ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫНЫҢ

Д.В.Сокольский атындағы «Жанармай,
катализ және электрохимия институты» АҚ

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
электрохимии им. Д.В. Сокольского»

NEWS

OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF KAZAKHSTAN
JSC «D.V. Sokolsky institute of fuel, catalysis
and electrochemistry»

SERIES
CHEMISTRY AND TECHNOLOGY

4 (436)

JULY-AUGUST 2019

PUBLISHED SINCE JANUARY 1947

PUBLISHED 6 TIMES A YEAR

ALMATY, NAS RK

Clarivate
Analytics

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Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

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«ҚР ҰҒА Хабарлары. Химия және технология сериясы».
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Меншіктенуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» Республикалық қоғамдық бірлестігі (Алматы қ.)
Қазақстан республикасының Мәдениет пен ақпарат министрлігінің Ақпарат және мұрағат комитетінде 30.04.2010 ж. берілген **№1089-Ж** мерзімдік басылым тіркеуіне қойылу туралы куәлік

Мерзімділігі: жылына 6 рет.
Тиражы: 300 дана.

Редакцияның мекенжайы: 050010, Алматы қ., Шевченко көш., 28, 219 бөл., 220, тел.: 272-13-19, 272-13-18,
http://chemistry-technology.kz/index.php/en/arhiv

Типографияның мекенжайы: «Аруна» ЖК, Алматы қ., Муратбаева көш., 75.

«Известия НАН РК. Серия химии и технологии».
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан» (г. Алматы)
Свидетельство о постановке на учет периодического печатного издания в Комитете информации и архивов Министерства культуры и информации Республики Казахстан **№10893-Ж,** выданное 30.04.2010 г.

Периодичность: 6 раз в год
Тираж: 300 экземпляров

Адрес редакции: 050010, г. Алматы, ул. Шевченко, 28, ком. 219, 220, тел. 272-13-19, 272-13-18,
http://chemistry-technology.kz/index.php/en/arhiv

Адрес редакции: 050100, г. Алматы, ул. Кунаева, 142,
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News of the National Academy of Sciences of the Republic of Kazakhstan. Series of chemistry and technology.
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Owner: RPA "National Academy of Sciences of the Republic of Kazakhstan" (Almaty)
The certificate of registration of a periodic printed publication in the Committee of Information and Archives of the Ministry of Culture and Information of the Republic of Kazakhstan N 10893-Ж, issued 30.04.2010

Periodicity: 6 times a year
Circulation: 300 copies

Editorial address: 28, Shevchenko str., of. 219, 220, Almaty, 050010, tel. 272-13-19, 272-13-18,
http://chemistry-technology.kz/index.php/en/arhiv

Editorial address: Institute of Organic Catalysis and Electrochemistry named after D. V. Sokolsky
142, Kunayev str., of. 310, Almaty, 050100, tel. 291-62-80, fax 291-57-22,
e-mail: orgcat@nursat.kz

Address of printing house: ST "Aruna", 75, Muratbayev str, Almaty

N E W S
OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN
SERIES CHEMISTRY AND TECHNOLOGY
ISSN 2224-5286 **https://doi.org/10.32014/2019.2518-1491.45**
Volume 4, Number 436 (2019), 66 – 73

UDC 542.941.7; 665.35

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HYDROGENATION OF PALM OIL ON Pt-CONTAINING CATALYSTS

Abstract. The article is devoted to the study of the catalytic hydrogenation of palm oil to obtain a high yield of fat without double bonds and trans-isomers. The catalysts used were platinum supported on various carriers - alumina, ball aluminosilicate, synthetic HY zeolite and activated carbon, among which ball aluminosilicate was the most active. The effect of process temperature and hydrogen pressure on the content of double bonds and trans-isomers of fatty acids was studied. In the temperature range of 80–200°C and pressures of 0.3–1.5 MPa, the content of C = C bonds decreases from 54 g I_2/100g, and the content of trans fats from 22 to 0%. On the Pt/aluminosilicate catalyst at 200°C and 1.5 MPa, double bonds and trans-isomers of fatty acids are completely absent in the reaction products. Using the BET, porometry and EM methods, a uniform formation of zero-valence metal particles on the catalyst surface was established, and nanoscale particles of Pt were determined.

