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IVANA BANKOVIĆ-ILIĆ[1]
OLIVERA STAMENKOVIĆ [2]
VLADA VELJKOVIĆ[3]

[1]University of Niš,
Faculty of Technology, Leskovac

[1]ivanabank@yahoo.com
[2]oliverastam@yahoo.com
[3]veljkovicvb@yahoo.com

ECO-FRIENDLY PROCESSES FOR BIODIESEL PRODUCTION FROM NON-EDIBLE OILS

Abstract: *At present, edible vegetable oils are most often used feedstocks for industrial biodiesel production. In this paper, the various non-edible oils and usually used processes for biodiesel production are analyzed from the ecological and economic aspects. The aim of the paper is to present the possibilities for improving the biodiesel synthesis by employing the heterogeneous catalysts, the novel catalysts obtained from waste materials and continuous process development, especially taking into account their impact on the environment.*

Key words: alcoholysis, biodiesel, non-edible oils.

INTODUCTION

The world's oil reserves depletion and environmental concerns increase caused a great demand for alternative sources of oil-based fuels. The most popular alternative energy sources fulfilling sustainability and economic feasibility criterions are biofuels particularly that made from readily available biomass feedstocks [1]. The best fossil diesel substituent is biodiesel, a clean renewable fuel that can be used in a compression ignition engine without or with small modifications. Comparing to fossil diesel, biodiesel is non-explosive, biodegradable, less toxic and with lower exhaust emission of smoke, dust, carbon (II) and (IV) oxides and hydrocarbon. Therefore, biodiesel is considered to be an environmentally friendly fuel [2].

Chemically, biodiesel is a mixture of alkyl esters of low aliphatic alcohols and high fatty acids obtained by the alcoholysis process of biological feedstocks rich in triacylglycerols (TAG). The most used alcohol for biodiesel production is methanol, so biodiesel is usually defined as a mixture of fatty acid methyl esters (FAME). Vegetable oils are the main feedstocks for biodiesel production. In current technologies, more than 95% of biodiesel production feedstocks come from edible vegetable oils such as rapeseed and sunflower seed oil in Europe, soybean oil in USA and palm oil in tropical countries [3]. However, this causes the competition at edible oil market, which increases both the price of edible oils and biodiesel [4]. Furthermore, environmentalists have discussed the negative impact of biodiesel production from edible oil on our planet, especially deforestation and destruction of ecosystem. Expansion of oil crop plantations for biodiesel production on a large scale has already caused deforestation in some countries [5].

In order to overcome these disadvantages, researchers have suggested using alternative oily feedstocks such as non-edible oils, used oils and fats, waste fats, side-stream products of refined edible oil production and algae oils. In this paper non-edible oils as feedstoks for biodiesel production and the alcoholysis processes for their conversion into biodiesel are analyzed. The aim is to present the advantages and disadvantages of non-edible oil using and the characteristics of commonly used processes for biodiesel synthesis along with a critical review in terms of environmental protection. The special attention is paid to the possibilities for improving the biodiesel production processes by using the heterogeneous catalysts.

NON-EDIBLE OILS AS FEEDSTOCKS FOR BIODIESEL PRODUCTION

Based on the previously investigation there are a lot of plant feedstocks, which can be exploited for biodiesel production: jatropha, karanja (pinnata), mahua, linseed, rubber seed, cottonseed, neem, tobacco seed, fodder radish, see mango, cardoon, putranjiba, moringa, castor, jojoba, crambe, tung, etc. [6]. Of these feedstocks, jatropha, karanja, mahua and castor oils are most often used in biodiesel synthesis. In terms of ecological and economical requirements, non-edible oil crop cultivation demands must be known. Non-edible oil crops such as jatropha, castor and karanja have unique ecological requirements and botanical features that make it suitable to be cultivated in lands that are unsuitable for food crops. For instance, jatropha plant can grow almost anywhere, even on gravely, sandy and saline soils and its cultivation is easy, without intensive care and minimal efforts. It has a healthy life cycle of 30–50 years, which eliminates the yearly re-plantation. Castor is another plant that is easily grown and has similar ecological requirements as jatropha. On the other hand, karanja is one of the few nitrogen-fixing trees that produce seeds with significant oil content. It can be cultivated to improve the soil quality, and the exhausted land can be reused for the agricultural purpose in future [5]. The biodiesel fuel characteristics depend on the fatty acid composition of the oily feedstocks. Generally, most of the fuel properties of biodiesel obtained from frequently investigated non-edible oil are within the standard limits [6].

