IRSTI 55.30.03, 55.30.31, 55.30.13 DOI: https://doi.org/10.26577/JMMCS.2022.v113.i1.09

A.N. Mussulmanbekova*, **A.T. Khabiev**, **A. Adilkhan**, **A.B. Baratova**, **M.K. Zhumadillayev**
U. Joldasbekov Institute of Mechanics and Engineering, Kazakhstan, Almaty
*e-mail: a.mussulmanbekova@gmail.com

AUTOMATION OF OBTAINING VINYL ACETATE IN A MICROREACTOR

Due to the lack of domestic manufacturers, the production of a microreactor with a control system for chemical processes will remain relevant. The reason for this is the demand for automated solutions and, in general, automation and research into the optimal conditions for promising chemical processes in companies and in production. Moreover, the microreactor proposed with a chemical process control system will be able to save money and time on the development of effective chemical processes for the production of a wide variety of substances. In addition, the project provides for the use of composite and polymer materials for work, which in turn will reduce the cost of manufactured products, and at the same time increase the competitive attractiveness. The aim is to develop a microreactor and a control system for chemical processes. This goal is achieved by justifying the choice of the direction of research, analysis of existing equipment for carrying out microreactor synthesis, application for their creation lightweight, composite and other technical materials, as well as through the development of technology for creating microreactor equipment using 3D printing, milling and engraving of light metals, composite and polymer materials. The article presents microreactors and the development of a microreactor for the production of vinyl acetate, a detailed description of the methodology for the development of this complex device, including a microreactor, a SCARA type robot and a control unit. Methods for the production of vinyl acetate and the possibility of automating this process with a complex device were also studied.

Key words: automation, microreactor, SCARA type robot, vinyl acetate.

А.Н. Мусулманбекова, А.Т. Хабиев, А. Әділхан, А.Б. Баратова, М.К. Жумадиллаев
Академик Ө.А. Жолдасбеков атындағы механика және машинатану институты, Қазақстан, Алматы қ.
*e-mail: a.mussulmanbekova@gmail.com

Винил ацетатын микрореакторда алуды автоматтацизациялау

Отандық өндірушілердің болмауына байланысты химиялық процестерді басқару жүйесі бар микрореактор өндірісі өзекті болып қала береді. Мұның себебі – автоматтандырылған шешімдерге сұраныс және жалпы алғанда, автоматтандыру және кәсіпорындардағы және өндірістегі перспективалы химиялық процестердің оңтайлы шарттарын зерттеу. Сонымен қатар, химиялық процестерді басқару жүйесімен ұсынылған микрореактор әртүрлі заттарды алу үшін тиімді химиялық процестерді әзірлеуге ақша мен уақытты үнемдеуге мүмкіндік береді. Сонымен қатар, жоба жұмыс үшін композиттік және полимерлі материалдарды пайдалануды қарастырады, бұл өз кезегінде өндірілген өнімнің өзіндік құнын төмендетуге, сонымен қатар бәсекеге қабілеттілікті арттыруға мүмкіндік береді. Мақсат – микрореактор мен химиялық процестерді басқару жүйесін жасау. Бұл мақсат зерттеу бағытын таңдауды негіздеу, микрореакторлық синтезді жүргізу үшін қолданыстағы жабдықты талдау, оларды жеңіл, композициялық және басқа да техникалық материалдарды жасауға қолдану, сондай-ақ 3D көмегімен микрореакторлық жабдықты құру технологиясын әзірлеу арқылы қол жеткізіледі. жеңіл металдарды, композициялық және полимерлі материалдарды басып шығару, фрезерлеу және ою.

Мақалада микрореакторлар мен винилацетат өндірісіне арналған микрореактордың дамуы, микрореакторды, SCARA типті роботын және оның басқару блогын қоса алғанда, осы күрделі құрылғыны жасау әдістемесінің толық сипаттамасы талқыланады. Сондай-ақ, винилацетат алу әдістері және осы процесті күрделі құрылғымен автоматтандыру мүмкіндігі зерттелді.
Түйін сөздер: автоматика, микрореактор, типті SCARA робот, винилацетат.

