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ОСОБЕННОСТИ ГИДРОГЕНИЗАЦИИ 2-ХЛОР-4-НИТРОАНИЛИНА НА НАНЕСЕННЫХ ПАЛЛАДИЕВОМ И ПЛАТИНОВОМ КАТАЛИЗАТОРАХ В 2-ПРОПАНОЛЕ И ЭТИЛАЦЕТАТЕ

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Статья посвящена исследованию влияния природы растворителя и катализатора на кинетические закономерности протекания гидрогенизации 2-хлор-4-нитроанилина на нанесенных палладиевых и платиновых катализаторах в водном растворе 2-пропанола азеотропного состава и этилацетате. Известно, что восстановление галогензамещенных нитробензолов может сопровождаться побочной стадией гидрогенолиза. В связи с этим, разработка объективных представлений о методах селективного управления стадийности превращений изучаемой реакции представляет чрезвычайно важную задачу. В работе обсуждены влияние природы растворителя и катализатора на выход хлорзамещенного амина. Следует отметить, что вид кинетических кривых по водороду при гидрировании в этилацетате и 2-пропаноле свидетельствует о значительном влиянии природы растворителя и сложной взаимосвязи структура катализатора – растворитель. Установлено, что побочным процессом, снижающим селективность реакции по отношению к 2-хлор-1,4-фенилендиамину, является его дегалогенирование. В случае использования в качестве растворителя этилацетата отмечено отсутствие в гидрогенизате продукта дегалогенирования 2-хлор-1,4-фенилендиамина при реализации процесса как на палладиевом, так и на платиновом катализаторах. Установлено, что скорости поглощения водорода в этилацетате на палладиевом катализаторе в восемь раз выше, чем на платиновом. Данный эффект может быть связан с меньшим вкладом диффузионного торможения по водороду на палладиевом катализаторе. Очевидно, что подобные эффекты могут быть обусловлены структурой носителей. Вероятно, платина при нанесении на подложку распределена на внутренней поверхности глубоких пор носителя, тогда как распределение фазы палладия скорее отвечает корочковому распределению. Данное предположение также объясняет и факт более высокой активности палладиевого катализатора, что сопровождается высокими степенями дегалогенирования в 2-пропаноле.

Ключевые слова: 2-хлор-4-нитроанилин, нанесенный палладиевый катализатор, нанесенный платиновый катализатор, скорость, гидрогенизация, степень дегалогенирования, 2-пропанол, этилацетат

FEATURES OF HYDROGENIZATION OF 2-CHLORO-4-NITROANILINE ON SUPPORTED PALLADIUM AND PLATINUM CATALYSTS IN 2-PROPANOL AND ETHYL ACETATE

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The article is devoted to studying the solvent and catalyst nature influence on the 2-chloro-4-nitroaniline hydrogenation kinetic regularities on supported palladium and platinum catalysts in an aqueous solution of azeotropic composition and ethyl acetate. It is known that the halogen-substituted nitrobenzenes reduction may be accompanied by a secondary stage of hydrogenolysis. In this regard, the development of objective ideas about the selective control methods of the studied reaction the transformations staging is an extremely important task. The paper discusses the influence of the solvent and catalyst nature on the chlorine substituted amine yield. It should be noted that the form of kinetic curves for hydrogen in hydrogenation in ethyl acetate and 2-propanol indicates a significant influence of the solvent nature and a complex relationship between the catalyst structure and the solvent. It was established that a side-effect process that reduces the selectivity of the reaction with respect to 2-chloro-1,4-phenylenediamine is its dehalogenation. In the case of using ethyl acetate as a solvent, the absence of the 2-chloro-1,4-phenylenediamine dehalogenate product in the hydrogenation during the process, on both palladium and platinum catalysts, is noted. It is established that the hydrogen absorption rate in ethyl acetate on a palladium catalyst is eight times higher than on platinum. This effect may be associated with a smaller contribution of diffusion braking on a palladium catalyst. It is obvious that such effects may be due to the structure of the carriers. Probably, platinum, when applied to a substrate, is distributed on the inner surface of the carrier deep pores, while the distribution of the palladium phase is more likely to correspond to the crust distribution. This assumption also explains the fact of the palladium catalyst higher activity, which is accompanied by dehalogenation high degrees in 2-propanol.

