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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

**SYNTHESIS OF BIODIESEL FROM VEGETABLE OILS IN
THE PRESENCE OF HETEROGENEOUS CATALYSTS,
PREPARATION OF COMPOSITIONS OF DIESEL FUELS
ON ITS BASE AND THEIR INVESTIGATION**

Speciality: 2314.01 - Petrochemistry

Field of science: Chemistry

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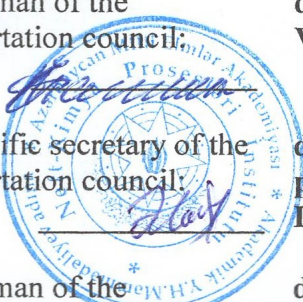
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GENERAL CHARACTERISTICS OF THE WORK

The relevance and the approbation of the subject. The intensive development of science and technology and the toughening of competitive economic environment in the world have also contributed to the rapid growth of environmental problems that will have a decisive impact on the future of humanity, and have made their solutions very relevant.

On the one hand, the intensive development of society and industrial production, on the other hand, the rapid increase in the number of fuel-efficient heating appliances, the ecological imbalance, and the possibility of the exhaustion of crude oil, which is the main source of fuel - all of these global problems of the modern world have been in the focus of public attention.

According to the literature, after 30 or 40 years crude oil, the main source of heat electric energy, is expected to be depleted. Analyses show that at maintaining the current speed of use today's 156.7 billion tonnes of oil will be exhausted after 41 years¹.

Therefore since the end of the twentieth century, interest in the search for alternative ways to obtain fuel from non-oil products has significantly increased. One of such alternative methods is biodiesel production from various vegetable oils (sunflower, corn, cotton, rape, soybean, palm, canola).

The synthesis and application of "green" catalysts in the process of obtaining biodiesel is one of the pressing problems of the present day.

The presented dissertation work is devoted to the synthesis of new effective catalysts for high-yield biodiesel production from vegetable oils and the development of ecologically safe and high-quality fuel compositions based on the obtained biodiesel.

¹ В.М. Аббасов, Т.А. Мамедова, М.И. Рустамов, А.Г. Талыбов, Н.К. Андрищенко. Биодизельные топлива из растительного сырья Азербайджана / Материалы конференции посвященной 100-летию акад. М.Ф. Нагиева, НАНА, Институт Химических Проблем, Баку, 2008, С. 198.

The object and the subject of the research. The main object of research is the production of biodiesel from vegetable oils with the participation of heterogeneous catalysts and the development of fuel compositions based on. The subject of the study is selection of various catalysts in the process of obtaining biodiesel by the transfer reaction of vegetable oils and the study of the operational properties of compositions based on the biodiesel.

The purpose and objectives of the research work. The main purpose of the research work is the preparation of nano CaO catalysts; $\text{La}_2(\text{SO}_4)_3/\text{TiO}_2$ catalysts and $\text{CeO}_2/\gamma\text{-Al}_2\text{O}_3$ catalyst containing 5-25% of CeO_2 and their application in the process of obtaining biodiesel from various vegetable oils, development of fuel compositions with the use of the obtained biodiesel and diesel fuel and study of their operational properties.

Research methods. The structure of the nano CaO particles synthesized by the new method has been studied by use of Atomic Force Microscope (C3MY-JИД5), and the structure of organic substances by modern analysis methods (IR-, ^1H , ^{13}C NMR-spectroscopy). The anti-wear lubrication properties of the prepared fuel compositions were determined in four-wheel friction machines (ЧТМ - 1) under 196 N load in accordance with ГOCT 9490-75, the corrosion aggressiveness of diesel fuel and the anti-corrosion protective effect of the fuel compositions by using the Pinkevich device (in accordance with ГOCT 5162-49) by modified contact method, microbiological tests were conducted according to ГOCT 9.023-74.

Basic provisions for defense. To achieve the purpose, the following research activities have been performed:

- Preparation of nano CaO; $\text{La}_2(\text{SO}_4)_3/\text{TiO}_2$ catalysts and $\text{CeO}_2/\gamma\text{-Al}_2\text{O}_3$ catalyst containing 5-25% of CeO_2 , study of their catalytic activity in the process of obtaining biodiesel from the reaction of vegetable oils with C_1 and C_2 alcohols;
- The determination of parameters and the study of the optimal conditions of the reaction of transesterification in the presence of nano CaO, $\text{La}_2(\text{SO}_4)_3/\text{TiO}_2$ and $\text{CeO}_2/\gamma\text{-Al}_2\text{O}_3$ catalysts in the process of

obtaining biodiesel from vegetable oils;

- Clean, loss-free separation of glycerol obtained along with biodiesel from reaction mixture and study of its new application areas;

- For the first time a dihydroxide derivative has been synthesized by oxidizing C=C bond of the ester (biodiesel) synthesized by the transesterification reaction, with KMnO_4 .

- Preparation of fuel compositions based on biodiesel and traditional diesel fuel and study of their operational properties (cetane number, lubricating and corrosion inhibition properties);

- Development and study of a new biostable fuel composition to increase the biodiesel resistance against microorganisms.

The scientific novelty of the research work. For the first time:

- Nano CaO catalyst has been developed by the new method and used in the transesterification reactions to obtain biodiesel fuel from vegetable oils (sunflower, corn, cotton).

- It has been found that in ethanolamine obtained nano CaO particles are smaller than in ethylene glycol. In the presence of nano CaO catalyst, yields of the biodiesel obtained in the transesterification reaction of vegetable oils with methyl and ethyl alcohols makes 90 to 95% accordingly.

- The transesterification reaction of vegetable oils with C_1 and C_2 alcohols in the presence of $\text{La}_2(\text{SO}_4)_3/\text{TiO}_2$ "green" catalyst has been studied. It has been established that the specified catalyst in comparison with other catalysts, can be used during several (3 - 5) cycles and regenerated.

- $\text{CeO}_2/\gamma\text{-Al}_2\text{O}_3$ catalyst containing 5 - 25% of CeO_2 has been prepared and applied in the transesterification reaction of vegetable oils. It has been shown that in spite of weak activity this catalyst can be used during several cycles compared to alkali, alkaline oxides, HCl and H_2SO_4 acids. One of advantages of the catalyst is the rapid separation from biodiesel and absence of necessity to rinse with water.

