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ABSTRACT

of the dissertation for the degree
of Doctor of Philosophy

**RESEARCH ON IMPACT OF DIFFERENT PHYSICAL
FACTORS IN PROCESS OF PRODUCTION
OF BIODIESEL FUELS**

Speciality: 3321.01 – Oil - gas - coal processing and technology

Field of science: Technical

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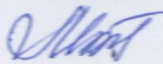
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GENERAL DESCRIPTION OF WORK

Relevance of the topic. Oil resources are now the basis of the world's energy supply and the largest consumer of these types of energy carriers is automobile transport, which is growing day by day. However, depletion of natural energy resources as well as during their operation, harmful combustion is emitting environmental pollution of products, in addition having considered that during the use of energy sources combustion of environmentally hazardous products into the atmosphere leads to pollution of the environment and severe climate changes, for this reason it is very important to switch to alternative energy sources. There are certain programs in order to switch alternative energy resources and they are currently implementing by the world leading countries. As an example of this, the USA 25/25, and in the European Union 20-20-20 plans could be shown. According to the transition to alternative energy sources, first and foremost, these programs are implemented for this purpose motor gasoline, diesel, bio-ethanol and biodiesel based on renewable raw materials are being developed as a fuel types. This research work aimed at improving the process of economic affordable, environmentally efficient products which does not take the side of the search and a variety of catalysts, are aimed at speeding up the process with the use of low-energy waves.

Given the rapid increase in the number of diesel engines, chemists are focused on the purchase of biodiesel-based alkyl esters based on vegetable oils and the rectification reaction with simple alcohols. Studies to improve this process are aimed at accelerating the process with the search for catalysts that are economically viable, environmentally friendly and do not allow the production of by-products and the use of various low-energy waves.

From this point of view, the use of new ammonium catalysts in the process of obtaining biodiesel fuel based on favorable plant resources in Azerbaijan, as a catalyst for the synthesis and transduction of ionic compounds that meet the principles of "green chemistry", as well as electrification of these processes. And the study under the influence of hydro-dynamic cavities is very relevant and awaits its solution. At the same time, the acquisition of new fuels and diesel

components for glycerol based on glycerol, which is a by-product of the conversion of fatty acids to simple alcohols, further enhances the relevance and value of the dissertation.

The object and subject of research is the production of diesel fuel based on renewable raw materials of Azerbaijan in accordance with modern environmental requirements and the principles of “green chemistry”.

The purpose of the work. The choice of new amino catalytic catalysts for the production of biodiesel based on renewable plant materials in Azerbaijan, the synthesis of ionic liquids based on the principles of “green chemistry” as catalysts for transduction, magnetic, twisted and electric fields. Investigation of the effect of cavitation and recommendations for the production of plant-derived fatty acid alkyl esters, as well as glycerin, and the use of extracts as fuel and fuel components.

Investigation methods. In the process of the dissertation, the quality of the resulting target products, as well as their purity, were determined by the relevant standards according to ГOCT and ASTM, as well as methods of infrared spectroscopy and ^1H and ^{13}C NMR.

The main provisions submitted to the defense. Selection and synthesis of new types of catalysts for the development of biodegradable diesel fuels or fuel components under low-energy effects based on Azerbaijani plant resources (cotton, sunflower, maize, soybeans and palm oils), as well as obtaining of multifunctional supplements to mineral diesel fuel on the basis of by-products purchase of incoming glycerin.

The scientific novelty of the work is that, for the first time:

- the process of transesterification of cotton, sunflower, corn, soy and palm oils with methyl, ethyl, isopropyl and butyl alcohols. Diphenylamine (DFA), phenylenediamine (FDA), trimethylamine (TEA), isopropyl amine (IPA), tretbutylamin (TBA) amino compounds and tetramethylammoniumdihydrophosphate (TMADHF), tetra-methylammoniumacetate (TMAA), diethylaminodihydrofosphat (DEADF), diethylaminohydro-sulphat (DEAHS) and diethylaminotrifluorborat (DEATFB) was used as catalysts in the process of obtaining a magnetic field of 0.2-0.9

- T, ultrasonic waves of 20 kHz, methyl, ethyl, isopropyl and butyl ethers of vegetable oils under the influence of a rotating electric field and hydrodynamic cavitation is investigated;
- it was found that the tested ammonium compounds form the series FDA > TEA > DFA > TBA > IPA in their activity as a catalyst for the conversion of vegetable oils during transduction with simple alcohols and they are approximately in the same activity as TMADHF, TMAA, DEADHF, DEAHS, DEATFB;
 - it was found that alcohol under traditional conditions: a fat ratio of 6 : 1, ammonium compounds are listed during the 6-hour process and 3.0 % by weight as a catalyst for ionic liquids. (in oil), methyl, ethyl and butyl fatty acid esters using 94.6-99.3 % by weight, isopropyl ethers 87.7-96.7 % by weight, with the same amount of catalysts as IPA and TA. These yields make up 84.5-95.8 and 80.2-87.9 %, respectively;
 - it was found that the process of extraction of the target products during the extraction of alkyl esters of vegetable fatty acids in a magnetic field with a strength of 0.2 T, ultrasonic cavitation of 20 kHz, twisted electric field and hydrodynamic cavitation is from 1.2 to 2.0 hours and 5-12 minutes. 90.7-99.5 % under reduced conditions. In this regard, the required temperature is obtained from the effects of ultrasonic cavitation, torsional electric field and hydrodynamic cavitation;
 - For the first time, oleic and acetate esters of glycerol, a by-product of monoalkyl ether of vegetable fatty acids (MAEFA), as well as components of commercial diesel fuel, combustion modifiers and food additives, were freely studied;
 - It was found that glyceryl triacetate and glyceryl monooleate esters were used in diesel fuel containing 2.5 % and 5.0 % of their compounds in combination with PamYMetE. At this time, the low-temperature properties of mixtures are improved, the set is increased by 0.5 units and saves the added amount of ethers to the traditional reserves of diesel fuel;
 - it was also found that the number of fatty acid alkyl esters in diesel fuel increased by 10, 15 and 20 %, respectively, the amount of carbon monoxide in the exhaust gas 25.0-29.0; 42.0-46.0; and

