

COPPER ALLOY CATALYSTS IN THE HYDROGENATION OF ANTHRAQUINONE: A TECHNOLOGICAL STUDY

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Abstract. The hydrogenation of anthraquinone is a critical step in the synthesis of hydrogen peroxide, an important industrial oxidant. Traditionally, solid noble metal catalysts such as nickel and palladium have been used for this reaction, but the exploration of alternative catalysts such as copper alloys offers potential advantages in cost and availability, as well as opportunities to tune the catalytic properties. The choice to study anthraquinone hydrogenation using a copper alloy catalyst was driven by the need to overcome the problems associated with noble metal catalysts, including cost and scarcity. This study focuses on the hydrogenation of anthraquinone using a copper-aluminum alloy catalyst. The catalyst was prepared by alloying copper and aluminum, with added elements such as chromium, iron, and silicon. The objective was to improve the catalytic activity and selectivity in the hydrogenation process, which is crucial for the industrial production of hydrogen peroxide. The catalyst was treated with 20% NaOH, which facilitated the exposure of active centers, leading to enhanced hydrogen desorption. The results demonstrated that the catalyst with 50% aluminum and 45% copper exhibited the highest catalytic performance, achieving hydrogen peroxide yields of up to 30.3% at 60°C and 1 MPa pressure. The catalysts containing chromium, iron, and silicon showed 1.6-1.9 times increase in productivity compared to the prototype. Optimal performance was observed at a temperature of 100°C, where the hydrogenation rate reached its peak. These findings indicate that the Cu-Al-FCC-75 alloy is highly effective for anthraquinone hydrogenation, providing high activity and selectivity.

Key words: anthraquinone, hydrogen peroxide, hydrogenation, organic synthesis, catalyst.

Introduction. Anthraquinone and its derivatives are important products of organic synthesis [1-2]. They are used in the production of dyes [3], hydrogen peroxide [4], pharmaceuticals [5], as catalysts for wood delignification [6], etc. Existing industrial methods for the synthesis of anthraquinone derivatives have long been outdated. They are usually multi-stage and accompanied by abundant waste, in particular acidic effluents. One possible method for obtaining substituted anthraquinone is diene synthesis. It is based on the reaction between 1,4-naphthoquinone and substituted 1,3-butadienes [3]. It is also possible to use naphthoquinones with the same substituents in the 6, 7 and (or) 5, 8 positions. Adducts are obtained in organic solvents (during the synthesis of anthraquinone, the process is carried out under a pressure of 1,3-butadiene of 0.3-2 MPa; organic acids are used to reduce the pressure). Adducts of diene synthesis can be further oxidized to anthraquinone by strong oxidizers in an acidic medium (CuCl_2 , H_2O_2 or NaClO_3 [7]) or by atmospheric oxygen in an alkaline medium [8]. Thus, at the present time, the problem of developing an environmentally friendly process for obtaining anthraquinone and its substituted derivatives is very relevant.

Hydrogenation of anthraquinone in the presence of a metal catalyst is a key reaction in the synthesis of H_2O_2 via the anthraquinone route [9]. Considering the increasing demand for H_2O_2 , developing a catalyst with excellent performance and relatively low price is one of the most important issues to meet the requirements of efficiency and economy. It is well known that supported bimetallic catalysts have attracted much attention in the field of catalysis, since such a combination of metals can often lead to excellent catalytic performance due to synergistic effects [10]. H_2O_2 has an environmentally friendly profile because it decomposes to form only water and oxygen as the only reaction products, making it one of the cleanest and most versatile chemical oxidants available (Figure 1).

