

THE PRODUCTION OF BIODIESEL AND RELATED FUEL ADDITIVES

Editors:

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**Recent Advances in Renewable
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(Volume 6)

***The Production of Biodiesel and
Related Fuel Additives***

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PREFACE

Progress in fuel and energy has always been the subject of interest in the world economy, transportation, and fuel market. The interest is growing exponentially due to the limit of traditional fossil fuels, out of many renewable energies. Biofuels are just one source, but they still count as an important one.

This book “Recent Advances in the Production of Biodiesel and Related Fuel Additives” deals with the many new developments along with substantial limitations in the production as well as use on a global level. Besides the basic introductory part, the book covers current approaches and plausible future approaches to be addressed. This book contains 12 chapters providing a comprehensive and all-encompassing reference about biodiesels which will be helpful to students, teachers, researchers, and professionals. In this book, readers will find fundamental concepts of important biofuels and the current state-of-the-art technology for their production.

Chapter 1 gives basic information about biodiesel and related fuel additives. The history, availability, and viability of biodiesel, as well as its relevance, technical properties, and prospective replacement for diesel fuel in internal combustion engines along with the biodiesel fuel additives, are discussed in this chapter.

Chapter 2 deals with the utilization of soybean oil as a source of biodiesel. Soybean oil has been widely used for oil production and the leftover is commonly used as animal meal. The chapter includes a detailed discussion of the catalysts used for biodiesel production from soybean oil feedstock.

Chapter 3 shares the production of biodiesel from palm (Arecaceae) oil. Different types of catalysts developed for the transesterification of palm oil are well-audited in this chapter. Meanwhile, chapter 4 discusses the details of biodiesel production from *Jatropha curcas* oil. This chapter describes the *Jatropha* system, oil extraction, and catalytic conversion by traditional and modern processes, downstream processes, and final product characteristics.

Chapter 4 discusses the production of biodiesel from *Jatropha curcas* oil. *Jatropha* oil is found in a wide range of fatty acid methyl esters profile, and free fatty acids content. Consequently, the adjustments of upstream processes must be considered. *Jatropha curcas* biodiesel is basically focused on small or medium-scale production. This chapter describes the *Jatropha* system, oil extraction, and catalytic conversion by traditional and modern processes, downstream processes, and final product characteristics.

Chapter 5 focuses on catalytic pyrolysis of biomass to biodiesel. This chapter focuses on recent catalytic methods for the conversion of biomass to biofuels namely biodiesel along with the pros and cons of the methods.

Chapter 6 describes biodiesel biocrude production from algae as a feedstock. The algal biocrude produced using hydrothermal liquefaction is a drop-in kind of biocrude. It can be processed, independently or in combination with fossil-based crude, in existing refinery infrastructure to produce green biofuel.

Chapter 7 deals with the homogeneous and heterogeneous catalysis in biodiesel production. The efficiency, limitations, advantages of all kinds of catalysts, comparison of their properties, and suitability in the transesterification are also discussed.

Chapter 8 emphasizes microwave-assisted synthesis of biodiesel and related fuel additives. This book chapter covers a broad spectrum of scientific and instrumental aspects of microwave radiation methodology in chemical synthesis, the practical approach of the microwave reactor design, the production of different biofuels and additives using microwave techniques, along with the advantages and several limitations of this methodology.

Chapter 9 throws light on the ultrasound-assisted production of biodiesel. The use of ultrasounds in biodiesel production has a growing interest due to several advantages; as it significantly reduces the reaction time and avoids the use of heating, reaching similar or higher FAME yield. This chapter explores the basis of ultrasounds and their use in biodiesel production, its main features, and challenges.

Chapter 10 provides an in-depth analysis of several cooking oils as a biodiesel feedstock, including their salient qualities and most common pests.

Chapter 11 emphasizes the waste-derived catalysts for biodiesel production. This chapter gives brief knowledge about such heterogeneous catalysts derived from waste in particular.

Chapter 12 deals with the synthesis of solketal: a potent fuel additive from glycerol, a by-product of biodiesel industries. This chapter gives new insight into the researcher to find a new pathway to utilize the by-product in an environmentally friendly manner.

Chapter 13 provides a brief review of the catalytic production of biodiesel specifically the esterification and transesterification processes. The advantages of solid acid catalyst, its activity, performance, efficacy, and reusability are well discussed in this.

With the high spirits of hope, we are serving this book to society and our all stakeholders. But at the same time, we are open and thankful for the comments and suggestions that will be received from our readers towards the improvement of this book.

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CHAPTER 1**Biodiesel and Related Fuel Additives: A Brief History****Supongsena Ao¹ and Samuel L. Rokhum^{1,*}**¹ *Department of Chemistry, National Institute of Technology Silchar, Silchar 788010, India*

Abstract: The discovery of the diesel engine by Rudolf Diesel in the mid-19th century is where the origin of what finally came to be known as “biodiesel” lies. Since then, numerous approaches have been suggested to utilize pure or blended, straight vegetable oils or their derivatives for the production of biodiesel. The availability and viability of biodiesel, as well as its relevance, history, technical properties, and prospective replacement for diesel fuel in internal combustion engines including various types of biodiesel fuel additives, are all discussed in this chapter.

Keywords: Bioglycerol, Fuel additives, Glycerol carbonate, Homogeneous and heterogeneous catalyst, Solketal.

1. INTRODUCTION

The demand for more ecologically friendly and sustainable means to power modern economies has recently been fueled by the rise in global energy consumption, as well as the depletion of fossil fuel supplies and increasing CO₂ emissions [1, 2]. In light of this, the search for alternative renewable energy has been sparked by worries about the depletion of fossil fuels and the degradation of the environment. Researchers are interested in biodiesel as a potential alternative fuel among the contenders. The biofuel biodiesel, which emits less carbon monoxide (CO), sulfur dioxide (SO₂), and unburned hydrocarbons (HC) than petroleum diesel and is non-toxic, has the potential to replace petroleum diesel as an alternative energy source at present [3]. Several countries now have laws and policies that promote the use of alternative fuels, such as biodiesel, by providing incentives and setting stringent limits on the usage of each fuel. Here, in this chapter, we will discuss about the insights into biodiesel, its properties, production techniques, and various types of fuel additives.

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1.1. Background of Biodiesel and Related Fuel Additives

1.1.1. Biodiesel

Although the term “biodiesel” was first used in 1988, the practice of substituting vegetable oil for diesel as a fuel dates back to 1900. The invention of the diesel engine by Rudolf Diesel laid the foundation for what eventually came to be known as “biodiesel” [4]. In the year 1900, the Paris World Fair hosted the first diesel engine demonstration for those nations growing oil crops, especially those in Africa in the 1940s, knowing that vegetable oils could be used to power the diesel engine which gave them a sense of energy independence [5, 6]. This was particularly true during the years of World War II when cottonseed oil exports were outlawed even in Brazil so that it might be utilized as a diesel alternative. In China, kerosene and gasoline were produced using tung oil and other vegetable oils [7]. In addition, India and the USA also did research on the conversion of a range of vegetable oils to diesel in response to the lack of fuel during World War II [8]. A passenger bus that ran between Brussels and Louvain in 1938 used palm oil ethyl ester as fuel, and vegetable oil was used as fuel by many nations, including Brazil, Argentina, China, India, and Japan, during World War II (1939–1945), when petroleum fuel supplies were disrupted. However, when the war was over and petroleum was once more accessible and affordable, vegetable oil fuel was forgotten. In Europe, commercial production of biodiesel began in 1992, with Germany being the biggest producer [4]. In Kansas City, Missouri, biodiesel was first produced commercially in the United States in 1991 [4]. Since then, the output of biodiesel increased globally from 1 billion liters in 2001 to 6 billion liters in 2006 [9]. One of the biggest non-governmental organizations for the development of standards, American Society for Testing and Materials (ASTM) International released ASTM Standard D6751 in 2001 as a biodiesel standard. This gave fleet owners and engine manufacturers confidence that biodiesel would meet their standards for quality.

1.1.2. Fuel Additives

With the exponential growth in biodiesel production, a significant amount of crude glycerol has been produced as a byproduct around the world. Glycerol could be a building block with many useful derivatives. For every 100 kg of biodiesel, glycerol accounts for 1 kg (w/w) or, 1.05 pounds of glycerol are created for every gallon of biodiesel produced [10]. This means that a factory producing 30 million gallons of glycerin annually will produce around 11,500 tonnes of 99.9% pure glycerin. In 2019, the market for glycerol was estimated to be worth USD 2.6 billion [11]. Additionally, from 2020 to 2027, it is predicted to rise at a compound annual growth rate of 4.0% [11, 12]. Therefore, finding new

applications for both crude and refined glycerol is vital for researchers. Value-added chemicals such as solketal, glycerol carbonate, acetin, can increase sales for the existing biodiesel industry and improve the sustainability of the process [13, 14].

1.1.2.1. Solketal

The very first solketal production occurred in 1895 when glycerol and acetone were combined in a batch reactor under acidic circumstances with hydrogen chloride as a catalyst [15]. In the years that followed, fresh ideas for achieving greater output in less time were put forth. In the beginning, homogeneous catalysts and strong acids such as sulfuric, hydrochloric, hydrofluoric, and phosphoric acids were used in the process, while para-toluene sulphonic acid (pTSA) was also utilised by Newman and Renoll back in 1945 [16]. The issues with using homogeneous catalysts are well documented and include difficult product separation, equipment degradation, and significant worries regarding effluent disposal [17, 18]. The use of heterogeneous catalysts adheres to several of the Green Chemistry principles, including safer solvents, accident prevention through auxiliary, naturally safer chemistry, and less hazardous chemical synthesis [19]. Since then, many production techniques for the production of solketal in batch and continuous methods of operation have been proposed, with the majority of studies concentrating on batch techniques, particularly in relation to overcoming the thermodynamic limitation [20, 21].

1.1.2.2. Glycerol Carbonate

Another value-added glycerol derivative that is popular for industrial use is glycerol carbonate (GC). Glycerol 1,2-carbonate is a naturally occurring substance that is environmentally benign, harmful, and biodegradable. Its chemical name is 4-(hydroxymethyl)-1,3-dioxolane-2-one [22, 23]. Due to its two separate functional groups, hydroxyl and cyclic carbonate, it is a five-membered cyclic carbonate with distinctive chemical reactivity. Vieville *et al.* [24], reported the first study on the carboxylation of glycerol utilizing zeolites and ion exchange resins as catalysts in the presence of supercritical CO₂. Zeolite Purosiv, zeolite 13X, and Amberlyst A26 were utilised as the catalysts, and they successfully accelerated the reaction of glycerol adsorption onto the solid catalyst and ethylene carbonate dissolution in a supercritical CO₂ solution. Glycerol and supercritical CO₂ are combined with methanol to achieve 100% solubility, which promotes a more homogenous process and increases yield [25]. Since then, considerable research on the carboxylation of glycerol utilising homogeneous and heterogeneous catalysts has been explored.

