ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫНЫҢ
Satbayev University

Х А Б А Р Л А Р Ы

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
Satbayev University

N E W S

OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF KAZAKHSTAN
Satbayev University

SERIES
OF GEOLOGY AND TECHNICAL SCIENCES

1 (445)

JANUARY – FEBRUARY 2021

THE JOURNAL WAS FOUNDED IN 1940

PUBLISHED 6 TIMES A YEAR

ALMATY, NAS RK

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«ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы».

ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)

Меншіктенуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» РҚБ (Алматы қ.).

Қазақстан Республикасының Ақпарат және қоғамдық даму министрлігінің Ақпарат комитетінде 29.07.2020 ж. берілген **№ KZ39VPY00025420** мерзімдік басылым тіркеуіне қойылу туралы куәлік.

Тақырыптық бағыты: ***геология және техникалық ғылымдар бойынша мақалалар жариялау.***

Мерзімділігі: жылына 6 рет.
Тиражы: 300 дана.

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

Типографияның мекенжайы: «NurNaz GRACE», Алматы қ., Рысқұлов көш., 103.

«Известия НАН РК. Серия геологии и технических наук».

ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)

Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан (г. Алматы).

Свидетельство о постановке на учет периодического печатного издания в Комитете информации Министерства информации и общественного развития Республики Казахстан **№ KZ39VPY00025420**, выданное 29.07.2020 г.

Тематическая направленность: ***публикация статей по геологии и технических наукам.***

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://www.geolog-technical.kz/index.php/en/

Адрес типографии: «NurNaz GRACE», г. Алматы, ул. Рыскулова, 103.

News of the National Academy of Sciences of the Republic of Kazakhstan. Series of geology and technology sciences.

ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)

Owner: RPA "National Academy of Sciences of the Republic of Kazakhstan" (Almaty).

The certificate of registration of a periodical printed publication in the Committee of information of the Ministry of Information and Social Development of the Republic of Kazakhstan **No. KZ39VPY00025420**, issued 29.07.2020.

Thematic scope: ***publication of papers on geology and technical sciences.***

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://www.geolog-technical.kz/index.php/en/

Address of printing house: «NurNaz GRACE», 103, Ryskulov str, Almaty.

N E W S
OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN
SERIES OF GEOLOGY AND TECHNICAL SCIENCES
ISSN 2224-5278
Volume 1, Number 445 (2021), 157 – 163 **https://doi.org/10.32014/2021.2518-170X.22**

U. Shokirov[1], S. Turabdjanov[1], H. Kadirov[2], F. Badriddinova[2], B. Kedelbaev[3]

[1]Tashkent State Technical University named after Islam Karimov, Tashkent, Uzbekistan;
[2]Tashkent Institute of Chemical Engineering, Tashkent, Uzbekistan;
[3]South-Kazakhstan State University named after M. Auezov, Shymkent, Kazakhstan.
E-mail: umid52@mail.ru, tur_sad@mail.ru, tkti.kodirov@mail.ru,
badritdinova_f@mail.ru, kedelbaev@yandex.kz

CALCULATION AND DESIGN OF AN INDUSTRIAL REACTOR FOR PYRROLES SYNTHESIS

Abstract. Industry of pyrroles synthesis on the basis of acetylene, ammonia and amines over polyfunctional catalyst can be organised only when for its recovery the rational industrial technology allowing to synthesize this product in necessary quantity and demanded quality cheap enough from accessible raw materials is developed. Considering the issues related to the development of a new reactor with high productivity and economic efficiency, the processes running in the reactor were studied.

Phase characteristics have been investigated in response to changes in acetylene conversion rate due to the height of the catalyst layer. As a result, high productivity of acetylene production with zinc-chromaluminum catalyst at the temperature range of 340-440^{o}C is achieved for 92% when the layer height is 1200 mm from the reactor top point. Based on the mass balance as well as the experimental result, a reactor for the industrial synthesis of pyrroles has been proposed.

Key words: pyrrol, acetylene, ammonia, amines, catalyst, dehydration, heterocyclization.

Introduction. The present-day level of development of innovative technologies promotes research in the field of chemistry of heterocyclic compounds [1-3]. Condensation of acetylene with ammonia and amines is of great theoretical and practical interest, which opens a perspective for the synthesis of complex organic nitrogen-containing compounds of pyrrole and its derivatives, which are currently of practical importance [4,5].

