An incisive discussion of biofuel production from an economically informed technical perspective that addresses sustainability and commercialization together

In Biodiesel Production: Feedstocks, Catalysts, and Technologies, renowned chemists Drs. Rokhum, Halder, Ngaosuwan, and Assabumrungrat present an up-to-date account of the most recent developments, challenges, and trends in biodiesel production. The book addresses select feedstocks, including edible and non-edible oils, waste cooking oil, microalgae, and animal fats, and highlights their advantages and disadvantages from a variety of perspectives. It also discusses several catalysts used in each of their methods of preparation, as well as their synthesis, reactivity, recycling techniques, and stability.

The contributions explore recently developed technologies for sustainable production of biodiesel and provides robust treatments of their sustainability, commercialization, and their prospects for future biodiesel production.

- A thorough introduction to the various catalysts used in the preparation of biodiesel and their characteristics
- Comprehensive explorations of biofuel production from technical and economic perspectives, with complete treatments of their sustainability and commercialization
- Practical discussions of the development of new strategies for sustainable and economically viable biodiesel production
- In-depth examinations of biodiesel feedstocks, catalysts, and technologies

Perfect for academic researchers and industrial scientists working in fields that involve biofuels, bioenergy, catalysis, and materials science, Biodiesel Production: Feedstocks, Catalysts, and Technologies will also earn a place in the libraries of bioenergy regulators.

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Biodiesel Production
Biodiesel Production: Feedstocks, Catalysts, and Technologies

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Preface

This book *Biodiesel Production: Feedstocks, Catalysts, and Technologies* includes the contribution of leading researchers in the fields of biodiesel, which will serve as a valuable source of information for scientists, researchers, graduate students, and professionals alike. It focusses on several aspects of biodiesel productions, technologies employed, and sustainability. It consists of 20 chapters, grouped together in three parts, in different technological aspects as follows.

The utilization of conventional and novel feedstocks for biodiesel production will be presented in Chapters 1–4.

Chapter 1 emphasizes on the conversion of several edible vegetable oils and animal fats to biodiesel. Different catalysts used and several factors that affect the overall biodiesel production are comprehensively discussed.

Chapter 2 provides the perspective of the biodiesel production from waste cooking oil via the conventional and modern technologies to bolster competitiveness of biodiesel with petrodiesel.

Chapter 3 addresses the state of the art and future perspectives of nonedible oils for biodiesel production. It provides several important aspects such as cultivation information, fatty acid composition, extraction, and conversion method for biodiesel production.

Chapter 4 proposes the important strategy of microalgae cultivation for the large-scale optimization of lipid accumulation as a potential sustainable approach for biodiesel production.

In the next part, Chapters 5–14, the various types of homogeneous and heterogeneous catalysts for biodiesel production will be discussed.

Chapter 5 reviews the utilization of homogeneous catalysts for various feedstocks under optimum conditions to serve the growing demand of biodiesel as a cost-effective production process.

Chapter 6 summarizes the development of metal oxide catalysts from various sources for biodiesel production. The reaction mechanistic pathways and causes of catalyst deactivation are discussed.

Chapter 7 focuses on the catalytic activity enhancement of metal oxides with particular focus on the role of supporters, their synthesis methods, and physicochemical properties to achieve eco-friendly and economically viable processes of biodiesel production.

Chapter 8 highlights the development of new mixed metal oxides with a variety of novel acidic, basic, and bifunctional catalysts from various feedstocks for enhancing their
catalytic performance. Their stability, catalyst regeneration techniques, and recommendation for full scale biodiesel production are also addressed.

Chapter 9 presents the outlook of using nanotechnology-based catalysts for the development of more efficient, economically viable, durable, and stable nanocatalysts, targeting at achieving higher biodiesel quality and yields.

Chapter 10 reveals the advantages and issues of using ion-exchange resins catalysts for both cation and anion exchange resins especially in continuous biodiesel production.

Chapter 11 discusses the solid bifunctional catalysts with acid–base and Lewis–Bronsted functionalities. The preparation methods, their characterization results, and the optimum condition for biodiesel production were addressed.

Chapter 12 proposes the green concept for biodiesel production using catalysts derived from renewable resources. Essential information on their preparation methods, physico-chemical properties, and catalytic activities as well as the challenges are discussed.

Chapter 13 exploits the usage of the promising ionic liquid catalyst to replace homogeneously catalyzed biodiesel production concurrently with techno-economic analysis, life cycle assessment, environmental impact assessment, and scale-up technologies.

