



Biodiesel production using supercritical alcohols with a non-edible vegetable oil in a batch reactor

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ABSTRACT

In the present work, the transesterification of non-edible oil with methanol and ethanol is studied. The reactor phase transition are directly observed in a double windowed cylindrical reactor and the conversions to fatty esters is measured. The optimization of the process conditions was carried out based on a statistical design of experiments where the key process variables were studied over different ranges to obtain a reliable model for the efficiency of the reaction as a function of reaction time, temperature, pressure and alcohol to oil molar ratio. From direct observations and the modeling of the phase behavior, a better understanding of the supercritical alcohol transesterification process is obtained as well as the confirmation of the phase equilibrium predictions based on the CCA-005 model.

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1. Introduction

The production of fatty acid methyl and ethylesters is of great industrial interest because of their direct use as biodiesel. Most of the biodiesel currently produced uses rapeseed or soybean oil, methanol and an alkaline catalyst. The main products of the reaction are fatty esters and glycerin. The high value of vegetable oils as food products makes production of a cost-effective fuel very challenging [1]. However, crude oils, animal fats and waste cooking oils can also be converted into biodiesel. Some problems with processing these low cost oils are related to the presence of water and free fatty acids [2]. The transesterification of vegetable oils using supercritical alcohols is an alternative for biodiesel industrial production. Recent experimental studies of non-catalytic transesterification by Saka and Kusdiana [3,4] have shown that the process is not sensitive to both free fatty acids and water contents, and high reaction rates are observed at conditions close to the critical properties of methanol.

Non-edible vegetable oils constitute another attractive option for biodiesel production. In the present work, the transesterification of fodder radish—*Raphanus sativus* L. var. *oleiferus* Stokes oil (a non-edible vegetable oil) in supercritical alcohols is studied in order to find optimum operating conditions. Fodder radish oil

has been regarded as an interesting option to produce biodiesel in Brazil, since the seeds have high oil content and the oil can be easily recovered by pressing. The plant has rapid development and has been used for green fertilization [5]. The study of the supercritical transesterification with ethanol, besides like that of methanol, is justified considering the large production of ethanol from renewable resources in South America.

In the present work, a phase equilibrium engineering analysis is applied to the transesterification of fodder radish oil in supercritical ethanol and methanol to study the influence of the pressure and a single phase condition in the transesterification reaction. Phase equilibrium engineering is the design of the phase condition required for a given process. This design is made on the basis of modeling of phase equilibria, knowledge of the phenomenological behavior or experimental information. Examples of phase equilibrium engineering applied to reaction and separation process can be seen elsewhere [6,7]. In this study, direct observations and modeling of the phase behavior is carried out in order to find feasible homogeneous operating conditions at moderated pressures. Then, the different variables affecting the non-catalytic transesterification reaction are evaluated to set the operating conditions and to demonstrate the feasibility of the operating conditions proposed previously. The best values of the variables in the experimental range studied are determined by applying a Doehlert design of experiments [8] and response surface methodology to determine how these variables influence the reaction. The results obtained are also interesting from the point of view of the continuous process

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Ashley D'Ann Koh



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Process Synthesis and Process Intensification Ben-Guang Rong,2017-09-25 Process synthesis and process intensification are becoming state of the art scientific fields that provide the methods and tools to improve process technologies in terms of high energy efficiency low capital investment low emissions improved safety and less hazardous byproducts to achieve sustainable products and processes The book covers manufacturing processes from both fossil and biomass based feedstocks for graduate students

Handbook of Research on Advancements in Supercritical Fluids Applications for Sustainable Energy Systems Chen, Lin,2020-08-28 Supercritical fluids are increasingly being used in energy conversion and fluid dynamics studies for energy related systems and applications These new applications are contributing to both the increase of energy efficiency as well as greenhouse gas reduction Such research is critical for scientific advancement and industrial innovations that can support environmentally friendly strategies for sustainable energy systems The Handbook of Research on Advancements in Supercritical Fluids Applications for Sustainable Energy Systems is a comprehensive two volume reference that covers the most recent and challenging issues and outlooks for the applications and innovations of supercritical fluids The book first converts basic thermo dynamic behaviors and abnormal properties from a thermophysical aspect then basic heat transfer and flow properties recent new findings of its physical aspect and indications chemical engineering properties micro nano scale phenomena and transient behaviors in fast and critical environments It is ideal for engineers energy companies environmentalists researchers academicians and students studying supercritical fluids and their applications for creating sustainable energy systems