А.Н. Мусулманбекова, А.Т. Хабиев, А. Адилхан, А.Б. Баратова, М.К. Жумадиллаев
Институт механики и машиноведения имени академика У.А. Джолдасбекова, Казахстан, г. Алматы
*e-mail: a.mussulmanbekova@gmail.com

Автоматизация получения винилацетата в микрореакторе

Из-за отсутствия отечественных производителей производство микрореактора с системой управления химическими процессами останется актуальным. Причина этого - потребность в автоматизированных решениях и в целом автоматизации и исследованиях оптимальных условий для перспективных химических процессов на предприятиях и на производстве. Более того, предложенный микрореактор с системой управления химическим процессом позволит сэкономить деньги и время на разработке эффективных химических процессов для производства самых разных веществ. Кроме того, проектом предусмотрено использование в работе композиционных и полимерных материалов, что, в свою очередь, снизит стоимость производимой продукции и одновременно повысит конкурентоспособность. Целью является разработка микрореактора и системы управления химическими процессами. Данная цель достигается за счет обоснования выбора направления исследований, анализа существующего оборудования для проведения микрореакторного синтеза, применения для их создания легких, композиционных и других технических материалов, а также за счет разработки технологии создания микрореакторного оборудования с использованием 3D. печать, фрезерование и гравировка легких металлов, композитных и полимерных материалов. В статье рассматриваются микрореакторы и разработка микрореактора для получения винилацетата, подробное описание методики разработки данного комплексного прибора, включающего микрореактор, робот типа SCARA и блок управления. Также были изучены методы получения винилацетата и возможность автоматизации данного процесса комплексным прибором.

Ключевые слова: автоматизация, миниатюризация, микрореактор, робот типа SCARA.

1 Introduction

Markets demand products that are smart, feature-rich, communicative, clean, safe, portable, lightweight and self-contained. The production of these products requires miniaturization of components and systems, which can be accomplished using microelectronic technologies developed in recent decades. This allowed not only to reduce the size of components and sensors, but also to increase their density in integrated circuits.

Microsystems are typically less than a millimeter in size. They can be produced using manufacturing technologies related to microelectronics. In this case, it is possible to combine sensors, actuators, and microelectronic components. This technology has contributed to the development of microreactors for controlling chemical reactions.

Leading companies in the world, for example, Lonza, DSM, Sigma Aldrich, Bayer, Astra Zeneca, Novartis, Eli Lilly, GlaxoSmithKline, Pfizer, MSD and research institutes: IMM – Institute of Microtechnology Mainz / Institute of Chemical Technology (Germany), TNO – Government Institute of Applied Research (Holland), MIT – Massachusetts Institute of Technology (USA) is already implementing all these solutions in industry, primarily at the pilot and semi-industrial level, less often at the industrial level [1].

Microreactors have provided a better understanding of the effects of miniaturization on flows and transport phenomena in the chemical industry.

Microreactor technology currently represents a serious alternative to conventional macroscopic production. Microreactors are reactor systems that include structures for the transfer or containment of a gas or liquid, in which at least one size is measured in micrometers and does not exceed 1 mm [2].

For the design of microreactors, various technologies are used: lithography, electroplating, casting, etc. Silicon, quartz, polymers, metals are used as structural materials [3].

Microreactor synthesis of active substances belongs to modern breakthrough technologies of chemical synthesis, which allow the production of complex substances with much lower operating costs. When using micro-reactor technologies, it is possible to provide the following technological advantages: guaranteed process safety, energy efficiency, compliance with regulatory standards, modular design, the possibility of its accelerated scaling, reproducibility of the technological process, compactness and high selectivity [4].

Moreover, microreactor technologies are able to reduce the costs of implementing the synthesis process, since they do not require huge premises and a large staff of specialists. At the same time, the use of a microreactor allows providing all the necessary conditions for the correct course of chemical processes, continuous operation under full automatic control of all parameters [5].