Production of Biodiesel from Soybean Oil

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Abstract: The necessity for clean, green, and renewable energy resources has gained tremendous attention from industries and academia. This is due to the alarming depletion of fossil fuels and the growing environmental concerns associated with their extensive use. Petroleum reserves are on the verge of extinction, and biodiesel is a promising alternative with better fuel properties compared to petroleum diesel. Various oils and fats have been employed as feedstock to produce biodiesel. Soybean oil is highly regarded as the most appealing feedstock due to its extensive cultivation for oil production as well as its widespread use as an animal meal. In previous years, various studies have been carried out to produce biodiesel with the use of various technologies and methods. Among the number of methods, transesterification is the most common method of biodiesel synthesis. This paper typically reviews the catalytic transesterification of soybean oil for biodiesel production and its fuel quality. This review also explores the effectiveness of various catalysts in converting soybean oil to biodiesel. Several reactors have been utilized by numerous researchers to optimize reaction parameters, which is also thoroughly highlighted in this review.

Keywords: Enzyme, Homogeneous, Heterogeneous, Reactor, Soybean oil, Transesterification.

1. INTRODUCTION

An alternative is a solution to every problem. Among the various alternative sources of renewable energy, biodiesel competes with petrodiesel as an alternative to depleting fossil fuels. The extensive usage of fossil fuels for petroleum and coal to meet the increased demands for industrialization and urbanization has led to the reduction of carbon energy sources, alteration of the earth's outer layers, and sinking of the ground surface [1]. The burning of fossil fuels results in air pollution, acid rain, enhanced CO₂ levels, and climate change. Considering the

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environmental impact of burning fossil fuels and the increase in price along with its demand, biodiesel has garnered significant attention in the last few decades. Biodiesel is regarded as renewable, environmentally benign, biodegradable, and compatible with diesel engines. Biodiesel is advantageous to petrodiesel as it has a high flash point, high cetane number, low viscosity, and good lubricity. Biodiesel reduces CO, HC, and particulate matter emissions in the exhaust gas as it contains 10% to 11% oxygen in comparison to diesel fuel [2]. Also, biodiesel is stated to emit 70% less CO₂ than petrodiesel and 15% reduced CO₂ emission with the addition of 20% biodiesel to petrodiesel [3]. Shortages in food crops and high processing costs are some of the demerits associated with the production of biodiesel.

Biodiesel contains fatty acid alkyl esters produced from oils or fats. Biodiesels are obtained *via* dilution, pyrolysis, micro-emulsification, and transesterification methods. The transesterification method is the most commonly used method, which constitutes the chemical reaction between triglycerides (present in oils or fats) and alcohol in the presence of a suitable catalyst that improves the reaction rate and yield. Transesterification can significantly alter the physical properties of triglycerides, which are inclined to induce deposition on the engine, injector-coking, and piston ring sticking [4]. The most commonly used alcohol for this reaction is methanol as it is cheap and available. However, various studies had been carried out using ethanol, butanol, and propanol which were efficient in the reaction. Various kinds of catalysts are employed for the transesterification reaction. These catalysts belong to homogeneous (acidic or basic), heterogeneous (acidic or basic), and biological sources. They are effective in completing the reactions within a specific time duration, using energy and capital to generate purified products.

Various kinds of edible and non-edible feedstocks are available. Edible or vegetable oils consist of more unsaturated fatty acids and low-temperature fluidity, which makes them suitable for biodiesel production. Among the varying vegetable oils, Soybean (*Glycine max* L.) oil is the most widely used feedstock for biodiesel synthesis. Soybean oil is produced from the soybean tree, which is also called soya bean and belongs to the pea family (known as Fabaceae or Leguminosae). It is high in protein, essential for diet, and provides necessary ingredients for various chemical products. There are varieties of soybean plants consisting of various shapes and sizes. They can grow up to 2^{1/2} feet in height. The plant bears about 80 pods, which weigh about 27.2155 kg. Soybeans have been demonstrated to have originated in Southeast Asia, but they were first cultivated by the Chinese at around 1100 BC. Eventually, by AD, Japan and other countries began to cultivate soybeans. Soybeans are mostly used as oil seeds as they contain

18% oil. It requires a warm season and loamy well-drained, fertile soil for cultivation.

According to the soybean outlook (October 2021) [5], Brazil holds the top position as the largest producer of soybeans followed by America, Argentina, and China. As per the report, India ranks as the fifth largest producer, with 11.22 million tons of soybean produced in 2019-20. India contributed 3.98% to the world's production in 2019-20, which is represented in Fig. (1). Madhya Pradesh, Maharashtra, Rajasthan, Karnataka, and Telangana are the leading soybean-producing states in India, which is estimated to have acquired 121.76 lakh hectares of land during 2021-22. The extraction of soybean oil from the soybean oilseed is mainly done by the pressing process, where the soybean is heated and the oil is extracted (Fig. 2). Another method is the direct solvent extraction method, which is one of the most used methods. However, implementing this process requires a significant capital investment. Gerde *et al.* [6] mentioned that in commercial processes, soybean oils have been extracted using petroleum distillate fractions of acetone, carbon disulfide, ethanol, trichloroethylene, and water. However, among the various organic solvents, hexane is the most commonly used solvent as it is cost-effective. Also, using water as an aqueous solvent for soybean oil extraction is a good method if environmental and human health are to be considered, but using water results in low oil recovery (60%). This is due to emulsification resulting from the interaction of protein with the cotyledons. Another extraction method is the enzyme extraction method, which is mentioned by Cheng *et al.* [7], which requires the demulsification of emulsion and hydrolyzation of the protein to generate high oil recovery (90%). Enzyme recovery is desirable as this technique does not require post-processing processes that are required in hexane extraction and screw pressing processes.

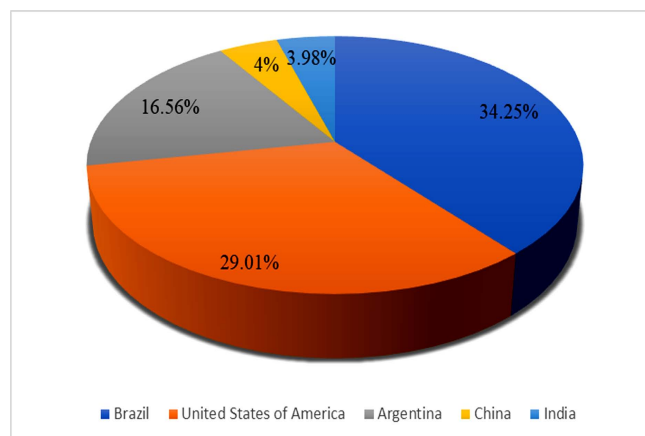


Fig. (1). Pictorial representation of world soybean production in 2019-20 [5].

Production of Biodiesel from Palm (*Arecaceae*) Oil

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Abstract: Biodiesel from palm (*Arecaceae*) oil, is a fuel that can be useful in compression start motors and, further, in diesel-based motors without any mechanical modification. It is an effective and promising feedstock to produce biodiesel for advanced generations. It also contains different phytonutrients that can be isolated earlier for biodiesel generation. So far, *Arecaceae* biodiesel transformation using the catalytic pathway has been well investigated. Among these catalysts, homogeneous base catalysts are the most commonly used, even though they face serious issues when FFA (Free Fatty Acid) content becomes high as observed in the case of CPO (Crude Palm Oil). The alternative tactic to produce *Arecaceae* biodiesel eco-friendly is using advanced catalysts such as heterogeneous (acid and base), enzymatic, and supercritical processes. However, these strategies have never been promptly accessible at the industrial site as the catalysts get deactivated easily, and thus such strategies demand extra high efforts. This chapter reviews the generation of biodiesel from *Arecaceae* oil, offering an eco-friendly pathway.

Keywords: *Arecaceae* oil, Biodiesel, Crude Palm Oil, Free Fatty Acid, Transesterification.

1. INTRODUCTION

Biodiesel being “a clean and renewable source” offers a promising fuel substitute that can be used in any direct injection motor without modifying the existing operating technology. Biodiesel is synthesized from the plant feedstock with higher biodegradability, increased lubricity, and insignificant sulfur content than traditional fossil fuel [1]. Biodiesel is made from a blend of green oils and diesel fuel [2]. According to Edmunds, its production has been expanded from 25 million gallons in 2005 to 1.7 billion gallons in 2013. Nowadays, biodiesel is mixed at a rate of 5% or less into almost all the diesel fuels that are sold within the United States. This attracts more and more researchers towards increasing its

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efficiency and sustainability. The essential source for biodiesel within the United States is soyabean oil, even though it is also produced from “ Yellow oil” (utilized edible oil from cooking), Green growth, Canola, and animal fat [3, 4], which in turn offers accountable employment. The biofuels can be categorized as biochar, green oils, biodiesel, bioethanol, biogas including biohydrogen [5]. Biodiesel synthesized from diverse triglycerides is an effective fuel and burns more efficiently and smoothly, whereas, petro-diesel with similar synthesis provides more energy, and low efficiency in burning. The American Society for Testing and Materials (ASTM) characterized biodiesel as a mono-alkyl ester produced from different lipid feedstock such as green oils, animal fats, etc. Besides this, biodiesel (recognized as a fuel-added substance) is also enrolled with the United States Environmental Protection Office (EPO) [6].

With the excessive use of fuel worldwide and the subsequent rise in fuel cost, biodiesel offers a more viable solution in the petroleum area [5, 7]. Advance studies have confirmed that excessive utilization of bio-oils and animal oils as fuel is commercially not viable because of their high atomic mass, low stability, increased kinematic thickness, higher viscosity, jellying, and staying of the oil [8] which lessens their usage in motors. To overcome such issues, strategies like mixing with petrol diesel, micro emulsification, thermal breakdown, and transesterification which are mentioned in Table 1 [8, 9] were employed.

Table 1. Production of biodiesel utilizing diverse technology [10-14].

Methodology	Key Method	Merits	Demerits
Mixing	Bio or animal oils were first preheated and mixed with fuel in 10 - 40% w/w. This blend was connected and converted into a diesel machine.	<ul style="list-style-type: none"> • Simple implementation • Non-polluting • Specialized adjustment is not required 	<ul style="list-style-type: none"> • Unstable • High thickness • Low instability • Deficient fuel combustion • Difficult to monitor • Destitute atomization
Microemulsion	The vegetable or animal oils were mixed in a solvent and wetting agent, for the specified desired consistency.	<ul style="list-style-type: none"> • Contamination free • Easy procedure 	<ul style="list-style-type: none"> • Low stability • Deficient combustion • Deficient carbon testimony • High thickness

(Table 1) cont....

Methodology	Key Method	Merits	Demerits
Pyrolysis	The vegetable or animal oils were heated to decay at an elevated temperature above 355°C.	<ul style="list-style-type: none"> • Successful method • Easy procedure • Contamination free 	<ul style="list-style-type: none"> • Need high heat-up • Costly apparatus required • Generate small yield of biodiesel
Trans-esterification	The animal oils and fats were treated with alcohol and a soluble base or acid catalyst, to obtain biodiesel and glycerol, which needs further refinement.	<ul style="list-style-type: none"> • High Transformation • Cost Effective • Mild reaction condition • Appropriate for Industrial level generation 	<ul style="list-style-type: none"> • Broad division and a large number of refinement steps • Plausible outcomes

Fats and oils are triglycerides, which are essentially water-insoluble, hydrophobic substances containing 1:3 moles of glycerol: fatty acids. The % weight of fatty acids found in vegetable oils is summarized in Figs. (1 and 2) highlights its chemical properties like acid value, phosphorus value, and peroxide values.