Key words: 2-chloro-4-nitroaniline, supported palladium catalyst, supported platinum catalyst, rate, hydrogenation, dehalogenation degree, 2-propanol, ethyl acetate

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INTRODUCTION

The synthetic fuel production from renewable hydrocarbons is one of the possible alternatives to the existing methods of processing fossil energy sources [1, 2]. Aromatic hydrocarbons are represented in petroleum by benzene and its homologues [3]. Therefore, the most optimal option for the aromatic hydrocarbons transformation is to obtain aromatic amines using catalytic methods. Aromatic amino compounds are known to possess high detonation resistance. They are widely used as a replacement for antiknock additives containing lead and manganese. In addition, the products of halogen substituted amines liquid-phase hydrogenation are widely used in the manufacture of pesticides, herbicides, synthetic dyes, fibers, pharmaceuticals and other substances. It should be noted that for different processes both a high and a low target compounds dehalogenation degree are necessary. This leads to considerable interest in optimizing catalytic systems involving halogen substituted amines. One of the most significant parameters of the catalytic system determining both the rate and hydrogenation selectivity is the solvent and the catalyst nature [4-6]. In this regard, the studies, aimed at clarifying the influence of active metal nature in supported catalysts and the solvent nature on the hydrogenation rate and the dehalogenation degrees of 2-chloro-4-nitroaniline (2C4NA), as well as the depth of the process and the selectivity of this compound hydrogenation with respect to to halogen-substituted amine, are relevant.

This investigation purpose is to study the solvent and catalyst nature influence on the hydrogenation kinetics of 2-chloro-4-nitroaniline on supported palladium (10% Pd/C) and platinum (0.5% Pt/C) catalysts.

EXPERIMENTAL PART

During the experiment, the kinetics of 2C4NA hydrogenation on deposited palladium and platinum catalysts with a metal content of 10% and 0.5%, respectively, in 2-propanol (0.68 mole fraction) and ethyl acetate aqueous solution was studied.

According to the literature data, in the reduction of chlorine-substituted nitro benzenes in low-grade aprotic solvents, and in proton-containing solids, high degrees of dehalogenation are observed [7]. In this regard, the study of the 2C4NA liquid-phase hydrogenation kinetics in ethyl acetate and 2-propanol is a practically significant task.

When carrying out the kinetic experiment, a static method was used to carry out the hydrogenation reaction in a semi-closed system at a constant temperature (303 ± 1 K) and hydrogen pressure with intensive liquid phase mixing (3000 rpm). Such an experimental scheme allows one to determine the observed reaction rates with high reliability, and the experimental conditions ensured the elimination of the external mass transfer influence on the observed reaction rates.

The catalysts used were 10% palladium catalyst deposited on carbon grade ARD, as well as 0.5% platinum catalyst supported on coal grade OBB. The surface morphology of the catalysts was studied using a Mira 3LMH, TESCAN high resolution scanning electron microscope and a VEGA 3SBH scanning electron microscope, TESCAN, Czech Republic.

The main process characteristics were the observed reaction rate constants for hydrogen. They were calculated by processing the kinetic curves, examples of which are shown in Fig. 1 and 2. The values of the observed reaction rate constant for hydrogen in each case corresponded to the tangent of the slope of the initial linear portion of the kinetic curve, indicating a zero order.

According to the data obtained, during the transition from an aqueous solution of 2-propanol to ethyl acetate, there was a decrease in the observed reaction rate by almost four times, both on palladium and platinum catalysts. It is worth noting that in 2-propanol aqueous solution on a palladium catalyst, the hydrogen absorption was observed in excess of the stoichiometrically necessary amount for the conversion of 2C4NA to 2-chloro-1,4-phenylenediamine (2CPhDA).