58.0-61.0 % of the bulbs. And 19.5-20.3 smoke; 40.8-42.0; and 55.0-56.0 % of the bulbs. Reduced in a result. The composition of the combustion products containing 2.5-5.0 % by weight of glyceryl triacetate ethers in commercial diesel fuel is 27.3 % more in carbon monoxide and 30.0-35.0 % in the same concentrations of glycerol monoolein ether were made;

- the lubricating properties of MAEFA in diesel oil were investigated and it was found that the addition of 2.0 % by weight of ethyl, isopropyl and butyl ethers of vegetable fatty acid methyl esters per 850 micrometers. It caused a decline in the diameter of the food stain from 850 microns to 420, 430, 452 and 415 microns, respectively. At the same time, the addition of 2.0 % cottonseed mixed fatty acid esters resulted in a decrease in edible spot diameter of 440 microns;
- the lubricating properties of glycerol monooleate and monoacetate on commercial diesel fuel have been studied and defined that, when glycerol monoacetate ester 75 mH and glycerol monoacetate ester 125 g, wear spot diameter (WSD) are 440 and 452 μm , respectively.

The theoretical and practical significance of the scientific work presented is the development of traditional fuels based on Euro-4 in Azerbaijan, the development of vegetable fatty acids and glycerol alkyl ether extracts based on Azerbaijan's renewable raw materials and the use of these fuels as diesel fuel. It is defined by the recommendations for the purchase of diesel fuel that meet the requirements of EN-590.

Publication: 26 scientific papers, including 10 articles, 1 patent, 15 abstracts of reports in 15 different international conferences and congresses.

Approbation of the work. The main results of the dissertation work has been discussed in several international national conferences, as well as symposiums, including: International conference “Catalysis for renewable sources: fuel, energy, chemicals” St. Peterburg, Tsars Village, 2010, IX Международная научно-техническая конференция “Энерго и материалосберегающие экологически чистые технологии” Гродно 20-21 октября 2011 г, The 1st International Conference on Chemistry and Engineering,

dedicated to the 90th anniversary of the birth of the National Leader H. Aliyev 2013, Baku, II International Scientific Conference "Ecology: problems of nature and society", dedicated to the 105th anniversary of academician Hasan Aliyev 7-8 November 2012, Baku, ECO 2014 2nd International Conference on Energy, Regional Integration, and Socio-Economic Development Baku, Azerbaijan, 2014, IX Republican scientific conference of doctoral students, masters and young scientists "Actual problems of chemistry" Baku 2015, Scientific And Practical Conference Concerning The 92th Anniversary Of National Leader Heydar Aliyev, Ganja 2015, Republican Scientific Conference "Lubricants, Fuels, Special Liquids, Additives and Reagents", dedicated to the 50th anniversary of the Institute of Additive Chemistry named after Academician A. Guliyev, XXIX Научно-техническая конференция «Химические Реактивы, реагенты и процессы малотоннажной химии» «Реактив-2015» г. Новосибирск, International scientific-practical conference on "Emergency situations and safe life", devoted to the 10th anniversary of the Ministry of Emergency Situations of the Republic of Azerbaijan 10 December 2015, Baku, IX Бакинская Международная Мамедалиевская Конференция По Нефтехимии 4-5 октября 2016.

Place of dissertation. The work was performed in accordance with the work program of the IPCP ANAS 4/2016, registration number 0106Az00018.

Author's personal participation. The purpose and direction of the research, the implementation of experiments, and the analysis of the results were carried out with the participation of the author.

The structure and scope of the dissertation. The dissertation is presented on 196 pages, consists of an introduction, 6 chapters, including 52 tables, 20 figures, 20 charts, a list of references from 216 items and is 176065 characters (without tables, figures, and list of literature), including the introductory part 19311, Chapter I – 50215, Chapter II – 18315, Chapter III – 46493, Chapter IV – 13090, Chapter V – 14377, Chapter VI – 14264 characters.

Introduction. This chapter contains information on the relevance, purpose, scientific novelty and practical significance of research conducted in the dissertation.

Chapter I examines published information in the international literature on alternative sources of energy, including the acquisition, current use, and prospects of alternative motors, and the main objectives of the dissertation work.

Chapter II presents the qualitative characteristics of the raw materials used, the characteristics of IQ, ^1H -, ^{13}C -NMR products and their proposed products, as well as a description of laboratory equipment for experiments.

Chapter III of the dissertation consists of ammonium derivatives of diphenylamine, phenylenediamine, trimethylamine, isopropyl amine, tributylamine, selected as catalysts based on cotton, sunflower, corn, soy and palm oils, as well as methyl, ethyl, isopropyl and butyl alcohols and traditional conditions also a magnetic field with a strength of 0.2-0.9 T, ultrasound at a frequency of 20 kHz, an electric field, as well as a study of the process of acquiring hydrodynamic cavitation.

Chapter IV of the dissertation is devoted to tetramethyl ammoniumdihydrogenphosphate (TMADHF), tetramethylammoniumdihydrophosphate (TMADHF), tetramethylammonium dihydrophosphate (TMADHF), synthesized as catalysts based on cotton, sunflower, corn, soybean and palm oils, and also methyl, ethyl, alkyl esters of vegetable oils using ionic liquids (DEAHS) and diethylamino-fluorofluorophosphate (DEATFB) are affected by normal conditions, as well as a magnetic field with an intensity of 0.2-0.9 T and ultrasonic cavitation of 20 kHz.

Chapter V of the dissertation determines to the study of acids of cotton, sunflower, corn, soy, and palm oil as a fuel and fuel component for methyl, ethyl, isopropyl, and butyl ethers, as well as glyceryl triacetate and glyceryl monoethylene compounds.

Chapter VI researches on the effect of cotton, corn, sunflower, palm, soy acids, their methyl, ethyl, redopil and butyl ethers, as well as glyceryl monoacetate and glyceryl monoete ethers on lubricating properties.

The dissertation work is completed with the main results and a list of references cited in the dissertation.