- For the first time a dihydroxide derivative has been synthesized by oxidizing C=C bond of the ester (biodiesel)

synthesized by the transesterification reaction, with KMnO_4 .

- It was developed a new method for separating glycerol as an intermediate product in the transesterification reaction from the reaction mixture as pure and without loss and aminomethyl and ester derivatives based on 1,3-dioxolanes of glycerol have been synthesized and studied as additives (antiwear and anti-corrosion, lubricating and biocide properties).

- Fuel compositions based on biodiesel and traditional diesel fuels have been developed and operational characteristics (increase the cetan number, properties of lubrication and corrosion inhibitors) have been studied;

- To ensure the resistance of compositions against to microorganisms the biological stability is ensured by adding the studied biocide additives (α -phenyl- β -nitroethen and α -furyl- β -nitroethen).

Theoretical and practical significance of the research work.

Studies have shown that biodiesel with high yield (90 - 95%) can be obtained from the transesterification of vegetable oils with methyl and ethyl alcohol in the presence of the new nano CaO catalyst.

With the presence of solid super "green" acid catalysts there is no need in water washing the biodiesel obtained from the transesterification reactions. This also demonstrates the practical importance of the work by saving clean water and without harming the environment.

Clean (99%) and loss-free separation of natural glycerol obtained from transesterification reactions is one of the main problems of the present day.

Experiments show that cetane number of biodiesel obtained from vegetable oils (47 - 49) is higher than the same indicator of normal diesel fuel (42 - 45).

It has been found that biodiesel, unlike diesel fuel, has high lubricating and corrosion inhibition properties as an additive.

Approbation and application of dissertation materials. The results of the dissertation have been reported and discussed at the following national and international scientific conferences:

VII-Baku International Mamedaliyev Conference on Petrochemistry, dedicated to the 80th anniversary of the Institute of Petrochemical Processes of the National Academy of Sciences of Azerbaijan (Baku - 2009); Proceedings of the V Republican Scientific Conference of doctoral students, masters and young researchers dedicated to the 88th anniversary of national leader Heydar Aliyev (Baku - 2011); Republican Scientific Conference dedicated to the 100th anniversary of academician A.M. Guliyev (Baku - 2012); II Republican Scientific Conference "Modern problems of chemistry of monomers and polymers", dedicated to the 50th anniversary of Sumgait State University (Sumgait - 2012); Alternative sources of raw materials and fuel. Abstracts of the IV, V, VI, VII International Scientific and Technical Conference (Minsk -2013; 2015; 2017; 2019); Republican Scientific Conference "Lubricants, fuels, special liquids, additives and reagents", dedicated to the 50th anniversary of academician A.M. Guliyev Institute of Chemistry of Additives. Abstracts (Baku - 2015); Abstracts of the IX Baku International Mammadaliyev Conference on Petrochemistry (Baku - 2016); International Scientific and Technical Conference dedicated to 100th Anniversary of acad. B.Q. Zeynalov on "Synthesis of Petrochemistry and Catalysis in Complex Condensed Systems" (Baku - 2017); Republican Scientific and technical conference dedicated to the 90th anniversary of Professor S.A. Sultanov on "Fuel, fuel components, special liquids, oils and additives". Abstracts (Baku - 2017); Proceedings of the scientific conference "Nagiyev's Readings", dedicated to the 110th anniversary of acad. M. Nagiyev (Baku - 2018).

The name of the organization where the dissertation work is performed. The work was performed at academician A.M. Guliyev Institute of Chemistry of Additives of Azerbaijan National Academy of Sciences.

The author's personal involvement. The main ideas included in the dissertation, the introduction and implementation of the tasks, experimental experiments, summarizing, reporting and publication of the dissertation were personally carried out by the author. Participation in co-authored scientific works included the direction of

the research, the selection and justification of the questions and methodology of the experts.

Publications on the dissertation work. 29 research papers have been published as a result of the dissertation work: 9 of them are articles, 2 of them are conference material and 15 are theses of reports. On the results of the dissertation work three patents of the Republic of Azerbaijan have been obtained.

Structure and volume of the dissertation work. The dissertation work consists of 155-page computer text: an introduction, 3 chapters, results and a reference list with 225 bibliographical names, including 44 tables, 14 figures and 7 graphics.

In the introduction section the relevance of the topic, the purpose of the dissertation, the scientific novelty of the work, its practical significance, the validity of the results, the approbation, structure and volume of the work are justified and the essence of the chapters is summarized.

The first chapter analyzes articles and patents on biodiesel production, proves the need for conducting research in this area and argues that the synthesis of biodiesel by the "Green Chemistry", and clean separation of glycerol as an intermediate substance are urgent problems.

The second chapter describes the preparation of nano CaO particles and "green" catalysts ($\text{La}_2(\text{SO}_4)_3/\text{TiO}_2$ və $\text{CeO}_2/\gamma\text{-Al}_2\text{O}_3$) and their application in obtaining the biodiesel and glycerol by transesterification reaction of vegetable oils with C_1 and C_2 alcohols, separation of pure glycerol and obtaining aminomethyl and ester derivatives, development of new fuel compositions based on biodiesel and traditional diesel fuels and study of their operational properties.

The third chapter provides analysis of the results of the conducted studies.

The results present the conclusion of the conducted research.

At the end of the dissertation the results of the research, including a list of references, were presented.

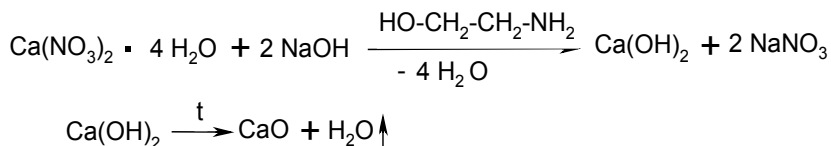
BASIC CONTENT OF THE RESEARCH WORK

The main purpose of the work is obtaining high-yield biodiesel from transesterification of vegetable oils with methyl and ethyl alcohols in the presence of the new synthesized catalysts and developing environmentally friendly fuel compositions with high-performance properties based on them.

One of the main problems in the synthesis of biodiesel is the purification of the obtained natural glycerol from non-reacting mixture of oil, di- and monoglyceride compounds. Currently there is a great demand for pure glycerol (99%) in the medical, cosmetic, food and chemical industries². This topical problem is also solved in the presented dissertation.