The pyrrole ring is contained in the molecules of blood - hemoglobin and green substances of plants - chlorophyll, a number of antibiotics, and in many compounds [6,7]. The pyrrole fragments contained are potentially important as optical electron-active materials [7]. Plasma copolymers of polypyrrole and polyethylene glycol are used to produce an implant that promotes neuroprotection and the restoration of a compound in the spinal cord after damage [8]. If the pyrrole structure contains two bound rings, then a high degree of planarity is observed and as a result leads to improvement of optical and electronic properties [9].

For the synthesis of pyrrole and its homologs there is a large number of methods [10-16], which we will conditionally divide into three main groups:

- Synthesis of pyrroles from ketoximes and acetylene.
- Synthesis of pyrroles from ketoamines.
- Synthesis of pyrroles from acetylene and amines.

However, the known diverse methods of synthesis of the pyrrole cycle are multistage. The lifetime of the catalysts used is limited and there are difficulties in separating the resulting complex mixture.

We have studied the reaction of synthesis of pyrrol and N-vinylpyrrol together with catalytic heterocyclization of acetylene, ammonia and amines in the presence of zinc-chrome-aluminum (ZCA) and cadmium-fluorine-zinc-chrome-aluminum (CFZCA) catalysts [17-19].

Methods. In laboratory environment, heterocyclization reactions were performed at the temperature range of 340 - 440 °C, the volumetric flow rate of gases 140-150 hour^{-1}. Processes running in the reactor are determined by the phase state of the initial reagents and reaction products (phase characteristic), type of catalyst (solid or liquid), reaction heat, respectively, thermal regime (energy and thermodynamic characteristics), process dynamics, the nature of movement of reagents and reaction products, as well as system hydrodynamics.

In order to select optimal process and reactor parameters, we have conducted a series of experiments with different reactor designs. The reactor design must ensure that the following basic process parameters are maintained [20-22]:

[1] reaction period;
[2] temperature: at various points of the reaction zone;
[3] reactor pressure;
[4] mass transfer rate to the active surface of the catalyst and between phases;
[5] catalyst activity.

Heterocyclization reaction was carried out in tubular reactors of size $\boldsymbol{d \cdot \ell = (\mathscr{b}.mm)}$

21×500;	21×1000;	21×1500
30×500;	30×1000;	45×1000

Results and discussion. Reactors with fixed granular catalyst bed are the most common type of devices for carrying out catalytic processes. Granular bed is a set of randomly stacked catalyst particles at intervals in which the acetylene flows, i.e. a kind of coarse-grained medium, which must be strongly exposed to various random factors related to the heterogeneity of particle-particle packing and distribution of the reaction mixture flow.

The relationship of acetylene conversion rate change to catalyst layer height has been studied. It has been established that the acetylene conversion rate changes in direct proportion to the catalyst layer height (table).

Relationship of acetylene conversion rate change to catalyst layer height ZCA at 340 - 440 °C
Reactor d×l = 21x1500 mm; V = 140 - 150 hour^{-1}

No.	Layer height from the top point of the reactor, mm	Acetylene conversion rate, %
1	400	41.0
2	600	57.0
3	800	76.0
4	1000	86.0
5	1200	92.0
6	On reactor outlet	96,0 - 99,0

The table shows that the reaction takes place in the inner diffused area. We considered the kinetics of catalytic reactions, in which the speed was determined by chemical processes inside of reactor: adsorption, surface reaction, desorption. The ZCA catalysts used by us are porous bodies. Heterogeneous reactions of acetylene and ammonia are rather fast, the speed of the total catalytic action is the diffusion of reagents in the pores of the ZCA catalyst grain, i.e. inside of the diffusion region.

The internal surface of porous catalyst grains can exceed 99 % of the total catalyst surface. Catalyst grain is described as a medium in which the substance is transferred with an effective diffusion coefficient, and the catalytic reaction of acetylene and ammonia proceeds with an effective speed constant, referred to grain volume unit, and these constants are constant throughout the volume of grain. Thus, the reaction in the reactor proceeds somewhat homogeneously in the mass of the catalyst, and the reagents come from another phase [23,24].

High efficiency of heterogeneous processes is achieved by the right choice of the reactor device, due to the required selectivity of chemical transformations of the initial substances, as well as the productivity of the target product.

The reactor design must ensure the maintenance of the following basic process parameters:

[1] reaction time;
[2] temperatures at various points of the reaction zone;
[3] reactor pressure;
[4] mass transfer rate to the active surface of the catalyst and between phases;
[5] catalyst activity.

For acetylene and ammonia catalytic reactions we proposed a catalyst layer.