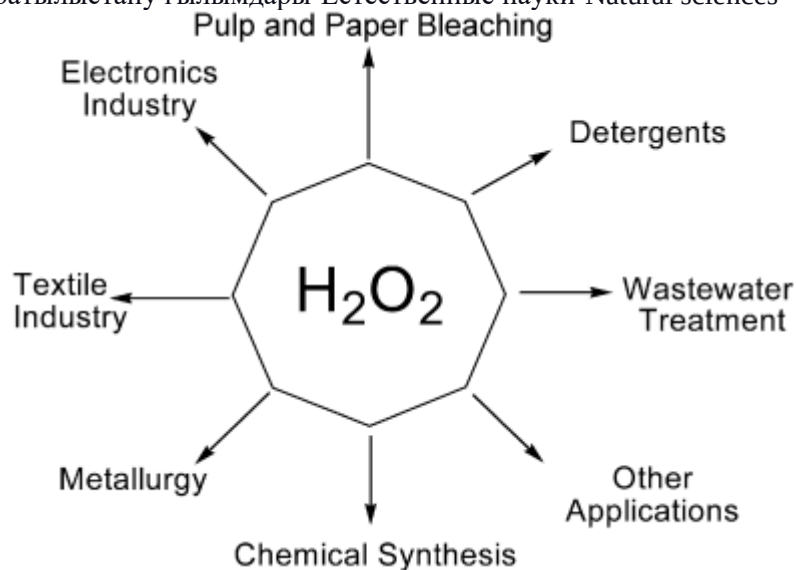


Figure 1 – Main areas of application of hydrogen peroxide

Hydrogen peroxide (H_2O_2) is a green and environmentally friendly molecular, widely used in food, paper, textile, and wastewater treatment industries. Its redox product is water and thus will not cause secondary pollution to the environment. Since the early twenty-first century, the application of H_2O_2 in producing some bulk chemicals, such as caprolactam, propylene oxide, and epichlorohydrin, has significantly promoted the growth of H_2O_2 demand. Recently, with the improvement of the environmental protection requirements, the replacement of non-green oxidants will be compelled. Thus, the need for H_2O_2 surely will further increase year by year [11].

These characteristics make hydrogen peroxide an environmentally friendly oxidant for many large-scale applications such as pulp and paper bleaching, textile manufacturing, detergent applications, wastewater treatment, and chemical oxidation processes. In the recent COVID-19 era, the demand for medical products is increasing because H_2O_2 has high antiseptic properties, which directly increases its market value. H_2O_2 is used as an antiseptic, which is usually applied to cuts and bruises of the skin to prevent infection. In addition, considering the recent COVID-19 pandemic, the demand for H_2O_2 has increased significantly worldwide due to its high consumption in disinfectants. H_2O_2 is the main ingredient in hand sanitizers, the demand for which has simultaneously increased worldwide [5].

Material and research methodology. The catalysts for anthraquinone hydrogenation were prepared by melting metals in a melting furnace. The melting process was carried out in a graphite beaker. The chemical composition of the primary castings and catalysts was determined by local X-ray spectral analysis on a Camebax microanalyzer from Samesa. X-ray structural analysis of the ingots and catalyst was carried out in the laboratory of physical and chemical studies of the Karaganda State University using an iron anode on a Wirs-50 IM and Dron-0.5 diffractometer. The specific surface area of the catalyst was determined by argon thermal desorption. The porous structure of the catalysts was studied using a volumetric adsorption unit.

Results and discussion. The study analyzed the catalytic activity and selectivity of a copper-aluminum alloy catalyst used for the hydrogenation of anthraquinone. The catalyst used for hydrogenation of anthraquinone based on leaching of copper-aluminum mixture contains chromium, iron and silicon in the form of ferroalloy-ferrosilicochrome in the following mass ratio: copper 40-49; chromium 0.209-2.09; iron 0.051-0.51; silicon 0.735-7.35; aluminum - the rest. Table 1 shows the control of the catalytic activity of the prepared catalyst with different amounts of caustic. Table 2 shows the chemical analysis of the catalyst in the form of a copper ingot alloyed with metal

Table 1 - Control of the catalytic activity of the prepared catalyst with different amounts of caustic soda

Mass fraction of alkali, %	Weight of the catalyst, g	Amount of hydrogen removed, l	Degree of destruction of aluminum, %	
			by weight	by H ₂
5	126.5	12	5	14
10	113.7	34	25.6	40.3
15	112.1	35	26.7	41.2
20	107.8	45	35.1	40.3