Preparation of nano CaO catalyst

Nano CaO catalyst was prepared according to the following scheme:



Equimolar mixture of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and NaOH in freshly distilled ethanolamine is rapidly stirred with a mechanical stirrer at room temperature for 30 min. After keeping the mixture for 5 hours formed $\text{Ca}(\text{OH})_2$ precipitate is filtered and washed and dried in a vacuum exciter.

The calcination process of $\text{Ca}(\text{OH})_2$ is carried out in a muffle furnace equipped with a special contact thermometer. Depending on the calcination temperature (up to 500°C) and time it is possible to

² Pereira, B.R., Carvalho, R.M., Caetano, S.T., Cosmetic production from glycerine the biodiesel // Periodico Tche Quimica, Brazil: – 2018. volume 15, № 30, – p. 185-192.

obtain 10 - 147 nm CaO particles.

The structure of nano CaO particles was studied in semiconductor mode (C3MY-JI15) with use of Atomic Force Microscope (Baku State University) [10].

Figure 1 presents the 2D and 3D projections of nano CaO particles.

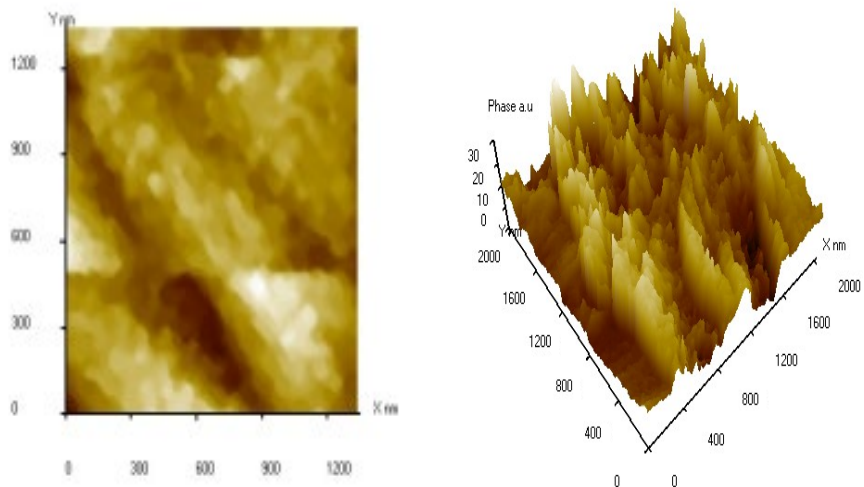


Figure 1. 2D and 3D projection of Nano CaO particles

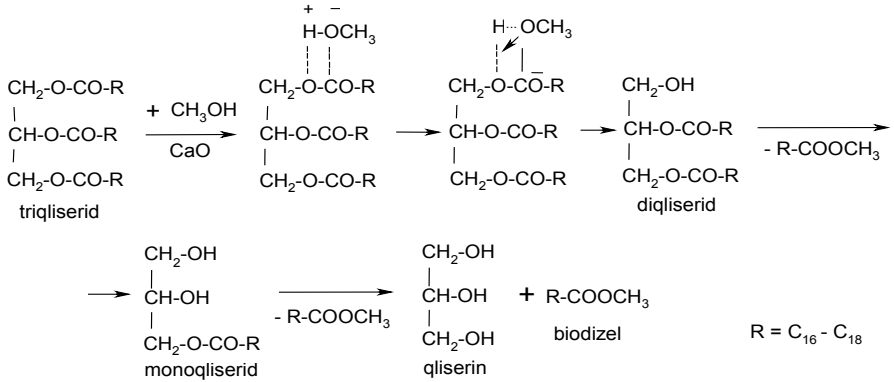
Synthesis of biodiesel and glycerol from the transesterification of sunflower oil with methyl alcohol catalysis of nano CaO

The transesterification reaction of vegetable oils can be divided into three stages. At the first stage biodiesel-diglyceride is obtained, at the second stage biodiesel-monoglyceride, at the third stage biodiesel-glycerol.

During the experiment the effects of nano CaO content, oil to alcohol molar ratio, temperature and reaction time on the biodiesel yield obtained from transesterification of sunflower oil with methanol were studied and the optimum condition was found: oil : CH_3OH = 1: 6, t = 60- 70°C, τ = 2 h, size of nano particles 10 -

15nm, content - 1.5%. Under this optimal condition the yield makes 95% [7].

The transesterification reaction proceeds according to the following scheme:



The effect of nano CaO catalyst content on biodiesel production is given in Figure 2. As can be seen from the graphic, increase in the nano CaO content from 0.5 to 1.5% causes rise of the biodiesel yield up to 90 - 95%. However, the further increase in nano CaO content reduces the biodiesel yield. The accepted optimum variant is 1.5%.

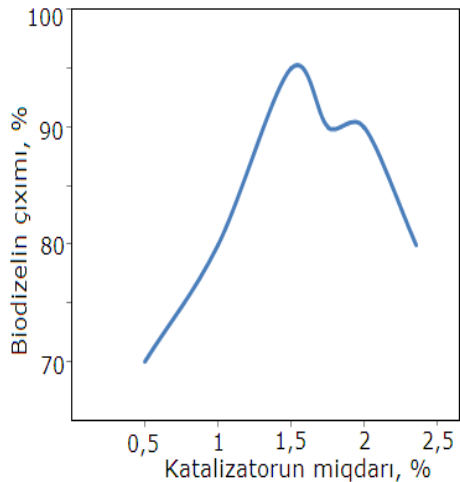
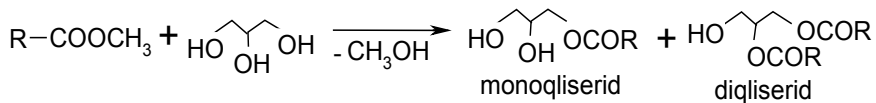


Figure 1. The effect of the concentration of nano CaO to biodiesel yield

It can be assumed that more than 1.5% of nano CaO causes the initiation of heteroreaction after the completion of transesterification, i.e. secondary formation of mono- and diglycerides as a result of the

partial reaction of the obtained biodiesel (methyl ester of C₁₆ - C₁₈ acids) and glycerol.

Increasing the kinematic viscosity and specific weight of the obtained biodiesel confirms the validity of this hypothesis:



It is known that sunflower oil contains triglycerides of the following acids: 55 to 65% linoleic, 33 to 36% oleic, 5 - 10% mixed palmitic and stearic acids. The biodiesel synthesized from this oil is also a mixture of the methyl esters of the above mentioned saturated and unsaturated C₁₆ - C₁₈ acids.

