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ИЗВЕСТИЯ

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АО «Институт топлива, катализа и
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THE KINETICS OF SELECTIVE HYDRAZINOLYSIS OF MALEIC ACID ON THE ACID CATALYST

Abstract. For the first time, kinetics and the mechanism of the reaction of hydrazinolysis of maleic acid in the presence of cation exchanger resin KU-2-8 in H-form have been studied. The experiments were carried out in a static system in a thermostat glass reactor. It was found that cation exchanger shows high catalytic activity in the studied process - maleic acid conversion was 93%, and maleic hydrazide yield was 90%. The conversion selectivity of maleic acid to maleic hydrazide was 97,8%. The reaction rate was determined from the accumulation of maleic hydrazide. The apparent reaction rate constant (k) was calculated from the second-order reaction rate equation. The effect of initial concentrations of maleic acid and hydrazine hydrate, the temperature on the reaction rate was studied. The first order of maleic acid and hydrazine hydrate is determined. Activation energy of the process found from the Arrhenius dependence is 32,1 kJ/mol. On the basis of kinetic and IR spectroscopic methods, a probable reaction mechanism involving polymer-bound hydrogen ions is proposed.

Keywords: maleic acid, hydrazinolysis, hydrazide, catalyst.

Introduction. Among biologically active derivatives of unsaturated 1,4-dicarboxylic acids, substituted hydrazides of maleic, citraconic and fumaric acids having antimicrobial, fungicidal, herbicidal activity occupy a special place [1-3]. For example, maleic acid hydrazide is used as a plant growth regulator and an effective herbicide [4]. Maleic acid hydrazide is obtained by reacting maleic anhydride with a hydrazine sulfate solution. A significant disadvantage of this method is the need to use a large molar excess of maleic anhydride and concentrated sulfuric acid, the difficulty of separating the desired product from solutions.

Recently, ion exchanger catalysts based on styrene and divinylbenzene copolymers [5-9] have been successfully used in the synthesis of hydrazides of carboxylic acids. However, the kinetics and reaction mechanism of the hydrazinolysis of unsaturated dicarboxylic acids in the presence of ion exchanger resin is not clear.

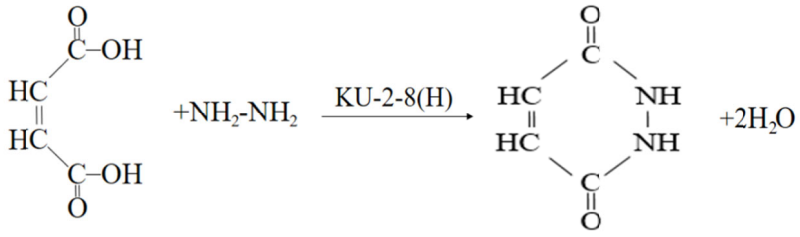
The present paper studies the kinetics and mechanism of the reaction of maleic acid with aqueous hydrazine on cation exchanger KU-2-8 using kinetic and IR spectroscopic methods.

Experimental part. The commercial synthetic cation exchange resin KU-2-8 was used as catalyst. The cation exchange resin was conditioned and converted into H-form by a standard method [10,11]. Experiments are carried out in static conditions. In a three-neck round-bottom flask with a capacity of 250 ml, equipped with a mechanical stirrer, a thermometer and a reflux condenser, 1 g (0,0086 mol) of maleic acid, 1,44 g (0,0288 mol) of hydrazine hydrate, 2 g of cation exchanger KU-2-8 in H -form, 16 g (0,889 mol) of water are introduced. The reaction mixture is heated on a water bath, stirring for 2 hours at a temperature of 343-368 K. The mixture was periodically sampled and analyzed. The analysis of maleic acid hydrazide is carried out by the photocolorimetric method [12]. The melting point of hydrazide is 571-573 K. Its

composition was determined by elemental analysis. For $C_4H_4N_2O_2$ calculated, %: C 42,84; H 3,57; N 24,99. Found , %: C 42,83; H 3,59; N 24,98.

The IR spectra of the starting materials and reaction products were recorded using an Impact- 410 spectrometer (USA) at 400-4000 cm^{-1}. The reaction rate was determined from the accumulation of maleic hydrazide. The apparent reaction rate constant (k) was calculated from the second-order reaction rate equation.

Results and discussions. We found that maleic acid readily reacted with hydrazine hydrate in the presence of the H-form of the cation exchanger , to yield maleic hydrazide.



It was found that cation exchanger shows high catalytic activity in the studied process – the conversion of maleic acid was 93%, and the maleic hydrazide yield was 90%. The conversion selectivity of maleic acid to maleic hydrazide was 97.8%. The optimal conditions for the synthesis are the following: the mass ratio of maleic acid: hydrazine hydrate: cation exchanger: H_2O = 1: 1.44: 2:16, temperature 368 K and reaction duration 2 h. Table shows the effect of initial concentrations of maleic acid and hydrazine hydrate, the temperature on the reaction rate.