Phase Equilibrium Engineering Esteban Brignole,Selva Pereda,2013-04-02 The application of the principles of phase equilibrium engineering to the development of two innovative technologies for the production of biofuels is discussed in this chapter The first technology is the production of biodiesel by transesterification of vegetable oils with supercritical methanol the second the extraction and dehydration of alcohols by near critical dual effect solvents that exhibit good solvent power to extract alcohols and water entrainment effect to dehydrate the extracted alcohol In the first case the complexity of the reacting system the large size asymmetry and strong molecular interactions of the mixture components methanol vegetable oils fatty esters and glycerin precluded the design and analysis of the process conditions based on thermodynamic model predictions Therefore in this case a systematic approach based on experimental studies was used to unveil the phase scenario and the physical properties required for the design and optimization of this technology The conceptual design of extraction and dehydration of alcohols by near critical solvents followed a different path The process development was initially based on very limited experimental information In this case an equation of state for highly nonideal systems was the main tool for exploration of the process conditions over a wide range of pressures temperatures and compositions This equation of state was based on a group contribution approach GC EOS that allowed extrapolating the scarce experimental information available not only in pressure

temperature and composition but also in molecular structure The basic conceptual design was later confirmed by experimental information and pilot plant studies In this case the design of the experimental studies was guided by the process conceptual design The experimental results provided key information for the upgrading of the thermodynamic model

Recent Trends for Enhancing the Diversity and Quality of Soybean Products Dora Krezhova,2011-10-28 This book presents new aspects and technologies for the applicability of soybean and soybean products in industry human food livestock feed oil and biodiesel production textile medicine as well as for future uses of some soybean sub products The contributions are organized in two sections considering soybean in aspects of food nutrition and health and modern processing technologies Each of the sections covers a wide range of topics The authors are from many countries all over the world and this clearly shows that the soybean research and applications are of global significance Biofuels Handbook James G Speight,2015-10-09 Petroleum based fuels are well established products that have served industry and consumers for more than one hundred years However petroleum once considered inexhaustible is now being depleted at a rapid rate As the amount of available petroleum decreases the need for alternative technologies to produce liquid fuels that could potentially help prolong the liquid fuels culture and mitigate the forthcoming effects of the shortage of transportation fuels is being sought The dynamics are now coming into place for the establishment of a synthetic fuels industry the processes for recovery of raw materials and processing options have to change to increase the efficiency of oil production and it is up to various levels of government not only to promote the establishment of such an industry but to recognise the need for available and variable technology This timely handbook is written to assist the reader in understanding the options that available for the production of synthetic fuel from biological sources Each chapter contains tables of the chemical and physical properties of the fuels and fuel sources It is essential that the properties of such materials be presented in order to assist the researcher to understand the nature of the feedstocks as well as the nature of the products If a product cannot be employed for its hope for use it is not a desirable product and must be changed accordingly Such plans can only be made when the properties of the original product are understood The fuels considered include conventional and unconventional fuel sources the production and properties of fuels from biomass crops wood domestic and industrial waste and landfill gas

Reactive Separation for Process Intensification and Sustainability Carlos Ariel Cardona Alzate,Mariana Ortiz Sanchez,Pisarenko Yury Andrianovich,2019-12-23 This book describes analyses and discusses the main principles phenomena and design strategies of reactive separation processes with an emphasis on the intensification as a basis of the sustainability Different reactive separation processes are explained in detail to show the phenomena and with the purpose of understanding when their use allows advantages based on the output results Case examples are analysed and the perspective of these processes in the future is discussed The overall sustainability of reactive separation processes in the industry is also explained separately *Biohydrogen* Ayhan Demirbas,2009-06-30 *Biohydrogen For Future Engine Fuel Demands* covers the

production purification storage pipeline transport usage and safety of biohydrogen Hydrogen promises to be the most significant fuel source of the future due to its global availability and the fact that water is its only by product Biofuels such as bioethanol biodiesel bio oil and biohydrogen are produced using technologies for thermochemically and biologically converting biomass Hydrogen fuel production technologies can make use of either non renewable sources or renewable sources such as wind solar and biorenewable resources Biohydrogen For Future Engine Fuel Demands reviews all of the modern biomass based transportation fuels including bioethanol biodiesel biogas biohydrogen and fuel cells The book also discusses issues of biohydrogen economy policy and environmental impact Biohydrogen looks set to be the fuel of choice in the future replacing both fossil fuels and biorenewable liquid fuels

Soybeans Lawrence A. Johnson, 2008 This comprehensive new soybean reference book disseminates key soybean information to drive success for soybeans via 23 concise chapters covering all aspects of soybeans from genetics breeding and quality to post harvest management marketing and utilization food and energy applications U S domestic versus foreign practices and production methods The most complete and authoritative book on soybeans Features internationally recognized authors in the 21 chapter book Offers sufficient depth to meet the needs of experts in the subject matter as well as individuals with basic knowledge of the topic