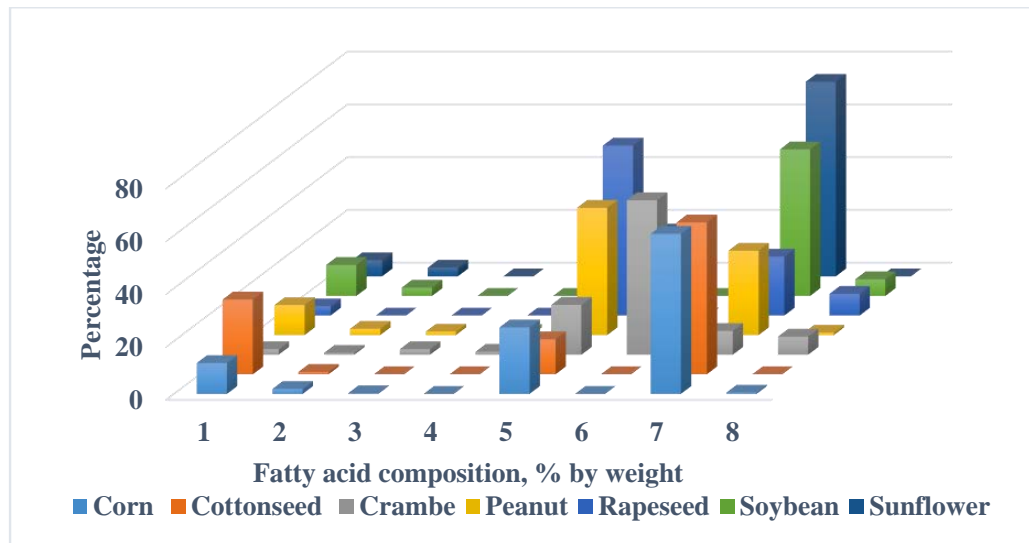


Fig. (1). Fatty acid composition of vegetable oils.

Production of Biodiesel from *Jatropha curcas* Oil

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Abstract: In biodiesel production, first-generation fuel faces the problems of using food as a feedstock which has its limitations like the destruction of vital soil resources, deforestation and negative impact on biodiversity, and the use of much of the available arable land. At the same time, the high cost of feedstock also restricts its widespread use. Keeping all those points in mind, researchers are focusing their attention on the production of biodiesel from non-edible vegetable oils such as *Jatropha curcas* L. The present chapter deals with the harvesting methods of *Jatropha curcas* L, extractions, and properties of the blends used in detail.

Keywords: Enzymatic extraction, Glycerolysis, *Jatropha curcas* L, Supercritical CO₂ Subcritical fluid extraction, Sedimentation.

1. INTRODUCTION

One of the existing concerns in the modern world revolves around the feasibility of each country being able to establish itself as self-sufficient for its energy demand and to produce energy sustainably. For this reason, the search for alternative fuels is still a challenge today. The production of energy from biomass as biofuels has a remarkable impact on the social, economic, legal, and environmental spheres. For several years, there has been interest not only in their use but also in their production. This has led to an evolution of the regulatory legislation of the energy sector concerning this subject.

Transesterification is the process of turning large, branching triglycerides into smaller, straight-chain methyl esters (FAME) in the presence of a solvent, using an alkali, acid, or enzyme as a catalyst. The main target of the transesterification process is the reduction of oil viscosity to levels close to those of standard diesel fuel and to meet the regulatory standards in all related issues.

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Biodiesel, mainly composed of FAME is a sustainable liquid biofuel that might be used to totally or partially replace standard diesel fuels. It has the potential to reduce pollutant emissions and may be used without the modification of compression ignition engines. It has been shown to have a positive impact, mainly as a partial substitute for diesel fuel in engines, being used as a blend.

The quality of this biofuel is affected by different factors. One factor is related to the fact that transesterification is a step reaction; another has to do with the inherent properties of alkyl esters that make up biodiesel. The presence of foreign materials and fuel storage conditions can also change the properties of the fuel and therefore its quality. All of these issues are addressed through specification limits reported in international biodiesel standards. The most widely used are the ASTM (American Society for Testing and Materials) D6751 standard and the European EN (European Committee for Standardization) EN14214.

In the field of production and use of biofuels, there are four generations. The first generation of biodiesel feedstocks is focused on edible oils, which have been used at large scale for the production of biofuels; in the case of biodiesel, the main sources are rapeseed and soybean oils. However, their use at large scale has raised concerns such as the permanent debate about food vs. fuel, environmental concerns such as the destruction of vital soil resources, deforestation and negative impact on biodiversity, and the use of much of the available arable land to produce them.

On the other hand, the cost of feedstock is often assumed to contribute to 75-80% of the entire cost of biodiesel produced. Consequently, much effort is being invested into developing biodiesel from non-edible vegetable oils such as *Jatropha curcas* L, assumed as a second-generation biofuel since it produces non-edible oil (in most cases) and does not need good quality soil to grow, among other features to be analyzed in the present chapter.

1.1. Main Characteristics of *Jatropha curcas* L. Variability and its Influence on Biofuel Properties

Jatropha curcas or physic nut (JC), native of tropical America, has been later introduced into Africa and Asia and is now cultivated worldwide. *Jatropha* is a genus of approximately 175-200 plants, shrubs, and trees, from the family of Euphorbiaceae. It is resistant to drought and produces seeds containing up to 40% mass of oil [1, 2]. *Jatropha* seeds have oval geometry with their size ranging from 1.5 to 2.0 cm in length and from 1.0 to 1.3 cm in width.

The worldwide use of *Jatropha curcas* L. to produce biodiesel is not widespread yet. Nonetheless, there is an interest yet to produce biodiesel from it since the

Jatropha oil seed has a high content of non-edible oil (considering most of the *Jatropha* strains). The presence of some anti-nutritional factors such as toxic phorbol esters and a high content of stearic acid render *Jatropha* oil unfit for edible purposes [3].

Jatropha curcas oil contains more than 75% unsaturated fatty acid, which is reflected in the pour and cloud point of the oil. Some of the physical and chemical properties of *Jatropha curcas* L oil and its derived FAME are shown in Table 1. The most important parameters contained in the main international biodiesel standards are also presented in Table 1. The information compared in Table 1 clearly shows that the conversion of *Jatropha curcas* oil to biodiesel has a significant impact on the physical properties of the biofuel, closing its features to the standard requirements.

Table 1. Physical and chemical properties of *Jatropha curcas* oil and derived FAME.

Physical Properties	JC oil	JC FAME	ASTM D7751	EN 14214
Density (g/m ³)	901 [2]	880 [3]	-	860-900
Melting point (°C)	5 [2]	-	-	-
Kinematic viscosity (mm ² /s at 40°C)	34.6 [2]	4.8 [3]	1.9-6.0	3.5-5.0
Cetane number	41-51 [2]	50-56 [4]	47 min	51 min
Iodine number	103 [2]	93-106 [4]	115 max	120 max
Heat of combustion	39.63 [2]	39-41 [4 - 6]	-	-
Flashpoint (°C)	225 [7]	162-188 [3, 4, 8]	93 min	120 min
Pour point (°C)	1 [7]	2-6 [3, 6]	-	-
Sulfur content (%)	0.0002 [9]	0.011-0.004 [3, 4]	0.05 max	0.01 max
Acid Value (mg KOH/g)	25-28 [7]	0.4 [3]	0.5 max	0.5 max

Nevertheless, attention must be paid to the influence of the variability in FAME profile found in the JC oil among different strains or soil and climatic conditions, which could contribute to significant changes in certain fuel properties [2, 10] such as cetane number, with a high impact on combustion.

Ewunie *et al.* [11] demonstrated in a comparison of JC oil and biodiesel from different regions of the world that properties or parameters such as cetane number, density, viscosity, flash point, pour point, acid value, and calorific value have significant variability with the source. This behavior is not particular to *Jatropha curcas* but to many biomasses. In this respect, it is important to point out that according to this, *Jatropha curcas* oil or the biodiesel produced from it cannot be

Recent Advancements in Catalytic Thermochemical Conversions of Biomass into Biofuels: A Comprehensive Review

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Abstract: The expeditious increase in population has led to prioritizing the use of biological sources as biofuels. The biofuels have been converted into different fuels by the virtue of green and sustainable approach. Other than the conventional sources of raw materials, specialized energy crops, some varieties of algae, seaweed and microalgae have been reported to be potential sources of biofuels. In recent years, various methods of conversion of organic matter into biofuels have been reported. High energy investment along with the added cost of solvent or catalyst is included in the thermochemical methods. Meanwhile, the biochemical route suffers from the drawbacks of lengthy cycle period and comparatively reduced efficiency in the bulk breakdown of the recalcitrant biomass. Hydrothermal routes have been employed to improve the overall efficiency of the biochemical process. The integration of thermochemical and biochemical routes also may lead to inhibition of microorganisms by the catalysts or mediated solvents. This review paper focuses on the recent catalytic methods for the conversion of biomass into biofuels namely biodiesel along with the pros and cons of the methods.

Keywords: Biochemical process, Biofuels, Conventional sources, Catalytic methods, Hydrothermal routes.

1. INTRODUCTION

The ever-increasing demand of energy and power due to the expeditious rise in population has increased the pressure on the existing sources of fossil fuels and added to the problem of environmental pollution by their combustion. In view of the various challenges like environmental pollution, limited energy resources,

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poor quality of air, greenhouse effect and associated global warming, the need for research and development of renewable sources of energy, maximisation of the utilization of non-conventional sources of energy into potential sources of energy, and the development of biofuels by catalytic conversion of biomass will be a sustainable and promising solution. Nevertheless biomass, unlike other sustainable energy sources is capable of dual production of energy and chemicals. In the past, energy production *via* thermochemical conversion technologies has not been developed to its full potential due to the abundance and easy availability of fossil fuels. Recently these technologies are now being developed to their maximum potential in view of the environmental concerns caused by the combustion and depletion of the fossil fuels. The global demand for energy generation from the biomass-based sources of energy have become popularised by the virtue of their renewability, cost-effectiveness, eco-friendliness and maximum abundance. The most fundamental thermochemical conversion technologies include pyrolysis, gasification, Torre faction, and hydrothermal liquefaction (HTL). It has been surveyed that biomass meets the demand of primary energy sources for nearly half of the world's population [1]. The never-ending availability and easy accessibility of biomass ensures sufficient energy production for the future generations. With the advancement of technology, the utilization of biomass as processed biofuels is also on pace. The biofuels have been converted into different fuels like biogas, biodiesel, bioethanol, biohydrogen, *etc.* by the virtue of green and sustainable approach. The raw source of biofuels comprises forest produce, farming waste, industrial pulp waste and sewage, biodegradable waste, waste wood from furniture, waste from construction sources, crop stubbles, wild vegetation, *etc.* Most recently, specialized energy crops, some varieties of algae, seaweed and microalgae have been reported to be potential sources of biofuels. Biofuels can be widely classified as per their constitution and physical forms into solid, liquid, and gaseous biofuels. The untreated raw biomass and their residues are generally referred to as solid biofuels. The finished products after proper chemical treatment of solid biofuels yield liquid biofuel like biodiesel and gaseous biofuel like syn-gas. In the recent years, various methods of conversion of organic matter into biofuels namely gasification, pyrolysis, fermentation, thermochemical approach, hydrothermal approach, *etc.* have been reported. Every method has its own advantages and disadvantages. High energy investment along with added cost of solvent or catalyst is included in the thermochemical methods. Meanwhile, the biochemical route suffers from the drawbacks of lengthy cycle period and comparatively reduced efficiency in the bulk breakdown of the recalcitrant biomass. Hydrothermal routes have been employed in the pre-treatment stage for the preparation of the appropriate biomass feedstock to improve the overall efficiency of the biochemical process. The integration of thermochemical and biochemical routes also may lead to the