The mass balance of the main parameters of flow-type reactor for pyrrole synthesis was calculated [25,26].

For plug-flow reactor, the mass balance equation for the reacting substance is as follows:

$$-\frac{\omega}{S_{ov}} \cdot \frac{d(UA)}{dx} = W + \frac{\omega}{S_{ov}} \cdot \frac{dA}{dt} \quad (1)$$

where $\omega = \frac{S_1}{S_2}$, S_1 is reactor cross-sectional area free of catalytic material; S_2 is reactor cross-sectional area; S_{ov} is catalyst volume unit area, m^2/m^3; A is current acetylene concentration, $mole/m^3$; U is reactor gas mixture rate, m/sec.; W is acetylene flow rate, $mole/m^2 \cdot sec.$; X is the coordinate of the volume element for which the process is described.

When the reactor reaches a steady-state condition

$$\frac{dA}{dt} = 0$$

and the equation is as follows:

$$W = \frac{\omega}{S_{ov}} \cdot \frac{d(UA)}{dx} \quad (2)$$

The rate of acetylene consumption reaction is described by the equation:

$$W = K \cdot e^{-\frac{E}{RT}} \cdot A$$

where, E is activation energy, J; $\hat{E}$ is catalyst activity parameter, m/sec.

In the process of the catalyst activity it is deactivated due to deposition of high-molecular carbon products on its surface.

We assume that the deactivation speed is proportional to the acetylene conversion rate.

Based on these assumptions, the equation reflecting the catalyst deactivation process becomes as follows:

$$\frac{dK}{dt} = -K^1 \cdot W \quad (3)$$

where K^l is catalyst deactivation constant, m/mole·sec.;

To describe the dependence of the gas mixture rate in the reactor on the current concentration of acetylene, the flow continuity equation is used:

$$\rho_0 U_0 = \rho U \quad (4)$$

where, U is feed rate of acetylene and ammonia mixture into the reactor, m/sec.; ρ_0 is gas mixture density at the reactor inlet, kg/m^3; ρ is reactor gas mixture density, kg/m^3.

It follows from equation (4) that

$$U = \frac{\rho_0 U_0}{\rho}$$

To get the ratio ρ_0/ρ, let's write down the stoichiometric balance equation for the pyrrole synthesis process:

$$b = b_0 \frac{1}{3}(a_0 - a)$$

$$d = h \frac{1}{3}(a_0 - a) \quad (5)$$

where, a_0, b_0 is the amount of acetylene and ammonia molecules entering the reactor per unit of time, mole/sec.

a, b, d, h is the amount of acetylene, ammonia, hydrogen and pyrrole molecules passing through the reactor section in a unit of time, mole/sec.

Let's denote

$$A_0 = \frac{a_0}{V_0}; B_0 = \frac{b_0}{V_0}; B = \frac{b}{V}; H = \frac{h}{V}; D = \frac{d}{V} \quad (6)$$

where, V_0 is volume of gas mixture passing through the reactor cross section in a unit of time, m^3/sec. V is the volume of gas mixture passing through the reactor cross section in a unit of time, m^3/sec.

Considering that the pressure along the length of the reactor remains virtually unchanged, we can write down

$$A_0 + B_0 = A + B + H + D \quad (7)$$

based on (5) and (7) we obtain

$$B = B_0 + \frac{V_0}{V} - \frac{1}{3}\left(A_0 \cdot \frac{V_0}{V} - A\right) \quad (8)$$

$$D = \frac{1}{3}\left(A_0 \cdot \frac{V_0}{V} - A\right) \quad (9)$$

$$H = \frac{1}{3}\left(A_0 \cdot \frac{V_0}{V} - A\right) \quad (10)$$

Using the law of mass conservation, we obtain:

$$\rho_0 U_0 = \rho U \frac{\rho}{\rho_0} = \frac{U_0}{U} \quad (11)$$

Considering (7), (8), (9), (10), (11)

$$\frac{U_0}{U} = \frac{\rho}{\rho_0} = \frac{3A_0 + 3B_0 - 2A}{A_0 + 3B_0}$$

$$U = U_0 \cdot \frac{A_0 + 3B_0}{3A_0 + 3B_0 - 2A} \quad (12)$$

To control the recording, the following designations are entered:

$$\beta = (A_0 + 3B_0) \cdot U_0$$

$$\gamma = 3A_0 + 3B_0 \quad (13)$$

then $U = \frac{\beta}{\gamma - 2A}$.