Keywords: palm oil, catalyst, hydrogenation, platinum, aluminosilicate.

Introduction

More than 60-70% of the oil and fat industry in the world is based on hydrogenated oils. Unhydrogenated fats are rancid, get oxidized due to double bonds, they have an unpleasant smell and taste. Hydrogenation slows down these processes and, in addition, allows from cheap oils to produce more valuable solid fats, which is the basis for the production of food margarines and technical hydrogenated fats.

Since the 2000s, the world has introduced a ban on trans fats, called "fat-killers". The following negative consequences of eating trans fats have been established: weakened immunity, reduced resistance to stress, the risk of depression, the birth of children with pathologically low weight, the risk of developing diabetes, a negative effect on the joints and connective tissue, the enzyme cytochrome oxidase, neutralizing carcinogens, they lead to atherosclerosis, ischemic heart disease, cancer, obesity, visual impairment, etc. [1-2].

In this regard, the content of trans-isomers in oil-fat products got stringent prohibition. In developed countries, their content in products is minimized or completely banned. As early as 2003, WHO recommended that the content of trans-fats in the caloric content of the diet should not exceed 1%. Legislative limitations of trans fat content of no more than 1-2% in products are introduced in the USA, Canada and all European countries. The FDA agency (USA) legally permits the content of trans fats below 0.5 g per 100 g of product [3-4].

According to the requirements of the Technical Regulations of the Customs Union (CU) "Technical regulations for fat and oil products" (TR CU 024/2011), the content of the isomers of fatty acids from 01.01.2018 for hard, soft and liquid margarines, spreads and mixtures should not exceed 2% [5]. In Kazakhstan, currently in widely consumed oil-fat products, the content of trans fats is up to 40-50%. Therefore, reduction of trans-isomers in edible oil-fat products is an actual and urgent problem in our country, since from 2018 onwards it would be necessary to produce products in accordance with the Technical Regulations of the Customs Union. The urgency of the problem of developing new catalysts for the hydrogenation of vegetable oils is also determined by the fact that the demand for hydrogenated fats is high, and the existing catalysts do not meet the requirements of modern technology in a number of parameters.

The problem of hydrogenation of fats is covered in many studies of scientists and technologists. Such studies are conducted in the developed countries of Europe, America and Asia [6-7]. In general, all studies are devoted to the partial hydrogenation of various vegetable oils and cetylated fatty acid esters, which makes it possible to form trans-isomers as more stable, and to reduce the degree of isomerization in order to reduce the content of trans isomers. The most studied and effectively used in developed countries are nickel catalysts and catalysts based on platinum metals.

When hydrogenating sunflower oil on a Ni/SiO_2 catalyst using 3 texturally different silica gel materials, the authors achieved the optimal fat composition [8]. It is shown that the high activity of the catalyst results in a greater number of reaction routes and side chemical reactions.

Hydrogenation of sunflower oil in cis-C18:1 was tested on heterogeneous catalysts - palladium and platinum deposited on Al_2O_3, ZrO_2 and TiO_2 [9]. It was found that Pd-catalysts are much more active than the industrial Ni-catalyst and the lowest yields of trans fats were obtained. Pt-catalysts were also applied to carriers of different nature: ZSM-5 with platinum nanoparticles [10], diatomite from the Mugodzhar deposit of Kazakhstan [11], Al_2O_3, ZrO_2, CeO_2, TiO_2, MgO, $BaSO_4$ and MoO_3 [12].

The reduction of the $Pd\text{-}B/\gamma\text{-}Al_2O_3$ catalyst with potassium borohydride (KBH_4) affects the structure of the catalyst, which leads to high conversion and reduces the selectivity for trans-isomerization compared to the unreduced catalyst [13].