THE MAIN CONTENT OF THE WORK

1. The study of amino acids DFA, FDA, TMA, IPA, UBA as catalysts in the process of trans-fermentation of vegetable oils with simple alcohols

First of all, the amino compounds DFA, FDA, TEA, IPA, UBA are catalyzed during the catalysis of cotton (CotO), sunflower (SunO), corn (CorO), soybean (SOY) and palm (PaLY) oils with methyl, isopropyl and butyl alcohols in normal conditions and under the influence of a magnetic field of 0.2-0.9 T, ultrasound at a frequency of 20 kHz, an electric field, as well as hydrodynamic cavitation. As a result of the studies, the ammonium compounds listed in the usual processes for the extraction of teran comprised 98.0-99.5 % of fatty acid alkyl esters formed by TEA > FDA > DFA > TBA > IPA (chart 1a) due to their activity as catalysts, optimal access conditions for: catalyst (the number of obtained oil) 3 % by weight, alcohol: oil ratio 6:1 and the duration of the process is determined 6 hours. (chart 1 b).

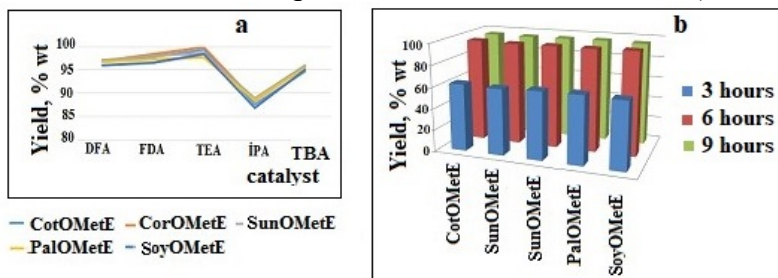


Chart 1. The dependence of the yield of esters on the nature of the catalysts and the time of the process by using TEA FDA TBA IPA as catalysts

To study the influence of the magnetic field on the biodiesel process, the transduction process was carried out under the above optimal conditions and a magnetic field of 0.2-0.9 T. It was found that the increase in either yield during the process at room temperature 0.2 T and even at room temperature is 12-28 %, while the maximum yield is achieved at 0.2 T of the magnetic field and boiling points and in the most active catalysts presence is 98-99,5 %. In this case, the duration of the process varies from 1.4 to 2.2 hours

instead of 6-8 hours (table 1) which is 4.2-3 times less than traditional ones.

Table 1
Dependence of the yield of monoalkyl esters of fatty acids of vegetable oils on the duration of the process with a magnetic field intensity of 0.2 T

Indicators	Yield of ester, % wt	Process time, h	Yield of ester, % wt	Process time, h	Yield of ester, % wt	Process time, h	Yield of ester, % wt	Process time, h	Yield of ester, % wt	Process time, h
catalyst	TEA		FDA		DFA		IPA		TBA	
PamY MetE	98,5	1,4	99,0	1,4	97,5	1,5	92,0	1,7	96,5	2,0
GünY MetE	98,0	1,6	98,5	1,5	96,3	1,6	92,4	1,7	95,8	1,8
QarY MetE	97,6	1,3	98,7	1,5	97,0	1,5	93,0	1,8	96,0	2,0
PalY MetE	99,0	1,4	97,9	1,5	96,8	1,5	92,8	1,7	97,0	2,3
SoY MetE	98,6	1,4	98,0	1,5	98,0	1,6	92,6	1,7	97,0	2,3
PamY EtE	98,0	2,1	97,2	1,6	95,0	1,7	86,7	1,9	96,3	2,4
GünY EtE	97,6	2,0	96,8	1,6	94,8	1,7	85,8	2,0	95,4	2,3
QarY EtE	97,2	2,0	96,4	1,5	94,5	1,7	85,4	2,0	95,8	2,4
PalY EtE	98,2	2,3	97,0	1,6	95,3	1,8	86,0	2,1	96,0	2,4
SoY EtE	98,1	2,2	97,5	1,7	94,8	1,7	86,4	1,9	96,7	2,5
PamY IprE	97,0	2,5	90,4	2,0	90,1	2,0	86,7	2,4	94,5	2,5
GünY IprE	96,8	2,5	89,6	2,0	90,5	2,0	85,4	2,5	93,8	2,5
QarY IprE	96,3	2,5	89,8	2,0	89,7	1,9	85,7	2,5	93,2	2,3
PalY IprE	97,4	2,4	90,7	2,4	89,0	1,9	86,4	2,4	94,1	2,5
SoY IprE	97,5	2,6	91,4	2,5	90,8	2,1	86,0	2,4	94,4	2,5
PamY ButE	99,0	1,5	99,0	1,5	98,4	1,8	92,3	2,0	97,0	1,9
GünY ButE	98,6	1,5	98,4	1,5	97,6	1,8	91,6	2,9	96,5	1,8
QarY ButE	98,4	1,6	98,7	1,5	97,0	1,7	92,5	2,1	96,8	1,9
PalY ButE	99,1	1,5	99,4	1,6	98,8	1,8	92,8	2,2	97,4	2,0
SoY ButE	99,0	1,5	99,5	1,6	97,9	1,7	91,7	2,0	98,1	2,2

An increase in the intensivity of the magnetic field strength to 0.65-0.9 T has a negative effect on the process and the yield of alkyl esters caused by fatty acids is reduced by 7-8 %.

At the next stage, the process of transpiration of vegetable oils used for the production of biodiesel fuels with simple alcohols was investigated in the vortex electric field. The process was carried out by placing a rotating electromagnetic field inductor with a reactor asynchronous electric motor (2.2 kW, feeding voltage 380 V). The magnetic induction of the reactor in the working area was 0.08-0.095 T (altitude) and 0.093-0.095 T (highest cut). As ferromagnetic elements, carbon steel cylinders 15 mm long and 1.5 mm in diameter were used. Ferromagnetic elements are not loaded into the reactor when in experiments, only the effect of the resulting magnetic field is studied. When studying only the experimental effect of a magnetic

field, ferromagnetic elements are not loaded into the reactor. In this case, the temperature of the reaction mass increases from 18-20 °C to 28-30 °C due to exposure to an electromagnetic field for 2 minutes. The obtained results are given in the table 2.