Contemporary GX analysis of the biodiesel (Agilent Technologies 7890A GC system) (Figure 3) shows that the content of saturated and unsaturated acid esters in its composition corresponds to the ratio of acids in oil. Based on it may be said that the various acid glycerides in sunflower oil produce almost the same amount of esters as methyl alcohol.

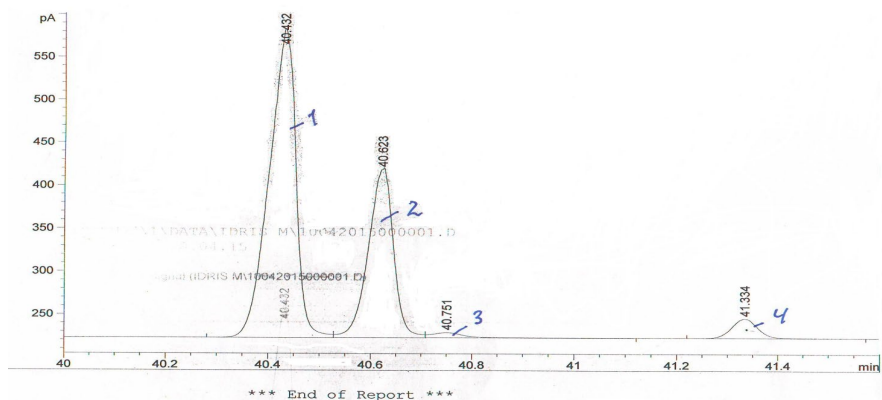


Figure 3. Chromatogram of biodiesel

In contrast to the spectrum of sunflower oil, in the H¹ - NMR spectrum of the biodiesel (Figure 4) there are no observable peaks (4.2-4.5 ppm) of protons belonging to the glycerol fragment -OCH₂-

CH(O)-CH₂O-. The peak of the -OCH₃ group is observed at 3.60 ppm in the spectrum. The protons of the biodiesel CH₃ and HC = CH fragments are observed at 0.85 and 5.30-5.40 ppm as intensive peaks.

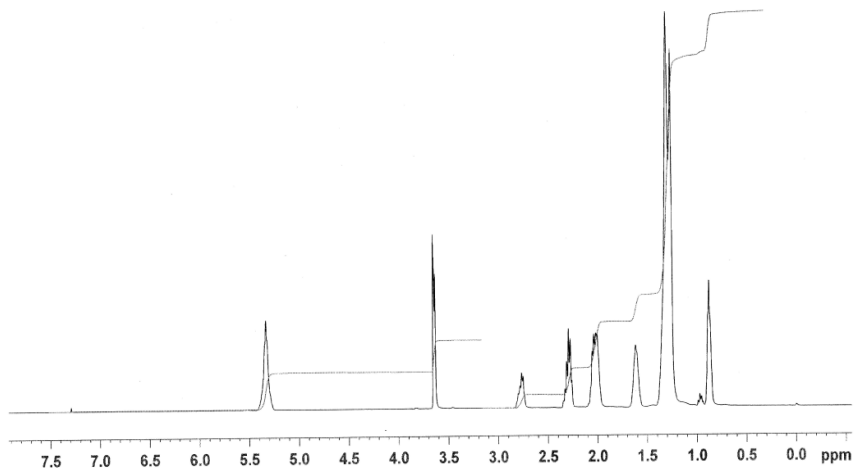
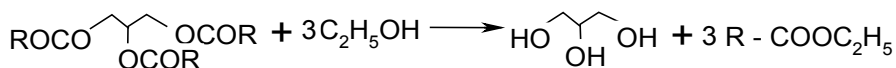


Figure 4. The ¹H-NMR spectrum of biodiesel

The structure of the biodiesel was also studied by using the Varian 3600 FT-IR spectrometer. In the spectrum, 1120 cm⁻¹, 1190 cm⁻¹ absorption bands correspond to the C-O bond, 1620 cm⁻¹ absorption bands to the C = C bond, 1740 cm⁻¹ absorption bands to the C = O bond. In the spectrum, 2750 - 3000 cm⁻¹ absorption bands correspond to valence vibrations of CH₂ v_{as} CH₃ groups.

By transesterification of sunflower oil with ethyl alcohol with nano CaO catalyst biodiesel with good yields (85 - 95%) was synthesized.



During the experiment it was found that ethyl alcohol also in the optimal condition found for the reaction with methyl alcohol (oil : C₂H₅OH = 1 : 6, t = 60 - 70°C, τ = 2 hours, catalyst - 1.5% (relative

to oil)) reacted with sunflower oil to form biodiesel with 90 - 95% yield.

The structure of biodiesel with ethyl radical was also studied by ^1H - NMR and ^{13}C - NMR spectroscopy (Figure 5).

The CH_3 group of the biodiesel carbonyl chain is observed in the form of triplet at 0.9 ppm, whereas the CH_3 group of the $-\text{CH}_3-\text{CH}_2\text{O}-$ (ethoxy) fragment is resonated as triplet at 1.1 ppm ($J = 7.2$ Hz), and the CH_2O group in the form of quadruplet at 4.15 ppm ($J = 7.2$ Hz). Protons of the $-\text{HC}=\text{CH}-$ bound in the biodiesel are observed at 5.30 - 5.50 ppm. In the ^{13}C -spectrum of biodiesel, carbon atom of the CH_3 group is observed at 15 ppm, $\text{C} = \text{C}$ atoms at 126, 129 ppm, and C atom of the $\text{C} = \text{O}$ group at 210 ppm.