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TRADITIONAL PROCESSES FOR NON-EDIBLE OIL BIODIESEL PRODUCTION

Homogeneously catalyzed vegetable oil methanolysis is the subject of most FAME synthesis researches and is a widely used process for industrial biodiesel production. Homogeneous base catalysts are attractive due to its higher activity in mild reaction conditions and a high FAME yield obtained in a short reaction time. Base catalysts used most frequently are sodium and potassium hydroxides and alkoxides [7]. Acid catalysts are rarely investigated but they are suitable for feedstocks with high free fatty acid (FFA) content.

The type of a catalyst for the biodiesel production from non-edible vegetable oils mostly depends on the FFA content in the oily feedstock which is influenced by the oil source, the type of cultivation and storage mechanism. A wide range of acid value data of non-edible oil has been reported in the literature. For example, for jatropha oil acid value varies from 0.92 mg KOH/g [8] to 28 mg KOH/g [9]. Therefore, researchers suggest a different catalyst type for non-edible oils alcoholysis. As expected, base catalysts are preferable in the case of oil with a lower FFA content. FFAs in the presence of base catalysts form soaps which reduce FAME yield, cause catalyst loss and complicate phase separation. Acid catalysts have low susceptibility to the presence of FFA in the starting feedstock due to their ability to simultaneously catalyze FFA esterification and TAG alcoholysis reactions. On the other hand, the acid catalyzed alcoholysis reaction is slow and long time is required to obtain high FAME yield, so acid catalysts have been rarely applied. Table 1 summarizes the catalyst type and optimal reaction conditions for some homogeneously catalyzed alcoholysis of non-edible oils.

To take advantages of both base and acid catalysts, two-step processes for the biodiesel production from the oils with the high FFA content have been developed. The two-step process, consisting of acid catalyzed FFA esterification and base catalyzed TAG alcoholysis is an effective way to achieve a high biodiesel yield within a short reaction time and at mild reaction conditions comparing to acid catalyzed process. A review of some two-step alcoholysis processes employing different non-edible oils is presented in Table 2.

From an ecological point of view, the use of homogeneous catalysts have many disadvantages Working problems are related to the catalyst type (potassium and sodium hydroxide) because they are hazardous, caustic and hygroscopic [10]. Additionally, in order to meet the specified product quality the process involved a number of washing and purification steps producing a large amount of wastewater, which is environmentally unfavourable and required appropriate treatment. The high amount of water used in washing and consequent treatment of the resulting effluent increased the overall process cost. For these reasons, homogeneously catalyzed alcoholysis could be considered as a traditional method for biodiesel synthesis, and alternative methods have been developed.

Table 1 *A review of the homogeneously catalyzed alcoholysis processes using different non-edible oils*

Feedstock	Alcohol	Type, volume of reactor, cm^3 / Type of agitator, agitation intensity, rpm	Alcohol:oil molar ratio, mol/mol	Catalyst / loading, %wt to the oil	Temperature, ºC	Yield (Conversion), % / Time, min	Ref.
Jatropha oil	Methanol	Batch reactor, -/ -, 300	9:1	NaOH / 0.8	45	(96.3) / 30	[11]
Jatropha oil	Methanol	Batch reactor, 224 / Mechanical, 900	6:1	KOH / 1	50	97.1 / 120	[12]
Jatropha oil	Methanol	Flask, 1000 / -	5.6:1	NaOH / 1	60	98 / 60	[13]
Jatropha oil	Methanol	Flask, 100/ Magnetic, -	6:1	KOH / 1	45	(≈100) / 30	[14]
Karanja oil	Methanol	-	10:1	KOH / 1	60	(92) / 90	[15]
Mahua oil	Methanol	Flask, 100 / Magnetic,	6:1	KOH / 1	45	(95) / 180	[14]
Castor oil	Ethanol	Flask, 250 / Magnetic, 600	16:1	$NaOC_2H_5$ / 1	30	93.1/30	[16]
Castor oil	Methanol Ethanol	Batch reactor, 250/ Magnetic, -	6:1	$KOCH_3$ / 0.2[a] $NaOCH_3$ / 0.2[a]	60 80	85 / 60 80 / 360	[17]