Table 2

Comparative yields of methyl, ethyl, isopropyl and butyl ethers of sunflower oils under the influence of a magnetic field and a magnetic-impulse cavitation

Esters	Condition of experiments		
	Only magnetic field (0.08-0.093 T)	Magnetic field + ferromagnetic particles	
	Duration of the process, min		
	2	2	5
Catalyst TEA			
GünY MetE	27,2	99,0	99,2
GünYEE	26,4	90,7	98,6
GünYİpr E	10,2	82,6	97,8
GünYBut E	27,8	98,7	98,0
Catalyst FDA			
GünY MetE	28,6	98,7	99,0
GünYEE	27,3	90,5	97,6
GünYİpr E	12,5	87,1	97,5
GünYBut E	28,0	98,6	99,0
Catalyst DFA			
GünY MetE	21,2	97,8	98,5
GünYEE	20,8	85,3	98,0
GünYİpr E	9,3	80,7	97,3
GünYBut E	19,3	96,0	99,0
Catalyst IPA			
GünY MetE	13,6	74,6	97,3
GünYEE	12,1	75,0	95,4
GünYİpr E	6,8	63,4	95,0
GünYBut E	12,4	80,2	96,7
Catalyst TBA			
GünY MetE	28,2	93,4	99,0
GünYEE	22,4	87,6	98,0
GünYİpr E	11,6	78,4	97,6
GünYBut E	27,0	94,6	98,4

In the process involving the addition of ferromagnetic particles with cavitation effect, the yields of alkyl esters of sunflower oil acids are 90.5-99.0 % mass with the application of TEA, FDA, DFA və ÜBA catalysts, but the yield of the i-propyl esters are 78.4- 82.6 %. Duration of this process is 2-5 minutes.

The same cavitation effect also appears with the influence of ultrasonic waves and in the process of transesterification of cotton,

sunflower, maize, soybean and palm oils with methanol, isopropanol and butanol 98.5-99.2 % alkyl esters of fatty acids are obtained as a yield within 5 minutes (table 3).

Table 3

Optimal time of obtaining methyl, ethyl, i-propyl and butyl ethers from vegetable oils under ultrasonic waves

Esters	Catalyst														
	DFA			FDA			TEA			IPA			TBA		
	Process time, min														
	2	5	7	2	5	7	2	5	7	2	5	7	2	5	7
PamYMetE	87,6	99,2	99,3	97,6	98,0	99,0	97,7	99,0	-	64,3	88,1	98,3	86,8	99,0	99,0
GünYMetE	86,5	98,7	98,7	96,1	98,2	99,1	96,5	99,0	-	61,2	88,3	98,6	87,1	98,5	98,8
GarYMetE	88,0	99,1	99,4	98,2	98,6	99,2	98,7	99,0	-	64,4	88,4	98,4	87,5	98,7	99,1
Pal YMetE	88,3	99,1	99,2	98,2	99,3	99,2	98,7	99,0	-	66,5	88,2	98,8	88,1	99,0	99,1
SoYMetEE	87,8	99,0	99,2	97,9	99,2	99,3	97,6	99,0	-	69,6	88,4	98,9	87,2	99,0	99,0
PamYEE	78,5	98,5	98,5	80,5	99,0	-	82,6	98,1	98,4	60,9	82,7	98,5	76,5	98,3	98,5
GünYEE	77,4	98,3	98,4	81,2	99,0	-	83,4	98,4	98,6	61,4	82,4	98,5	76,3	98,1	98,4
GarYEE	80,5	98,1	98,7	80,8	99,0	-	83,9	98,4	98,4	61,7	83,6	98,1	77,2	97,9	98,7
Pal YEE	79,6	98,7	98,7	79,8	99,0	-	84,0	99,1	99,0	62,0	84,0	98,0	77,4	97,7	98,7
SoYEEE	77,4	98,1	98,4	80,4	99,0	-	83,5	99,0	99,0	62,0	84,0	98,0	76,8	98,3	98,4

Almost the same regularity is also observed in the synthesis of other ethers (Chart 2). From the data presented, it is clear that when catalyst concentrations is 1 % the highest increase is observed for methyl esters from cotton fatty acids and when the concentration is 2 % increase in both methyl and ethyl ethers are detected.

Hydrodynamic cavitation occurs when fluids move at high speed and in a changing path. Variable trajectory refers to the movement of fluids in variable diameter pipes. Unlike ultrasonic and electromagnetic cavitators, no special reactors are needed to create hydrodynamic cavitation, which is why it is more economically viable.

In this thesis research, studied process of transesterification of cotton, sunflower, maize, soybean and palm oil with methyl, ethyl, i-propyl and butyl in hydrodynamic cavitation medium under the above-mentioned optimum conditions where amin based compounds like TEA, FDA, DFA, IPA, UBA used as catalysts. The obtained results are presented in Table 4.

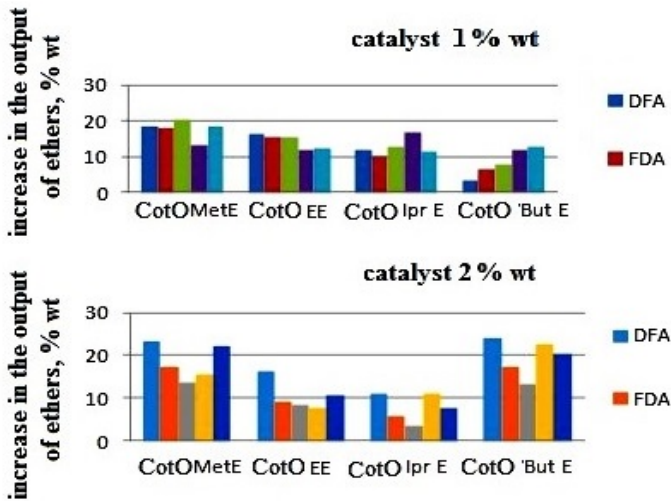


Chart 2. Observed increase in the yield of monoalkyl esters of cotton oil acids with 1 and 2 % of catalysts under the effect of ultrasound cavitation

**Table 4
Optimal conditions for obtaining of methyl, ethyl and i-propyl ethers of fatty acids under the influence of hydrodynamic cavitation**