The effect of the nature of the precursor of platinum on the activity of catalysts has been studied [14]. The catalyst prepared from $Pt(AcAc)_2$ is more active than H_2PtCl_6. The effect of a change in the Pt content (0.7-4.6% wt) on a new mesoporous SiO_2 for the hydrogenation of sunflower oil was studied. It is shown that the smaller the platinum, the lower the content of trans-isomers, which is due to the smaller catalyst particles.

Modification of catalysts also leads to a decrease in the formation of trans-isomers. Silver-modified Mg-Ni/diatomite catalysts have shown a decrease in trans-isomerization during the hydrogenation of soybean oil [15]. Partial hydrogenation of sunflower oil on $Pd/\gamma\text{-}Al_2O_3$, modified with magnesium glycine revealed a minimal formation of trans-isomers.

Studies show that on catalysts based on noble metals with the hydrogenation of vegetable oils the yields of trans fatty acids are lower than those from nickel catalysts. Ni-catalysts are not very active below 120°C, and platinum ones are active at temperatures up to 70°C. Pt, Pd and Ru are the most active catalysts for the hydrogenation of vegetable oils, but on Pt-catalysts there're the minimum amount of trans fatty acids.

Monolithic catalysts based on anodized aluminum $Pd/Al_2O_3/Al$ showed high activity when hydrogenating sunflower oil with 50% conversion C=C [16]. These structured catalysts may be a good alternative in the partial hydrogenation reaction.

In addition to conventional catalytic methods for carrying out hydrogenation processes, new ones are proposed, such as electrocatalytic hydrogenation, hydrogenation in supercritical conditions, hydrogenation using membranes and cold plasma, which have shown promising results in reducing trans fatty acids. But these alternative methods are not brought to the introduction for industrial production of fats without trans-isomers yet.

Hydrogenation of sunflower oil in supercritical propane and dimethyl ether 2% Pd/C showed the best profile of hydrogenated products, supercritical conditions significantly affect the melting characteristics compared to conventional hydrogenation [17].

A significant decrease in the content of trans fatty acids in soybean oil is demonstrated by a low-temperature electrochemical process [18]. Compared to the commercial process of hydrogen gas hydrogenation, the application of this method leads to a decrease in trans fatty acid by 80%.

Membrane reactors are proposed in [19], which address the drawbacks of the process of conventional three-phase hydrogenation, such as the insufficient provision of the catalyst with hydrogen, the slow diffusion mass transfer of reactants from the gas phase through the liquid to the active sites of the catalyst.

High-voltage atmospheric cold plasma (HVACP) was studied to produce partially hydrogenated soybean oil without the formation of trans fatty acids [19]. This study is the first study demonstrating an alternative technology to conventional catalytic hydrogenation.

Despite a large number of studies in this field, industries of almost all the countries of the world, including in Kazakhstan, use the method of classical hydrogenation on nickel catalysts, in which the double bonds do not completely hydrogenate and a sufficient number of trans isomers is brewed. In particular, Johnson Mattey company's catalysts PRICAT 9910 and 9920 (nickel catalyst based on silicon dioxide with a content of 22% wt) are used in Kazakhstan.

Disadvantages of industrial processes and catalysts are a short service life (one-time catalyst), a laborious filtration stage for separating the catalyst from the target product and significant losses of fat and active catalyst phase. But the main disadvantage of these catalysts is that nickel is partially present in the hydrogenated fat, which is harmful to the human body. Therefore, harmless hydrogenation catalysts, such as platinum-containing catalysts, are needed.

Usually, hydrogenation of double bonds of vegetable oils on metal catalysts leads to the formation of trans-isomers, as the most stable substances. It is generally accepted that platinum catalysts produce the least amount of trans-fatty acids upon hydrogenation.

The aim of this work is to develop active selective palm-oil hydrogenation catalysts for the production of fat-free high-yield grease without double bonds and trans-isomers.

