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Д.В. Сокольский атындағы «Жанармай,
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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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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.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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CATALYTIC SYNTHESIS OF A LINE BY ACETYLENE HYDRATION

Abstract. Acetone is a valuable chemical product. It can be obtained by oxidative dehydrogenation of isopropyl alcohol, oxidation of propylene, decomposition of acetic acid and ethyl alcohol, oxidation of cymene and others. Among the known processes for the production of acetone, the most promising is the synthesis by hydration of acetylene in the presence of catalysts. The advantage of this method is the possibility of carrying out the process in existing plants for the production of acetic aldehyde. On the other hand, the process of simultaneously producing acetaldehyde and acetone under the influence of multifunctional catalysts and carrying out the process using flexible technology is promising. The vapor-phase hydration of acetylene with the formation of acetone on polyfunctional catalysts was studied. Process parameters are found that provide acetone with high selectivity and acetylene conversion. At present, acetic aldehyde is mainly obtained by two methods - hydration of acetylene and oxidation of ethylene. The process of hydration of acetylene to acetic aldehyde in the presence of catalysts has been studied quite well. Numerous catalysts have been proposed for this process. Among the known catalysts for hydration of acetylene to acetic aldehyde, the most active was the cadmium calcium phosphate catalyst (CCF), which is recommended for industrial use. However, cadmium calcium phosphate catalyst is not without drawbacks. The average yield of acetaldehyde in one pass of acetylene does not exceed 7.0%. The CCF catalyst is very sensitive to temperature changes, its service life before regeneration does not exceed 72-76 hours.

Keywords: acetone, propylene, hydration process, catalyst, crosslinking, multifunctional properties.

Introduction. Acetylene hydration in the presence of a catalyst can be carried out in order to obtain aceton. The advantage of this method is the possibility of carrying out the process in existing plants for the production of acetic aldehyde. Replacing cadmium calcium phosphate catalyst with a zinc-containing catalyst allows obtaining acetone with a good yield with minor changes in technology [1-5]. Recently, the sol-gel method has been intensively used for the synthesis of inorganic and oregano-inorganic matrices at low temperatures.

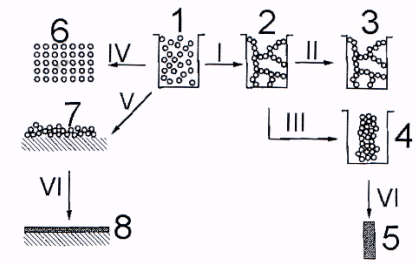

Figure 1 - General scheme for the production of nano catalysts by the sol-gel technology

This method has a number of advantages: the simplicity of the equipment used, efficiency, environmental safety, low cost, adaptability of technology and others. Figure 1 shows the general scheme for the preparation of Nano catalysts by the sol-gel technology.

The main processes that occur in the sol-gel transition and the products that can be obtained by the sol-gel method are shown in figure 1. I-maturing of the sol and gel formation: sol (1) → gel (2); II-drying under super critical conditions or washing the gel with solvents: gel (2) → airgel (3); III-drying under normal conditions: gel (2) → xerogel (4); IV-deposition of nano particles: sol (1) → powder (6); V-absorption of a sol by a rod substance: sol (1) → a thin layer xerogel (7); VI - tempering (cooking): xerogel (4) or a thin layer of xerogel (7) → monolithic glass and ceramics (5) or a thin layer and shell. Nano catalysts exhibit high catalytic activity, selectivity, as well as stability (stability).

Firstly, with decreasing particle sizes, most of the atoms are located on the surface; therefore, a catalyst consisting of nano particles has a large surface and becomes very active in heterogeneous reactions.

Secondly, most of the properties of nano particles are related to their size (size efficiency), therefore, by changing the size of nano particles one can control not only activity, but also selectivity. With a decrease in the particle size of the catalyst, the reaction rate sharply increases.