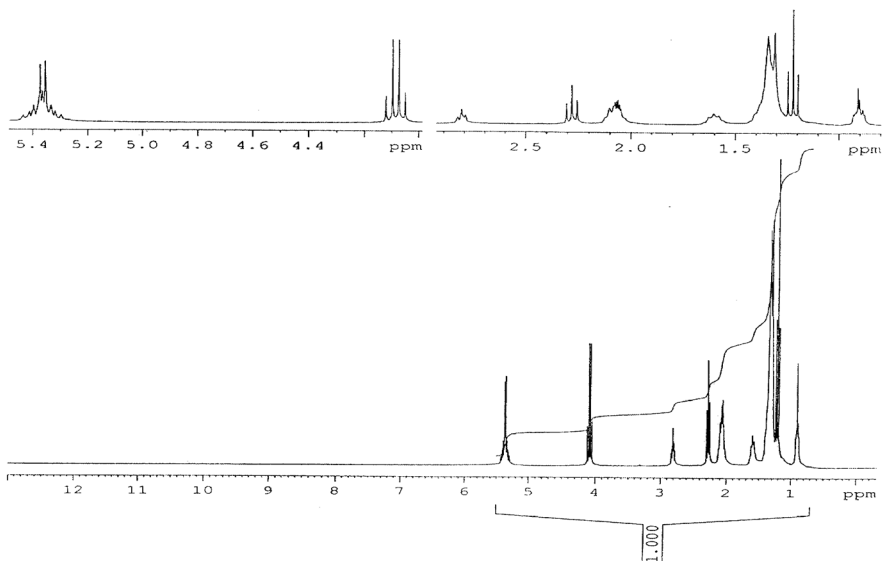


Figure 5. The ^1H -NMR spectrum of ethyl-biodiesel

It was found that corn and cotton oils with the presence of nano CaO also produced biodiesel with 80-90% yield in the transesterification reaction with methyl and ethyl alcohol under the optimal conditions determined for sunflower oil. It should be noted that the percentage of biodiesel extracted from cotton oil was lower

than in case of other two oils (80 - 82%). This is due to the fact that cotton oil contains more aminophospholipids than the other two oils.

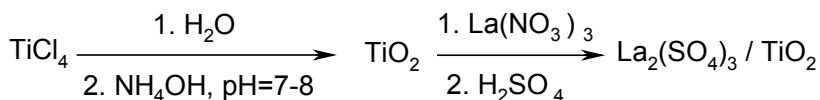
The physico-chemical properties of the biodiesel obtained from the transesterification of vegetable oils with C₁ and C₂ alcohols in the presence of nano CaO were studied.

It should be noted that alkaline and alkaline-earth metal oxides, basics and salts, as well as mineral acids (HCl, HNO₃, H₂SO₄) are used as a catalyst in the transesterification reaction during only one cycle and obtained biodiesel has to be washed with water several times. It is economically disadvantaged. Therefore, the synthesis of heterogeneous catalysts and their application in the reaction of transesterification is a pressing problem.

Preparation of La₂(SO₄)₃/TiO₂ catalyst and application in the reaction of transesterification of vegetable oils with C₁ and C₂ alcohols

The La₂(SO₄)₃/TiO₂ catalyst is a solid heterophilic acid of the Keggin type. There is some information in the literature on the use of the catalyst only in the esterification reaction³.

In the presented work, La₂(SO₄)₃/TiO₂ solid heterophilic acid was used as a catalyst for the production of biodiesel from the reactions of vegetable oils with the methyl and ethyl alcohols [14]. The catalyst was synthesized according to the following scheme known in the literature:



The effect of vegetable oil to alcohol ratio, catalyst content, temperature and time on biodiesel production was studied during the experiment. With the presence of La₂(SO₄)₃/TiO₂ catalyst, in the

³ Yanq, L., Guo, H., Huanq, Y. Synthesis of ethyl oleate catalyzed by solid superacid SO₄⁻²/TiO₂/La⁺³ // Chemical Researches of Chinese Universities. – 2010. №26(1), – p. 92-97

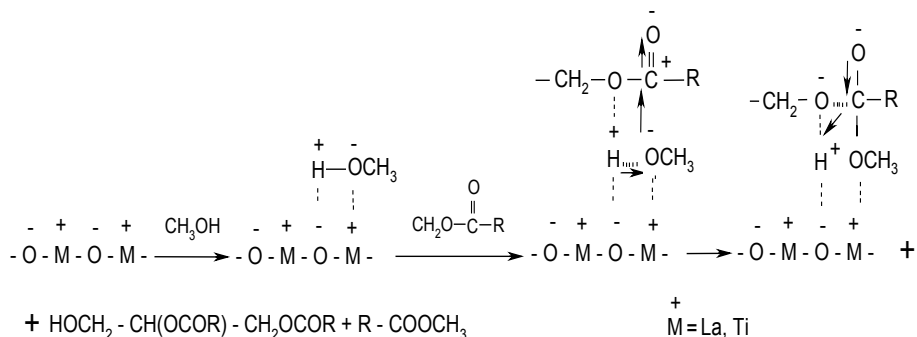
ratio of sunflower oil to methyl alcohol as 1 : 6 and reaction time 4 hours biodiesel yield makes 40%.

When the reaction time lasts for up to 6 hours, the yield rises to 69%. But after 8 hours the yield remains stable. It has been established that an increase in the catalyst by up to 6% (relative to oil) leads to an increase in the biodiesel yield. However, further growth doesn't affect the biodiesel production.

It should be noted that with the presence of $\text{La}_2(\text{SO}_4)_3/\text{TiO}_2$ catalyst biodiesel was synthesized under the above mentioned optimum conditions from corn and cotton oils too.

One of the advantages of this catalyst is its regeneration after the experiment.

As with the Keggin type catalysts, the mechanism of the transesterification reaction of vegetables oils with the methyl and ethyl alcohols with the presence of the $\text{La}_2(\text{SO}_4)_3/\text{TiO}_2$ catalyst is as follows.



As can be seen from the scheme, methyl alcohol is mixed with the catalyst. At this time the hydrogen atom of methyl alcohol on the surface of the catalyst forms a weak bond with the oxygen, while the methoxy fragment of the catalyst forms a weak bond with positive metal oxide and due to these bonds the methyl alcohol is polarized.

At the next stage, the polarized methoxy group on the surface of the catalyst attacks the nucleophile in the carbonyl group in the oil, the negative charge of the methoxy group is attached to the carbonyl

group >C=O , and the hydrogen atom bound with the oxygen atom of the catalyst combines with the oxygen atom of the glycerol in the oil. As a result of redistribution of electron charges in the complex formed by methyl alcohol, vegetable oil and catalyst, a biodiesel and a diglyceride molecule form from one oil (triglyceride) molecule.

At the end of the reaction, two layers are formed: the upper layer consists of a mixture of biodiesel, a small amount of oil, a mixture of mono- and diglyceride, and the bottom layer is a mixture of catalyst and crude glycerol.