Experimental part

At preparation of catalysts was used $H_2PtCl_6 \cdot 6H_2O$ of "chemically pure" mark. Solutions of this compound was applied by the adsorption method on the prepared carriers: alumina, ball aluminosilicate, synthetic HY zeolite and activated carbon. The catalyst samples were filtered off and dried at 100-110°C to constant weight. The reduction of supported catalysts was carried out in a quartz tube with electrical heating in a hydrogen stream at 200°C for 4 hours, then the catalysts were cooled in a hydrogen stream until room temperature.

The hydrogenation process was carried out by Amar Equipment Ltd autoclave in the isobaric-isothermal regime in the temperature range of 80-200°C and hydrogen pressure of 0.3-1.5 MPa. The analysis of the reaction products was carried out for the content of the double bond (method of iodine number) and content of trans-fats by gas chromatography on Crystal-5000 chromatograph.

The catalysts were studied by physicochemical methods of analysis: measurement of the catalyst surface (BET), electron microscopy (EM). The surface of the catalysts was examined by the BET method on nitrogen adsorption by "Accusorb" instrument. Electron microscopy of samples was studied by means of electron microscope EM-125K by replica with extraction using microdiffraction (160,000 magnification).

Results and discussion

The influence of the nature of the support on the content of double bonds in the salomas (Table 1) was studied during the hydrogenation of palm oil on Pt-catalysts. ShAS - ball aluminosilicate carrier brand ShAS-2, alumina, activated carbon and HY zeolite were used as supports.

When hydrogenating palm oil on platinum catalysts on the listed supports at 1.5 MPa and 200°C, the content of double bonds varies depending on the type of support and decreases from 54% in the original palm oil to 0%. The content of trans-isomers also varies depending on the support - from 21 to 0%. The most active catalyst is Pt/ShAC, on which the content of C = C bonds and trans-isomers is 0.

The effect of temperature and pressure of hydrogen on the double bond content in the lard (Tables 2 and 3) was studied during the hydrogenation of palm oil on the most active catalyst - Pt/ShAS. With an increase in temperature from 80 to 200°C, the content of C = C bonds decreases from 46 to 0 g I_2/100g. The content of trans-isomers of fatty acids with increasing temperature increases from 1% in the initial oil to 22%, with a further increase in temperature to 200°C, the trans-isomers completely disappear as all double bonds (Table 1).

Table 1 - The content of double bonds and trans-isomers of fatty acids at hydrogenation of palm oil on the Pt/support catalysts at 200°C and 1.5MPa

Catalyst	Content of double bonds, g I_2/100g	Content of trans-isomers, %
Initial oil before treatment	54	1
Pt/C	47	21
Pt/Al_2O_3	5	3
Pt/HY	21	10
Pt/ShAC	0	0

Table 2 - The content of double bonds and trans-isomers of fatty acids at hydrogenation of palm oil on the Pt / ShAC catalyst at 0.3 MPa and different temperatures

Temperature, °C	Content of double bonds, g I_2 / 100g	Content of trans-isomers, %
Initial oil before treatment	54	1
80	46	15
120	28	22
150	5	3
200	0	0

Table 3 - The content of double bonds and trans-isomers of fatty acids at hydrogenation of palm oil on a Pt /ShAC catalyst at 120°C and various hydrogen pressures

Pressure, MPa	Content of double bonds, g I_2 / 100g	Content of trans-isomers, %
Initial oil before treatment	54	1
0,3	28	22
0,5	21	15
1,0	15	11
1,5	8	3

Similar to the effect of temperature, as the pressure rises, the content of double bonds decreases in catalyzate from 54 in the initial palm oil mode to 8g I_2/100g at 1.5 MPa. The content of trans-isomers first increases to 22%, while at 1.5 MPa it decreases to 3% (Table 3). This is due to the complete hydrogenation of the double bonds of palm oil.

When the hydrogen pressure is changed from 0.3 to 1.5 MPa, the time of the catalytic treatment of palm oil decreases from 65 to 32 minutes (figure 1). The absence of C=C bonds in this case after the experiment was observed at 1.5 MPa. The content of trans-isomers is also equals 0.