Based on the foregoing, it follows that the study of the possibility of using nano-catalysts obtained on the basis of metals Zn, Mn, Fe, V and other elements with multifunctional properties for the hydration reaction of acetylene and its derivatives is an urgent task [6-9].

Methods. The experiments on the catalytic hydration of acetylene and its derivatives in the gas phase were carried out in a reactor with a diameter of 25 mm, a height of 1000 mm, made of stainless steel under stationary conditions. The qualitative and quantitative composition of the reaction products was studied by gas-liquid chromatography under the following conditions: stationary phase 15% Apiezon-M in Color chromium, column thermostat temperature 80 0C, carrier gas flow rate helium 60 cm^3/min., DIP detector. Quantitative analysis was carried out using the internal standards method.

The specific surface area, crushing strength, total pore volume and ash content of the samples were determined.

The specific surface area was determined by thermal desorption of nitrogen in a carrier gas flow - helium at the boiling point of liquid nitrogen, the experimental data were processed using the BET equation [10-13].

The mechanical strength of the granules for crushing was measured on a device “Durably measures PK-1”, designed to test granular materials for mechanical strength under static conditions by compression. The arithmetic average of 25 individual tests was taken as the result of the analysis.

The total pore volume of the granules was calculated by the formula:

$$V = \frac{1}{p_k} - \frac{1}{p_n}$$

where, pk, pn are the apparent and psychometric density of the granules, g/ml, respectively.

The apparent density of the granules was determined by measuring their volume without taking into account the internal pores. The volume of the granules was found by immersing them in solid powder (silica sand with a particle size of 0.063-1.1 mm).

The phase composition of the samples was determined by x-ray diffractometry, the survey was carried out on a DRON-3M diffract meter using CuKα radiation with a Ni filter, and the length of the x-ray radiation.

The specific surface area of the obtained catalyst was calculated by the BET method, the average mesopore size by the VUA method. The dispersed properties of the catalyst were studied using a scanning electron microscope (JSM-6510 LV). The catalytic activity of the obtained sample was studied on the hydration reaction of acetylene [14-17].

Acetaldehyde and acetone were synthesized as follows. Acetylene was saturated with water at a temperature of 70-80 ^{0}C and at a ratio of water: acetylene = (1: 3) - (1: 5) moll was passed through a catalyst bed at 360 ^{0}C with a space velocity of 180-200 h-1.

The gas-vapor mixture leaving the reactor was cooled in the refrigerator. The reaction products were captured in water. Catalysis contains acetaldehyde, acetone, cretonne aldehyde, steam aldehyde and others. In order to maintain the degree of acetylene conversion not lower than 80%, every 20 hours the

reaction temperature was raised by 10 ^{0}C. After 96-120 hours, the degree of conversion of acetylene is reduced to 75-70%. Then the reaction was stopped and the catalyst was regenerated in a known manner [18-21].

Further, the effect of various parameters (temperature, space velocity, acetylene-water ratio) on the conversion of acetylene and the yield of acetone was studied.

Results. Because of studying the effect of temperature on the acetone yield, it was found that in the temperature range 360-500 ^{0}C the dependence between the reaction yield, temperature is extreme, and at 450 ^{0}C, the yield was considered maximal.

Based on the results on the qualitative and quantitative composition of the reaction products on this catalyst, we propose the following acetone formation mechanism:

In the reaction of acetone formation, acetaldehyde is first formed then 3-hydroxybutanal is formed as a result of aldol condensation.

$$CH{\equiv}CH + H_2O \rightarrow CH_3CHO,$$

$$2CH_3CHO \rightarrow CH_3CHOHCH_2CHO.$$

Upon hydration of 3-hydroxybutanal, a dihydric alcohol is formed and upon further dehydrogenation, acetoacetic acid is formed:

$$CH_3CHOHCH_2CHO + H_2O \rightarrow CH_3CHOHCH_2CH(OH)_2$$

$$CH_3CHOHCH_2CH(OH)_2 \rightarrow CH_3COCH_2COOH + 2H_2$$

In turn, acetoacetic acid under these conditions is decarboxylase and acetone is formed.

$$CH_3COCH_2COOH \rightarrow CH_3COCH_3 + CO_2$$

We have proposed a technological scheme for producing acetaldehyde, acetone (or a mixture thereof) is shown in figure 2.