[a] *- molar ratio catalyst: oil*

Table 2 *A review of the two-step (acid/base) homogeneously catalyzed alcoholysis of different non-edible oils*

Feedstock	Alcohol	Type, volume of reactor, cm^3 / Type of agitator, agitation intensity, rpm	Step[a]	Alcohol:oil molar ratio, mol/mol	Catalyst / loading, %wt to the oil	Temperature, ºC	Yield (Conversion), % / / Time, min	Ref.
Jatropha oil	Methanol	Flask, - / Magnetic, 1000	I	6:1	H_2SO_4 / 0.5	45	(93)[b]/ 120	[18]
			II	9:1	KOH / 2	60	95 / 120	
Jatropha oil	Methanol	Flask, 1000 / Mechanical, 600	I	8:1	H_2SO_4 / 0.4	60	(92)[b]/ 30	[19]
			II	6:1	KOH / 1	60	86.2 / 30	
Karanja oil	Methanol	Flask, - / Magnetic, 1000	I	6:1	H_2SO_4 / 1	50	(94)[b] / 45	[20]
			II	9:1	KOH / 0.5	50	80/ 30	
Tobacco sedd oil	Methanol	Flask, - / Magnetic, 400	I	13:1	H_2SO_4 / 2	60	(97)[b] / 50	[21]
			II	6:1	KOH / 1	60	91 / 30	

[a]– I- first step: acid pretreatment, II- second step: base-catalyzed alcoholysis; [b] – FFA conversion

HETEROGENEOUSLY CATALYZED PROCESSES FOR NON-EDIBLE OIL BIODIESEL PRODUCTION

Attempts at improving the biodiesel production process have been ongoing for years. Development of biodiesel production methods has aimed at reducing the biodiesel production cost in environmentally favorable processes. Among them, special attention has been attributed to the heterogeneously catalyzed alcoholysis which is referred as green process. The use of heterogeneous catalysts improves biodiesel synthesis for several reasons. These catalysts can be separated more easily from the reaction mixture, and thus the process does not demand catalyst neutralization in reaction products. Accordingly, the purification of the products is much more simplified and does not require a large amount of water, which makes the process environmentally and economically advantageous. The additional benefit of the solid catalysts using is their easy regeneration and reuse that further make the biodiesel synthesis process cost-effective. The disadvantages of heterogeneously catalyzed processes are the formation of three phases leading to diffusion limitations, thus lowering the reaction rate, and the complex catalyst preparation in some cases.

The catalytic activity of a heterogeneous catalyst depends on its nature, specific surface area, pore size and volume and active site concentration. Their catalytic activity could be improved by catalyst support on the carriers, which provide a higher specific surface area [22] or by applying the appropriate treatment in order to increase the catalyst acidity or basicity [23, 24].

A short review of the catalyst type and reaction conditions applied in heterogeneously catalyzed alcoholysis of non-edible oils is given in Table 3. The type of a heterogeneous catalyst for the biodiesel production from non-edible vegetable oils depends on the FFA content in the oily feedstock. Solid acid catalysts have been more often used in alcoholysis of non-edible oils with the high FFA content because they contain a variety of acid sites with different strength of Bronsted or Lewis acidity. Some researchers also employ the modified catalysts which exhibit dual basic and acidic sites [25] or the mixture of acid and base catalysts [26], which allow oil conversion to biodiesel in a one-step process of simultaneous esterification and alcoholysis. Previous researches of heterogeneously catalyzed alcoholysis of non-edible oils have been aimed at developing a catalyst with a high catalytic activity, which is generally, attributed to the presence of the large amount of strong basic [27, 28], acid [29] or both basic and acid [25] sites.