Next, we will consider microreactors of different generations for modeling chemical reactions. Currently, 3 generations of microreactors are known. These are microreactors of the first generation (1G), microreactors of the second generation (2G), as well as microreactors of the third generation (3G).

2 First generation microreactors (1G)

The first generation microreactors made it possible to measure frictional pressure loss. Unfortunately, the asymmetry of the drilled hole in the glass relative to the surface of the well was too great to provide a good seal at the fluid-microreactor junction.

The first generation of microreactors is made from silicon and glass. For this, a 2 mm high drilled glass plate must be coated with etched silicon one millimeter thick (Figure 1) [6].

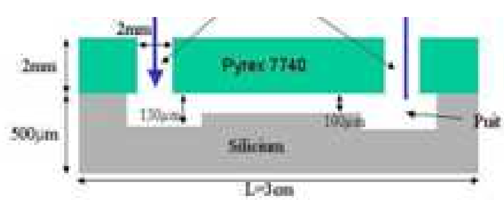

Figure 1: Superposition of silicon and glass plates on top of each other (in the figure, the arrows indicate the entrance and exit).

Next, the second generation microreactors will be presented. (Стекло7740-glass, silicon).

3 Microreactors of the second generation 2G

Second generation microreactors are made from polydimethylsiloxane. The manufacturing process for second generation microreactors includes 3 stages:

- in the first stage, a layer containing channels is obtained by pouring polydimethylsiloxane into a mold (20 g of polymer makes a layer 1.5 mm thick after application to a silicon wafer with a diameter of 4 inches);
- in the second stage, holes with a diameter of 2 mm are drilled in this layer using a punch, at the level of the tanks;
- in the third stage, the sealing plate is made by casting polydimethylsiloxane on a solid silicon substrate without a pattern.

The two plates (stages 1 and 2) are then brought into contact after being exposed to oxygen plasma for 20 seconds at a plasma power of 200 watts (Figure 2).

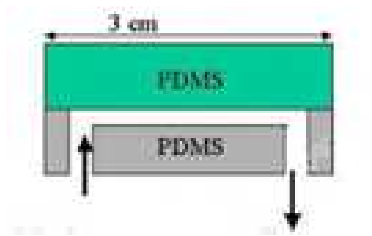

Figure 2: Microreactor in the second generation (PDMS-polydimethylsiloxane).

Next, consider the third generation microreactors.

4 Microreactors of the third generation 3G

Microreactors of the third generation are made of silicon and glass with features of liquid access from the silicon side. These microreactors have exactly the same geometry as the 2G microreactors [7].

Figure 3: Microreactor of the third generation 3G.

Next, we will consider a technique for creating a complex device for vinyl acetate production.

5 Method of creating a complex device for vinyl acetate production

To create a complex device, it is necessary to design and assemble a microreactor, SCARA robot, install a microreactor and SCARA type robot control system, connect with them peristaltic pumps for pumping liquids and gases, a refrigeration device and a thermostat for cooling and temperature control in the microreactor. Further in Figure 4, a diagram of a microreactor and a SCARA type robot in a complex will be presented.

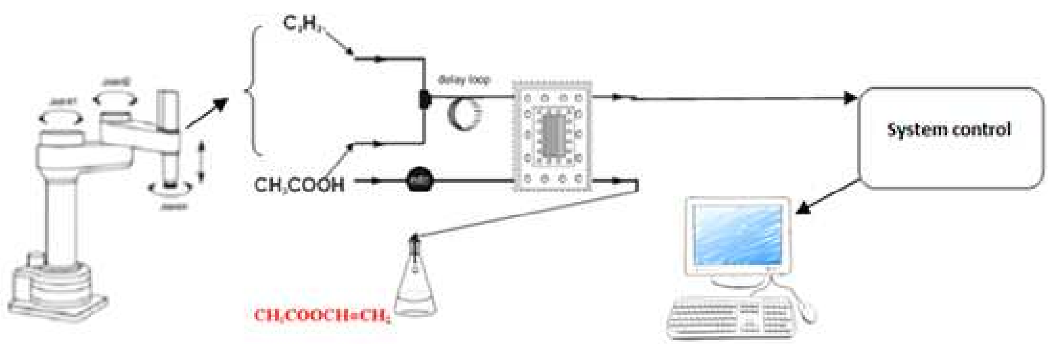

Figure 4: Scheme of a microreactor and a SCARA type robot in a complex.