inhibition of microorganisms by the catalysts or mediated solvents [2]. With the development of new technologies, there has been a spike in the development of biofuels *via* non-food biomass, primarily by catalytic conversion of lignocellulosic biomass. Lignocellulose to date is the most abundant and affordable source of raw material to be employed to produce advanced biofuels that are greener and ecologically safer. Pyrolysis is greatly used for its simpler approach and better productivity. Unfortunately, bio-oil suffers from many disadvantages like substandard quality, corrosiveness due to low pH, high viscosity, non-miscibility with other fuels, and incompatible for storage due to chemical instability. These drawbacks hinder the commercialization of bio-oil and consequently bring down its economic value. These important limitations can be subjugated by modifying the elemental composition of the bio-oil. Catalytic pyrolysis has arisen in recent years to be one of the most promising techniques for upgrading bio-oil into advanced biofuels. Till date the development of renewable production technologies *via* the use of nanoscience is the biggest challenge. Nanoparticles are known for their large surface to volume ratio which makes them efficient potential catalysts. Catalysts are known for their metal content, porosity, and behaviour towards different substances [3, 4]. Many methods have been reported by several researchers regarding the development of nanoparticles with different functionalities. These materials may be used directly or in combination as oxides, carbon, or zeolites on solid surfaces. Impregnation, chemical vapor deposition, precipitation and electrochemical deposition are some of the general methods used for the synthesis of nanoparticles. Although the conventional methods of precipitation and impregnation are well explored, they suffer from the problem of controlling the particle size. Chemical vapor deposition despite being a costlier method is a method of choice in the electronics industries. Electrochemical deposition is widely used by virtue of its inexpensive approach without any specificity in temperature and concentration. This ensures particle size control and the chemical nature of the newly deposited dimensional nanomaterial [5, 6]. Conventional methods like the impregnation process and precipitation have been largely used for the synthesis of catalysts *via* biomass. The other controlling factors in terms of the size and functionality of the nanoparticles are the pH of the solution, concentration of biomass and the reducing agent, and calcination temperature [7]. Recent upgradations in the process of biomass gasification have increased syngas production by reducing the tar formation. Furthermore, the desirable characteristics namely increased catalytic efficiency and greater specific surface area have given a boost to the preparation of heterogeneous catalysts for biodiesel production *via* biomass. Thus, with respect to the background of the development of catalytic techniques, our review paper focusses on the recent advanced methods for the conversion of biomass to biofuels along with the pros and cons of each method (Fig. 1).

Algal Biocrude as Drop-In Feedstock for Green Fuel and Green Chemicals

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Abstract: Algal biocrude, derived through hydrothermal processing of algal biomass, is a drop-in feedstock and can be processed in the refining and petrochemical infrastructure developed for fossil crude. Algal biomass, the raw material for algal biocrude, has lipids, proteins, and carbohydrates as main constituents. It does not have lignin/cellulose. The presence of lignin/cellulose in non-algae biomass makes downstream processing difficult in the existing fossil-based infrastructure. Algal bio crude has the potential to be the source of biogenic feedstock, not only for making green fuel but also for making numerous chemicals. The research work on algae for food products and energy by fuel began as early as the 1940s. However, even after years of efforts, the algal technology for low-value, high-volume commodity products, such as fuel, is not yet commercialised mainly due to economic reasons. This chapter provides insight and a balanced perspective on commercialization of the algae-based pathways for green fuel and green chemicals.

Keywords: Biofuel, Commercialization, Drop-in biofuel, Microalgae, HTL, Scale-up.

1. INTRODUCTION

Algal oil has the distinct advantage of high-density energy storage in liquid form. This feature makes it the most promising feedstock for sustainable biofuel, particularly in hard-to-abate aviation sector. The algal biocrude, obtained through hydrothermal liquefaction (HTL) of algal biomass, can be co-processed and refined in the existing as well as new refining facility developed for fossil crude processing. The algal oil obtained after refining of algal biocrude, can be used in the existing engines, independently or in a blended form. This drop-in characteristic of algal biocrude, enabling the use of existing processing infrastructure including engine, makes the algal oil truly a sustainable fuel.

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Algal biomass, the raw material for algal biocrude, grows much faster (5x to 10x) than the land-based plants. The productivity of genetically modified and engineered microalgae is claimed to be as high as 20x times than any existing terrestrial crop [1, 2]. Algae can be grown and produced using abundant solar energy, climate-unfriendly CO₂, non-potable water, and non-arable land. The photoautotrophic algae use light to grow and produce biomass. There are heterotrophic algae, which use sugars as an energy source and grow in the dark, without light. Mixotrophic algae make use of both, light and sugar to grow and produce biomass. Algae are typically categorised as microalgae, and macroalgae. Microalgae include cyanobacteria (similar to bacteria, generally known as blue-green microalgae) and green, red, and brown algae. Microalgae are recognised as the potential raw material for fuel applications [4 - 6]. Main constituents of algal biomass are lipids (*i.e.*, polyunsaturated fatty acids), proteins, carbohydrates, nucleic acids, and natural pigments. Besides these main components, algal biomass also contains inorganic ash and metals, which influence the quality of the algal biocrude and its downstream refining. Most wild microalgae species are rich in protein, with concentration ranging from 30% to 71% on moisture and ash free (MAF) basis. The carbohydrate and lipid concentration range from 4% to 58% and 2% to 25%, respectively [7 - 9]. Efforts have been underway to increase the lipid content of algae to as high as 80%, using various techniques including genetic modification of wild algae [10].

Algal biomass can be processed to develop various products, such as nutraceuticals, human foods, animal feeds, pigments, fertilizers, biofuels and bioplastics. Realising the potential of algae as a source of food and fuel, exploratory research on algae began in the 1940s [11]. By the 1960s, commercial production of *Chlorella* and *Spirulina* (*Arthrospira platensis*) started for human consumption as nutritional supplements, after almost two decades of development before achieving the commercial viability. Even today, microalgae are produced commercially only for high-value, low-volume human nutritional products (*e.g.*, *Chlorella*, *Spirulina*), and not for any low-value and high-volume commodity products, such as fuel oil or proteins.

The algal biomass is typically cultivated in open raceway ponds (ORP), photobioreactors (PBR) on large scale and in fermenters on a relatively small scale for niche products [12, 13]. Algal culture, when grown in ORP and PBR cultivation systems, has more than 99% water, with algal biomass concentration of less than 0.1 wt%. Harvesting microalgae biomass from extremely dilute growth media is very different and difficult than harvesting land-based terrestrial plants. Achieving the concentration of even 20% algae in a slurry, after harvesting, dewatering has been found to be challenging. This is mainly due to almost the same density of biomass as that of growth medium, and extremely

small size of algal cells [14]. This limitation in dewatering algae slurry poses challenges in downstream processing of algae to fuel and bio-products. For example, extracting oil or proteins from algae slurry after dewatering and drying is excessively energy intensive and uneconomical for commodity products.

Historically, oil extraction from dry seeds of terrestrial plants is done using mechanical expeller, solvent extraction, or a combination thereof. Drying algae to remove over 80% water and then extracting oil from algae, particularly low-lipid algae (2 to 25% lipid content), using conventional oil extraction technologies, is very energy-intensive process and impractical at large scale [15-17]. Extracting oil from wet slurry without drying has been studied to overcome the limitation of oil extraction from dried algae. Wet extraction methods such as extraction using microwaves, pulsed electric field, and ultrasonic techniques are found to be promising from long-term perspective. However, these methods of lipid extraction from wet algae are yet to be developed beyond the laboratory scale [18]. Further investigation is essential to scale up these technologies to enable them to be deployed commercially for extracting the lipids from wet algal biomass.

Another limitation of extracting only lipids from algae is the overall low yield of oil. Even assuming a lipid content of 25% (upper end of low-lipid algae) and extraction efficiency of 80% (practical limit) to 90% (aspirational), the overall oil yield would be 16% to 23% [19 - 21]. The significant amount of algal biomass remaining after lipid extraction in wet algae slurry will need further processing to monetise the full value of algal biomass. In view of the above challenges, the concept of maximising the biofuel yield by processing whole algal biomass attracted attention in the 2010s [22 - 24]. Pyrolysis, though it is a proven process, needs dried biomass. This rules out its possibility of algal oil application. HTL is acknowledged as a process of choice for its technical robustness and feedstock flexibility over competing technologies [25, 26].

HTL is a thermo-chemical process that converts biomass, waste biomass, and any organic waste, wet or dry, at elevated pressure and temperature, to liquid drop-in fuel. The process uses water at near-critical temperature and pressure, as a reaction medium. Initial research on HTL started early in the 20th century, with well-documented work in the 1940s to process terrestrial biomass for fuel oil. In the 1970s, after the worldwide oil crisis, research on HTL again increased and a few pioneering pilot plants were installed and tested for woody biomass with limited success. HTL development got a boost with algal research in the 2000s, wherein HTL was found to be the process of choice for the conversion of wet and whole algae biomass without drying to drop-in algal biocrude [27, 28].

Homogeneous and Heterogeneous Catalysis in Biodiesel Production

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Abstract: One of the prospective alternative sources of energy is biodiesel, which is obtained from conventional and substandard sources *via* various methods. One of them is transesterification in the presence of a catalyst. The catalyst may be either harmonized or varied. This chapter will give detailed information about the various catalysts used in biodiesel synthesis. The chapter focuses on the efficiency, limitations, and advantages of all kinds of catalysts and their properties, and appropriateness in the transesterification method. An extensive study has been carried out on the usage of homogeneous and heterogeneous catalysts for biodiesel production. The data reviewed reflects that those homogeneous catalysts are proficient in converting oil with low FFA and feedstock that contains water. On the other hand, heterogeneous catalyst gives a range of selectivity on high FFA content and water adaptability. It is known that the numbers of acidic or basic sites control the properties of heterogeneous catalysts. Zirconia and Zeolites-based catalysts by some modifications, can be used as both basic and acidic catalysts. Heterogeneous catalysts derived from waste have received an important role in biodiesel production. Lately, high catalytic activities under optimum operating conditions have been recognized of Nanocatalysts. This review article gives elaborated information on various materials used as catalysts.

Keywords: Biocatalyst, FFA, Homogeneous catalyst, Heterogeneous catalyst, Nanocatalyst, Transesterification.

1. INTRODUCTION: ABOUT ENERGY CONSUMPTION

As the global population is constantly increasing, the requirement for energy is rising simultaneously. Energy in all forms is the sustaining factor for the entire world. If we see globally, by the year 2021, China will be the biggest buyer of the primary source of energy and India stands third in the position. The major portion of the total energy utilized is used in industries, power generation, transportation

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(by all means), households, *etc.* A large portion of the energy is obtained from fossil fuels such as coal, oil, and fracked gas [1]. But all aforesaid sources are non-conventional sources so a substitute energy source is needed presently. The energy expenditure globally by various conventional and non-conventional fuel sources in 2019 is shown using a bar graph in Fig. (1) below.