Thus, for mathematical description the following equations were used: mass balance on reacting substance for the reactor of ideal displacement (2), reflecting the process of catalyst deactivation (3), flow continuity (4), stoichiometric balance (5), as well as the equation derived from the Avogadro law for isothermal process (9).

With substitution (13) in equation (2) we obtain

$$\frac{dA}{A(2A-\gamma)^2} = \frac{K \cdot \exp\left(-\frac{E}{RT}\right)}{K_2^1 \cdot \beta \cdot \gamma} \cdot dx \quad (14)$$

where $K_2^1 = \frac{\omega}{S_{ov}}$.

Thus, the system of equations of mathematical model of the process in the isothermal case has the form (14).

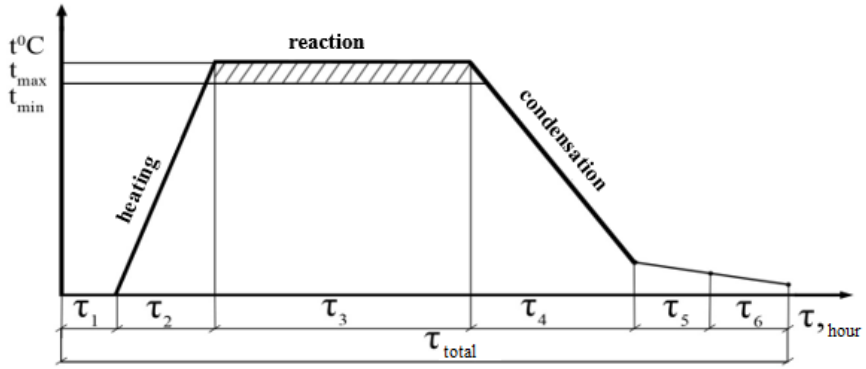
Expenses of initial components of acetylene, ammonia and ammines at the reactor inlet and final components at the outlet have been calculated and mass balance has been compiled.

Based on data from laboratory, experimental installations and mass balance we have proposed a reactor for industrial synthesis of pyrroles.

The reactor is a vertical cylindrical device in the form of a heat exchanger with an external diameter of 3800 mm and a height of 8400 mm. Inside the cylinder are fixed pipes with dimension of 38×2,5 filled with a catalyst in the amount of 1765 pcs. The reaction pipes are heated with nitrogen. A mixture of acetylene and ammonia is supplied to the upper parts of the reactor and hot nitrogen is supplied to the lower parts [27-29].

The nature of movement of initial reagents and reaction products is largely determined by the volume of output products. Depending on the production volume, periodic processes are used.

For periodic processes during time, we can distinguish the following separate stages (figure).



Process steps in the reactor

Where

is loading of raw materials,	τ_1
is mixing and heating,	τ_2
is chemical transformation of raw materials,	τ_3
is reactor heat removal,	τ_p
is reaction mass cooling,	τ_4
is reaction mass unloading,	τ_5
is reactor cleaning and inspection,	τ_6
is length of operation,	τ_{total}
is reaction temperatures	t_{max} t_{min}

Such processes have the following advantages: they are characterized by great flexibility (different products can be obtained in the same reactor).

Conclusion. Thus, we have selected the design and calculated the mass balance of the process on the basis of laboratory and experimental installations. A model has been developed for the case of the reaction proceeding in the reactor of ideal displacement under isothermal conditions taking into account the change of steam mixture rate in the reactor and taking into account deactivation of the catalyst surface. The obtained data can be used for the development of the pyrrole synthesis unit in industrial conditions.

У. Шокиров[1], С. М. Турабджанов[1], Х. Кадиров[2], Ф. Бадриддинова[2], Б. Ш. Кедельбаев[3]

[1]Ислам Каримов атындағы Ташкент мемлекеттік техникалық университеті, Өзбекстан;
[2]Ташкент химия-технологиялық институты, Өзбекстан;
[3]М. Әуезов атындағы Оңтүстік Қазақстан мемлекеттік университеті, Шымкент, Қазақстан

ПИРРОЛДАРДЫ СИНТЕЗДЕУГЕ АРНАЛҒАН ӨНЕРКӘСІПТІК РЕАКТОР ҚҰРЫЛЫМЫН ЕСЕПТЕУ ЖӘНЕ ТАҢДАУ

Аннотация. Ацетилен, аммиак және аминдер негізіндегі пиррол синтезін көпфункционалды катализаторлардың қатысуы арқылы өндіру үшін ұтымды өнеркәсіптік технология жасалғанда ғана ұйымдастыруға болады, бұл өнімді қолжетімді шикізаттан арзан және қажетті сапада алуға мүмкіндік береді.