Table 2 - Chemical analysis of the catalyst in the form of a copper ingot alloyed with metal

The composition of the charge ingot, weight, %	Chemical composition of the catalyst, %				
	Cu	Me ₁	Me ₂	Me ₃	Al
Cu-Zn-Al	86.4	—	—	—	13.6
Cu-Al-FCC-75	81.1	4.4	—	—	14.5

The catalyst treated with 20% NaOH solution showed high activity. Such treatment facilitated the opening of active centers of the catalyst surface, which in turn accelerated the process of hydrogen desorption. According to the results of the studies, the activity and stability of the catalyst reach the highest value at a leaching depth of 15-18%.

Table 3 presents the results of X-ray spectral microanalysis of the chemical composition of the catalyst, where the percentage composition of copper, aluminum and additional metallic elements (Me₁, Me₂, Me₃) in various catalysts was analyzed.

Table 3 - X-ray spectral microanalysis of the chemical composition of the catalyst and castin

Compound	Sample	Component, % by weight				
		Ni	Me ₁	Me ₂	Me ₃	Al
Cu-Al	Mixture	50.0	—	—	—	50.0
	Alloy	48.6	—	—	—	51.2
	Catalyst	95.7	—	—	—	3.2
Cu-Zr-Al	Mixture	45.0	5.0	—	—	50.2
	Alloy	43.2	4.8	—	—	52.0
	Catalyst	82.0	9.5	—	—	6.5
Cu-Nb-Al	Mixture	45.0	5.0	—	—	50.0
	Alloy	42.2	4.9	—	—	50.6
	Catalyst	82.0	8.9	—	—	8.0
Cu-Al-FCC-75	Mixture	45.0	5.0	—	—	50.0
	Alloy	44.2	4.8	—	—	51.0
	Catalyst	81.0	9.9	—	—	9.0

According to Table 3, copper and aluminum-based catalysts exhibit high activity and selectivity. Additional elements (Me₁, Me₂, Me₃) affect the stability of the catalyst, its surface properties and chemical activity. Table 4 presents the results of anthraquinone hydrogenation with a multicomponent copper catalyst (50% Al) in a benzene-propanol mixture at 60°C and a pressure of 1 MPa.

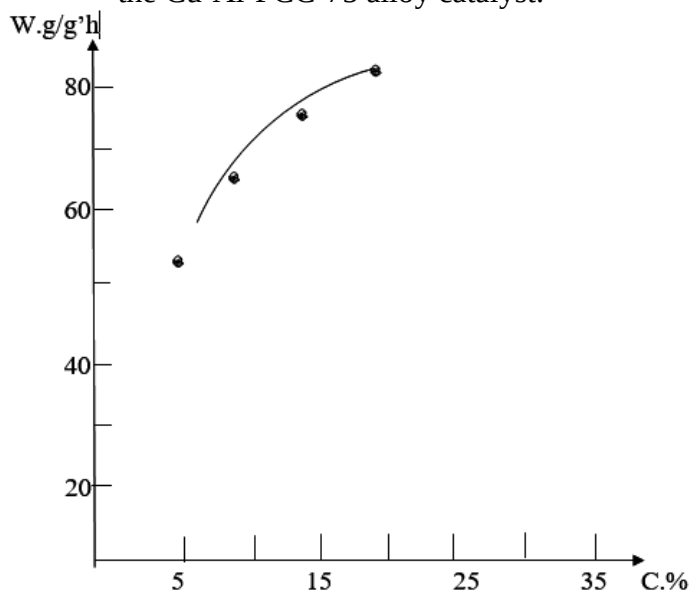
Table 4 - Results of anthraquinone hydrogenation with a multicomponent copper catalyst (50% Al) in a benzene-propanol mixture at 60°C and a pressure of 1 MPa.