The catalyst separated from the glycerol layer can be again used after water washing and regenerating in the muffle furnace at 250°C for 3 hours. However, in the following cycles, the biodiesel yield decreases. Decrease in the catalyst activity can be explained by partial separation of lanthanum sulfate from the catalyst during water washing.

During the experiment it was found that with the presence of the $\text{La}_2(\text{SO}_4)_3/\text{TiO}_2$ catalyst it is possible to synthesize biodiesel with 50-60% yield as a result of transesterification reaction of the corn and cotton oil with C_1 and C_2 alcohols.

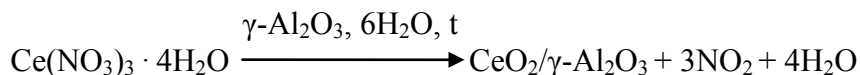
Preparation of $\text{CeO}_2/\gamma\text{-Al}_2\text{O}_3$ catalyst and application in the reaction of transesterification of vegetable oils with C_1 and C_2 alcohols

It is known from literature that CeO_2 and Al_2O_3 separately don't exhibit catalytic activity in the transesterification of vegetable oils with alcohols without modification with alkaline metal oxides and alkalines. Adding a certain amount of CeO_2 to the composition of Keggin type solid catalysts significantly increases their activity.

After calcination of the mixture of these oxides to a certain proportion (10-25%) the catalytic activity of oils in the transesterification of vegetable oils with alcohols allows one to think that a certain synergistic effect is produced.

For this purpose, the process of precipitation of 5-25% CeO_2 on

γ -Al₂O₃ was performed by the following equation:



In the beginning calculated amount of Ce(NO₃)₃ · 4H₂O and γ -Al₂O₃ are mixed in distilled water at 80°C until the homogeneous system is formed. After separating water on the “Rotopar” device it is dried in the muffle furnace at 350-400°C for 4 hours.

It was found that in the presence of CeO₂/ γ -Al₂O₃ catalyst containing 5% CeO₂, biodiesel with 10% yield is obtained from the reaction of sunflower oil with methyl alcohol (1:6 ratio). However, in the presence of catalyst CeO₂/ γ -Al₂O₃ containing 25% of CeO₂ (relative to 8%) in the optimum condition (t=90°C, τ =8h) the biodiesel yield makes 60-66%.

The subsequent increase in the amount of the catalyst doesn't affect the biodiesel yield. This fact shows that a certain proportion of CeO₂ and γ -Al₂O₃ mixtures has a "synergistic" effect [5].

The CeO₂/ γ -Al₂O₃ catalyst is also easily separated from the mixture after the reaction as the La₂(SO₄)₃/TiO₂ catalyst. In the presence of this catalyst also biodiesel was synthesized with 40 - 50% yield from corn and cotton oils.

However, after the regeneration process the activity of the serum-containing catalyst is significantly reduced compared to the lanthanum catalyst. This is due to the greater separation of CeO₂ from the catalyst during water washing.

Transesterification reaction of vegetable oils with alcohol in the presence of both serum and lanthanum catalysts takes place by the same mechanism.

Separation of pure (99%) glycerol from biodiesel mixture obtained in the transesterification reaction and its some conversion reactions

It is known that glycerine which is an integral part of biodiesel is obtained in the reaction of transesterification of vegetable oils and

animal fats with low molecular ($C_1 - C_2$) alcohols in the presence of alkaline, alkaline metal oxides and their salts.

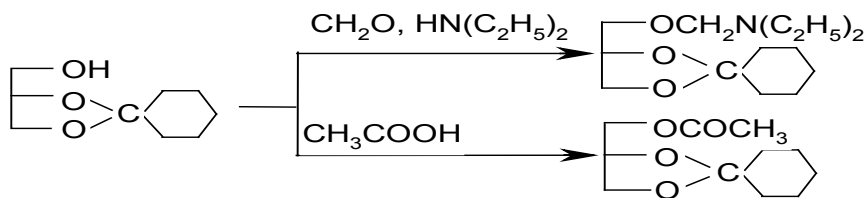
Recently, the process of pure and loss-free separating of the glycerol which in the process obtained in parallel with biodiesel from the reaction mixture and search of its new application areas has been actualized according to the rapidly increasing demand for biodiesel and the technology of biodiesel production modernization.

If taken into account that natural glycerin (99%) is a precious chemical product which of widely used in medicine, perfumery, cosmetics, food, petrochemical and other industries, its clean separation from mixture by the method of efficiently and economically advantageous it will be clear great scientific and practical significance as a topical problem.

It was found that under the influence of 5% H_3PO_4 on glycerol layer obtained from the transesterification reaction of sunflower oil with methanol in the presence of nano CaO , substances contained in the mixture (CaO and $Ca(OH)_2$) gravitate in the form of surfactants calcium salts of fatty acids ($Ca_3(PO_4)_2$).

Because of reduction in the mixture density the mixture of mono-, di- and triglyceride is easily separated from the glycerol layer. After distillation of the glycerol layer by the NMR - spectroscopy method its 99% purity was determined [16].

Some of the glycerol derivatives (4-diethylamino-methoxymethyl-2,2-pentamethylene-1,3-dioxolan and 4-acetoxy-methyl-2,2-pentamethylene-1,3-dioxolan based on 1,3-dioxolans) were synthesized under the following scheme and their properties were studied [22].



By adding in various concentrations of synthesized additives to traditional diesel fuel compositions were developed and tests have been carried out for the research of their operational properties. The tests were accomplished both at the laboratory conditions and at the engine test station. The properties as additives (antiwear lubricating, anti-corrosion, biocide in oil products) of the synthesized compounds to diesel fuel have been comprehensive studied.

It was found that during the exploitation of the composition prepared by adding aminomethyl derivative to diesel fuel improved the anti-wear properties of lubrication properties of engine components by 36 to 40% and is protected from corrosion by 86%. In addition were tested antimicrobial activity that damaging the oil products and were determined to have bactericidal properties. It is effective against bacteria in both diesel fuel and M-8 oil.

Anti-wear properties of lubrication properties of the composition prepared by adding this compound to diesel fuel is improved 16 - 32%, prevents corrosion by 87% and the cetan number increases. It is also considered satisfactory for diesel fuels.