Мақалаларда өнімділігі мен экономикалық тиімділігі жоғары жаңа реакторды әзірлеуге қатысты мәселелер қарастырылған. Реактордағы үдерістер де зерттелді.Ацетиленді конверсиялау дәрежесінің өзгеруіне және катализатор қабатының биіктігіне байланысты фазалық сипаттамалар зерттелді. Нәтижесінде 340-440 °С температурада мырыш-алюминий катализаторы бар ацетиленнің жоғары өндірістік жылдамдығы реактордың жоғарғы жағынан қабаттың биіктігі 1200 мм болғанда 92%-ға жетеді. Материалдық тепе-теңдіктің, сондай-ақ эксперимент нәтижесінің негізінде пиролольдердің өнеркәсіптік синтезіне реактор ұсынылады.

Түйін сөздер: пиррол, ацетилен, аммиак, аминдер, катализатор, дегидратация, гетероциклизация.

У. Шокиров[1], С. М. Турабджанов[1], Х. Кадиров[2], Ф. Бадриддинова[2], Б. Ш. Кедельбаев[3]

[1]Ташкентский государственный технический университет им. Ислама Каримова, Ташкент, Узбекистан;
[2]Ташкентский химико-технологический институт, Ташкент, Узбекистан;
[3]Южно-Казахстанский государственный университет им. М. Ауезова, Шымкент, Казахстан.

РАСЧЕТ И ВЫБОР КОНСТРУКЦИИ ПРОМЫШЛЕННОГО РЕАКТОРА ДЛЯ СИНТЕЗА ПИРРОЛОВ

Аннотация. Производство синтеза пирролов на основе ацетилена, аммиака и аминов в присутствии полифункциональных катализаторов может быть организовано только тогда, когда для его получения разработана рациональная промышленная технология, позволяющая достаточно дешево из доступного сырья получать этот продукт в необходимом количестве и требуемого качества.

В статье рассматриваются вопросы, связанные с разработкой нового реактора, обладающего высокой производительностью и экономической эффективностью. Были также изучены процессы, протекающие в реакторе.

Исследованы фазовые характеристики в ответ на изменение скорости конверсии ацетилена в зависимости от высоты слоя катализатора. В результате достигается высокая производительность производства ацетилена с цинк-хромалюминиевым катализатором в интервале температур 340-440 ° с на 92% при высоте слоя 1200 мм от верхней точки реактора. На основе баланса масс, а также результатов эксперимента предложен реактор для промышленного синтеза пирролов.

Ключевые слова: пиррол, ацетилен, аммиак, амины, катализатор, дегидратация, гетероциклизация.

Information about authors:

Shokirov Umidjon Utkur ogli, PhD student, Tashkent State Technical University, Tashkent, Uzbekistan; umid52@mail.ru; https://orcid.org/0000-0003-4233-2508

Turabdzhanov Sadritdin Maxamatdinovich, Doctor of Technical Sciences, professor, rector of Tashkent State Technical University, Tashkent, Uzbekistan; tur_sad@mail.ru; https://orcid.org/0000-0001-8963-5763

Kadirov Xasan Ikromovich, Doctor of Technical Sciences, assistant-professor of Tashkent Chemical Technological Institute, Tashkent, Uzbekistan; tkti.kodirov@mail.ru; https://orcid.org/0000-0003-0770-6965

Badritdinova Farida Maxamatdinovna, Candidate of technical sciences, professor-assistant of Tashkent Chemical Technological Institute, Tashkent, Uzbekistan; badritdinova_f@mail.ru; https://orcid.org/0000-0002-5909-5927

Kedelbaev Bakhytzhan Shilmirzaevich, doctor of technical sciences, professor of the department "Biotechnology" M.Auezov South Kazakhstan State University, Shymkent, Kazakhstan; kedelbaev@yandex.ru; https://orcid.org/0000-0003-0322-9743

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www:nauka-nanrk.kz

ISSN 2518-170X (Online), ISSN 2224-5278 (Print)

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Редакторы *М. С. Ахметова, Д. С. Аленов, А. Ахметова*
Верстка *Д. А. Абдрахимовой*

Подписано в печать 01.02.2021.
Формат 70x881/8. Бумага офсетная. Печать – ризограф.
12,75 п.л. Тираж 300. Заказ 1.

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