Ingot composition, wt. %	H ₂ O ₂ yield over time (%)				W 10 ² g/g h by H ₂ O ₂
	0	20	40	60	
1. Cu-Al-FCC-75, (49-50-1.0)(Cu-49; Al-50; Fe-0.051; Cr- 0.209; Si- 0.735, P ^x -0.005)	3.5	7.8	17.8	26.4	8.6
2. Cu-Al-FCC-75, (47-50-3.0)(Cu-47; Al-50; Fe-0.133; Cr- 0.627; Si- 2.205 P ^x -0.015)	4.5	10.0	21.5	30.3	9.9
3. Cu-Al-FCC-75, (45-50-5.0)(Cu-45; Al-50; Fe-0.255; Cr- 1.045; Si- 3.675 P ^x -0.025)	4.2	9.0	17.2	24.5	8.0
4. Cu-Al-FCC-75, (43-50-7.0)(Cu-43; Al-50; Fe-0.357; Cr- 1.463; Si- 5.145 P ^x -0.035)	3.7	6.7	17.0	19.4	6.3
5. Cu-Al-FCC-75, (40-50-10.0)(Cu-40; Al-50; Fe-0.51; Cr- 2.09; Si- 7.35 P ^x -0.05)	2.5	6.0	12.3	17.0	5.5
6. Prototype Cu-Al=50-50	0.0	1.8	6.0	10.5	3.4

P^x- compounds (C, P, S).

According to the results of the table, it was determined that the catalyst in the mixture of 50% aluminum and 45% copper had the highest catalytic activity. The amount of additional elements in the composition of this alloy increased the structural stability of the catalyst and increased the ability to adsorb hydrogen. In the hydrogenation process, when the aluminum content in the catalyst was 50%, it was observed that the yield of hydrogen peroxide reached 26.4-30.3%. These indicators showed that the catalysts in the mixture of copper and aluminum have a high hydrogen-absorbing capacity and provide complete hydrogenation of anthraquinone into anthrohydroquinone during the reaction. Catalysts enriched with additional elements (Cr, Fe, Si) showed productivity 1.6-1.9 times higher than the prototype.

Figure 2 - Dependence of the anthraquinone hydrogenation rate on the NaOH concentration in the Cu-Al-FCC-75 alloy catalyst.



It is evident from Fig. 1 that the concentration of NaOH has a significant effect on the activity of the catalyst. A NaOH concentration of 15-25% is an effective way to achieve the highest activity of the catalyst, significantly increasing the rate of hydrogenation.

Figure 3 shows the temperature dependence of the anthraquinone hydrogenation rate of the Cu-Al-FCC-75 alloy catalyst obtained during the experiment.