Esters	Catalyst														
	DFA			FDA			TEA			IPA			TBA		
	Process time, min														
	5	10	12	5	10	12	5	10	12	5	10	12	5	10	12
PamYMetE	82,4	99,3	-	87,4	99,0	-	88,2	99,0	-	83,4	96,0	98,6	85,7	99,1	-
GünYMetE	81,2	98,8	-	86,3	98,5	-	87,6	99,0	-	82,8	94,5	98,4	84,9	98,7	-
GarYMetE	83,3	99,0	-	88,5	98,4	-	88,1	99,0	-	84,5	93,8	98,5	84,3	98,7	-
Pal YMetE	83,1	99,0	-	88,4	99,0	-	88,5	99,0	-	85,7	95,0	98,2	86,2	98,2	-
SoYMetEE	82,4	99,0	-	87,6	99,0	-	87,7	99,0	-	85,4	94,6	98,2	85,4	98,4	-
PamYEE	72,1	98,5	98,7	74,5	98,0	-	72,4	98,4	98,7	55,6	89,5	98,0	72,3	97,1	-
GünYEE	71,8	98,1	98,4	73,9	98,0	-	73,2	98,4	98,7	54,8	87,5	98,0	71,9	98,6	-
GarYEE	71,6	98,4	98,7	74,7	98,0	-	73,7	98,4	98,7	55,6	86,4	98,0	72,4	98,2	-
Pal YEE	71,4	98,7	98,7	75,0	98,0	-	74,5	99,1	99,4	54,4	74,8	98,0	72,6	97,9	-
SoYEE	71,5	98,5	98,4	75,1	99,0	-	73,5	99,0	99,4	55,0	75,6	98,0	72,5	98,3	-
PamYlprE	50,6	94,3	98,7	60,8	94,8	98,0	62,5	96,0	98,0	40,7	89,0	98,5	62,5	96,0	97,8
GünYlprE	51,4	92,9	98,0	61,2	92,6	97,5	62,8	95,9	98,0	41,9	90,1	98,6	62,4	95,2	97,6
GarYlprE	52,0	91,5	97,8	62,6	93,4	97,7	62,8	93,4	98,0	41,6	91,2	98,7	63,3	95,4	97,0
Pal YlprE	51,7	93,5	98,1	62,7	92,6	98,0	63,0	94,1	98,0	42,2	91,3	98,5	62,8	95,8	97,1
SoYlprE	52,2	93,1	98,4	62,5	94,7	98,1	63,2	94,2	98,0	42,4	91,0	98,5	63,0	95,0	98,0

2. Investigation of TMADHF, TMAA, DEADHF, DEAHF, and DEAUFB ionic fluids as catalysts for vegetable oil transesterification reactions with methyl, ethyl, iso-propyl, and butyl alcohols

First of all, the alkyl ethers of fatty acids was determined by varying the amounts of the ionic fluids listed above by 1-3 %, with the change in the ratios molar ratio of alcohol : fat within 4 :1-10 : 1, the reaction time in 6-10 hours range and the reaction temperature corresponds to the boiling temperature of the alcohols used. The optimal performance of the process achieved in 6 hours under normal conditions with 3 % catalyst and 6:1 alcohol:fat ratio.

Unlike the amin based catalysts studied in the first section, all the used ionic liquids have nearly the same and high catalytic activity (Chart 3).

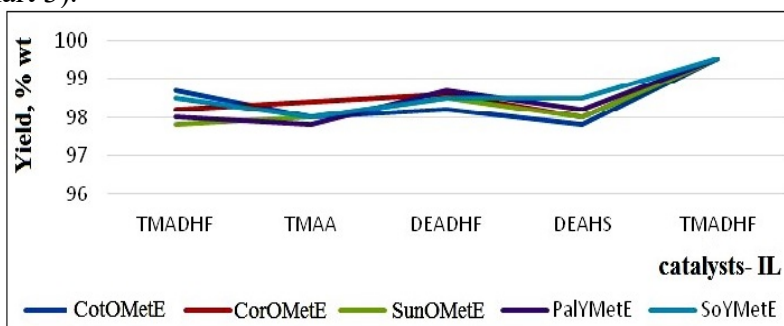


Chart 3. Comparative yield of methyl esters of cotton, sunflower, corn, palm and soybean depending on the ionic liquids used

At the next stage, oils transesterification process with simple alcohols under the effect of magnetic field and ultrasonic waves in determined optimum condition where listed ionic liquids used as catalyst was studied.

As expected, under the effect of magnetic field a 1.8-2.5-fold reduction in the duration of the processes compared to the conventional conditions observed. It was found that the used ionic fluids had higher activity compared to amine-based compounds (Chart 4).

As a result of studies conducted under the influence of ultrasonic cavitation, the optimum conditions of the process were determined.

Process duration 5 minutes, alcohol : fat ratio 6 : 1, the content of ionic liquids 2-2.5 %.

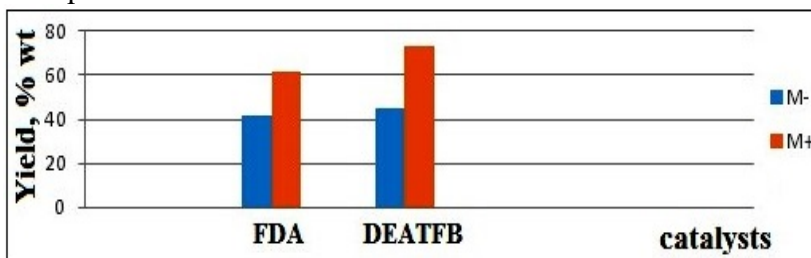


Chart 4. Comparative yields of methyl esters from sunflower oil in the presence (M +) and absence (M-) of magnetic field with FDA and DEAUFB catalysts

3. Investigation of methyl, ethyl, iso-propyl and butyl ethers of cotton, sunflower, maize, soybean and palm oil acids as a fuel and fuel components

Based on the research done, an advanced pilot device using ultrasonic or magnetic field is proposed for the production of biodiesel fuel (Figure 1).