Chapter 14 demonstrates the effective acid/base metal–organic frameworks (MOFs) catalysts for both transesterification and esterification reactions to intensify biodiesel production based on their catalytic synergy.

The strategies in terms of upstream, mainstream, and downstream process to fulfill the economical and sustainable for biodiesel production will be addressed in Chapters 15–21.

Chapter 15 scrutinizes the strategies for upstream biodiesel production dealing with the advanced feedstocks like waste cooking oil, waste animal fats, nonedible oils, or genetically engineered oils based on the appropriate catalyst, reaction conditions, and the following downstream processes.

Chapter 16 approaches the operating key parameters of mainstream strategies in terms of the novel reactor for biodiesel production based on the scientific and practical viewpoints to achieve efficiency and sustainable concept.

Chapter 17 discloses the downstream strategies to accomplish biodiesel standard as well as operating cost reduction using methanol recovery and glycerol by-product refining. The integration of bioenergy systems to produce antioxidant additives for improving biodiesel quality is also encouraged.

Chapter 18 addresses the conversion of bio-glycerol to value-added chemicals especially acrylic acid to boost alternative sustainable routes for biodiesel production.

Chapter 19 introduces the sustainability in the production and use of biodiesel, which is mainly dependent on the types of feedstocks and government policy as the incentives of using biodiesel.

Chapter 20 discusses the advanced, sustainable technology with respect to the diversified feedstock and design of the novel efficient catalytic system for production of biodiesel and its commercialization.
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An Overview of Biodiesel Production

The advent of the industrial revolution had many benefits such as increases in wealth of the average masses, upgrade in living standards, and vast improvements in production of goods (both in quality and in quantity), which reduced prices drastically. Technological advancements also occurred in the transport sector, which enabled ease in travel, while the use of coal and petroleum skyrocketed: an example of this would be the 20-fold increase in coal imports between 1550 and 1700 in Newcastle, England. Consequently, a proportional increase in mining of these fossilized reserves had to be done as far as from the early nineteenth century. Since then, the energy demand per capita has increased manifold to the point where current consumption trends cannot be supported without exhausting the remaining global reserves – alternative energy sources must be sought. Additionally, large areas of forest land had been cleared for fuelwood, which served as the primary energy source for cooking and heating in rural households. Widespread deforestation led to a rapid rise in global temperature since less trees are available for climate modulation. Also, upon using wood and other fossilized sources as fuel, huge amounts of particulate matter, smoke, and other noxious gases (NOXs, SOXs, CO, and CO2) are emitted, and thus their continued emission for the last few centuries has led to global warming, harmful impacts on terrestrial and aquatic life (through acid rain, aquatic pollution resulting in eutrophication), and changes in weather patterns, which has even impacted the overall health and life expectancy of humans (lung diseases caused by air pollution, water pollution leading to chronic diseases, etc.).

In order to combat or gradually reverse the effects of such a global situation where arable land and potable water are scarce, alternative energy sources that have no or negligible environmental impacts must be sought. Thus, renewable energy research over the last few decades has been steadily increasing and is now capable of changing an entire country’s energy consumption trend. A good example is Brazil, which runs entirely on “sustainable” fuels, having produced 26.1% (a staggering 26.72 billion liters) of the global ethanol being used as fuel in 2017, and many countries have tried to replicate the so-called “Brazilian ethanol model.” Among the wide variety of renewable energy sources available, feedstock for biofuels such as biodiesel and bioethanol are limited to a few varieties. Vegetable oils (edible or nonedible) cannot be directly used in engines due to their incompatible physicochemical properties. This had been tested by Dr. Rudolph Diesel who used peanut oil for
his internal combustion (IC) engine and reported many problems in required performance when run for extended durations. Thus, such oils are converted into esters that are the main component of biodiesel, a fuel suitable for use in diesel engines with minor modifications. To convert vegetable oils as well as other potential feedstock such as microalgal lipids, animal fats and greases, waste oils, and other miscellaneous sources, various approaches may be used with different conversion efficiencies. The most efficient conversion process, however, is transesterification, which may or may not be coupled with an esterification pretreatment stage depending on the free fatty acid content of the oil.