Chemical Engineering Progress, 2008 **Biodiesel Production Using Supercritical Methanol** Dhaval Nitin Modi, 2010 The catalyst free transesterification of oil triglycerides in supercritical methanol at a temperature and pressure above 239 C and 8 MPa respectively provides a new way of producing biodiesel High reaction temperature and pressure help to accelerate the transesterification because the supercritical methanol becomes non polar and has enhanced contact with oil The supercritical transesterification of soybean oil was carried out in a 170 ml volume high pressure batch reactor The pressure within the batch reactor was selfgenerated from heating the contents since no pumps were used Transesterification of soybean oil with supercritical methanol readily produced biodiesel Since the supercritical transesterification is carried out without a traditional acid base catalyst and does not produce any saponified byproducts the recovery of pure biodiesel product becomes much simpler After the reaction the products readily separated into two phases of biodiesel fatty acid methyl esters and glycerol Process variables such as pressure volume and temperature were studied Also the effect of supercritical CO₂ as a potential co solvent was investigated Several 7 ml volume mini reactors were fabricated and used to evaluate the supercritical transesterification reaction and develop an analytical method for lipid composition determination Reaction temperature reaction time and the methanol to oil molar ratio were studied The effect of water free fatty acids hexane co solvents and reaction with different alcohols n propanol isopropanol allyl alcohol were also studied A sequential analytical process was developed by extracting lipids directly from oil seeds using hexanes and converting them to fatty acid esters using supercritical alcohol Abstract p iv

Two-step Biodiesel Production Using Supercritical Methanol and Ethanol Ashley D'Ann Koh, 2011 Current industrial biodiesel production utilizes an alkali catalyst

that can participate in saponification side reactions The side reactions are reduced by using highly refined vegetable oil feedstocks Also the catalyst must be extracted from the final product in a washing step A catalyst free alternative for the production of biodiesel was developed It involves two reaction steps 1 triglyceride hydrolysis fat splitting at subcritical conditions to separate glycerol from fatty acids and 2 fatty acid esterification in supercritical alcohol to form fatty acid alkyl esters The catalyst free process can potentially be used with a variety of low cost vegetable and animal fats without undesired side reactions The focus of this project was on the esterification reaction Experiments were carried out with methanol and ethanol in a batch reaction system at supercritical conditions High conversions could be attained at short reaction times It was determined that the reaction followed second order reversible kinetics In addition a novel Raman spectroscopic method was developed for the analysis of esterification reaction products

23 European Symposium on Computer Aided Process Engineering Fernando I. Gomez-Castro, Juan G. Segovia-Hernandez, Salvador Hernandez-Castro, Vicente Rico-Ramirez, Zeferino Gamiño-Arroyo, Irene Cano-Rodríguez, 2013-06-10 On the last years there have been proposals for using supercritical conditions to produce biodiesel fuel from vegetable oils and or animal fats without a catalyst Different schemes have been proposed the most popular consisting on the use of supercritical methanol as reactant Other alternatives involve the use of methyl acetate or acetic acid as reactants The potential of those processes may be established in terms of their total annual cost and environmental impact Thus in this work the production of biodiesel fuel by using different reactants is studied Four processes are considered the one step supercritical methanol process Saka process the two steps supercritical methanol process Saka Dadan process a process with methyl acetate as reactant and a process with acetic acid as reactant Possible flowsheets for the reaction and separation stages are proposed The processes are analyzed and compared in terms of energy consumption pollutant emissions and total annual costs It has been observed that in terms of energy the one step methanol process has the lowest energy requirements Nevertheless a higher temperature for the steam supplied is required thus that process has high values of CO₂ emissions Furthermore methyl esters are obtained at higher temperatures which may have a negative impact on its quality

Analysis and Optimization of a Biodiesel Production from WCO Roland Kalonji, 2018-02-06 Project Report from the year 2017 in the subject Engineering Industrial Engineering and Management language English abstract The conventional approach of biodiesel production is transesterification using oil and alcohol in the presence of a catalyst with glycerol as a by product of the reaction Product quality is dependent on the type and amount of catalyst type of oil feedstock alcohol to oil ratio etc In terms of the best process currently the alkali catalyzed process is the most profitable while the enzymatic based one is even more promising due to the lower consumption of energy and water however it requires that the enzyme cost is reduced The reason that biodiesel is not utilized widely around the world is due to the high cost of raw materials To overcome this one can use lower quality oils such as Waste Cooking Oil WCO A lot of research has been carried out on the production of biodiesel from