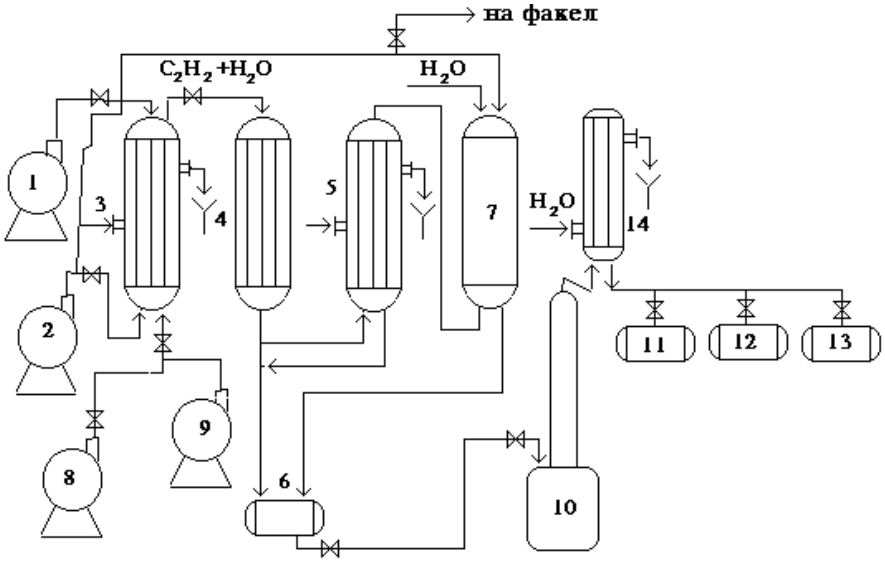

Figure 2 - Technological scheme of hydration of acetylene:
1-pump; 2-acetylene gas holder; 3-heat exchanger; 4-reactor; 5-heat exchanger; 6-capacity for catalysis;
7-absorption column; 8-blower; 9-gas holder for nitrogen; 10-distillation column; 11-container for acetaldehyde;
12-capacity for acetone; 13-tank for bottoms; 14-heat exchanger

We have studied the process of catalytic vapor-phase hydration of acetylene and its derivatives in the presence of complex mixed polyfunctional catalysts. In the process of producing acetaldehyde, cadmium fluoride on alumina promoted by aluminum fluoride was used as a catalyst. In the synthesis of acetone, zinc oxide supported on alumina, promoted with cadmium fluoride and aluminum, of the following composition, % weight: ZnO-20.0-25.0 was used as a catalyst; CoF_2-3.0-5.0; $A1F_3$-3.0-5.0; Al_2O_3-60.0-84.0.

The composition and properties of the synthesized catalysts for the synthesis of acetaldehyde and acetone are shown in table 1.

Table 1 - The composition and properties of the synthesized catalysts

№	Composition, mass %	Legend	Specific surface, m^2/g	Operating time before regeneration, hour	The yield of the target product, %	
					acetaldehyde	acetone
1	ZnO-20.0; AlF_3– 3.0 Al_2O_3 -77.0	ЦХ-1	135,0	72,0	10,0	82,0
2	CdF_2 – 20.0 Al_2O_3 – 80.0	КА-1	186,0	96,0	75,0	5,0
3	CdF_2 -18.0; AlF_3-3.0 Al_2O_3 – 79.0	КА-2	210,0	96,0	82,0	3,0
4	CdF_2-18.0; AlF_3-2.0 Cr_2O_3-5.0; Al_2O_3-75.0	КХА-1	225,0	120,0	83,0	1,5
5	ZnO-18.0; CdF_2-2.0 Cr_2O_3-5.0; Al_2O_3-80.0	ЦКХА-1	165,0	120,0	3,0	86,0

As can be seen from the table, catalyst No. 5 stably works for 120 hours (if the temperature rises by 100 ^{0}C every 20 hours), ensuring the degree of conversion of acetylene at the level of 91-95% and the yield of acetone is 86.0%.