This indicates that the process proceeds more deeply and under the conditions of 2C4NA hydrogenation, hydrogen reacts not only with the nitro group, but also with the halogen substituent.

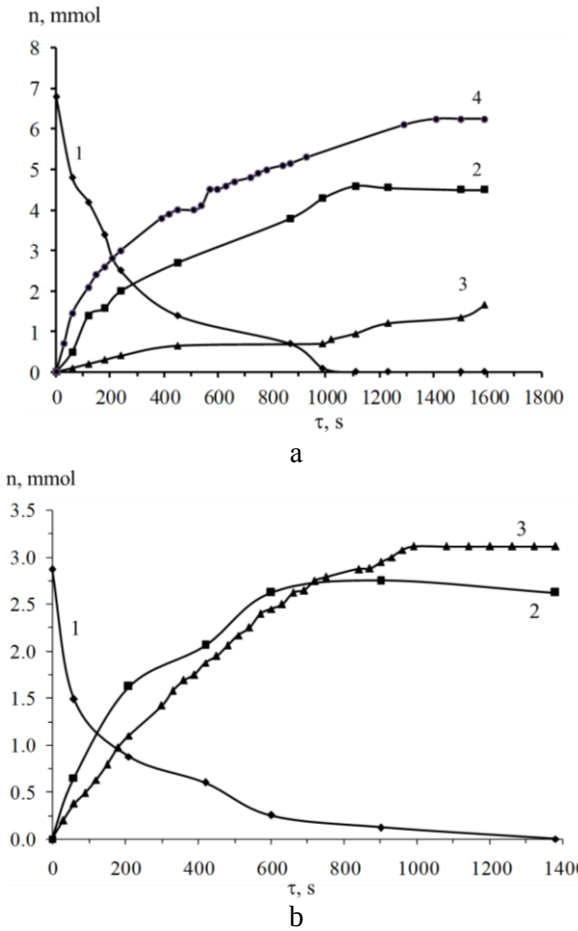
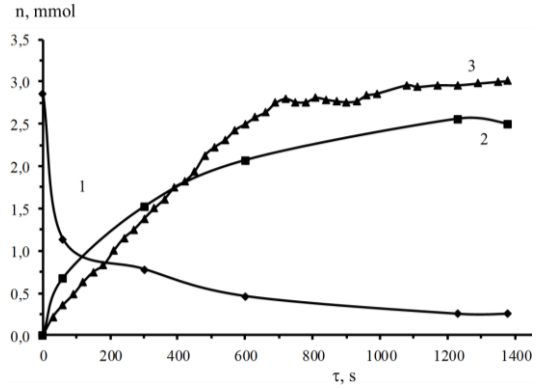


Fig. 1. Kinetic curves of hydrogen absorption for the 2C4NA hydrogenation in 2-propanol aqueous solution on 10% Pd/C (a) and 0.5% Pt/C (b): 1 - hydrogen, 2 - 2-chloro-4-nitroaniline, 3 - 2-chloro-1,4-phenylenediamine, 4 - 1,4-phenylenediamine

Рис. 1. Кинетические кривые поглощения водорода в гидрогенизации 2-хлор-4-нитроанилина в водном растворе 2-пропанола на 10 % Pd/C (a) и 0.5 % Pt/C (b): 1 – водород, 2 – 2-хлор-4-нитроанилин, 3 – 2-хлор-1,4-фенилендиамин, 4 - 1,4-фенилендиамин

In works [8-15] it is noted that upon completion of the reaction a mixture of substances is formed, containing 2-chloro-1,4-phenylenediamine and 1,4-phenylenediamine (PhDA). The ratio of products, both during the reaction and at its completion, may be different based on the characteristics of the selected catalyst, solvent, temperature and pressure.

The reaction media analysis, which was carried out using a scanning spectrophotometer “LEKI SS 2110 UV”, showed that in the 2-propanol aqueous solution, the integral selectivity in 2CPhDA and PhDA was 76% and 23%, respectively. On the contrary, during the 2C4NA hydrogenation in ethyl acetate by 10% Pd/C, the 2CPhDA selectivity was higher and was 89%. At the same time, no PhDA significant amounts were detected in the reaction mixture. The data illustrating this provision is shown in Table.