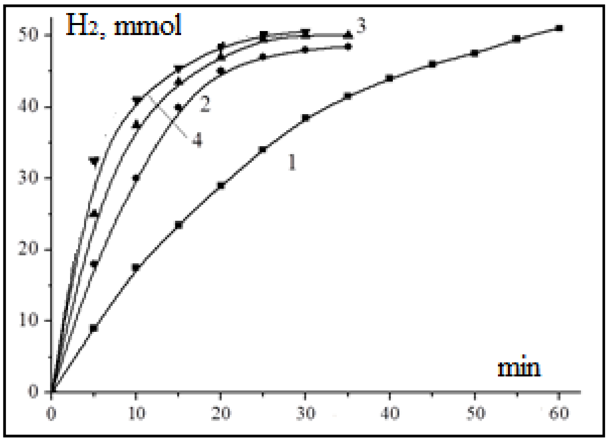


Figure 1 - Hydrogenation of palm oil at different hydrogen pressures on Pt/ShAS catalyst at 50°C. a - 1 – 0.3; 2 - 0.5; 3 - 1.0; 4 -1.5 MPa

An increase in temperature from 80 to 200°C increases the rate of hydrogenation of palm oil (Figure 2, b) and the apparent activation energy is 40.8 KJ/mol.

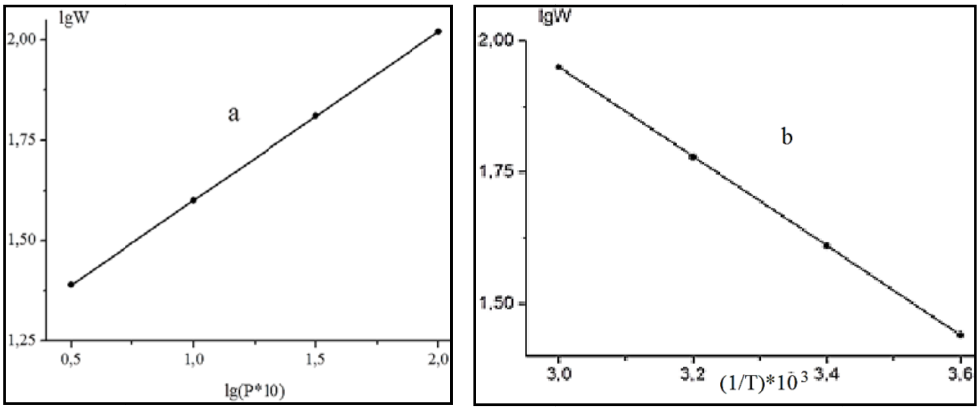


Figure 2 - Hydrogenation of palm on Pt/ShAS at 1.0 MPa. a - bilogarithmic dependence of rate from pressure; b - dependence of the logarithm of rate from reciprocal temperature

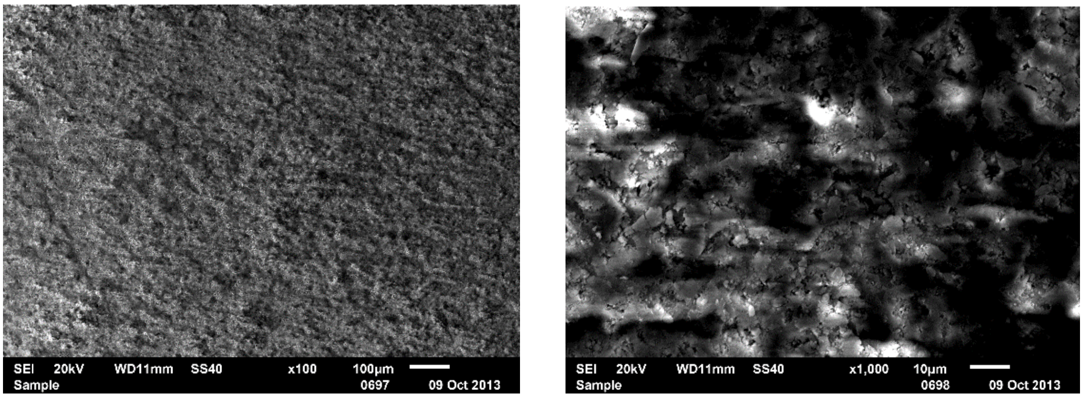
Thus, according to the results of Tables 2-3 and figures 1-2 on the Pt /ShAC catalyst, the optimal temperature and pressure are 200°C and 1.5 MPa, at which the double bonds and trans-isomers of fatty acids are completely absent in the reaction product.