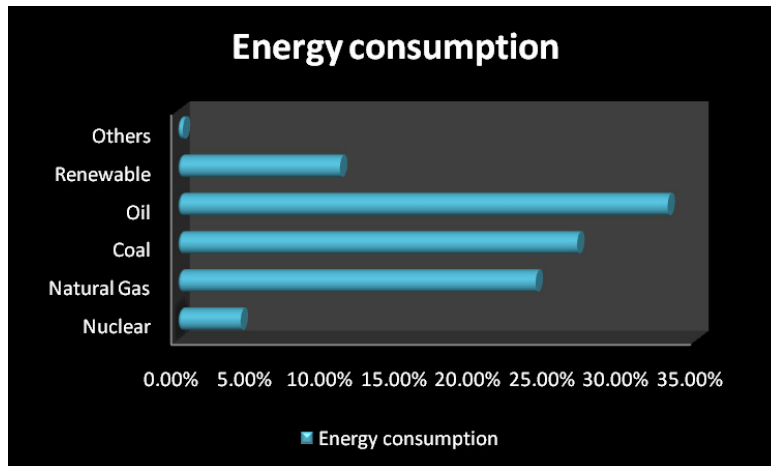


Fig. (1). Energy consumption globally by various fuel sources.

The increasing usage and demand of fossil fuels has led to many environmental issues and climate change, greenhouse effect is the most challenging one. The world's daily manufacture plus demand of crude oil in million barrels per day is shown below in Fig. (2) using a bar graph.

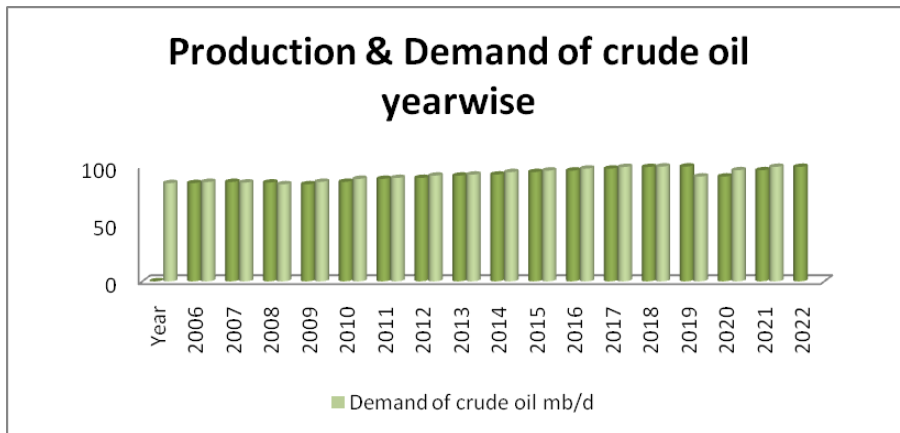


Fig. (2). World's daily manufacture plus the demand of crude oil in million barrels per day.

It is depicted from the graph that the raw oil manufacture has increased from 82.5 mb/d to 94.9 mb/d in 2019 and simultaneously the demand has also increased from 85.3 mb/d to 99.7 mb/d. As per the US energy information and administration, the abrupt decrease in the pattern of production and usage of crude oil in the year 2020 and 2021, respectively, is due to compact monetary activity related to covid-19 pandemic. Nevertheless, the oil delivery and demands are expected to raise to 99.4 and 104.1 mb/d with the current usages. If the statistics remains unchanged, then these natural resources will last 200, 70, and 40 years, respectively.

Excessive handling of conventional fuels has also led to the increase in Carbon-di-oxide emission as shown in Fig. (3) (Environmental Protection Agency, 2021). As per the World Health Organization report, Carbon-di-oxide emission has increased by 0.5% in 2019. This variation is less than half as compared to the average of the last decade, which is 1.1% per year. This emission occurs mostly from the combustion of fossil fuels required for transportation, and industrial use. This further leads to an increase in the human death rate due to heart strokes, heart attacks, lung cancer and chronic diseases in addition. People from developed and developing countries are more prone to such diseases as 91% of the total population resides in the area exceeding the WHO air quality guidelines [2]. Moreover, excessive usage of fossil fuels has also contributed to environmental concerns.

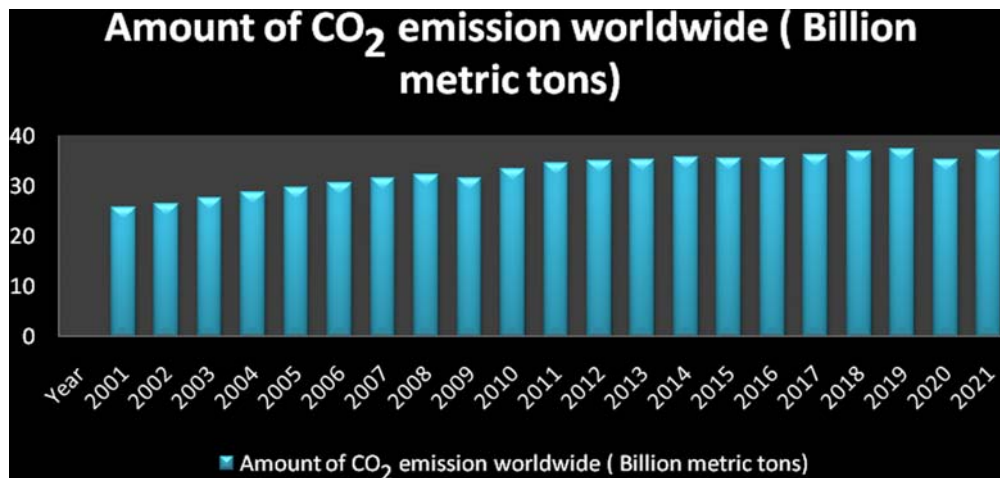


Fig. (3). CO₂ Emission in the last two decades.

It should be taken into consideration that for a sustainable and balanced environment on the earth, each of us is required to look for ways and means for

CHAPTER 8**Microwave-Assisted Synthesis of Biodiesel and Related Fuel Additives****Chandrashekhar Pandhurnekar^{1,*}, Himani Pandhurnekar² and Babita Yadao²**¹ *Department of Chemistry, Shri Ramdeobaba College of Engineering and Management, Ramdeo Tekdi, Gittikhadan, Nagpur, India*² *Department of Chemistry, Dada Ramchand Bakhru Sindhu Mahavidyalaya, Nagpur, India*

Abstract: With the rising cost of non-renewable petroleum fuels, growing environmental concerns, and energy shortages, industrial-scale production of biofuels and their additives using readily available resources has gained a lot of attention. The cost-effective and commercial development of clean energy sources is expected to be aided by various renewable biomasses for the synthesis of biofuels or fuel additives. Microwave techniques with various precursors could be one of the strategies for the synthesis of biofuels or gasoline additives, with advantages such as being very energy-efficient, less time-consuming, high selectivity, a greener approach, and high-yield producing procedures. This microwave effect is caused by microwave radiations interfacing with the molecules of solute, solvents, or catalysts throughout the reaction.

This book chapter covers a broad spectrum of scientific and instrumental aspects of microwave radiation methodology in chemical synthesis, the practical approach of the microwave reactor design, the production of different biofuels and additives using microwave techniques, and the advantages, and several limitations of this methodology.

Keywords: Biofuel additives, Biofuel, First-generation biofuel, Green chemistry, Microwave, Microwave synthesis of biofuel, Second-generation biofuel.

1. INTRODUCTION**1.1. Basics of Green Chemistry**

The whole world is presently under the carcinomatous grip of degrading conditions of soil, air, water pollution, and many more [1]. Due to the degradation

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of the biosphere, life on this planet, “The Earth” has become miserable [2]. This all happened due to the use of conventional chemical principles to satisfy various demands of ingredients with varied applications. Because of all these problems, there is a massive demand to minimize the practice of supplementary noxious materials. The faster progress in the field of science and technology, nowadays, is directed to economic development in the world, but due to this economic development, there is also emerging environmental degradation, which is revealed by climate change, like the issue of ozone holes, global warming, accumulation of non-destructive organic pollutant in all parts of biospheres, *etc* [3]. Since the 1940s, environmental problems started to emerge concerning the growth of industrial activities. Due to this rise in environmental problems and concerns, companies have changed their protocols for conventional production and product development ways *via* sustainable processes to the present [4]. The present situation demands a solution to balance the use of natural resources and environmental conservation. Only the way to achieve these is by transforming from grey chemistry to green chemistry [5, 6].

The term green chemistry was first introduced by Paul T. Anastas when a few decades into the 20th century the reduction or elimination of hazardous substances was ensured in a chemical process or the production of hazardous or toxic intermediates or products, such as feedstock, reagents, solvents, products, and by-products. It also includes the use of sustainable raw materials and energy sources for this manufacturing process [7 - 10]. The U.S. Presidential Green Chemistry Challenge, March 1995 [11], describes the concept of green chemistry as “It is the use of chemistry which is the mediator for pollution prevention, the maximum level of the danger for organization grading as mentioned in Pollution Prevention Act of 1990” [12]. The subject of green chemistry has a new concept of preparation, and a new methodology, and involves the use of chemical materials in such a way as to minimize danger to the health of humans and the environment as follows [13 - 15]:

1. Ecologically gentle chemistry
2. Clean Chemistry
3. Atom Economy

According to Sir Paul Anastas, the concept of green chemistry is based upon different 12 principles that include various aspects such as either minimizing or removing the utilization of any risky material in the designing, production, and application of chemicals. The branch of chemistry that we call green chemistry comprises tools and techniques that would help in research related to the formation of chemical products and processes, which include the elimination of harmful chemicals and also harmful and toxic products for the progress of eco-

friendly and development of more efficient products along with the less wastage. Because of all these reasons nowadays, green chemistry has become a very important tool in the field of chemistry [16]. One of the most significant aspects of Green Chemistry is also the concept of its design. Its design contains different parameters like novelty, planning, and systematic conception. The Twelve Principles of Green Chemistry include “design rules” which help chemists achieve the intentional goal of sustainability. Green Chemistry is employed by careful preparation of chemical synthesis and molecular design to decrease adverse consequences [17, 18]. The branch of Green Chemistry explains how chemists made the protocols for next-generation materials and methods, hence with this improvement, it will be beneficial for the health of humans and the environment.

The green chemistry concept has an approach that is governed by 12 principles given by Sir P. Anastas, which are still followed by chemists. The main aim of these concepts is to minimize the environmental and occupational hazards inherent in industrial activities [19, 20], which are listed below.

- i. Prevention,
- ii. Atom economy,
- iii. Less hazardous chemical syntheses,
- iv. Designing safer chemicals,
- v. Safer solvents and auxiliaries,
- vi. Design for energy efficiency,
- vii. Use of renewable feedstocks,
- viii. Unnecessary derivatization,
- ix. Catalysis,
- x. Design for degradation,
- xi. Real-time analysis for pollution prevention, and
- xii. Inherently safer chemistry for accident prevention.