Several studies deal with the use of heterogeneous catalysts in the two-step (acid-base) process, and they are reviewed in Table 4.

Table 3 *A review of the heterogeneously catalyzed alcoholysis of different non-edible oils*

Feedstock	Alcohol	Type, volume of reactor, cm^3 / Type of agitator, agitation intensity, rpm	Alcohol:oil molar ratio, mol/mol	Catalyst / loading, %wt to the oil	Temperature, °C	Yield (Conversion), % / Time, h	Ref.
Jatropha oil	Methanol	Round-bottom flask, 50 /	55:1	Mg-Zr mixed oxide with Mg/Zr weight ratios of 2:1 / 10	65	≈(100) / 0.75	[28]
Jatropha oil	Methanol	Flask, 100	9:1	CaO / 1.5	70	(93) / 2.5	[24]
Cottonseed oil	Methanol	Autoclave, 250 / Magnetic	9:1	SO_4^{2-}/TiO_2-SiO_2 / 3	200	92 / 6	[30]
Castor oil	Methanol	Round-bottom quartz flask, 250, Microwave / Magnetic, 600	12:1	55% H_2SO_4/C / 5	65	94 / 1	[31]
Rubber seed oil	Methanol	Teflon-lined steel autoclave, 100 / rotating, 50	15:1	$K_4Zn_4[Fe(CN)_6]\cdot 6H_2O\cdot 2$(tert-BuOH) / 3	170	(97) / 8	[29]
Jatropha oil	Methanol	Batch reactor Parr 4842, 300 / Mechanical	11:1	$Mg_{0.7}Zn_{1.3}Al_{2/3}O_3$ / 8.68	182	94 / 6	[25]
Jatropha oil	Methanol	Round-bottom flask / Mechanical, 300	6:1	CaO+$Fe_2(SO_4)_3$; Li-CaO+ $Fe_2(SO_4)_3$ / 5	60	≈100 / 3	[26]

Table 4 *A review of the two-step (acid/base) heterogeneously catalyzed alcoholysis of different non-edible oils*

Feedstock	Alcohol	Type, volume of reactor, cm^3 / Type of agitator, agitation intensity, rpm	Step[a]	Alcohol:oil molar ratio, mol/mol	Catalyst / loading, %wt to the oil	Temperature, °C	Yield (Conversion), % / Time, h	Ref.
Jatropha oil	Methanol	Flask, 1000 / Magnetic, 400	I	12:1	SiO_2·HF / 10	60	(96)[b] / 2	[23]
			II	6:1	NaOH / 1	60	≈99.6 / 2	
Jatropha oil	Methanol	Three-neck flask, 250 / Mechanical	I	20:1[c]	SO_4^{2-}/TiO_2 / 4	90	(≈97)[b] / 2	[32]
		Autoclave / Mechanical, 1500	II	6:1	KOH / 1.3	64	98 / 0.33	
Karanja oil	Methanol	Round-bottom flask, 1000 / Mechanical, 600	I	6:1	H_2SO_4 / 1.5 v/v	65	(≈91)[b] / 1	[33]
			II	8:1	CaO / 2.5	65	95 / 2.5	
Mahua oil	Methanol	Round-bottom flask, 1000 / Mechanical, 1000	I	6:1	H_2SO_4 / 1.5 v/v	55	(91)[b] / 1	[34]
			II	8:1	CaO / 2.5	65	95 / 2.5	

[a]– I- first step: acid pretreatment, II- second step: base-catalyzed alcoholysis; [b] – FFA conversion [c] – methanol:FFA molar ratio

Heterogeneous catalysts are used either to catalyze FFA esterification or TGA alcoholysis. According to best authors' knowledge a completely heterogeneous two-step process has not been developed yet.