fresh vegetable and animal oil sources but the use of Waste Cooking Oil such as palm oil etc has not been well documented Then the aim of this current project is to analyze and optimize the conditions for biodiesel production from Waste Cooking Oil by investigating interaction effects among process variables temperature oil to methanol molar ratio and catalyst loading using SPC and other tools Thus this project focuses on making biodiesel processes better and more efficient Biodiesel Production Technologies Jorge Mario Marchetti,2010 Biodiesel production is a very modern and technological area that is winning relevance and market due to its benefits such as that it is biodegradable a renewable and alternative source of fuel with less pollutants and less particle pollution Different studies have been carried out using various oils as raw material different alcohol as well as different catalysts homogeneous ones such as sodium hydroxide potassium hydroxide sulphuric acid and supercritical fluids and heterogeneous ones such as solid resins and enzymes as well as new technologies that are being developed every day This book discusses the global energy situation in regard to the biodiesel industry as well as a specific focus on operational conditions kinetics model and economic comparison in order to see if they could be used as profitable alternatives **Biodiesel Production from Tall Oil and Chicken Fat Via Supercritical Methanol Treatment** William Brent Schulte,2007 **Phase Behaviour Studies Related to Biodiesel Production Using Supercritical Methanol** Sultan Al-Habsi,2019 Production of Biodiesel from Non-Edible Sources A. Arumugam,2022-02-10 Production of Biodiesel from Non Edible Sources Technological Updates offers a step by step guide to the production of biodiesel providing comparisons of existing methods new and state of the art technologies and real world examples of implementation The book discusses all potential non edible feedstocks for biodiesel production providing their properties availability and processing including deeper insights into kinetic models and simulation of biodiesel fermentation Readers will gain knowledge of existing parameters and methods for biodiesel production optimization scale up and sustainability along with guidance on the practical implementation of these methods and techniques Finally environmental sustainability techno economic analysis and policymaking aspects are considered and put into the context of future prospects This book offers a step by step guide for researchers and industry practitioners involved in bioenergy renewable energy biofuels production and bioconversion processes Provides step by step guidance on key processes and procedures Reviews all the available non edible feedstocks for biodiesel production and presents their properties pros and cons Presents pilot and industry scale case studies on the implementation of biodiesel production from non edible feedstocks Addresses optimization environmental sustainability economic viability and policy issues to support commercialization **Advanced Technologies in Biodiesel** Aminul Islam,Yun Hin Taufiq-Yap,2015-04-24 The inadequacy of fossil fuel is the main driving force of the future sustainable energy around the world Since heterogeneous catalysis is used in chemical industry for biodiesel production achieving optimal catalytic performance is a significant issue for chemical engineers and chemists Enormous attention has been placed in recent years on the selection of heterogeneous catalyst in biodiesel industry where the catalyst could be facilitated highly

selective toward desired products easily handled separated from the reaction medium and subsequently reused This book stresses an overview on the contributions of tailored solid acid and base catalysts to catalytic biodiesel synthesis and the influences of heterogeneous catalyst properties on biodiesel yield in order to develop a better understanding of catalyst design for the green production process as well as practical applications in the biodiesel industry Biodiesel Margarita Stoytcheva, Gisela Montero, 2011-11-09 The book Biodiesel Feedstocks and Processing Technologies is intended to provide a professional look on the recent achievements and emerging trends in biodiesel production It includes 22 chapters organized in two sections The first book section Feedstocks for Biodiesel Production covers issues associated with the utilization of cost effective non edible raw materials and wastes and the development of biomass feedstock with physical and chemical properties that facilitate its processing to biodiesel These include Brassicaceae spp cooking oils animal fat wastes oleaginous fungi and algae The second book section Biodiesel Production Methods is devoted to the advanced techniques for biodiesel synthesis supercritical transesterification microwaves radio frequency and ultrasound techniques reactive distillation and optimized transesterification processes making use of solid catalysts and immobilized enzymes The adequate and up to date information provided in this book should be of interest for research scientist students and technologists involved in biodiesel production Biodiesel Production with Green Technologies Aminul Islam, Pogaku Ravindra, 2016-09-23 This book provides a single source reference to green technologies in advanced biofuel technology The main focus is on the description of the state of the art in catalytic processes for the green production of biofuels The authors describe two different practical approaches for catalysts which allow for effective and easy separation of the catalyst by simple filtration and enable reuse for several cycles Readers will gain understanding as to the mechanisms involved in the synthesis and structure formation of the catalyst in order to maximize yield of biodiesel production The authors also address the question of how catalytic material should be distributed inside a porous support to obtain optimal performance The effects of physicochemical and operating parameters are analyzed to gain insight into the underlying phenomena governing the performance of optimally designed catalysts br

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