The catalysts were studied by physicochemical methods of analysis by BET and EM. In table 4 the characteristics of used 4 catalysts are presented. Pt/ShAC has the largest surface 201 m^2/g and pore size 15-20 Å.

Table 4 - Surface area and pore size of the prepared supported catalysts

Catalyst	S^*, m^2/g	d^*, Å
Pt/C	175	12-25
Pt/Al$_2$O$_3$	164	4-14
Pt/HY	185	5-18
Pt/ShAC	201	15-20

The surface of catalysts was studied by scanning electron microscopy. In figure 3 of different increase of the Pt/ShAC catalyst is shown the uniform surface of the carrier, on which agglomerates of active metals are viewed.



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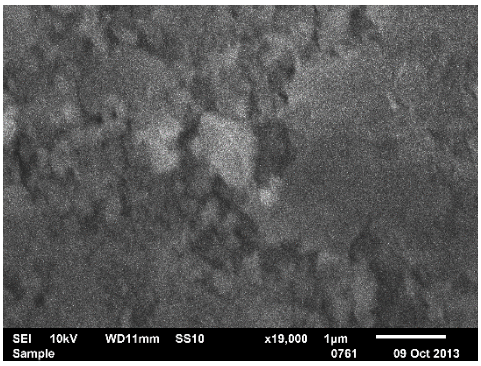

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Figure 3 - EM images of Rh-Pt (9:1)/Al_2O_3

The most active catalyst Pt/ShAC was investigated by transmission electron microscopy (TEM). On figure 4 Pt/ShAC-catalyst was presented by fine particles 2-2.5 nm in size and a small amount of more dense and large particles of 50 nm. Microdiffraction patterns of particles are represented by diffuse rings, corresponding to metals Pt^o which are most active in hydrogenation of double bonds of fatty acids of palm oil. Apparently, high catalyst activity is due to the uniform distribution of nanoscale particles on the surface of catalyst.

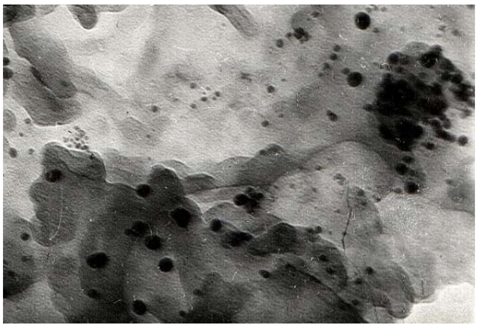

Figure 4 - TEM image of catalyst Pt/ShAC (increase 160,000)

A distinctive feature of the used catalysts and technology from the nickel catalysts widely used on an industrial scale is the complete hydrogenation of double bonds of fatty acids to 100% removal of trans-isomers [20]. In addition, the further application of the process of trans-esterification of hydrogenated fats with liquid vegetable oils that do not initially contain trans-isomers leads to optimum margarines, spreads and milk fat substitutes that are in accordance with the norms of the Technical Regulations of the Customs Union.

Conclusions

Platinum supported on various carriers - alumina, ball aluminosilicate, synthetic HY zeolite and activated carbon were prepared. Among carriers ball aluminosilicate was the most active.

The effect of process temperature and hydrogen pressure on the content of double bonds and trans-isomers of fatty acids was studied. In the temperature range of 80–200°C and pressures of 0.3–1.5 MPa, the content of C = C bonds decreases from 54 g I_2/100g, and the content of trans fats from 22 to 0%. For Pt/aluminosilicate catalyst optimal parameters were selected: 200°C and 1.5 MPa double bonds and trans-isomers of fatty acids are completely absent in the reaction products. Apparent activation energy for hydrogenation of palm oil is 40.8 KJ/mol.