1.2. Microwave Technology: Fundamentals, Advantages of Microwave-assisted Synthesis

Concerning the concept of green chemistry, one of the very important methods is microwave-assisted reactions. Microwave irradiation serves as a substitute for the classical conventional methods for heating or introducing energy into the reaction system. Microwave-conducted reactions are very fast, clean, economical, environmentally benign, and eco-friendly [21 - 24]. It is the best way to produce smart materials having the dimensions of nanoscale, which is at the speed of light and enables control of the matter at the nanoscale. Nowadays, researchers have started to think about reforming synthetic strategies that are essential requirements

Ultrasound Assisted Biodiesel Production

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Abstract: The production of biofuels has a great impact on the economy and society. Biodiesel is a sustainable liquid fuel used for partial or full replacement of standard diesel fuel, and its production generates valuable by-products. The use of ultrasound in biodiesel production has a growing interest due to several advantages; it significantly reduces the reaction time and avoids the use of heating, reaching similar or higher FAME yield. The application of ultrasounds in homogeneous and heterogeneous catalysis processes is reported to be technically feasible, but several issues are to be considered such as the corrosion effect on sonotrodes, and the effect of ultrasounds waves on solid catalyst surface and pores. Combining it with microwave irradiation might be an effective procedure for the intensification of biodiesel production, especially with heterogeneous catalysis. Technical challenges are associated with the design of large-scale reactors in which both types of energy could be applied concurrently with cost reduction. This chapter explores the basis of ultrasounds and their use in the production of biodiesel, its main features, and challenges.

Keywords: Biodiesel, Cavitation, FAME, Transesterification, Ultrasounds.

1. INTRODUCTION

The main energy source for power and transportation comes from fossil fuels. Particularly in the transportation sector, most of the energy used is based on liquid conventional fuels, establishing a strong dependency on fossil fuels, market instabilities, and the environmental impact generated by their combustion. The increasing energy demand has led to high production, and consumption, associated environmental problems, and global warming. In this context, biofuels such as biodiesel have been considered an important part of the energy mix in the transportation sector and are a technology implemented in the world transportation market. In this context, biodiesel has been for several years the ideal substitute for like-diesel fuels for engines.

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Among several advantages compared to diesel fuel, biodiesel is four times easier to degrade, emitting 86% fewer greenhouse gases, it is a renewable energy source, and it has proven lubricant properties, besides other associated benefits. Even in a future scenario of electric mobility, liquid biofuels will always be a part of the energy mix to consider since it is a proven way to solve the final disposal of Waste Cooking Oils (WCO), but also because biodiesel is an important bioenergy figure, produces energy but is also an invaluable source of biomaterials with multiple applications.

Currently, the large-scale and local-scale production of biofuels (mainly bioethanol and biodiesel) is very well established as already commercial technology and as an energy and economic sector. The development of more efficient and friendlier technologies considering the complete life cycle (from the cradle to the grave) allows us to assert that these liquid biofuels have a present and a future.

Biodiesel production generates a by-product (glycerol), which is even more valuable in society and the economy than biodiesel due to its wide industrial applications. One of the main features of bioenergy is that it is always a way to produce energy, biomaterials, and valuable chemicals. The glycerol worldwide marketed is refined and contains less than 5% impurities, which means that the product obtained from biodiesel production is not yet a commercially demanded product.

Crude glycerol is produced through different routes. These include processes such as transesterification of triglycerides, saponification, or hydrolysis, generating a product with impurities of 10-30% [1]. Its impurities are glycerides, alcohol, salts, catalysts, and water. There are many industrial applications for glycerol such as the food industry, pharmaceuticals, and cosmetics. Nevertheless, many of the products that include the use of glycerol in formulations are very sensitive to its quality and content. Due to this, the glycerol obtained from biodiesel production is in less demand in main industries without a refining process.

Instead of developing a glycerol refining process, it can contribute to finding effective outputs for this transesterification product. For instance, the in-situ use of crude glycerol in a local agro-industrial system is a good alternative. In this respect, its use as a fuel additive could be in part to tackle this problem. Another alternative to find a use for crude glycerol is its use as a fuel by direct combustion, which represents an additional way of using biofuels at the local scale.

One chemical route to use glycerol in the self-industry is to apply glycerolysis reaction, reaction or process, which is used to convert Free Fatty Acids (FFA) into acyl glycerol by esterification. This way, glycerolysis is the esterification process

in which glycerol acts as alcohol to convert FFA into glycerides. In this reaction, as a reaction product, water is also obtained. Glycerolysis is normally run when the FFA content is higher than 5% [1], nevertheless, the most traditional procedure to reduce the FFA content is a previous esterification process.

Biodiesel is obtained mainly by transesterification of vegetable oils or animal fats. In the transesterification chemical reaction, a triglyceride molecule reacts with three molecules of a short-chain alcohol (methanol or ethanol), in the presence of a catalyst. In this process, three reactions occur consecutively, degrading the triglyceride to di, mono, and finally glycerol. As a result of the chemical reaction, three molecules of fatty acid methyl or ethyl esters (FAME or FAEE), depending on the alcohol used, and one molecule of glycerol are produced.

Esterification occurs between FFA and methanol (MetOH) in the presence of a catalyst. The last one could be a strong acid or base, and the result is the obtaining of biodiesel (FAME) and water. It constitutes the first step when biodiesel is prepared using a feedstock that has a relative or high content of FFA and a homogeneous catalyst. On the other hand, transesterification is the main process in biodiesel production. The main transesterification mechanism is shown in Fig. (1).

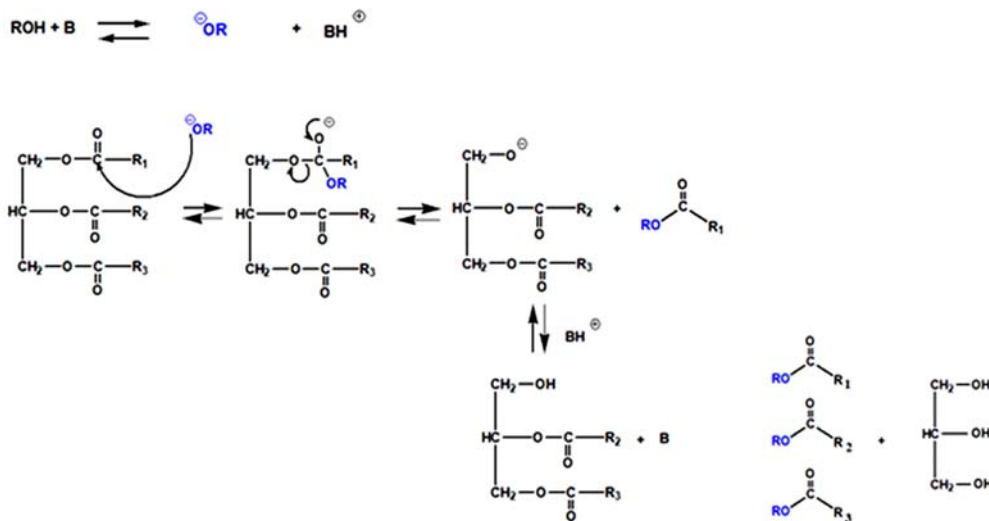


Fig. (1). Transesterification reaction mechanism.

The main alcohol used in transesterification is methanol, but others can also be used. Nevertheless, the best reaction rate and FAME yield are correlated to the use of methanol. Alternatives to the use of methanol are ethanol, n-butanol, and isopropanol. The proper selection of alcohol may take several issues into account,

Waste Cooking Oil to Biodiesel - A Review

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Abstract: The phrase “Biodiesel from Waste Cooking Oil” refers to a broad wide range of unconventional fuels generated from different kinds of oils and fats. The American Society for Testing and Materials (ASTM) defines biodiesel as “monoalkyl esters of long chain fatty acids,” which can be produced by the transesterification of vegetable oil, animal fat, or recycled cooking oil. The key factor leading to fossil fuel reserves being depleted is the increasing demand for these resources. Increasing the development of biomass fuels like biodiesel might help get us out of this jam. Oil molecules are reacted with alcohol and a catalyst to produce methyl esters in the transesterification process during biodiesel production from cooking oil. In Colombia, palm oil and methanol are used to produce biodiesel and it shares the second place with Colombia as Latin America's top ethanol producer.

Waste cooking oil disposal causes several environmental issues. In addition, sewer overflows and the subsequent spread of illness might be the consequence of years of pipe wear and tear. As a renewable and biodegradable biofuel, biodiesel has the potential to reduce environmental damage by displacing the need for fossil fuels. Palm biodiesel, either on its own or blended with diesel fuel, is effective in lowering carbon dioxide (CO₂) and nitrogen oxide (NO_x) emissions, respectively.

This chapter discusses the transesterification process as a method of creating biodiesel. It consists of three sequential and reversible reactions. It begins with a conversion from triglyceride to diacylglycerol, then continues to monoglyceride and glycerin. In particular, this chapter provides an in-depth analysis of several cooking oils, including their salient qualities and the most common pests. Most biodiesel originates from oilseed plants, such as palm, rapeseed, canola, sunflower, soy, and animal fats. The creation of biodiesel, however, may utilize anything that includes triglycerides. Used oil from the kitchen may be recycled into biodiesel at a low cost.

Keywords: Biodiesel, Chemical catalyst, Esterification, Transesterification, Waste Cooking oil (WCO).

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1. INTRODUCTION

The fast expansion in the global population and the concomitant growth in economic activity have resulted in a dramatic rise in the need for new forms of energy. With rising concerns about global warming and diminishing fossil fuel reserves, there is a pressing need to identify sustainable and renewable fuel alternatives. As a result, several sectors are competing for customers to keep up with the demand. It is becoming more desirable to construct these fuel alternatives despite the unsustainable nature of the procedures now used to produce, transport, and use energy derived from fossil fuels throughout the globe. Ethanol, biodiesel, and methane are all biomass-based diesel substitutes that are gaining popularity due to their low production costs, high regenerative potential, and low environmental impact. In addition, they may be made with little effort in rural regions, which have a high need for modern forms of energy but a low supply [1].

A kind of alternative fuel that is made close to home from sustainable resources and burns cleanly is biodiesel. Edible and non-edible oils from a variety of plants are utilized extensively in India's biodiesel industry. Biodiesel may be produced from many types of edible vegetable oils, including soybean, canola, peanuts, corn, jatropha seeds, olives, cotton seeds, sunflowers and maize. Research suggests that these oils might be utilized as effective diesel replacements [1 - 3]. Several studies have demonstrated that these oils may effectively replace diesel [4 - 6]. However, as a direct result of their high demand in the food business, it is difficult to use these oils to manufacture biodiesel on a large scale due to the high cost of production [7]. There can be no assurance of biodiesel's long-term economic sustainability without first ensuring that the cost of feedstock is reduced [8]. Using less expensive feed sources, such as waste cooking oils (WCO) and vegetable oils (VO), which are either inedible or take less labor to acquire, can lower the price of this fuel. One alternative is to consume less gasoline in general. The exciting idea of WCO as a possible alternative for edible vegetable oil (EVO) is supported by the fact that it can be acquired for a lot less money than EVO made specifically for human use [9].