The influence of various factors on the kinetics of maleic acid hydrazinolysis

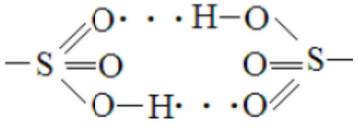
c_0 , mol/L	$c_0^{'}$, mol/L	T,K	$k \cdot 10^{-3}$, L/ (mol·min)
0,28	2,70	363	6,6
0,36	2,70	363	
0,44	2,70	363	
0,44	2,54	363	6,2
0,44	2,92	363	
0,44	2,70	343	2,7
0,44	2,70	353	2,9
0,44	2,70	368	3,1

The change in the initial concentration of the maleic acid (c_0) within the studied range does not affect the reaction rate constant, which indicates the first order of reaction for this component (table). When the initial concentration of hydrazine hydrate ($c_0^{'}$) changes, it is shown that the values of the reaction rate constant also remain constant. The reaction is of the first order according to the second component - hydrazine hydrate.

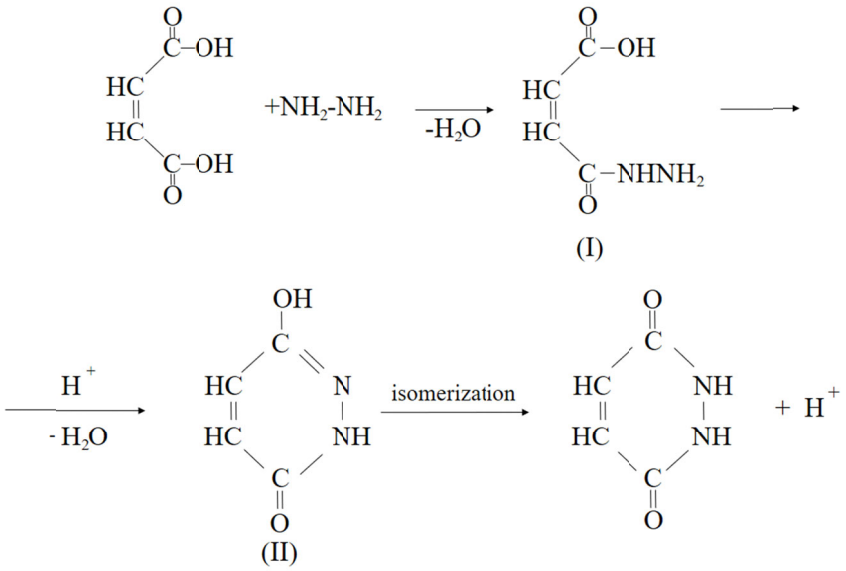
The dependence of the maleic hydrazide concentration on time at different temperatures was studied. As the temperature rises, the value of the reaction rate constant increases. In the temperature range 343-368 K, the activation energy denermined from the Arrhenius equation equaled 32,1 kJ/mol.

In view of the obtained kinetic parameters (low value activation energy, first reaction rate orders with respect to both maleic acid and hydrazine hydrate) it could be suggested that the reaction occurred via the formation of surface transition complexes.

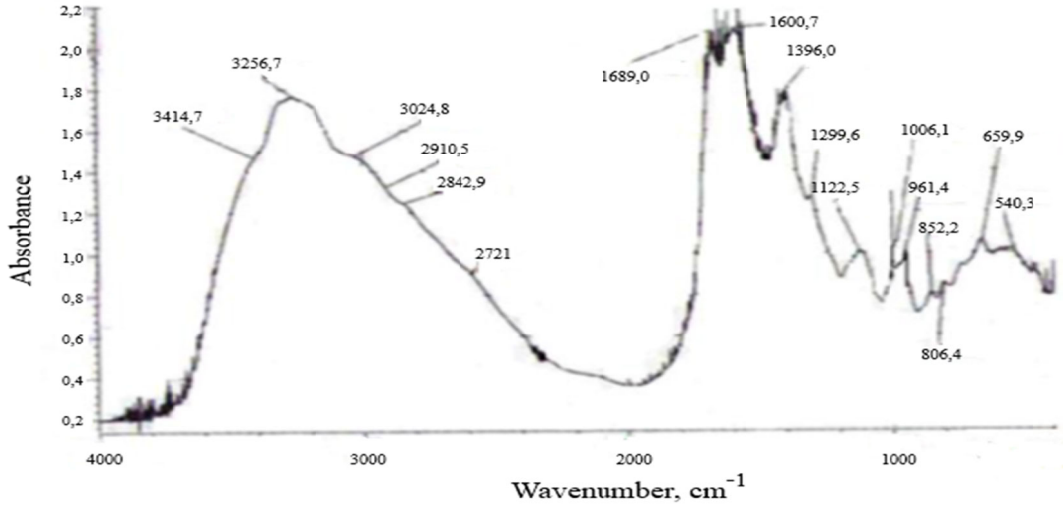
The discussed kinetic data were in line with the results of the IR spectroscopy study. The literature [13, 14] suggests that the catalytic active sites of the KU-2-8 cation exchange resin in the hydrazinolysis of maleic acid are polymer-bound sulfonium ions and hydrogen ions:



The transporters across the phase interface in cation exchange resin are protons. Therefore,the adsorption on the cation exchange resin is carried out as a result of proton transfer. It can be assumed that the hydrazinolysis of maleic acid in the presence of cation exchanger KU-2-8 in the H-form is carried out in several stages: first, maleic acid reacts with the hydrazine molecule by the mechanism of nucleophilic addition, with the to formation of the corresponding monohydrazide (I). In the next step, monohydrazide under the action of the acid catalyst (H^+) enters into a cyclodehydration reaction. **T**he resulting product is isomerized to form maleic acid hydrazide and regenerating the catalyst.