6 Microreactor design technique

It is planned to create two entrances in the microreactor being designed. Acetylene and acetic acid will flow through these outlets [8]. The material for creating a microreactor is aluminum. Aluminum is a material that is resistant to corrosion and acids. For the manufacture of the microreactor body, polymeric materials will be used, for example, acrylonitrile / butadiene / styrene copolymers and polylactide. For the manufacture of the microreactor, a diagram of its body was created using the Autocad program. This diagram is shown below in Figure 5.

To create a prototype, the following components were used, which are presented in Table 1:

Next, the SCARA type robot will be discussed.

7 SCARA type robot - an integral part of a complex device

3D EXPERIECE Solidworks software was used to design the type SCARA robot. The following is a 3D model of a SCARA type robot (Figure 6).

The robot has 4 degrees of freedom and is driven by four stepper motors. In addition, it includes a servo motor for end-grip control. To create the robot, most of the body parts were designed in Solidworks and will be printed on a 3D printer. The SCARA robot and microreactor will be monitored by a microcontroller.

To control the SCARA type robot, a graphical user interface was created, in which there is control of forward and reverse kinematics. With forward kinematics, each joint of the robot

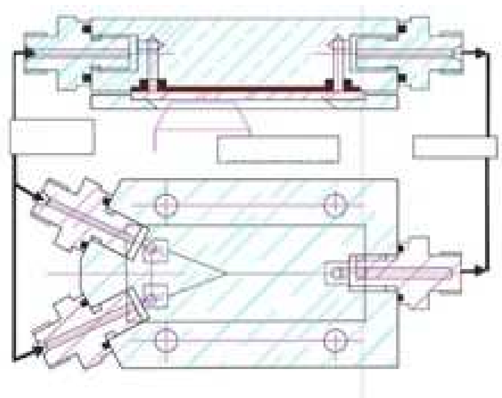

Figure 5: Diagram of the microreactor housing.

Table 1: Name of elements and materials for the microreactor

No	Name of equipment, device, elements and materials of the microreactor	Appointment of equipment, device, inventory	Parameters
1	refrigeration device, thermoelectric in the form of a Peltier plate element	for cooling the microreactor	4-stage multistage refrigeration unit
2	digital temperature controller	for temperature control	12 B, 24 B
3	aluminum heatsink with tight teeth	water cooling system	The dense teeth aluminum radiator has a bottom plate thickness: 4.6 mm and tooth thickness: 1.0 mm and the number of teeth: 27 tablets
4	transparent silicone tube	for bay from a probe	transparent flexible silicone tube size 0.5mm x 1mm non-toxic
5	peristaltic pump	for automatic dosing pump	cylinder automatic titration pump
6	tripod	for collecting probes for sending analyzes	tripod made of material PLA
7	sample collection container	to collect data	container for collecting material

can be manually moved to obtain the desired position [9]. Using the sliders on the left side, you can set the angle of each joint. The final position of the end gripper, the value of its X, Y and Z positions are calculated and printed [10]. On the other hand, using inverse kinematics, you can set the desired position, and the program will automatically calculate the angles for each joint: in order for the robot to get to the desired position [11]. The joint angles and their X, Y and Z values of the end clamp are linked and always present on the screen.