For both esterification and transesterification, the reactants are the feedstock and an alcohol, which in the presence of a catalyst are converted into their esters, producing either water or glycerol as by-products. Depending on the reaction conditions (based on the approach used), catalysts may not be required, although a multitude of catalysts have been developed and tested with varying degrees of efficiency. Such catalysts range from the simplest mineral acids, enzymes, or bases, which are added for achieving a homogeneous system and discarded with every use to simple heterogeneous catalysts that rely on solid metal oxides or the use of inert carbonaceous or siliceous biomass doped with the required catalytic groups (including transition metals) or enzymes, as well as nanocatalysts that have increased efficiency (when compared with inert microporous support-based catalysts), while specially designed catalysts based on resin supports or metal organic frameworks have also been developed and can be very efficient but may be difficult to commercialize due to high development costs and unavoidable losses in each cycle of use. Strangely, processes such as supercritical fluid technology or superheated vapor technology can function reliably even without the use of catalysts, although the use of catalysts can augment the process, which may require a cost-to-benefit analysis before commercialization.

The process of biodiesel commercialization does not simply end at its production, since there are many stages that need to be considered for downstream processing as well as the consideration for treatment of hazardous materials generated (such as biodiesel wastewater that contains spent catalyst or leached ions) and the recovery of spent alcohol and the valorization of generated glycerol. Additionally, the produced fuel must have an acceptably long shelf life, and since biodiesel is prone to auto-oxidation (it contains high oxygen content that helps in reducing pollution due to complete fuel combustion), such additives are essential for storage. Such processes generally increase the cost of available fuel, which has made it necessary to consider these hurdles that are yet to be overcome before the complete utilization of biodiesel is feasible as an environment-friendly and affordable alternative to petrodiesel.

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Part 1

Biodiesel Feedstocks
1

Advances in Production of Biodiesel from Vegetable Oils and Animal Fats

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1.1 Introduction

Currently, the energy requirements of the world are mainly met through fossil fuel resources, such as gasoline, petroleum-based diesel, and natural gas. Such fossil-derived resources are too limited to fulfill the future energy demands and meet the challenges of rapid human population growth coupled with technological developments [1]. Presently, research is progressively more directed toward exploration of alternative renewable fuels. Several types of biofuels, such as vegetable oil/animal fat (raw, processed, or used), methyl esters from vegetable oil/animal fat, and ethanol or liquid fuels from biomass (bioethanol and biomethanol), have been investigated as a replacement for gasoline and petrodiesel [2].

At present over 197.97 million metric tons of 10 major vegetable oils are produced worldwide [3]. Vegetable oils are commonly derived from various oilseed crops. In a vegetable oil, almost 90–95% is glycerides, which are basically esters of glycerol and fatty acids (FAs) [4]. The vegetable oils can be considered as a feasible alternative for diesel fuel as the heating value of vegetable oils is comparable to that of diesel fuel [5, 6]. However, the uses of vegetable oils in direct injection diesel engines are restricted due to some unfavorable physical properties, particularly the viscosity. The viscosity of vegetable oil is roughly 10 times higher than the diesel fuel. Therefore, the use of vegetable oil in direct injection diesel engines creates poor fuel atomization, incomplete combustion, and carbon deposition on the injector [7, 8].

Several techniques are employed to bring down the physical and thermal properties of vegetable oils close to mineral diesel, by which these oils and fats can be used in internal combustion engines as fuel. This mainly requires improvement in viscosity of the vegetable oil. The possible treatments employed to improve the oil viscosity includes dilution with a suitable solvent, microemulsification, pyrolysis, and transesterification [9, 10].

The uses of biodiesel (BD) as a renewable, biodegradable, nontoxic, and eco-friendly neat diesel fuel or in blends with petroleum-based fuels are fascinating [11, 12]. “Biodiesel,” termed as the monoalkyl esters of long-chain FAs, is derived from vegetable oils or animal fats.
Numerous types of conventional and nonconventional vegetable oils and animal fats including those of used oils from the frying industry, soybean oil, rapeseed oil, tallow, rubber seed oil, and palm oil have been investigated to produce BD [13–15]. The production of BD involves the conversion of vegetable oils/animal fats using methanol or ethanol and a catalyst to produce fatty acid methyl esters (FAMEs) and crude glycerin as by-product through a process termed as “transesterification” [16].