Using the BET, porometry and EM methods, a uniform formation of zero-valence metal particles on the catalyst surface was established, and nanoscale particles of Pt were determined.

ӘӨЖ 542.941.7; 665.35

А.Т. Масенова, А.С. Сасс, О.А. Фролова, Н.Р. Кензин, М.К. Калыкбердиев, А.Байкен

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ҚҰРАМЫНДА Pt БАР КАТАЛИЗАТОРЛАРДЫ ПАЛЬМА МАЙЫНДА ГИДРЛЕУ

Аннотация. Мақала саломастың қос байланыссыз және транс-изомерлерсіз жоғары өнімділігін алу үшін пальма майының каталитикалық гидрирленуін зерттеугe арналған. Катализаторлар ретінде әртүрлі тасымалдағыштар қолданылды – алюминий оксиді, шарикті алюмосиликат, синтетикалық цеолит НY және белсендірілген көмір қолданылған, олардың арасында ең белсенді шарикті алюмосиликат болды. Үрдіс температурасы мен сутегі қысымы май қышқылдарының қос байланыстары мен транс-изомерлерінің құрамына әсері зерттелді. С=С құрамы 80-200^{o}С температуралық интервал мен 0,3-1.5 Мпа қысымда 54 г-нан I_2/100 г дейін, ал транс-майлардың мөлшері 22-ден 0% - ға дейін азаяды. Pt/алюмосиликат катализаторында 200^{o}С және 1,5 МПа кезінде реакция өнімінде май қышқылдарының қос байланысы және транс-изомерлері болмайды. БЭТ, порометрия және ЭМ әдістерімен металл бөлшектері катализаторлардың беттік қабатына валенттіліктің нөлдік деңгейінде біркелкі орнатылған және Pt нано өлшемді бөлшектері анықталды.

Түйін сөздер: пальма майы, катализатор, гидрлеу, платина, алюмосиликат.

УДК 542.941.7; 665.35

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ГИДРИРОВАНИЕ ПАЛЬМОВОГО МАСЛА НА Pt-СОДЕРЖАЩИХ КАТАЛИЗАТОРАХ

Аннотация: Статья посвящена исследованию каталитического гидрирования пальмового масла для получения с высоким выходом саломаса без двойных связей и транс-изомеров. В качестве катализаторов использовались платиновые, нанесенные на различные носители – оксид алюминия, шариковый алюмосиликат, синтетический цеолит НY и активированный уголь, среди которых наиболее активным оказался шариковый алюмосиликат. Исследовалось влияние температуры процесса и давления водорода на содержание двойных связей и транс-изомеров жирных кислот. В температурном интервале 80-200^{o}С и давлений 0,3-1.5 Мпа содержание С=С связей уменьшается с 54 г I_2/100г, а содержание транс-жиров с 22 до 0 %. На катализаторе Pt/алюмосиликат при 200^{o}С и 1,5 МПа в продукте реакции полностью отсутствуют двойные связи и транс-изомеры жирных кислот. Методами БЭТ, порометрии и ЭМ установлено равномерное образование на поверхности катализаторов частиц металлов в нулевой степени валентности, определены наноразмерные частицы Pt.

Ключевые слова: пальмовое масло, катализатор, гидрирование, платина, алюмосиликат.

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ISSN 2518-1491 (Online), ISSN 2224-5286 (Print)

Редакторы: *М. С. Ахметова, Т. А. Апендиев, Аленов Д.С.*
Верстка на компьютере *А.М. Кульгинбаевой*

Подписано в печать 05.08.2019.
Формат 60x881/8. Бумага офсетная. Печать – ризограф.
5,25 п.л. Тираж 300. Заказ 4.

Национальная академия наук РК
050010, Алматы, ул. Шевченко, 28, т. 272-13-18, 272-13-19