The following is the dependence of the degree of conversion, the stability of the catalyst (catalyst No. 5) and the yield of the target product on the ratio of water: acetylene (table 2).

From the presented data, it follows that with an increase in the water: acetylene ratio, a gradual increase in the stability and selectivity of the catalyst, yield of the target product, and acetylene conversion are observed.

Table 2 - The dependence of the degree of conversion, the stability of the catalyst and the yield of the target product on the ratio of water: acetylene

The ratio of water: acetylene, mol	The average mileage of the catalyst before regeneration	The yield of the target product, on the reacted acetylene, %		Acetylene conversion, %
		acetaldehyde	acetone	
1	32,0	4,6	55,0	70,2
2	54,0	3,4	66,0	80,4
3	96,0	2,2	76,0	86,0
4	125	1,5	80,0	91,0
5	144	1,2	82,0	95,0
6	132	1,1	76,0	94,0

We also studied the effect of temperature, space velocity, the ratio of methyl acetylene-allen fraction: water and others on the yield of acetone. Under optimal conditions, acetone was obtained with a yield of 86.0% upon conversion of the methyl acetylene-allene fraction 92.0-94.0%.

The hydration reaction of acetylene to acetaldehyde was carried out in the presence of cadmium chromium aluminum catalyst - KHA in the temperature range 360-460 ^{0}C.

The effect of the ratio of acetylene: water on the selectivity of acetaldehyde formation in the range of acetylene: water = 1: 1-6 mol was studied. Moreover, it was found that in the range of the ratio of acetylene: water = 1: 1-1: 3, an increase in the proportion of side products is observed.

The yield of acetaldehyde does not exceed 26.0% of the reacted acetylene. At a ratio of -1: 4-1: 6, the yield of acetaldehyde and the conversion of acetylene reaches a maximum. A further increase in the ratio of acetylene water has no significant effect.

It was experimentally established that the yield of acetaldehyde and the conversion of acetylene substantially depends on the space velocity of acetylene.

The effect of the space velocity of acetylene on the acetaldehyde yield and its conversion was studied in the range of the space velocity from 50 to 120 hours (figure 3).

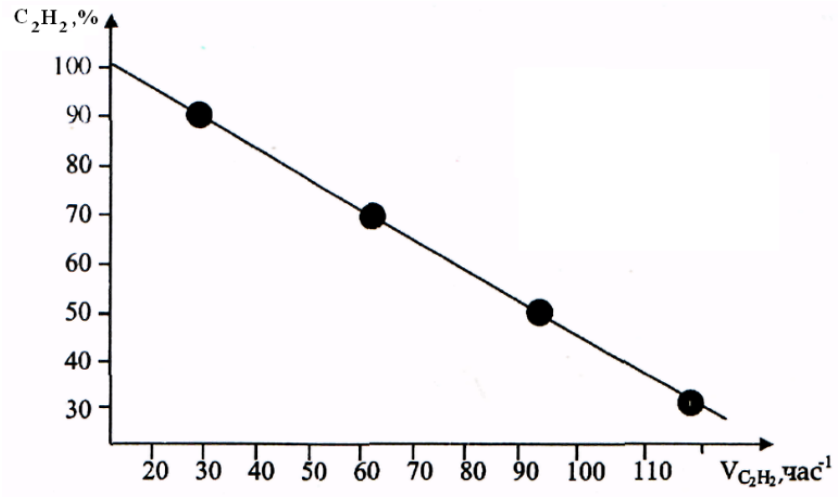

Figure 3 - The effect of the space velocity of acetylene on its conversion

The effect of temperature, grain size of the catalyst, reactor parameters, and catalyst bed height on the process parameters was studied. It was found that the reaction for the synthesis of acetaldehyde begins at 350 ^{0}C. In order to maintain the acetylene conversion at a level of 70-75%, the temperature of the reactor was raised by 10 ^{0}C every 10 hours. It was found that the optimum ratio of the height of the catalyst layer to the diameter of the reactor equal to 50-60, the space velocity of acetylene is 50-60 hours Under these conditions, the KXA-1 catalyst with constant activity lasts up to 120 hours and during regeneration within 16-24 hours completely restores its activity.