Process: Alcohol and catalyst mix (S / K) is loaded into T-1 mixer with a mixer and stirred for a while (about 5-10 minutes) until the solid catalyst is completely dissolved in alcohol (when the catalyst is liquid. happens). Prepared S / K blends from the T-1 capacity through the M-1 pump to the screw Q-1 mixer to the T-2 intermediate harvesting capacity. From the T-2 capacity, the S / K blend is directed to the Q-2 screwdriver using the M-2 pump. At the same time, vegetable oil is heated to 70-800 °C by turning the T-3 into an ID-I heater and transported to an ultrasonic cavitator reactor (UK) through a Q-K mixer with a M / 3 pump. Here, the mixture of S / K and vegetable oil enters the separator chamber (K-4) as a two-phase mixture (ie, the ether and glycerol phase), mixed with the ultrasonic frequency created by the cavity S-3 sonotrode for a few seconds.

Up to -850 °C only increases at the expense of the cavitation effect. In addition, the pressure in the system is increased by the evaporation of alcohol used to 0.3-0.5 MPa.

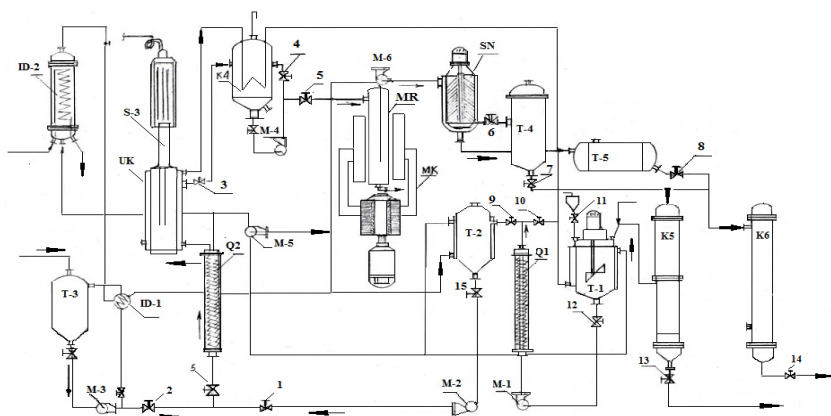


Figure 1. Pilot device for biodiesel fuel acquisition under ultrasonic cavitation and magnetic field. T-1- alcohol / catalyst mix, T-2 retention, T-3-vegetable oil capacity, Q-1, Q-2-teapot mixers, UK-ultrasonic cavitation reactor, S3-ultrasonic sonotrode, ID- 1, ID-2 heat exchangers, K-4-unit chamber, MR magnetic reactor, SN-separator, crude T-4, T-5 boosterizer and glycerin capacity, K5 and K6-purifier columns, 1-15 flaps

Alcohol is removed from the K-4 chamber, and the liquid phase is pumped to the SN separator via the M-4, and the corresponding alkyl ethers of the fatty acids obtained from it are added to the T-4 capacity and the glycerol to the T-5 capacity and finally to K5 and K6 KU-2-8 ions are removed from the catalyst residue through the columns filled with exchange resins.

To influence the process magnetic field, vegetable oils and S / K blends into the ID-2 heater after a Q2 screwdriver, where the temperature of the reaction solution is heated to the boiling temperature of the alcohol used and the M-5 pump in the magnetic field. here the process lasts 1.2-2.1 hours. When the process is complete, the reaction solutions pass through the separator SN to the capacities of T4 and T5, and the columns K5 and K6 are transferred to the final product capacity.

The process of obtaining the biodiesel fuel under the influence of ultrasound and magnetic field on the basis of cotton, corn oils and methanol in the presence of TEA catalyst in the above mentioned

material balance shown in Table 5. Mathematical optimization of the process was carried out on the basis of the initial input parameters, and the practical results obtained were confirmed.

Table 5

Material balance of the process of obtaining biodiesel fuel under the influence of ultrasound and magnetic field on the basis of cotton, corn oils and methanol in the presence of TEA catalyst

Indicators	Under the influence of ultrasound		Under the influence of the magnetic field	
	cotton oil	sunflower oil	cotton oil	sunflower oil
Taken, % wt				
Vegetable oil	77,5	77,5	77,5	77,5
Methanol	20,0	20,0	20,0	20,0
Catalyst TEA	2,5	2,5	2,5	2,5
Total	100	100	100	100
Received, % wt				
Biodiesel	76,8	77,1	76,0	75,8
Glycerin	8,6	7,0	8,2	6,8
Total mono and diglycerides	0,7	0,8	0,9	1,2
Alcohol	10,7	12,0	11,5	12,5
Losses	1,0	1,0	1,2	1,4
Catalyst TEA	2,2	2,1	2,2	2,3
Total	100	100	100	100

The alkyl ethers obtained from the process of transesterification of studied vegetable oils with simple alcohols were investigated as fuel (Table 6) and as a fuel component (Table 7).

Table 6

Quality indicators of synthesized alkyl ethers of cotton, sunflower, maize, soya and palm oils

Indicators	EN 1424	CotOMetE	SunOEE	CorOlprE	SoyOButE	PalOMetE
Cetan number	51	52	52	52	53	53
Kinematic viscosity, mm ² /sc	3,5-5,0	3,94	3,87	4,08	4,18	4,38
Density 15 °C, kg/m ³	860-900	888,9	886,0	884,8	886,1	885,9
Flash point in closed crucible, °C	120	125	125	132	132	130
Ash content, % wt	0,02	0,001	0,001	0,001	0,012	0,001
Content of esters, % wt	96,0	96,0	98,0-99,0	98,0-98,5	98,8-99,0	99,0
Coking ability 10 %-li of the remainder	0,3	0,1	0,1	0,2	0,14	0,1
Acid number mq KOH/1q	0,5	0,38	0,28	0,38	0,18	0,3
Jodine number, q J ₂ /100q	120	82,7	83,4	94,3	96,3	21,2
Content of alcohol, % wt	0,2	0,01	-	0,017	0,02	-
Content of monoglycerides, % wt	0,80	0,4	0,4	0,6	0,05	0,05
Content of diglycerides, % wt	0,2	0,1	0,12	0,1	0,1	0,1
Content of gliserides, % wt	0,2	0,1	0,03	0,15	-	-
Content of gliserine, % wt	0,25	0,18	-	0,11	-	-
Boiling range, °C	-	260-350	255-352	260-355	255-360	265-350

Analysis of the data clearly shows that the obtained methyl, ethyl, iso-propyl and butyl ethers of fatty acids fully meet the requirements of EN 14214 standard for biodiesel fuels and can be used as fuel.