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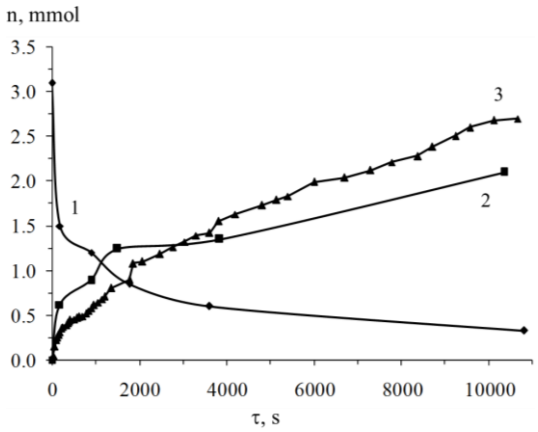

b

Fig. 2. Kinetic curves of hydrogen absorption (4) for the 2C4NA (1) hydrogenation in ethylacetate on 10% Pd/C (a) and 0.5% Pt/C (b): 1 - hydrogen, 2 - 2-chloro-4-nitroaniline, 3 - 2-chloro-1,4-phenylenediamine

Рис. 2. Кинетические кривые поглощения водорода в гидрогенизации 2-хлор-4-нитроанилина в этилацетате на 10 % Pd/C (a) и 0.5 % Pt/C (b): 1 – водород, 2 – 2-хлор-4-нитроанилин, 3 – 2-хлор-1,4-фенилендиамин

THE RESULTS AND THE DISCUSSION

The appearance of kinetic curves (Fig. 1 and 2) for hydrogen in hydrogenation in ethyl acetate and 2-propanol indicates a significant solvent nature influence and a complex relationship between the catalyst structure and the solvent. It should be noted that in 2-propanol for platinum and palladium catalysts the kinetic curves have the same type and the reaction follows the zero order in hydrogen.

When going from 2-propanol to ethyl acetate, the observed rates drop significantly. This may be due to the low mutual miscibility of ethyl acetate and water formed during the reduction of the nitro group. In this case, the separation of the liquid phase of ethyl acetate-water occurs and, as a result, the contribution of diffusion braking on hydrogen to the observed reaction rate increases. Due to the decrease in the solubility of the starting compound, its concentration in the surface layer increases, which leads to an increase in the resulting chloramine desorption rate. This assumption is consistent with the obtained data on the final 2CPhDA amounts in the reaction mixtures on platinum and palladium, where also no PhDA traces were detected (Table 1).

When using a palladium catalyst, an increase in chloramine selectivity becomes a side effect of diffusional drag on hydrogen. This may be due to the palladium ability to dissolve a larger amount of hydrogen as compared with platinum [16], which is able to participate in the nitro group reduction [17, 18].

Table

The observed 2-chloro-4-nitroaniline hydrogenation rate constants on supported palladium and platinum catalysts in 2-propanol and ethyl acetate

***Таблица.* Константы скорости по водороду и 2-хлор-4-нитроанилину на нанесенных палладиевом и платиновом катализаторах в 2-пропаноле и этилацетате**

Catalyst	0.5% Pt/C		10% Pd/C	
Solvent	2-propanol	ethylacetate	2-propanol	ethylacetate
$k_{H2}\cdot10^{-2}$ mmol/(s·g)	0.87 ± 0.04	0.23 ± 0.03	2.16 ± 0.04	0.55 ± 0.03
n_{H2}, mmol	3.30 ± 0.04	2.71 ± 0.03	3.22 ± 0.04	2.90 ± 0.03
n_{2CPhDA}, mmol	2.56 ± 0.05	2.02 ± 0.05	2.20 ± 0.05	2.57 ± 0.05
n_{PhDA}, mmol	–		0.68 ± 0.05	-

The hydrogen absorption rate in ethyl acetate at 10% Pd/C is in eight times higher than at 0.5% Pt/C. It can be assumed that the contribution of diffusional drag on the palladium catalyst is less than on platinum, which also provides 2C4NA conversion high degrees, as evidenced by the data in the Table.