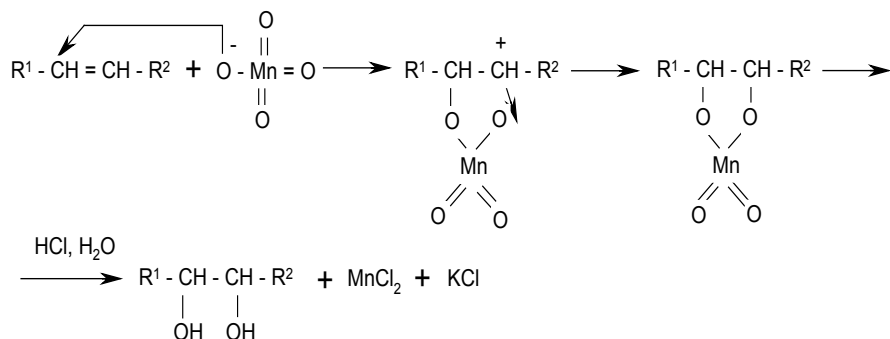
Investigation of the dihydroxylation reaction of double bonds of ester that derived from the transesterification reaction of sunflower oil with methyl alcohol by KMnO_4

The presence of one or more double bonds >C=C< and complex ester groups ($-\text{COOCH}_3$) in the molecule of complex ethers derived from the transesterification reaction of sunflower oil with methyl alcohol attracts attention as well as multifunctional reagent that improves the operational properties of diesel fuel.

Considering this, for the first time, the dihydroxylation reaction of the double bond of the ester that synthesized from the transesterification reaction of sunflower oil with methyl alcohol was carried out at room temperature with KMnO_4 aqueous solution [25].

Dihydroxylation reaction of the ester that synthesized from the transesterification reaction occurs by the following mechanism:

As shown in the equation, the MnO_4^- anion joins one of the double bond of ester to form a carboanion intermediate, and then the positive carbon atom forms an intermediate substance together with the oxygen atom of the manganese ion. At the end of the reaction, when this complex is broken down by weak HCl acid, dihydroxyl compound occurs .



The structure of the synthesized dihydroxybiodiesel was studied by IR–spectroscopy method. It was found that in the IR spectrum of dihydroxybiodiesel, there is a wide absorption band of the hydroxyl group is observed in the $3000\text{--}333\text{cm}^{-1}$ area, in contrast to the biodiesel spectrum.

Development of compositions based on biodiesel and diesel fuel and study of their operational properties

The fact that biodiesel has different chemical composition than that of hydrocarbons, the presence of a polar ester group with adsorption ability in the molecule, a double bond may give it a higher lubrication, corrosion inhibition and other beneficial properties. In this regard, the comparative study of lubrication, corrosion and setan number indicators of diesel fuel and diesel compositions contained various amount of biodiesel was conducted.

One of the main factors affecting to the exploitation qualities of diesel fuel is the problem of improving its anti-wear lubricating

properties.

The problem of wear of the fuel apparatus details as a result of friction occurs during the fuel is supplied to the combustion chamber with the help of the pump by oil burner (sprayer). In addition to the main function of fuel in this process, it must also perform lubrication. Otherwise, regulation of settings details of the pump that working under extreme conditions is disrupted, the useful coefficient of the pump decreases and operating time is shortened. Particularly, the lubricating properties is getting deteriorate at he high temperature conditions due to the viscosity of the fuel falls down, the corrosion effect is increases, insoluble solids are formed as a result of oxidation.

Taking into account the above-mentioned it becomes clear how effective additives that to ensure normal operation of apparatus for engine fuel are needed increasing the viscosity of fuel, improving its anti-wear lubricating properties.

For this purpose, fuel compositions have been developed in various variants by adding biodiesel as additives to diesel fuel and have been studied the properties of the anti-wear lubricating of metal particles during their operation.

The anti-wear lubrication properties of the compositions were determined in four-wheel friction machines (ЧТМ - 1) under 196 N load in accordance with ГОСТ 9490-75. It was found that the use of compositions improved the durability of engine components by 20-31% [17].

These results confirm the possibility that biodiesel may have a higher lubrication effect than oil-derived diesel fuel and justifies the possibility that the details in which the fuel compositions are in contact in the operation process may be longer.

One of the major problems arising during the exploitation of diesel fuels is the corrosion they are in contact with metall parts. Equipments exposed to corrosive out of order premature, great expense is required for their repair or replacement. During storage, transportation and operation of fuels, their corrosion activity increases. As a result, the emerging aggressive environment in

contact with metal of the fuel causes to accelerate the corrosion process. One of the method delaying the corrosion process in fuels, preventing it is the addition of special inhibitors. This is an economically favorable and efficient method, because added inhibitor contains only a small part of the cost of the fuels.

Taking into account the above-mentioned it one of the direction of researches dedicated to the study of the corrosion properties of fuel compositions based on biodiesel and traditional diesel fuels.

The corrosion aggressiveness of diesel fuel and the anti-corrosion protective effect of the fuel compositions that prepared by adding biodiesel to diesel fuels was studied by using the Pinkevich device (in accordance with ГOCT 5162 - 49) by modified contact method.

Tests were performed in the following order to obtain the effectiveness of the compositions and the accuracy of the results obtained. First of all, the degree of corrosion of the diesel fuel itself. The test result is taken as the average of the parallel trials. After determining the corrosion rate of the fuel, compositions based on biodiesel and diesel fuel are tested, the process is repeated, and the corrosion prevention effect is calculated.

Based on the test results, it was found that the compositions had a high inhibition (100%) properties and the metal parts contacting with them were not corrosive [8, 12].

The great attention is required to regulate the cetan number of diesel fuels during its production. Taking into account the importance and relevance of the effect to the exploitation properties of fuel, to study of the cetan number of the prepared compositions is considered appropriate. The most convenient and purposeful method to improve of the cetan number of the diesel fuel is to use special organic compounds.

Synthesized methyl and ethyl ethers (biodiesel) from the reaction of transesterification of sunflower oil with methyl and ethyl alcohol and traditional diesel fuels were used to carry out the experimental studies. For this purpose, the fuel compositions were prepared biodiesel by adding in different concentrations - 3, 5, 7% (mass) to diesel fuel and comparatively cetan number are studied. Based on the

results of our research, biodiesel can be considered as high-performance additive that improving the cetan number of diesel fuel.

The setan number of compositions was determined by of Y.Churshukov empirical formula and studied at the Baku Oil Refinery. It was established that the use of compositions led to increase in the setan number of diesel fuels and saving of the fuel [11].