Table 7
Properties of mixture of commercial diesel fuel (CDF)
with 5-20 % methyl, ethyl, iso-propyl
and butyl ethers of corn and palm oils acids

Indicators	EN-590 (2004)	CDF	CDF + 5 % MetE of		CDF + 10 % EE of		CDF + 15 % IprE of		CDF + 20 % ButE of	
			Palm	Corn	Palm	Corn	Palm	Corn	Palm	Corn
Density at 20 °C, kg/m ³ , no more than	860,0	848,7	850,6	850,2	849,1	848,8	853,0	852,4	855,1	854,4
Fraction composition, %										
50 % is expelled	280	280	280	280	280	280	280	280	280	280
90 % are expelled	350	335	342	340	346	344	349	346	350	350
96 % are expelled	360	358	360	355	360	355	360	360	360	360
The temperature of the ignition in the indoor tigel should not be less than, °C	55	75	77	77	77,5	77,8	78,5	78,7	79	79
Kinematic velocity at 20 °C, mm ² /sec, no more	2,0-6,0	3,40	3,55	3,47	3,68	3,60	3,91	3,91	4,83	4,57
Freezing temperatures, °C, should not exceed	-10(-35)*	-30	-28	-29	-26	-28	-23	-25	-20	-21
Cloud point, °C, should not exceed	-25(-10)*	-19	-17	-18	-15	-16	-13	-15	-11	-12
Copper plate test, 50°C, 3 hours	+	+	+	+	+	+	+	+	+	+
The amount of aromatic hydrocarbons, % wt	15,0	16,0	15,2	15,4	14,6	14,6	13,7	13,6	12,3	12,5
Acidity, mg KOH/100 cm ³ fuel, no more	5	1,2	1,35	1,42	1,34	1,45	2,23	2,54	2,87	2,84
Iodine number mg KOH/100 g fuels, no more	6	0	1,0	4,75	1,6	4,89	3,67	5,95	4,85	7,75
The total sulfur content, %, should not be much	0,005-0,001	0,0102	0,010	0,010	0,0096	0,0096	0,0092	0,0090	0,0080	0,0080
The actual amount of resin in 100 cm ³ of fuel should not be more than mg	25	20	18,4	18,6	17,1	17,4	16,6	16,0	15,1	15,3
Low burning temperature kC/kg	-	43480	42500	42465	42400	42380	41580	41465	40580	40380
Cetan is number, no less	51	46	47	47	47,5	47,5	48	48	51	51

Those facts that the cetane rating of synthesised biodiesel fuels are

52-53, self-ignition temperatures are 125-132 °C and sulfur compounds are absent, once again proves that these fuels are safe being fire-resistant and environmentally friendly. Alkyl esters of fatty acids obtained as a fuel component were studied by adding 5-20 % to the commercial diesel fuel.

As it is known, glycerol is obtained as a by-product of biodiesel fuels production and its conversion into useful products can make biodiesel production more economically viable.

With this in mind in this thesis, the synthesized glyceryl-triacetate and glyceryl-monooleate ethers can be studied independently as well as in combination with methyl ethers of palm oil as a fuel component and may be recommended as a resource-additive to fuel content (Table 8).

Table 8
Quality indicators of 2.5-5.0 % wt compounds
by glyceryl-triacetate and glyceryl-monooleate
ethers with commercial diesel fuels

Indicators	CDF + GTAE		CDF + GTAE/ CotOMetE		CDF + GMOE		CDF + GMOE / CotOMetE	
	2,5%	5,0%	2,5 %	5,0 %	2,5 %	5,0 %		
Density at 20 °C, kg/m ³ , no more than	852,6	853,8	851,0	851,6	849,7	850,3	851,2	851,8
Fraction composition, %								
50 % is expelled	279	277	280	280	279	278	280	280
90 % are expelled	340	342	341	342	345	349	341	342
96 % are expelled	355	355	355	360	355	360	355	360
The temperature of the ignition in the indoor tigel should not be less than, °C	75	74	75	76	77	78	77	78
Kinematic velocity at 20 °C, mm ² /sec, no more	3,97	4,02	3,55	3,64	4,1	4,28	3,95	4,10
Freezing temperatures, °C, should not exceed	-30	-31	-30	-29	-30	-31	-30	-29
Cloud point, °C, should not exceed	-19	-21	-18	-20	-19	-21	-18	-20
The amount of aromatic hydrocarbons, % wt	15,4	15,2	15,4	15,2	15,3	15,3	15,1	15,1
Acidity, mg KOH/100 cm ³ fuel, no more	1,85	1,92	1,76	1,80	1,42	1,58	1,32	1,51
Iodine number mg KOH/100 g fuels, no more	0	0	0	0	1,6	2,8	2,7	3,8
The total sulfur content, %, should not be much	0,0101	0,010	0,0101	0,010	0,010	0,010	0,010	0,010
The actual amount of resin in 100 cm ³ of fuel should not be more than mg	17,6	16,8	17,9	17,0	17,9	17,1	18,2	17,6
Low burning temperature kC/kg	41320	40165	41515	40690	41840	41135	41978	41580
Cetan is number, no less	46	46	46,5	46,5	46	46	46,5	46,5

Alkyl ethers of synthesized fatty acids and glycerol-monooleate and mono-acetate ethers were also investigated on commercial diesel fuel additives for enhancing the lubrication properties of diesel fuels and found that the fatty acids were less than 460 micrometers less than 2 % (Chart 5) of the fatty acids. monooleate and mono-acetate ethers are enough to add 75-125 mol.