Generally, heterogeneous catalysts could exhibit high activity but the reactions are slow and time consuming. Therefore, resent researches have been focused towards an ideal heterogeneous catalyst which is low cost, eco-friendly and with high catalytic activity. Such a novel catalyst could be prepared either from biomass or from waste generated in the households. Recently, high efficient catalysts for the methanolysis reaction with CaO as the main component have been obtained from waste egg, oyster, shrimp and lime stone. Using wastes as raw materials for catalyst synthesis could eliminate the wastes and simultaneously produced the catalysts enabling sustainable process development. The use of cheap and efficient catalyst makes the process economic and fully ecologically friendly. For example, CaO obtained from waste chicken eggshells was used as a methanolysis catalyst in the two-step biodiesel production process from karanja [33] and mahua [34] oil. The catalyst preparation involves eggshells washing, drying at 105ºC, powdering and finally calcinating at 900°C for 2 h. The FAME yields obtained in those processes were 95%. It is expected that further investigations could be aimed at applications of the high-effective heterogeneous catalysts derived from various natural or waste resources, development of continuous heterogeneous catalyzed processes and optimization on reaction parameters.

The possibility of reusing heterogeneous catalysts is another of its advantages, which enables the continuous process development. Continuous processes facilitate the larger biodiesel productivity and the improvements in the equipment design for the optimization of the biodiesel quality. Additionally, continuous processes could reduce the production cost, making the price of biodiesel competitive with respect to fossil fuels.

The laboratory scale continuous process of heterogeneously catalyzed alcoholysis of non-edible oil was developed by Sreeprasanth et al. [29]. The process was conducted in a fixed-bed, down flow reactor with $K_4Zn_4[Fe(CN)_6]\cdot 6H_2O\cdot 2$(*tert*-BuOH) as the catalyst. The unrefined rubber seed oil and *n*-octanol at the alcohol:oil molar ratio 15:1 were fed with the total flow rate 2 ml/h. The obtained rubber seed conversion was 89.2%, and no loss in the catalytic activity was noticed after 52 h [29]. Peng et al. [30] proposed a continuous biodiesel production process from cheap raw feedstocks by solid acid catalysis. The production process was carried out in a sequence of three reactors with the countercurrent flow of vaporized methanol. Using the proposed continuous process, a 10,000 tonnes/year industrial biodiesel demonstration product plant was built [30].

CONCLUSION

The mostly applied process for the industrial biodiesel production is homogeneously base catalyzed methanolysis of edible oils. Due to the competition at edible oil market and negative environmental impact of large scale edible oil crop plantations, investigations have been focused towards non-edible oils. The non-edible oil crop cultivation is generally easy and with minimal requirements, making them attractive as sources for biodiesel production. The biodiesel production development is directed towards simple, environmentally and economically advantageous processes; among them, heterogeneously catalyzed alcoholysis is the most promising method. Due to simple catalyst separation and reaction products purification heterogeneously catalyzed processes are referred as green processes. Novel high-performance catalysts have been prepared from waste materials allowing their utilization in a useful product. Continuous process technologies based on heterogeneously catalyzed process is expected to get a wider application in the future industrial biodiesel production.

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BIOGRAPHY

Ivana Banković Ilić was born in Leskovac, Serbia, in 1963. She took her M.Sc. degree and Ph.D. degree at the Faculty of Technology and Metallurgy, University of Belgrade in 1993 and at the Faculty of Technology in Leskovac, University of Niš in 1999, respectively, in the area of chemical engineering. She is currently working as a full professor at the Faculty of Technology in Leskovac, University of Niš.

EKOLOŠKI NEŠKODLjIVI PROCESI ZA PROIZVODNjU BIODIZELA IZ NEJESTIVIH ULjA

Ivana Banković-Ilić, Olivera Stamenković , Vlada Veljković

Rezime: *Jestiva biljna ulja predstavljaju danas najčešće korišćene sirovine za industrijsku proizvodnju biodizela. U ovom radu analizirana su ekološkog i sa ekonomskog aspekta različita nejestiva ulja, kao i uobičajeni procesi za proizvodnju biodizela procesi.Cilj rada je da predstavi mogućnosti za unapređenje sinteze biodizela upotrebom heterogenih katalizatora, novih katalizatora dobijenih od otpadnog materijala i da prikaže kontinuirani razvoj procesa, posebno uzimajući u obzir njihov uticaj na životnu sredinu.*

Ključne reči: alkoholiza, biodizel, nejestiva ulja.