Such effects may be due to the carrier's structure [19]. Probably, platinum, when applied to a substrate, is distributed on the carrier's deep pores inner surface, whereas the distribution of the palladium phase rather corresponds to the crust distribution (Fig. 3 and 4). It can also provide a higher activity of the palladium catalyst, which is accompanied by high dehalogenation degrees in 2-propanol, and the formation of PhDA is observed from the very beginning of the reaction (Fig. 1a).

Thus, it can be concluded that in ethyl acetate both on platinum and palladium catalysts, the 2C4NA

conversion degree does not reach 100%. As a result, the 2C4NA conversion degree on 0.5% Pt/C is 94%, and on 10% Pd/C – 88%. The process proceeds selectively to 2CPhDA at 10% Pd/C in ethyl acetate and at 0.5% Pt/C in a 2-propanol-water azeotropic composition.

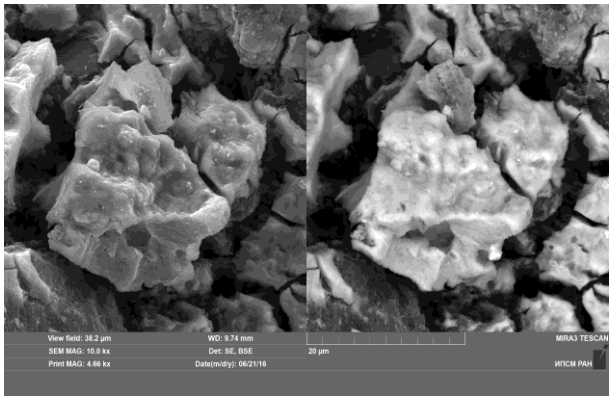

Fig. 3. Images of the catalyst surface of 10% Pd/C obtained with high-resolution scanning electron microscope Mira 3LMH, TESCAN
Рис. 3. Изображение поверхности катализатора 10 % Pd/C, полученное с использованием сканирующего микроскопа высокого разрешения Mira 3LMH, TESCAN

Therefore, supported catalysts are very promising. They are distinguished by versatility, sufficient wear resistance, relatively high activity and selectivity. However, disclosure of the relationship "catalyst structural characteristics - activity and selectivity in the reaction" is not yet possible due to the catalysts diversity and the scientifically lack based principles for their selection. To solve this problem, it is advisable to conduct similar studies with catalysts that have a longer service life, lower deactivation rate and greater activity compared to traditional deposited ones [20].

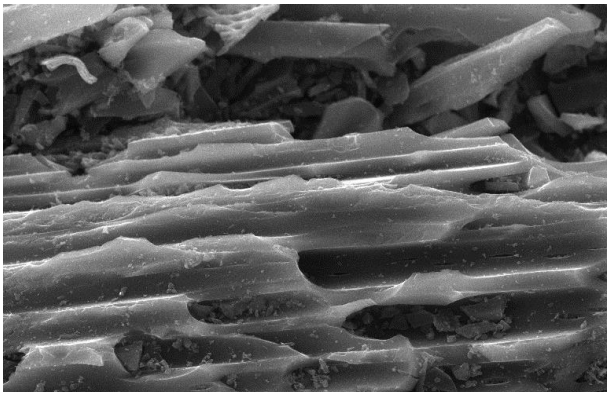

Fig. 4. Pictures of SEM VEGA 3SBH, TESCAN catalyst surface of 0.5% Pt/C obtained with electron microscope SEM VEGA 3SBH, TESCAN
Рис. 4. Картина поверхности катализатора 0.5 % Pt/C, полученная на сканирующем электронном микроскопе SEM VEGA 3SBH, TESCAN

Achievement of the goal for the synthesis of such catalysts will be provided, both using template methods for the synthesis of modified catalytically active surfaces, and the traditional sol-gel method.

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