Replacing cadmium chromium aluminum catalyst with zinc alumina fluoride or zinc cadmium chromium aluminum catalyst leads to the formation of acetone as the main product in the process of hydration of acetylene.

In order to establish technological parameters of the process for producing acetone by direct hydration of acetylene, we studied the effect of space velocity, temperature, the ratio of acetylene: water and others on the yield of acetone,

The effect of temperature was studied in the range of 250-500 ^{0}C in the presence of CCA-1 catalyst, with a ratio of acetylene water = 1: 4 moll, with a space velocity of acetylene of 80 hours1 (table 3).

Table 3 - The effect of temperature on the conversion of acetylene

Temperature, ^{0}C	Acetone yield on reacted acetylene, %	Acetylene conversion, %
250	15,2	18,0
300	20,6	26,0
325	33,4	34,0
350	63,0	62,0
375	70,6	80,0
400	82,0	84,0
425	84,0	90,0
450	65,0	94,0
500	52,0	98,0

As can be seen from the table, TsKKA-1 catalyst is inefficient up to 350 ^{0}C. Starting from 350 ^{0}C to 425 ^{0}C, there is a gradual increase in acetone yield and acetylene conversion. A further increase in temperature leads to a significant decrease in the yield of acetone due to the flow of side products.

Conclusion. The reactions of acetone synthesis were studied by direct hydration of acetylene on catalysts prepared from oxides and fluorides of zinc, cadmium, iron, chromium and aluminum, using a solution of acetic acid as a peptizing agent. The main texture and operational characteristics of the synthesized catalysts are determined.

Based on the study of the dependence of the degree of conversion, the stability of the catalyst and the yield of the target product on the water: acetylene ratio, it was revealed that catalyst No. 5 worked stably for 125-144 hours (provided that the temperature rose by 100 ^{0}C every 20 hours), ensuring the degree of conversion of acetylene at the level of 91-95% and the yield of acetone - 90-92%.

A technological scheme for producing acetaldehyde, acetone by catalytic hydration of acetylene is proposed.

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АЦЕТОН ЛИНИЯСЫНЫҢ КАТЕТИКАЛЫҚ СИНТЕЗІ

Аннотация. Ацетон – маңызды химиялық өнім. Оны изопропил спиртін тотығу арқылы дегидрлеу, пропиленнің тотығуы, сірке қышқылы мен этил спиртінің ыдырауы, куменнің тотығуы және басқалары арқылы алуға болады.

Ацетон өндірудің белгілі процестерінің ішінде катализаторлардың қатысуымен ацетиленнің гидратациясы арқылы синтездеу перспективалы сипатта болып келеді. Бұл әдістің артықшылығы қолданыстағы өсімдіктерде сірке альдегидін өндіру процесін жүргізу мүмкіндігімен байланысты. Екінші жағынан, көпфункционалды катализаторлардың әсерінен ацетальдегид пен ацетонды бір уақытта өндіру және икемді технологияны қолдану арқылы процесті жүргізу перспективалы сипатта болып келеді.

Ацетиленнің көпфункционалды катализаторларда түзілуімен бу фазалы гидратациясы зерттелді. Процесс параметрлері ацетонды жоғары селективтілікпен және ацетилен түрлендірумен қамтамасыз етеді.

Қазіргі уақытта сірке альдегиді негізінен екі әдіспен шығарылады: ацетиленнің гидратациясы және этиленнің тотығуы.