The transesterification process is accomplished by reacting vegetable oil with alcohol in the presence of alkaline or acidic catalyst. A catalyst is typically used to accelerate the reaction rate and yield. The stoichiometric equation requires 1 mol of triglyceride and 3 mol of alcohol to form 3 mol of methyl ester and 1 mol of glycerol [17]. Since the reaction is reversible, excess alcohol is used to shift the reaction equilibrium to the product’s side. The most preferred catalysts are sulfuric, sulfonic, and hydrochloric acids as acidic catalysts and sodium hydroxide, sodium methoxide, and potassium hydroxide as alkaline catalysts [18]. The product, fatty esters, have improved viscosity and volatility relative to the triglycerides. A dense, liquid phase rich in glycerol is the coproduct of this process. The separated fatty esters have cetane number and heating value close to that of the conventional diesel. The transesterification process for converting vegetable oils to BD is shown in Figure 1.1.

The “R” groups are the FAs, which are usually 12–22 carbons in length. The large vegetable oil molecule is reduced to about one third of its original size, lowering the viscosity and making it like diesel fuel. The resulting fuel can work like diesel fuel in an engine. The by-product “glycerin” produced in this process is valuable due to its diverse industrial applications [19].

Technically, BD is a fuel comprising of monoalkyl esters of long-chain FAs derived from vegetable oils or animal fat, which meets current EN 14214 and ASTM D 6751 BD standards of Europe and the United States, respectively. These standards are frequently employed as references to evaluate and compare the properties of other fuels.

Presently, the BD is commonly produced using a base-catalyzed transesterification reaction because it involves low temperature and pressure processing, high conversions, no intermediate steps, and lower costs of processing materials [20]. Alkoxides and hydroxides of potassium and sodium are often used as catalysts in the transesterification of refined oils and low FA greases and fats. However, acid esterification followed by transesterification of high free fatty acid (FFA) fats and oils is also applicable. The base catalysts have better efficiency than the acid catalysts [21]. The base-catalyzed transesterification reaction can be carried out at lower temperature, yet at room temperature, with the base catalysts, whereas acid catalysis required higher temperature (100 °C) and longer reaction time. During the process, basic catalyst breaks the FAs from the glycerin one by one. When a methanol molecule contacts an FA molecule, it will bond and form BD molecule. The hydroxyl group

\[ \text{CH}_2\text{OCOR}_1 \quad \text{R}_1\text{COOCH}_3 \quad \text{CH}_2\text{OH} \]
\[ \text{CH}_2\text{OCOR}_2 + 3 \text{CH}_3\text{OH} \leftrightarrow \text{R}_2\text{COOCH}_3 + \text{CH}_2\text{OH} \]
\[ \text{CH}_2\text{OCOR}_3 \quad \text{R}_3\text{COOCH}_3 \quad \text{CH}_2\text{OH} \]

Figure 1.1 General reaction for transesterification of vegetable oil.
from the catalyst alleviates the glycerol formation. The resulting product named as methyl esters (BD) has appreciably lower viscosity and increased volatility relative to the triglycerides present in vegetable oils [22–24].

The second usual method of producing BD involves the use of an acid as a substitute of a base catalyst. Any mineral acid can be employed to catalyze the process; the most used acids are sulfuric acid and sulfonic acid. Although yield is high, the acids, being corrosive, may cause damage to the equipment, and the reaction rate is also observed to be relatively low [9, 21]. Oil feedstocks containing more than 4% FFAs must pass through an acid esterification process to increase the BD yield [25]. Such feedstocks are filtered and preprocessed to remove water and contaminants and then fed to the acid esterification process. The catalyst (sulfuric acid) is dissolved in methanol and then mixed with the pretreated oil [26].

The alcohols employed in the transesterification are generally short-chain alcohols such as methanol, ethanol, propanol, and butanol producing esters named as methyl-, ethyl-, propyl-, and butyl-esters, respectively [9, 10]. It is reported that when transesterification of soybean oil using methanol, ethanol, and butanol was performed, 96–98% of ester’s yield could be obtained after an hour of reaction [27]. Though utilizing different alcohols presents little differences with regard to the kinetic of reaction, the final yield of esters remains unchanged. Thus, assortment of the alcohol is based on cost and performance consideration. Generally, reaction temperature is set at near the boiling point of the alcohol used [28].

Due to the reality that many vegetable oils, including soybean, canola (rapeseed) oil, and rice bran oil, have a major number of FAs with double bonds, oxidative stability is a problem, particularly when storing BD for longer period of time [29, 30]. This problem becomes severe due to improper storage conditions, which may include exposure to air and/or light, temperatures above ambient, and presence of extraneous materials (contaminants) with catalytic effect on oxidation. Some additives such as antioxidants might control the oxidation.