In the IR spectrum of maleic acid hydrazide, absorption bands in the region at 2910-2842 cm^{-1} that is due to the stretching vibrations of the C-H group. Bands with absorption frequencies in the region at 1396-1299 cm^{-1} and 1600 cm^{-1} assigned to the deformation vibrations of the C-H and C = C groups. The absorption bands at 1689, 3414 and 1122 cm^{-1} assigned to the stretching vibrations of the C=O , N-H and C-N groups, respectively (figure).



IR spectrum of maleic acid hydrazide

Against the background of the own bands of the cation exchanger, intense absorbtion bands at 1406, 1451, 1500 cm^{-1} and a number of bands in the region at 3100-3200 cm^{-1} appear, which can be assigned to symmetric and antisymmetric vibrations of the adsorption complex > NH_2^+ of the hydrazide ion with the sulfo group of the cation exchanger [15-20].

Conclusion. In summary, we investigated for the first time, the kinetics regularity of hydrazinolysis of maleic acid in the presence of the H-form of the cation exchanger KU-2-8. The second overall reaction rate order was found. Activation energy of the process were determined over the 343-368 K temperature range. On the basis of kinetic and IR spectroscopic studies, a probable reaction mechanism involving polymer-bound hydrogen ions is proposed.

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ҚЫШҚЫЛ КАТАЛИЗАТОРЫНДАҒЫ МАЛЕИН ҚЫШҚЫЛЫНЫҢ СЕЛЕКТИВТІ ГИДРАЗИНОЛИЗІНІҢ КИНЕТИКАСЫ

Аннотация. Алғашқы рет малеин қышқылының гидразинолиз реакциясының кинетикасы және механизмі Н-формадағы КУ-2-8 катионит қатысында зерттелді. Тәжірибелер статикалық жағдайда шыны реакторда жүргізілді. Қарастырылған реакцияда Н-формадағы катионит жоғары каталиттік активтілік көрсететіндігі анықталды, малеин қышқылының конверсиясы 93 %, ал малеингидразид шығымы 90 % болды. Малеин қышқылының малеингидразидке конверсиясының селективтілігі 97,8 % болды. Реакцияның жылдамдығын малеин қышқылының гидразидінің түзілуіне қарай бақылайды. Байқалатын жылдамдық константасы (k) екінші ретті реакцияның кинетикалық теңдеуі арқылы есептелді. Малеин қышқылының және гидразингидраттың бастапқы концентрацияларының, температураның реакция жылдамдығына әсері зерттелді. Реакция малеин қышқылы және гидразингидрат бойынша бірінші ретті екендігі анықталды. Аррениус тәуелділігінен анықталған процесс активтендіру энергиясының шамасы 32,1 кДж / моль болды. Кинетикалық және ИҚ-спектроскопиялық зерттеулер негізінде полимермен байланысқан сутегі иондарының қатысуымен жүретін реакция механизмі ұсынылды.

Түйін сөздер: малеин қышқылы, гидразинолиз, гидразид, катализатор.

С.А. Джумадуллаева[1], А.Б. Баешов[2], А.В. Колесников[3]

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КИНЕТИКА СЕЛЕКТИВНОГО ГИДРАЗИНОЛИЗА МАЛЕИНОВОЙ КИСЛОТЫ НА КИСЛОТНОМ КАТАЛИЗАТОРЕ

Аннотация. Впервые изучены кинетика и механизм реакции гидразинолиза малеиновой кислоты в присутствии катионита КУ-2-8 в Н-форме. Опыты проводили в статической системе в термостатированном стеклянном реакторе. Установлено, что катионит проявляет высокую каталитическую активность в изучаемом процессе – конверсия малеиновой кислоты составил 93 %, а выход малеингидразида 90 %. Селективность конверсии малеиновой кислоты в малеингидразид составил 97,8 %. За скоростью реакции следили по накоплению гидразида малеиновой кислоты. Наблюдаемые константы скорости (k) вычисляли по кинетическому уравнению второго порядка. Изучено влияние начальных концентраций малеиновой кислоты и гидразингидрата, температуры на скорость процесса. Определен первый порядок по малеиновой кислоте и гидразингидрату. Энергия активации процесса, найденная из аррениусовской зависимости, составила 32,1 кДж/моль. На основе кинетического и ИК-спектроскопического методов предложен вероятный механизм реакции с участием полимерносвязанных ионов водорода.

Ключевые слова: малеиновая кислота, гидразинолиз, гидразид, катализатор.

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