Катализаторлардың қатысуымен ацетиленді сірке альдегидіне дейін ылғалдандыру процесі жақсы зерттелген. Бұл процесс үшін көптеген катализаторлар ұсынылды. Ацетиленді сірке альдегидіне дейін ылғалдандыруға арналған белгілі катализаторлардың ішінде өнеркәсіптік қолдануға ұсынылған кадмий-кальций фосфатының катализаторы (CCF) белсенді болды. Алайда кадмий кальций фосфатының катализаторы кемшіліктерсіз болмайды. Ацетиленнің бір жолындағы ацетальдегидтің орташа шығымдылығы 7,0 %-дан аспайды. CCF катализаторы температураның өзгеруіне өте сезімтал, оны қалпына келтіруге дейінгі қызмет мерзімі 72-76 сағаттан аспайды.

Катализатордың қатысуымен ацетилен гидратациясын ацетон алу үшін жүргізуге болады. Бұл әдістің артықшылығы – қолданыстағы өсімдіктерде сірке альдегидін өндіру процесін жүргізу мүмкіндігінде. Кадмий-кальций фосфатының катализаторын цинк бар катализатормен алмастыру технологиядағы аздаған өзгерістермен жақсы шығымды ацетон алуға мүмкіндік береді.

Жақында бейорганикалық және органикалық-бейорганикалық матрицаларды төмен температурада синтездеу үшін сол-гель әдісі қарқынды қолданыла бастады. Бұл әдіс бірқатар артықшылықтарға ие: қолданылатын жабдықтың қарапайымдылығы, үнемділік, экологиялық қауіпсіздік, арзан баға, технологиялардың бейімділігі және басқалары.

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КАТАЛИТИЧЕСКИЙ СИНТЕЗ ЛИНИИ ГИДРАТАЦИЕЙ АЦЕТИЛЕНА

Аннотация. Ацетон является ценным продуктом химической промышленности. Он может быть получен окислительным дегидрированием изопропилового спирта, окислением пропилена, разложением уксусной кислоты и этилового спирта, окислением кумола и др.

Среди известных процессов производства ацетона наиболее перспективным является синтез гидратацией ацетилена в присутствии катализаторов. Преимуществом данного метода является возможность проведения процесса в существующих установках производства уксусного альдегида. С другой стороны,

многообещающим является процесс одновременного получения ацетальдегида и ацетона под действием полифункциональных катализаторов и проведение процесса по гибкой технологии.

Изучена парофазная гидратация ацетилена с образованием ацетона на полифункциональных катализаторах. Найдены параметры процесса, обеспечивающие получение ацетона с высокой селективностью и конверсией ацетилена.

В настоящее время уксусный альдегид в основном получают двумя методами - гидратацией ацетилена и окислением этилена.

Процесс гидратации ацетилена до уксусного альдегида в присутствии катализаторов изучен достаточно хорошо. Для этого процесса предложены многочисленные катализаторы. Среди известных катализаторов гидратации ацетилена до уксусного альдегида наиболее активным оказался кадмийкальций-фосфатный катализатор (ККФ), который рекомендован для промышленного применения. Однако кадмийкальцийфосфатный катализатор не лишен недостатков. Средний выход ацетальдегида за один проход ацетилена не превышает 7,0 %. ККФ катализатор очень чувствителен к изменению температуры, его срок службы до регенерации не превышает 72-76 часов.

Гидратация ацетилена в присутствии катализатора может быть проведена с целью получения ацетона. Достоинством данного метода является возможность проведения процесса в существующих установках производства уксусного альдегида. Замена кадмийкальцийфосфатного катализатора на цинксодержащий катализатор позволяет получить ацетон с хорошим выходом при незначительных изменениях в технологии.

В последнее время золь-гель метод интенсивно используется для синтезирования неорганических и органо-неорганических матриц при низких температурах. Этот метод обладает целым рядом преимуществ: простотой используемого оборудования приборов, экономичностью, экологической безопасностью, низкой себестоимостью, приспособляемостью технологий и другие. На Рис.1 приведена общая схема получения нанокатализаторов методом золь-гель технологии.

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