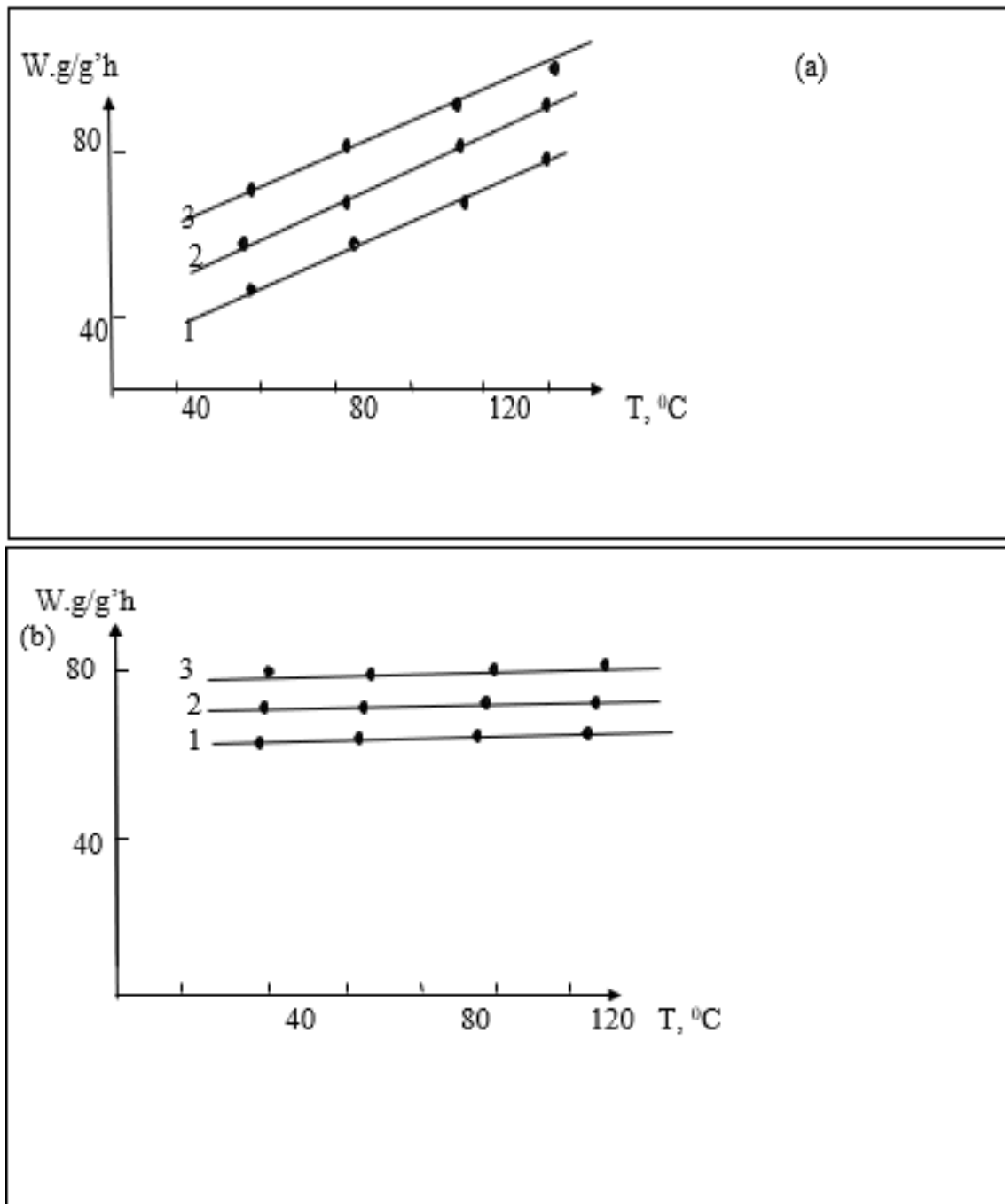


Figure 3 – Temperature dependence of the anthraquinone hydrogenation rate of the catalyst made of Cu-Al-FCC-75 alloy (a-6.0 MPa, b-10.0 MPa)

According to Figure 3, the Cu-Al-FCC-75 catalyst shows the highest hydrogenation rate at

temperatures from 40°C to 120°C, which proves its efficiency and stability. Upon reaching a temperature of 100°C, the activity of all catalysts reaches its maximum level and is maintained when the temperature increases to 120°C.

Conclusion. The duration of the catalyst leaching process, and the temperature regime increased the number of active sites on the catalyst, leading to an increase in the hydrogen peroxide formation rate. Experimental data showed that optimizing the amounts of chromium and silicon in the catalyst significantly improved its efficiency. The optimal temperature for the hydrogenation process was approximately 100°C, at which the catalyst demonstrated maximum activity. The hydrogenation rate of the Cu-Al-FCC-75 catalyst reached a maximum of 80 g/g/h at 100°C. Thus, the copper-aluminum alloy catalyst exhibited high activity and selectivity in the anthraquinone hydrogenation process, making it an effective solution for the industrial production of hydrogen peroxide.

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АНТРАХИНОНДЫ ГИДРЛЕУДЕГІ МЫС ҚОРЫТПАЛЫ КАТАЛИЗАТОРЛАР: ТЕХНОЛОГИЯЛЫҚ ЗЕРТТЕУ