Disposal of used cooking oils and fats presents serious problems in many parts of the globe. Used cooking oil, if properly used and managed as a fuel alternative, might be a part of the solution to this environmentally damaging issue that has the potential to be helpful to the economy as well as the environment. Putting the used cooking oil down the drain is the law in many modern nations. Countries like these have legislated this restriction into law for full enforcement. These laws are meant to discourage individuals from acting in such a way. According to the US government's "Energy Information Administration (EIA)," Americans dump 100 million gallons of spent CO every day, or nearly nine pounds per person annually.

This estimate was made using the statistic that the typical American weighs around 155 pounds. Estimates place Europe's daily CO₂ collection at between 0.4 and 0.7 million gallons [10].

1.1. Definition

In its broadest sense, the word “biodiesel” encompasses any fuels that may be thought of as replacements for diesel and which are produced from renewable biomass. Biodiesel is a cleaner alternative to diesel oil since it does not contain any Sulphur, is oxygenated, is non-toxic, is biodegradable, and is excellent for the environment. Biodiesel is an alternative fuel made from long-chain fatty acid mono-alkyl esters (FA). These FAS are obtained from non-exhaustible resources such as VO, animal fat, and used cooking oil that have been verified to be B100 compliant. Biodiesel must also follow all applicable regulations and standards, such as those established by the European Committee for Standardization and the American Society for Testing and Materials. Transesterification [11] is frequently employed in the production of biodiesel [12].

Vegetable oils have considerably different injection and atomization qualities compared to petroleum-based diesel fuels due to their much greater viscosity. In large part, this is because of the noticeable disparity in their viscosities. These engines use a fuel injection system that is very sensitive to gasoline viscosity changes. One solution is to reduce the viscosity of VO fuel, which might mitigate these issues. Biodiesel production from VO is a practical solution to all VO-related issues. The high viscosity of the fuel causes a number of problems, but four different methods; micro emulsification, dilution, pyrolysis, and transesterification are used to discover answers.

Transesterification is the most widely used method for converting VO and lipids into mono-alkyl esters, popularly known as “biodiesel.” The transesterification of VO results in a “methyl ester” with enhanced heating value, decreased viscosity, and a higher cetane number than the original VO. This is because the method works to reduce the methyl ester's viscosity. This may allow the fuel to be ignited sooner and consumed for a longer length of time, leading to reduced particle emissions.

1.2. Historic Background

In 1893, Dr. Rudolf Diesel is widely regarded as the creator of the first diesel engine [13]. If VO were to be used, this motor's power output would increase significantly. The idea of VO as a fuel source has been discussed widely ever since. In contrast, diesel engines were adapted to run on petroleum distillate, a fuel that was both cheap and abundant. The late 20th century saw an increase in

Waste-Derived Catalyst for Biodiesel Production

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Abstract: Due to its eco-friendly and renewable characteristics, biodiesel has become a promising alternative to energy sources. However, the issue associated with traditional biodiesel production is the expensive production cost on the industrial scale, which is primarily caused by raw materials. Thus, the catalyst plays a crucial role with the objective to speed up the overall biodiesel production rate and lower the production cost. Recently, numerous studies on different kinds of catalysts used in the production of biodiesel have been carried out. Therefore, this chapter offers a detailed overview of biodiesel production by analyzing the latest trends that utilize biomass waste-derived catalysts.

Keywords: Biomass-derived catalyst, Biodiesel, Renewable energy, Transesterification.

1. INTRODUCTION

Biodiesel is an excellent option for the replacement of conventional fuel because it is produced from renewable sources and emits fewer pollutants [1]. Around 150 000 000 GWh were utilized globally in 2015; the total primary energy consumed (TPEC) is predicted to increase by a startling 57% in 2050 [2]. Nowadays, non-renewable fossil fuels are used mostly in the transportation of products and services, which contributes significantly to the global economy [3, 4]. In terms of overall primary energy consumption, petroleum resources account for 80% of the energy used. To ensure sustainability, research has been performed to hunt for alternate fuels [5]. The major components of the oils were free fatty acids and triglycerides, which were converted into biodiesel through the transesterification and esterification processes [6]. When compared to fossil fuels like gasoline and

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diesel, biodiesel has many environmental benefits like low toxicity, low sulfur content, less emissions of CO and CO₂, as well as being biodegradable and renewable [7].

The utilization of homogeneous catalysts in the present biodiesel manufacturing, however, has various drawbacks, including unrecoverable catalysts, pricey separation stages, and substantial wastewater output [8, 9, 10]. There are various methods available today to manufacture biodiesel without the issues mentioned above, including enzymatic catalysts [11] and supercritical condition transesterification [12]. Due to the extremely high pressure and the costly enzyme's synthetic cost, which results in higher operational expenses, the economic viability of both approaches is severely hampered. Currently, heterogeneous catalysts are an option [13], particularly those made from organic materials like waste biomass, which are the subject of intensive research and show promising results [14, 15]. As they have demonstrated high catalytic activity, high surface area, high reusability, and reasonable temperature stability, in industrialized-scale biodiesel production, carbon-based heterogeneous catalysts are considered a good substitute for homogeneous catalysts [16]. While converting oil feedstock into biodiesel, the heterogeneous catalyst's activity increased to functionalize carbons with several groups of alkali metals, alkoxides, sulfonic acids, enzymes, and transition metals [2, 17]. The agricultural sector's waste biomass can be used to develop carbon-based catalysts for biodiesel production [18].

The current chapter summarizes extensive research on the synthesis of biodiesel and evaluates the most recent developments related to the application of catalysts made from waste biomass. To improve the biodiesel production framework, we have carefully reviewed a collection of experimental data from related literature. Moreover, our research is focused on improving the industrial economic viability of large-scale biodiesel production, which heavily depends on the catalyst's cost and effectiveness and further resolving issues with heterogeneous catalyst separation, preserving the environment while satisfying energy demand.

2. A SUMMARIZATION OF BIODIESEL (FAME) PRODUCTION

The transesterification process is a conventional method for biodiesel production [19]. This procedure is also known as “alcoholysis” [20]. Triacylglyceride and alcohol, typically methanol, are transesterified in the presence of a homogeneous or heterogeneous catalyst to produce fatty acid alkyl esters (biodiesel) and glycerol as a by-product [21]. Alcoholysis, another name for transesterification, is the method used to distinguish an alcohol from the constituent esters of a separate alcohol, this method is analogous to hydrolysis. The viscosity of triacylglyceride

has been decreased using this technique frequently. The methanolysis happens when the aforementioned transesterification procedure uses methanol as a solvent, the possible reaction is shown in Fig. (1). The reaction stoichiometry shows that methyl esters, with glycerol as a by-product, may be produced from alcohol (three moles) and triacylglyceride (one mole) [22]. The process of transesterification requires at least 1.6 times more alcohol because it can be reversed. When producing biodiesel on an industrial basis, CH_3OH and NaOCH_3 are frequently employed due to their cheaper costs and superior reactivity. However, as shown in Fig. (2), the esterification process, which results in ester formation when alcohols and carboxylic acids react, results in biodiesel production from all free fatty acids (FFA) contained in vegetable oil [23]. These esterification and transesterification processes are typically carried out in two pots [24]. Generally, in an acid catalyst-assisted esterification process, vegetable oils with a high FFA content are converted into ester fatty acid methyl ester (FAME), and last, triglycerides are transformed into FAME using a basic catalyst-aided transesterification procedure [25]. The simultaneous transesterification and esterification of vegetable oil (with high FFAs) into FAME are very desirable to speed up and lower the cost of biodiesel synthesis [26].

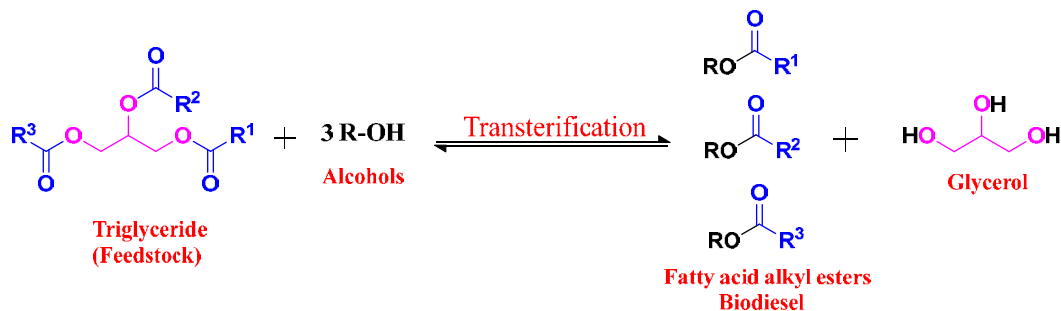


Fig. (1). Transesterification of soybean oil to produce biodiesel (FAME).



Fig. (2). Esterification of soybean oil to produce biodiesel (FAME).

3. DIFFICULTIES WITH EXISTING BIODIESEL PRODUCTION FROM CURRENT CATALYSTS

Reducing harmful emissions like greenhouse gas (GHG), which pose major dangers to human health and other environmental issues, was the fundamental reason for using biodiesel as a replacement fuel [27]. Traditionally, the production

Synthesis of Solketal: A Potent Fuel Additive from A Glycerol, A By-Product of Biodiesel Industries

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Abstract: To overcome the problem associated with conventional fuels and the need for alternative fuels, the production of biodiesel increased and was promoted by government policies and air pollution-controlled laws. The by-product glycerol produced from the biodiesel industry is in massive quantity and all of its quantity is not utilized by the pharmaceutical and cosmetic companies, therefore a huge amount of it is discarded as waste which is a disadvantage of biodiesel production. Hence this led the researcher to find a new path to utilize it in an environmentally friendly manner and therefore glycerol is being used to produce solketal which are fuel additives and enhance the properties of the fuel. Therefore, glycerol is employed as feedstock for the production of solketal. Generally, batch and continuous processes are used to synthesize solketal in which the continuous method is the most promising one. Various catalysts are also employed to increase the yield of solketal from glycerol. Thus, the reaction of glycerol with dimethyl ketone using various catalysts (homogeneous and heterogeneous) in different reactors takes place. This chapter gives insight into the development of biodiesel production and increased usage of propane-1,2,3-triol (glycerol) into more valuable product solketal using various advanced catalysts. The synthesis of solketal using continuous process is a vast area and one can find many more environmentally friendly methods to synthesize it with low cost at industrial and commercial scale.

Keywords: Biodiesel, Fuel additives, Glycerol, Ketalization, Solketal.

1. INTRODUCTION

Due to the decrease in fossil fuel reserves, there is a need for alternative fuels. Since conventional fuels are the main source of energy in developing countries and are the main cause of carbon dioxide emissions that result in global warming, therefore researchers have to find new alternative sources of energy, that lower greenhouse gas emissions and are also environmentally friendly [1]. Some alternative energy sources are geothermal, wind, and solar energy, and among all

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of them, biofuels have gained much importance as a renewable energy source because they decrease GHG emissions and the climate change-related impact from the transport sector [2, 3]. Biodiesel is one of the forms of biofuels, which is produced after the transesterification process carried out on oils and fats. Since biodiesel is degradable, not a dangerous and sustainable energy source and it has properties that are similar to diesel fuels and therefore the European Union suggests its blend form to be used and hence its production has increased. During the transesterification process, glycerol is obtained as a byproduct which has a variety of uses as lubricants, emulsifiers, humectants, and in cosmetics but all such utilization cannot decrease the high amount of propane-1,2,3-triol which is produced during the process [4, 5]. The estimated quantity of propane-1,2,3-triol produced by each volume of biodiesel is about 10.2 wt%. The biodiesel industry generated approximately 69% of glycerol, therefore it was assumed that by 2027 the growth rate of glycerol will be increased by 5.2% and hence a sustainable way has to be obtained for the utilization of glycerol. Glycerol has many applications [6]. It is widely used in dairy, polymer, medicinal and cosmetic industries. Different chemical processes like etherification, hydrogenation, dehydration, esterification, oxidation, oligomerization, acetalization, and pyrolysis are applied to convert glycerol into valuable products like fuel additives, propene, glycerol carbonate, acetins, *etc.* The versatility and chemical activity of glycerol are high because of three –OH (hydroxyl) groups and therefore, it can be converted into fuel additives and other chemicals. Glycerol has a high boiling point and high viscosity due to the presence of hydroxyl groups [7, 8]. Table 1 shows different types of glycerols and their applications.