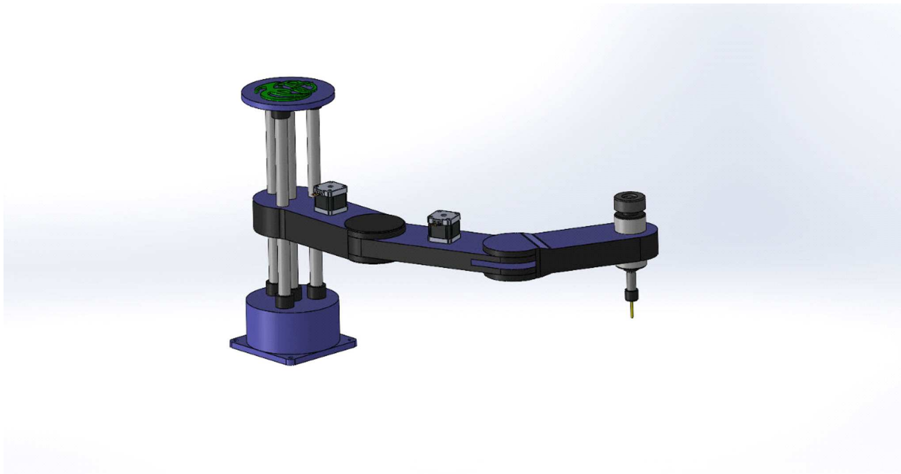

Figure 6: 3D model of the SCARA type robot.

8 Vinyl acetate production method

Vinyl acetate - obtained in a microreactor can serve as a raw material for the production of polyvinyl acetate. Vinyl acetate can be obtained in the process of catalytic vapor-phase synthesis based on the addition of acetylene and acetic acid:

$$2H2 + CH3COOH \rightarrow CH2 = CHOCOCH3$$

Zinc acetate supported on active carbons characterized by the presence of macro- and mesopores can be used as a catalyst [12].

Vinyl acetate can be obtained from acetic cystola and acytelene in two ways. This is a vapor-phase and liquid-phase method. The advantages of the vapor-phase method in comparison with the liquid-phase method are: ease of design, reduced corrosion, increased conversion of both ethylene and acetic acid, and increased selectivity of the process.

8.1 Vapor-phase method of vinyl acetate production

Vapor-phase vinylation is carried out with a large excess of acetylene. The higher the molar ratio of acetylene to acetic acid, the greater the conversion of the acid in one pass through the catalyst. The highest conversion is achieved at a molar ratio of acetylene to acid from 8: 1 to 10. However, due to the difficulty of the subsequent isolation of vinyl acetate from very dilute contact gases, it is necessary to carry out with a much lower excess of acetylene (4: 1). In this case, the degree of conversion in one pass decreases and the amount of unreacted acid increases, which is separated from the contact gases and returned to the process [13].

8.2 Liquid-phase method for producing vinyl acetate

The liquid-phase process for the production of vinyl acetate is carried out at $60 - 65°C$, passing at high speed an excess of acetylene through the reactor, which contains a mixture of glacial acetic acid and acetic anhydride containing dispersed mercury salts. Vinyl acetate, as it is formed, is removed from the reaction zone in the form of vapors entrained in excess acetylene. Vapors of vinyl acetate are condensed and sent to rectification. The acetylene separated from the liquid is returned to the production cycle [14]. Next, we will consider the method of obtaining vinyl acetate.

9 Technique for producing vinyl acetate in a microreactor

To obtain vinyl acetate, you must first obtain acetylene. To do this, take 5-10 grams of calcium carbide and place it in a 250 *ml* flask. Calcium carbide reacts violently with water. To slow down this reaction, you must use a saturated solution of sodium chloride. We will add a few drops of sodium chloride solution to the funnel [15]. Further addition of the solution is carried out so that a uniform gas flow is established at a rate that allows the formed bubbles to be counted. The evolved gas is acetylene and the second reaction product is calcium hydroxide.

To obtain vinyl acetate in a microreactor, the resulting acetylene must be mixed with acetic acid in a ratio of $3.5 - 5 : 1$. The mixture enters the microreactor and the reaction of the combination of acetylene and acetic acid occurs. The resulting vinyl acetate goes into a container for further analysis in the laboratory.