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Андатпа. Антрахинонды гидрлеу маңызды өнеркәсіптік тотықтырғыш агент болып табылатын сутегі асқын тотығын синтездеудегі маңызды қадам болып табылады. Дәстүрлі түрде бұл реакция үшін никель және палладий сияқты қатты асыл метал катализаторлары пайдаланылды, бірақ мыс қорытпалары сияқты балама катализаторларды зерттеу құны мен қолжетімділігі, сондай-ақ каталитикалық қасиеттерді реттеу мүмкіндігі бойынша ықтимал артықшылықтарды ұсынады. Мыс қорытпасының катализаторы арқылы антрахинонды гидрлеуді зерттеуді таңдау қымбат металдар катализаторларымен байланысты проблемаларды, соның ішінде құны мен тапшылығын жеңу қажеттілігінен туындады. Бұл зерттеу мыс-алюминий қорытпасының катализаторы арқылы антрахинонды гидрлеуге бағытталған. Катализатор мыс пен алюминийді хром, темір және кремний сияқты элементтерді қосу арқылы легирлеу арқылы дайындалды. Зерттеудің мақсаты сутегі асқын тотығын өнеркәсіптік өндіру үшін маңызды болып табылатын гидрлеу процесінде каталитикалық белсенділікті және селективтілікті жақсарту болды. Катализатор 20% NaOH-пен өңделді, бұл белсенді орталықтардың ашылуына ықпал етті және сутегі десорбциясының жоғарылауына әкелді. Нәтижесінде 50% алюминий және 45% мыс бар катализатор 60°C және 1 МПа температурада сутегі асқын тотығының 30,3% дейін шығымына қол жеткізіп, ең жоғары каталитикалық тиімділікті көрсетті. Құрамында хром, темір және кремний бар катализаторлар тәжірибелік үлгімен салыстырғанда өнімділіктің 1,6-1,9 есе артқанын көрсетті. Оңтайлы өнімділік 100°C температурада байқалды, онда гидрлеу жылдамдығы ең жоғары деңгейге жетті. Алынған нәтижелер Cu-Al-ФСХ-75 қорытпасы антрахинонды гидрлеу үшін өте тиімді, жоғары белсенділік пен селективтілікті қамтамасыз етеді.

Түйін сөздер: антрахинон, сутегі асқын тотығы, гидрлеу, органикалық синтез, катализатор.

КАТАЛИЗАТОРЫ НА ОСНОВЕ МЕДНОГО СПЛАВА В ПРОЦЕССЕ ГИДРИРОВАНИЯ АНТРАХИНОНА: ТЕХНОЛОГИЧЕСКОЕ ИССЛЕДОВАНИЕ

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Аннотация. Гидрирование антрахинона является критическим этапом в синтезе перекиси водорода, важного промышленного окислителя. Традиционно для этой реакции использовались твердые катализаторы из благородных металлов, такие как никель и палладий, но исследование альтернативных катализаторов, таких как медные сплавы, предлагает потенциальные преимущества в стоимости и доступности, а также возможности настройки каталитических свойств. Выбор изучения гидрирования антрахинона с использованием катализатора из медного сплава был обусловлен необходимостью преодоления проблем, связанных с катализаторами из благородных металлов, включая стоимость и дефицит. В этом исследовании основное внимание уделяется гидрированию антрахинона с использованием катализатора из сплава меди и алюминия. Катализатор был приготовлен путем сплавления меди и алюминия с добавлением таких элементов, как хром, железо и кремний. Целью было улучшение каталитической активности и селективности в процессе гидрирования, что имеет решающее значение для промышленного производства перекиси водорода. Катализатор обрабатывали 20% NaOH, что способствовало раскрытию активных центров, что приводило к усилению десорбции водорода. Результаты показали, что катализатор с 50% алюминия и 45% меди продемонстрировал наивысшую каталитическую эффективность, достигнув выхода перекиси водорода до 30,3% при 60°C и давлении 1 МПа. Катализаторы, содержащие хром, железо и кремний, показали увеличение производительности в 1,6-1,9 раза по сравнению с прототипом. Оптимальная производительность наблюдалась при температуре 100°C, где скорость гидрирования достигала своего пика. Полученные результаты свидетельствуют о том, что сплав Cu-Al-ФСХ-75 весьма эффективен для гидрирования антрахинона, обеспечивая высокую активность и селективность.

Ключевые слова: антрахинон, перекись водорода, гидрирование, органический синтез, катализатор.