Improvement of biocide properties of fuel composition based on biodiesel and diesel fuel

Although there are some positive feature during the storage, transportation and operation of fuel composition based on biodiesel and diesel fuel, one of the factors that adversely affecting the exploitation properties of compositions is their exposure to microorganisms.

The main disadvantage of biodiesel and diesel fuel is that it is exposed to microbiological damage as a result of microbe and bacteria effect after certain period of production (3 months). As a result, their hydrocarbon structure are changed, the physical and chemical properties of biodiesel are degraded, its acidity and corrosion aggression increase and it becomes useless as fuel. The speed of this process depends on the storage conditions of fuels. Thus, at high humidity and temperature microorganisms develop more intensively.

The most universal method of combating microbiological damage of fuels is the use of special bioside additives. It has been determined in researches that biodiesel has several advantages as a multifunctional additive compared to diesel fuel.

There is no information in the literature on microbiological studies of biostability of biodiesel and its composition with diesel fuel, because the synthesis of biodiesel from vegetables oils is a new research, although there is extensive information about the improvement of its biocide properties by adding bioside additives to diesel fuel. For the first time, the researches that improvement of the

biocide property by adding biocide additives to biodiesel was carried out by us [24].

α -Phenyl- β -nitroethene and α -furyl- β -nitroethane that both high-performance and easily obtainable have been used to protect biodiesel against microbiological damage as biocide additives. These biocides do not precipitate when they are present in the composition for a long time while finding out their effect to the basic physical, chemical and operational properties, it was found that biocides do not have a negative effect anti-corrosion, anti-oxidation properties, and the cetan number of the fuel compositions [18].

As a result of conducted studies it was found that by the addition of 0.1-0.5% of α -phenyl- β -nitroethene and α -furyl- β -nitroethene into the composition of biodiesel and diesel fuel it was completely protected from microbiological damage for a long time [26].

Microbiological tests were conducted according to GOST 9.023-74. The main group of microorganisms damaging oil fuels were used for testing. Based on the results of the experiments it was found that while 7% composition prepared on the base of biodiesel and diesel fuel were completely damaged by both bacteria and fungi on the second day of the test, the compositions containing 0.1 - 0.5% of α -phenyl- β -nitroethene and α -furyl- β -nitroethene had complete and long-term resistance to microbial damage. Although 8-oxyquinoline accepted as etalon at a concentration of 0.4% has anti-bacterial effect, but it has no effect on fungi.

Thus, to obtain biodiesel from vegetable oils, small-particle nano CaO and Keggin type "green" catalysts ($\text{La}_2(\text{SO}_4)_3/\text{TiO}_2$, $\text{CeO}_2/\gamma\text{-Al}_2\text{O}_3$) were developed. To increase the biodiesel resistance to microorganisms new compositions were developed and their operational properties were studied.

RESULTS

1. To obtain high-yield biofuel from vegetable oils (sunflower, corn, and cotton) for the first time highly effective nano CaO particles have been obtained by a new method, their sizes and 2D,

3D projections have been studied with use of atomic force microscope in semiconductor mode. It has been established that the smaller nano CaO particles (10-15nm) can be obtained by replacing ethyleneglycol with ethanolamine used as a solvent [9, 10].

2. Higher activity of small-sized nano CaO (10-15nm) in comparison with high-sized nano CaO (100-147nm) and normal CaO particles in the reaction of transesterification of vegetable oils with C₁ and C₂ alcohols have been established. The biodiesel yield makes 95% [2, 7].

3. By studying parameters of the transesterification reaction and optimal conditions it has been established that when using 1,5% (relative to oil) of nano CaO catalyst in the process of obtaining biodiesel from vegetable oils under oil : alcohol = 6 : 1 volumetric ratio, and at 60-70°C temperature and within 2 hours the yield of biodiesel reaches 95%.

4. For the first time, biodiesel has been synthesized from transesterification of vegetable oils with (C₁ and C₂) alcohols with the participation of "super" solid acid La₂(SO₄)₃/TiO₂ and CeO₂/γ-Al₂O₃ catalysts. It has been established that La₂(SO₄)₃/TiO₂ catalyst compared to homogenous catalysts has ability to participate in several cycles and to be reused being regenerated [14].

5. Although CeO₂ and γ-Al₂O₃ have no catalytic effect alone in the reaction of transesterification of vegetable oils with alcohols, their mixture in a certain proportion influence as Keggin-type catalysts. This is explained by "synergistic" effect between Ce and Al metals. The CeO₂/γ-Al₂O₃ double oxide which retains certain CeO₂ on the γ-Al₂O₃ surface, in comparison with γ-Al₂O₃ has higher effect as a catalyst in the transesterification reaction [5].

6. To separate pure (99%) and loss-free glycerol which is obtained as intermediate product in the transesterification reaction from the reaction mixture, for the first time an economically responsible method has been developed. Some conversion reactions of the obtained glycerol have been carried out and the obtained compounds have been studied as additive for diesel fuels [22, 29].

7. Dihydroxybiodiesel has been synthesized as a result of dihydroxylation of double bond of the ester (biodiesel), obtained by the transesterification reaction, with KMnO_4 . It has been found that when added dihydroxybiodiesel to the diesel fuel, its cetane number increases and other operational properties don't change [25].

8. Fuel compositions based on synthesized biodiesel and diesel fuel have been developed and studied. It has been established that by adding 3-7% (mass.) of biodiesel to diesel fuel the cetane number of the prepared composition increases and fuel is saved, anti-wear resistance of details improves by 20-31% during their operation on diesel engines, metal constructions contacted with fuel are not exposed to corrosion, introduction of the studied biocide additive into the composition provides biological stability of fuel against microorganisms, prolongs its storage period, and, most importantly, leads to the protection of the environmental ecology [18, 24, 26, 28].

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1. Movsumzadeh, M.M, Makhmudova, L.R, Aliyev, N.A. Transesterification of vegetable oils with catalysis of CaO nano particles // VII-Baku International Mammadaliyev Conference on petrochemistry dedicated to the 80th anniversary of the Institute of Petrochemical Processes of the National Academy of Sciences of Azerbaijan, – Baku: – 29 September – 2 October, – 2009, – p. 130. 29 сентября – 2 октября, – 2009, – с. 130.
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