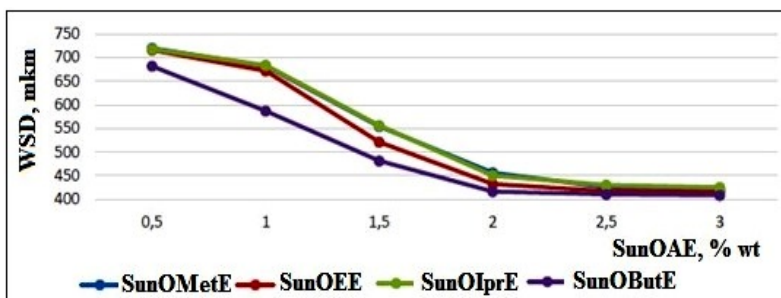


Chart 5. Dependence diagram diesel fuel wear spots diametrs (WSD)from the concentration of alkyl ethers of sunflower oil acid

Synthesized alkyl ethers were also investigated as a combustion modifier and it was found that the addition of fatty acids to alkali ethers in diesel fuel by 10-20 % resulted in 25.0-61.0 % reduction of CO content in exhaust smoke, and of 19.5 % reduction in smoke. The reduction of -56.0 %, combustion products of glyceryl triacetate ethers by 2.5-5.0 %, by 27.3 %, and the use of glycerol mono-olein ethers by 30.0-35.0 %.

CONCLUSION

1. The processes of transesterification of cotton, sunflower, maize, soybean and palm oil acids into methyl, ethyl, iso-propyl and butyl alcohols: diphenylamine (DFA), phenylenediamine (FDA), triethylamine (TEA), i-propylamine (IPA), butylamine (UBA) amino-compounds and tetramethylammonium dihydrofosphate (TMADHF), tetramethylammonium-acetate (TMAA), diethylaminehydrophosphate (DEADHF), diethylaminohydro-sulphate

(DEAHS) and diethylmethylene-diatenamine Investigated the process of obtaining methyl, ethyl, iso-propyl and butyl ethers of magnetic fields with an intensity of 0.2-0.9 T, 20 kHz ultrasonic waves, the effect of the twisted electric field and hydrodynamic cavitation were studied [11, 26].

2. It was found that in normal conditions alcohol:fat ratio 6: 1mol, process duration 6 hours, 3 % DFA, FDA and TEA utilization of fatty acids methyl, ethyl and butyl ethers as catalyst 94.6-99.3 % -propyl ether yield is 87.7-96.7 %, with the presence of catalysts IPA and UBA 84.5-95.8 and 80.2-87.9 % respectively; When the synthesis process of alkyl esters of vegetable fatty acids is carried out under a magnetic field with 0.2T intensity, the yield of the target products is 99.0-90.7 % even if the process duration is 4.8-5.4 times less [4, 18].
3. It was found that the process of obtaining alkyl esters from the mentioned vegetable fatty acids under the influence of ultrasonic cavitation at 20 kHz was carried out at 2 %, with the catalyst DPA, FDA TEA and UPA catalysts at 3 % and 99.5-95.8 % and the duration of the process is reduced to 5 and 7 minutes respectively, with the necessary temperature conditions being provided by the cavitation effect [17].
4. It was determined that the process of obtaining monoalkyl esters from the vegetable fatty acids under the influence of the turbulent electric field should be carried out with the alcohol ratio 6 : 1 and the yield of compound ethers in 5 minutes is 96.7-99.2 % [2].
5. It was found that the process of obtaining monoalkyl esters of vegetable fatty acids is 2.5 % of the required solubility of DFA, FDA TEA and UBA catalysts in hydrodynamic cavitation conditions, alcohol: fat ratio 6 : 1, and methyl and butyl ethers for 10-12 minutes, while the ethanol and i-propyl esters have a catalyst concentration of 3.0 % [9].
6. Use of TMADHF, TMAA, DEADHF, DEAHS and DEAUFB ionic liquids as catalysts in the traditional process of obtaining monoalkyl esters of vegetable oils: alcohol: fat ratio 6 : 1, the yield of ethyl and iso-propyl esters is 90.4-95.5 %. Duration of

the process is 1.2-2.0 s and 5 min, respectively, in the presence of these catalysts, with the high yields of the target products, but with activation at the magnetic field of 0.2 TL and ultrasonic cavitation at 20 kHz frequency [3, 6, 19].

7. Methyl, ethyl, iso-propyl and butyl ethers of cotton, sunflower, maize, soybean and palm oil acids, as well as glyceryl triacetate and glyceryl-monooleate ethers have been studied and as fuels and fuel components monoalkyl ethers meet the requirements of the EN-14214 standard, and when the diesel fuel is added by 5-15 %, the mixture meet the requirements of the EN-590 standard. At the same time, glyceryl triacetate and glyceryl monooleate ethers were investigated as a fuel component in addition to 2.5-5.0 % as well as methyl esters of cotton fatty acids and were identified as glyceryl triacetate and glyceryl mono-oleate ethers. It is advisable to use freely 2.5 % and 5.0 % with PamYMet ethers. During this time, the low-temperature properties of the impurities improve, the cetane number increases by 0.5 units, and saves diesel fuel in the amount added ethers [8].
8. Studied the methyl 10, 15 and 20 % increase in esters of fatty acids in combinations with methyl, ethyl, iso-propyl and butyl ethers, as well as glycerol-triacetate and glyceryl-monooleate ethers of fatty acids of cotton, sunflower, maize, soybean and palm oils results 25,0-29,0; 42,0-46,0 % and 58,0-61,0 % decrease in carbon monoxide content in the smoke gases and provides smoke reduction 19,5-20,3, 40.8-42.0 and 55.0-56.0 % respectively. In the combustion products of diesel fuel containing 2.5-5.0 % glyceryl triacetate ethers, 27.3 % reduction of carbon monoxide and at the same concentration of glyceryl mono-olein ether 30.0-35.0 % reduction of carbon monoxide was observed [7, 22].
9. Studied the lubrication properties of monoalkyl esters of vegetable fatty acids in diesel fuels and found that adding methyl esters 2.5 % ethyl, iso-propyl and butyl ethers of vegetable oils results in wear spot diameter reduction from 850 μm , to 420, 430, 452, and 415 μm . At the same time, the addition of 2.0 % of mixed acetate ethers of cottonseed acids leads to a reduction in edema

spot diameter to 440 μm . The lubrication properties of glycerol mono-oleate and mono-acetate ethers in commercial diesel fuels have been investigated and found that in compounds obtained from addition of 75 mol glycerol mono-oleate, and 125 mol of glycerol mono-acetate ether wear spot diameter is 440 and 452 μm respectively [12, 13].

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