Characterization of BD fuel properties and evaluation of its quality are the matters of great concern for the successful commercialization of this fuel. A high fuel value with no operational problems is a condition for market acceptance of BD. Accordingly, the analysis of BD and the monitoring of the transesterification reaction have been the subject of numerous publications [31, 32]. The constraints, which are used to define the quality of BD, can be divided in two groups [33]. One of them is also used for mineral diesel, and the second illustrates the composition and purity of fatty esters. The former includes, for example, density, viscosity, flash point, sulfur percentage, carbon residue, sulfated ash percentage, cetane number, and acid number. The latter comprises, for example, methanol, free glycerol, total glycerol, phosphorus contents, water, and esters content. Chromatography and spectroscopy are the mainly used analytical methods for BD analyses, but procedures based on physical properties are also available [34]. Furthermore, it is important to mention that in most chromatographic analyses, mainly gas chromatography (GC) has been applied to methyl and not to ethyl esters [29].

As the demand for vegetable oils for food has increased tremendously in recent years, hence, the contribution of nonedible oils such as jatropha, *Moringa oleifera*, rice bran oils, etc. can play an important role for BD production. In view of the limited petro-oil resources and rapidly growing energy demands of the world, there is an extensive need to take immediate initiatives for exploring alternative energy sources to meet the domestic needs and
reduce the dependence on imported fossil fuels. In view of the future perspectives of biofuels, the present book chapter was designed with the main purposes to assess the feasibility of BD production from multi-feedstock vegetable oil sources.

### 1.2 History of the Use of Vegetable Oil in Biodiesel

The idea to use vegetable oils as fuels for diesel engines dates back to more than one hundred years. Historically, Rudolf Diesel, the inventor of diesel engine, at the Paris Exhibition in 1900, conducted engine tests, for the first time, on peanut oil [22, 35]. At that moment Diesel said, “The use of vegetable oils for engine fuels may seem insignificant today. However, such oils may in course of time be as important as petroleum and the coal tar products of the present time.” Today, over a century later, the scientific community is working to fulfill his dream by considering potential benefits of BD as an alternative fuel to petrodiesel for future uses.

### 1.3 Feedstocks for Biodiesel Production

All over the world, the usual lipid feedstocks for BD production are refined vegetable oils. In this group, the oil of choice varies with location according to availability; the most abundant lipid is generally the most common feedstock. The bases for this are not only the desire to have an ample supply of product fuel but also because of the inverse relation between supply and cost. Refined oils can be comparatively costly under the best of conditions, compared with petroleum products, and the choice of oil for BD production depends on local availability and corresponding affordability. The four oil crops clearly dominate the feedstock sources used for worldwide BD production. With a share of nearly 85%, rapeseed oil is by far leading the field, followed by sunflower seed oil, soybean oil, and palm oil [36]. Apart from the “great four” – rapeseed oil, sunflower seed oil, soybean oil, and palm oil in BD production – other edible plant oils have also successfully been transesterified to produce BD.

The choice of raw material used for BD production in a specific region mainly depends on the respective climatic conditions. Thus, rapeseed and sunflower oils are mainly used in the European Union [37], palm oil predominates in BD production in tropical countries [38, 39], and soybean oil [40] and animal fats are the major feedstocks in the United States. FA ester production has also been demonstrated from a variety of other feedstocks, including the oils of coconut [41], rice bran [42], Thespesia populnea [43], safflower [44], palm kernel [45], *M. oleifera* [46], *Citrus reticulata* (mandarin orange) [47], *Jatropha curcas* [48], Ethiopian mustard [13], *Cynara cardunculus* [49], *Hibiscus esculentus* [50], maize [51], *Cyperus esculentus* (Barminas et al. [52]), *Prunus mahaleb* [53], kapok [54], tobacco [55], milkweed [7], *Yucca aloifolia* [56], *Oleum papaveris seminis* [57], *Pongamia* [58], *Brassica napus* [59], *Citrullus colocynthis* [53], rubber seed oils [60], palm FA distillate [61], the animal fats, tallow [7, 62], lard [63], and waste oils [64, 65]. As such, any animal or plant lipid should be a ready substrate for the production of BD. Such features as supply, cost, storage properties, and engine performance will determine whether a particular potential feedstock is actually acceptable for commercial fuel production.