented. The procedure employed for zinc chloride activation was adapted from [6] and the procedure for the physical activation at 800°C was carried out in a muffle furnace for 1 hour, with a heating rate of 10°C/min. The N_2 adsorption-desorption experiment were conducted at 77K using a commercial surface area and pore size analyzer (Quantachrome Instruments – Model Novatouch LX4). The samples were degassed at 120°C for 16 hours. The surface area

was obtained using the BET model (Brunauer-Emmett-Teller) and the pore size distribution was assessed by the DFT method (Density Functional Theory).
For the determination of free glycerol content in the biodiesel produced, the methodology developed by [7] was followed.
Adsorption kinetics was evaluated at temperatures of 25°C, 35°C and 45°C. For each batch, 80g of biodiesel, adsorbent concentration equal to 5%wt was used, and contact times of 5, 10, 15, 30, 60, 120, 240, 360, 1380 and 1440 min.

Results

The two most impacting parameters in the percentage of fatty acid ethyl esters (%FAEE) in the biodiesel produced are the alcohol:oil ratio and the temperature. The percentage of catalyst was not able to have an impacting influence on the yield. Below, in Figure 1, the contour surface for the study of the two parameters of greatest influence on the %FAEE.

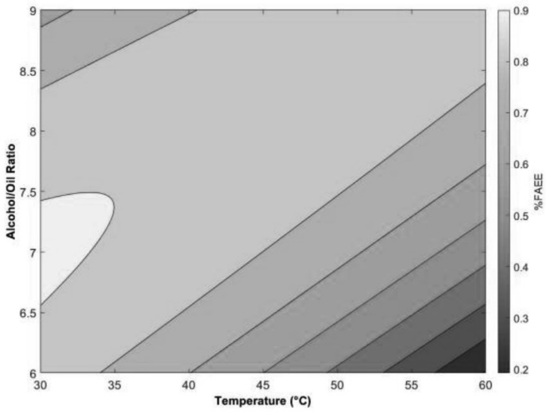


Figure 1. Contour Plot of %FAEE .vs. Alcohol:Oil ratio .vs. Temperature.

It was possible to observe the possibility of using waste cooking oil for the production of biodiesel. The optimal conditions for biodiesel production were determined as being 30°C, 7.5 alcohol/oil ratio and 1%wt of catalyst.
Table 1 shows the results of the analysis of the surface area for the activated carbons produced and the precursor used, olive pit. The production of activated carbons demonstrates promising textural properties, with two materials with surface areas greater than 300 m^2/g.

Table 1. Some textural properties of the adsorbents.

Adsorbent	Surface area (m^2/g)	Pore diameter (nm)
Precursor	1.3798	2.7436
H_3PO_4 - 500°C	171.7740	0.7742
KOH - 800°C	61.1832	1.5381
Physical - 800°C	374.5070	0.8260
ZnCl2 – 500°C	936.9390	2.1684

The activated carbons with the highest areas were physically activated at 800°C and chemically activated with zinc chloride, both were selected for further adsorption studies.
In Figure 2, it is possible to observe some results of the adsorption kinetics study, carried out at 3 different temperatures for physically activated carbon at 800°C. An equilibrium time close to 4 hours of adsorption was observed. An increase in the adsorption capacity according to temperature was also observed, which can be explained by the better mass transfer due to the decrease of biodiesel viscosity.

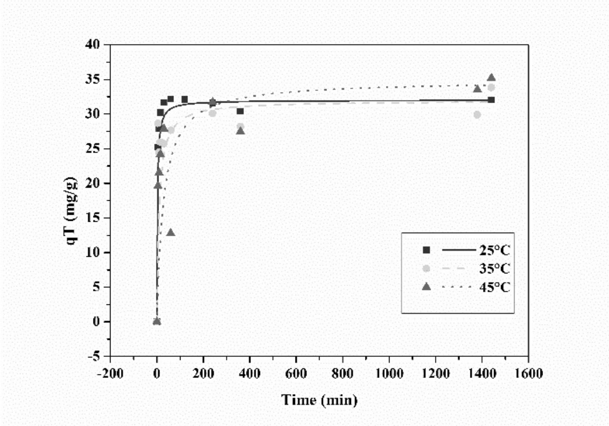


Figure 2. Kinetic study of 800°C-AC with 5% adsorbent concentration.

The pseudo-second order model (lines) was found to better fit to the experimental kinetic data (points). The obtained results are presented in Figure 2, and the related estimated kinetic parameters are shown in Table 2.

Table 2. Kinetic constants values.

T (°C)	q_e (mg/g)	K (g/mg.min)	R^2
25	32.01	0.01276	0.9998
35	31.94	0.00314	0.9942
45	34.86	0.00099	0.9946

Conclusions

The adsorption tests and analyzes demonstrated a good adsorption capacity for these materials, a moderate equilibrium time and therefore enabling the reduction or, with further studies, the complete avoidance of the use of water for the purification of the product, reducing the generation of effluents inherent to the conventional purification process. More research for the development of techniques can be carried out in the future, such as the study of adsorption in a continuous regime using a column packed with the adsorbent, and even a mixture of washing procedures with adsorption, in order to seek a drastic reduction on the consumption of water, and a simultaneous valorization of a residue as an adsorbent material.

Acknowledgements

The authors are grateful to the Foundation for Science and Technology (FCT, Portugal) for financial support through national funds FCT/MCTES (PIDDAC) to CIMO (UIDB/00690/2020 and UIDP/00690/2020) and SusTEC (LA/P/0007/2021).

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