Table 1. Types and application of glycerol [9].

S. No.	Types of Glycerol	Uses	Sources
1	Crude Glycerol	After removing the impurities, it can be applied for fermentation, oligomerization, oxidation, biomass liquefaction <i>etc.</i>	During the transesterification process produced as a by-product.
2	Purified Glycerol	Used for the textile, food and chemical industry	FCC and USP grade
3	Technical Glycerol	Industrial applications	Purified glycerol

It is observed from above Table 1 that to obtain valuable products, purification of crude glycerol is required and therefore there are various methods by which it can be purified such as filtration, adsorption, crystallization *etc.* It can then be applied for various industrial and commercial applications and usually glycerol 99.4-99.6% is sold dry [9].

The ketalization of propane-1,2,3-triol using various catalysts forms solketal which is an oxygenated compound and a universal solvent and used as an intermediate in the organic synthesis reactions. It is an odorless and colorless liquid and has less toxicity. When added to the gasoline obtained from catalytic cracking, it increases its octane number and decreases the gum formation [10, 11]. Therefore rather than 2-methoxy-2-methylpropane (MTBE), solketal can be added to gasoline in an environmentally friendly manner and since the price of raw materials like glycerol and acetone is low; therefore the synthesis of Solketal is economically more beneficial and has industrial importance. It decreases harmful emissions, and increases viscous flowability and oxidation stability of fuels. Solketal, which is also known as 1, 2-isopropylidene glycerol, is a value-added chemical and is used as a suspending agent and solubilizing in medicinal industries. Further, it has many applications like in paints it is used as a solvent to replace volatile organic compounds (VOCs), the formation of different formulations, industrial products like cooling and cleaning agents, inks, *etc* [12, 13].

Fig. (1) shows the routes of different products obtained by the different reactions of glycerol. Through propane-1,2,3-triol valorization paths, the application of crude and refined propane-1,2,3-triol has been suggested such as biofuel production methanol, hydrogen, and ethanol, fuel additives like glycerol ethers, and esters. Generally, the glycerol valorization takes place through the biotechnological process for its conversion into value added products [14].

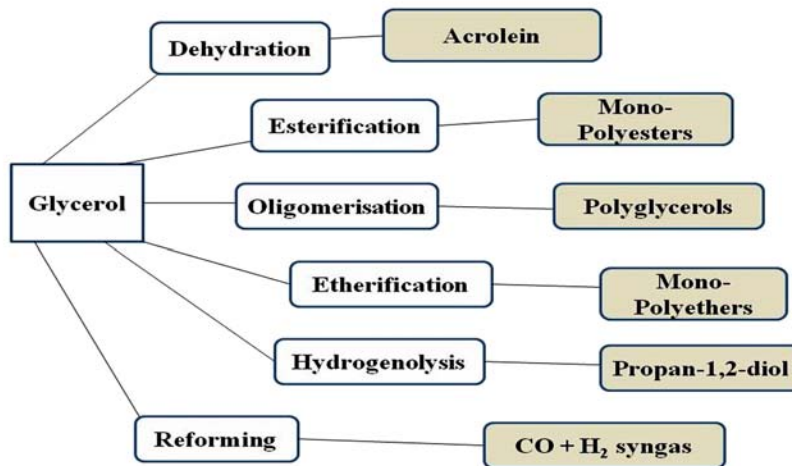


Fig. (1). Valorization reactions of glycerol [14].

Catalytic Production of Biodiesel *via* Simultaneous Esterification and Transesterification

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Abstract: Biofuels, a promising form of renewable energy, have the potential to replace fossil fuels and mitigate the impact of greenhouse gas emissions. In the presence of a catalyst, biodiesel, which is a biofuel, is produced through the process of transesterification by combining vegetable oils or animal fats with methanol or ethanol. The use of appropriate catalysts can improve the production efficiency of biodiesel, shorten the production time, and reduce the occurrence of side reactions. This chapter introduces different kinds of solid acid catalysts in the catalytic production of biodiesel, especially the advantages of the simultaneous catalytic esterification and transesterification reaction to produce biodiesel. This chapter introduces various solid acid catalysts used in the catalytic production of biodiesel, especially the advantages of simultaneous catalytic esterification and transesterification to produce biodiesel, as well as the challenges faced by current research.

Keywords: Biomass, Biodiesel, Esterification, Solid acid catalysts, Transesterification.

1. INTRODUCTION

Biodiesel, an environmentally friendly energy source with low sulfur content, has emerged as a prominent player among other renewable energy options, gaining widespread usage. It is not only recognized as a sustainable and green fuel but also as a valuable raw material or intermediate for generating fine chemicals like industrial solvents, surfactants, and lubricants [1 - 3]. With its notable advantages of excellent combustion performance, effective cold-start capabilities, superior lubrication properties, and high safety standards, biodiesel significantly reduces

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engine part wear and extends engine longevity [4, 5]. Therefore, biodiesel is regarded as a highly promising alternative to fossil fuels, playing a crucial role in the quest for global energy sustainability. The production of biodiesel has steadily increased over time and is projected to witness further growth in the future [6]. In 2008, approximately 36.8 million liters of biodiesel were produced worldwide, with a projected production volume of approximately 44 million liters by 2027 [7, 8]. Biodiesel, which consists primarily of fatty acid alkyl esters, is typically produced through the (trans)esterification process of oils or fatty acids with short-chain alcohols, utilizing acid or base catalysts [2, 5]. Currently, edible oils serve as the primary raw material for industrial biodiesel production in most regions. However, non-edible oils, which offer a cost-effective alternative, hold great promise for biodiesel production. The use of non-edible oils can not only reduce biodiesel costs, but also circumvent any competition with the production of food for human consumption.

It is worth noting that oils inherently contain varying levels of free fatty acids. The presence of these free fatty acids poses a challenge when using alkaline catalysts, as they may unexpectedly undergo saponification reactions. To avoid the complexities and expenses associated with the arduous separation and purification processes, it is crucial to pretreat oils when utilizing alkaline catalysts for biodiesel production [9, 10]. These processes often involve unwanted saponification reactions, which can be both intricate and cost-intensive. As a result, acid catalysts have gained greater prominence in this regard. Mineral acids, such as H_2SO_4 and HCl , are commonly employed as homogeneous catalysts in the production of biodiesel [11, 12], and can undergo simultaneous esterification and transesterification for catalytic production of biodiesel as well as avoid saponification. However, the use of homogeneous acid catalysts often leads to various drawbacks, including equipment corrosion, complex production processes, and challenging separation procedures. Conversely, heterogeneous acid catalysts offer notable advantages such as reusability, non-corrosive characteristics, and environmental friendliness [13 - 17]. Several common examples of heterogeneous acid catalysts include mixed metal oxides such as $\text{ZnO-La}_2\text{O}_3$ and Cu/ZnO [18, 19], sulfonic-based solid acids like $\text{TiSBA-15-e-PrSO}_3\text{H}$ and Amberlyst-15 [16, 20], functional polymers such as $\text{FDU-15-SO}_3\text{H}$ and ZIF-8/HPA [21, 22], zeolites (*e.g.*, ZSM-5 , and K/NaY) [23, 24], acidic montmorillonite with structures comprising tetrahedral SiO_4 and octahedral XO (where X represents various elements such as Sc^{3+} , Ti^{4+} , Sn^{4+} , *etc.*) [25], and hetero-poly acids (*e.g.*, $\text{Cs}_\alpha\text{H}_{(3-\alpha)}\text{PW}_{12}\text{O}_{40}$ and $\text{Cs}_\beta\text{H}_{(4-\beta)}\text{SiW}_{12}\text{O}_{40}$) [26], *etc.* Some other special heterogeneous acid catalysts include acid-base oxides (*e.g.*, Hydrotalcites and La_2O_3) [27], acid-base hybrids (*e.g.*, acid-base zeolites K-ITQ-6 and Ly_2HPW) [28], bi-functionalized oxides (*e.g.*, TiO_2/SO_4 and WO_x/ZrO_2) [29], bifunctional acidic ionic liquids (*e.g.*, $[\text{BSO}_3\text{HMIM}]\text{HSO}_4\text{-Fe}_2(\text{SO}_4)_3$ and $[\text{Ps-}$

MTH][CF₃SO₃]-FeCl₃) [29], bifunctional hetero-poly acids (Zn_{1.2}H_{0.6}PW₁₂O₄₀ and C₁₆TA-H₄TiPW₁₁O₄₀) [30, 31], and others. The utilization of heterogeneous acid catalysts can address the challenges arising from the use of homogeneous acids. The field of heterogeneous bifunctional catalysts has witnessed significant advancements in recent years. These catalysts are characterized by the presence of both acidic and basic active sites. This unique combination enables them to overcome the limitations associated with homogeneous catalysts, thereby making them a promising alternative. These catalysts have been shown to exhibit excellent catalytic performance in the esterification and transesterification reactions of high FFA-content raw materials [32, 33], which can simultaneously promote the esterification of FFAs and the transesterification of triglycerides, leading to improved conversion rates and selectivity. Table 1 summarizes the advantages and disadvantages of different types of transesterification catalysts.

Table 1. Advantages and disadvantages of different types of catalysts for transesterification reaction.

Type Of Catalysts	Examples	Advantages	Disadvantages	References
Homogeneous Alkali	KOH, NaOH	<ul style="list-style-type: none"> •Modest operational conditions •High catalytic activity <ul style="list-style-type: none"> •Low cost •Favorable kinetics •Faster reaction time 	<ul style="list-style-type: none"> •Highly sensitive to water and FFA •Saponification as a side reaction, <ul style="list-style-type: none"> •Soap formation •Low FF A requirement in the feedstock (<1wt%) •Difficult recovery of catalysts. •Equipment corrosion <ul style="list-style-type: none"> •High volume of wastewater 	[2, 5, 35, 36]
Acid	HCl, H ₂ SO ₄ , HF, p-sulfonic acid	<ul style="list-style-type: none"> •Catalyzed simultaneous esterification and transesterification reactions •Avoid soap formation •Insensitive to FFA and water content in oil 	<ul style="list-style-type: none"> •Long reaction time •Slow reaction rate •Equipment corrosion <ul style="list-style-type: none"> •High alcohol/oil requirement •Weak catalytic activity •Higher reaction temperature and pressure •Difficult recovery of catalysts 	[2, 5, 35, 36]

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