10 Conclusion

The possibility of using a microreactor and a SCARA type robot considered in the article has shown its applicability as a complex device for the production of vinyl acetate. A detailed description of the methodology for creating this complex device was developed, including a microreactor, SCARA type robot, control unit, pumps, tubes, thermostat and refrigerator. To implement the creation of a complex device in a real environment, it is necessary to assemble several prototypes and analyze the yield of vinyl acetate in the laboratory.

References

[1] Haswell S.J., Skelton V., "Chemical and biochemical microreactors", *TrAC Trends in Analytical Chemistry,* 19(6) (2000): 389–395 (ISSN 0165-9936, https://doi.org/10.1016/S0165- 9936(00)00012-1).

[2] Hernandez K., Eryanen K., Salmi T.O., Murzin D.Yu., "Gas-phase microreactors: a powerful tool for kinetic research", *J. Ros. Chem. Society named after D.I. Mendeleev,* 2 (2011): 16.

[3] Jensen K., *Chem. Eng. Sci.,* 56 (2006): 293.

[4] Aswathy K. Raghu, Niket S. Kaisare, "Thermally integrated microreactor for Sabatier reaction: Study of air-cooled and inert-diluted counter-current operation strategies", *Catalysis Today,* 2020 (ISSN 0920-5861, https://doi.org/10.1016/j.cattod.2020.08.025).

[5] Okamoto H., Ushijima T., Kito O., "New methods for increasing productivity by using microreactors of planar pumping and alternating pumping types", *Chemical Engineering Journal,* 101(1-3) (2004): 57–63 (ISSN 1385-8947, https://doi.org/10.1016/j.cej.2003.11.033).

[6] Hessel V., Hardt S., Löwe H., Schöfeld F., "Laminar mixing in different interdigital micromixers: I. Experimental characterisation", *AIChE J.,* 49(3) (2003): 566–577.

[7] Hassel V., Hardt S., Löwe H., "Chemical Micro Process Engineering: Fundamentals, Modelling and Reactions", *Wiley-VCH, Weinheim,* (2004): 5.

[8] Liu Y., Zhou W., Chen L., Lin Y., Xuyang Ch., Zheng T., Wan Sh., "Optimal design and fabrication of surface microchannels on copper foam catalyst support in a methanol steam reforming microreactor" , *Fuel,* 253 (2019): 1545–1555 (ISSN 0016-2361, https://doi.org/10.1016/j.fuel.2019.05.099).

[9] Gómez A., Lafuente P.D., Rebollar C., Hernéndez M.A., Olguín E.H., Jiménez H., Rodríguez J. "Design and construction of a didactic 3-dof parallel links robot station with a 1-dof gripper" , *Journal of Applied Research and Technology*, 12 (2014): 435–443.

[10] López M., Castelán F.J., Castro M., Peña R. Osorio. "Using object's contour, form and depth to embed recognition capability into industrial robots" , *Journal of Applied Research and Technology*, 11 (2013): 5–17.

[11] Surapong N., Mitsantisuk C. "Position and force control of the SCARA robot based on disturbance observer" , *Procedia Computer Science*, 86 (2016): 116–119.

[12] Omanov B.Sh., Khatamova M.S., Fayzullaev N.I., "Vinyl acetate production technologies" , *Innovative science,* 3 (2020): 10.

[13] Rihe A. *Fundamentals of the technology of organic substances* Per. with him. / Edited by D. D. Zykov (M.: State Scientific and Technical Publishing House of Chemical Literature, 1959): 531.

[14] Timofeev V.S. *Principles of the basic technology of organic and petrochemical synthesis: Textbook* Manual for universities V.S. Timofeev, L.A. Seraphimov. - 2nd ed., Perarab (M .: Higher. shk., 2003): 536.

[15] Temkin O.N., Shestakov G.K., Terer Yu.M. *Acetylene: Chemistry, mechanism effect, technology* (M: Chemistry, 1991): 416.