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THE SYNTHESIS, STUDY OF PHYSICOCHEMICAL PROPERTIES, AND EVALUATION OF CATALYTIC ACTIVITY OF A THREE-WAY CATALYST MODIFIER CONTAINING EUROPIUM (III) OXIDE**S.I. Niftaliev¹, F.M. Chiragov², I.V. Kuznetsova¹, A.D. Nikiforova¹, D.S. Sugatov¹,
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Modification of the applied Pt-containing catalysts with rare-earth metals is one of effective ways to increase their activity and resistance to thermal deactivation, which is associated with both an increase in thermal stability of the texture and structure of the carrier material and an increase in the degree of dispersion and resistance to sintering of the applied platinum metals (PM) when exposed to high temperatures. Conflicting data make it difficult to select a suitable modifier for the use as part of a three-way catalyst in the exhaust treatment system of gasoline-powered cars, whose function is to simultaneously convert CO, C_xH_y and NO_x. In this work, the effect of europium-modified Pt-containing γ -Al₂O₃-based catalyst on its physicochemical properties and activity in the oxidation of CO, C_xH_y, and NO_x reduction processes was evaluated. The catalytic activity was evaluated by passing a gas mixture simulating the exhaust of a gasoline-powered car through the catalyst applied onto the surface of a cylindrical honeycomb block with simultaneous measurement of the degree of conversion of toxic components into relatively non-toxic CO₂, N₂ and H₂O in the temperature range from 100⁰C to 400⁰C. The modification of the Pt/ γ -Al₂O₃ catalytic system with europium was shown to enhance the activity in all three reactions studied, indicating a great potential for the use as a modifier of a three-way catalyst.

Keywords: *three-way catalyst, alumina, europium oxide, platinum, conversion, catalytic activity.*

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Introduction

In the next decade, the total automobile production will exceed 1 billion units and 75% of them will be equipped with internal combustion engines (ICE), which must necessarily be fitted with an exhaust treatment system. The development of catalytic technologies for treatment of vehicle exhaust gases is stimulated by both tightening of environmental standards and ICE improvement. For example, the European standards for the dynamics of the introduction of emission requirements for vehicles have gone from Euro-1 to Euro-6. Euro-7 standards in Europe and their analogs in many countries should be introduced by 2025 [1].

A three-way catalyst contributes to the simultaneous conversion of CO, C_xH_y and NO_x into CO₂, N₂ and H₂O. Traditionally, it consists

of a ceramic or metal block of a cellular structure, on the inner surface of which a thin layer of catalytically active coating containing oxide carriers, PM (Pt, Pd and Rh) and modifiers is applied [2, 3].

Stringent requirements for the amount of harmful emissions and catalyst resources are a driving force to improve their efficiency. For example, placing the catalyst closer to the engine solves the “cold start” problem by accelerating the temperature rise required for catalyst operation. At the same time, this step leads to a significant increase in the operating temperature limit of the catalyst, which often leads to its rapid thermal deactivation under extreme engine operating conditions. One of the effective ways to increase the catalyst resistance to thermal deactivation is its modification with rare

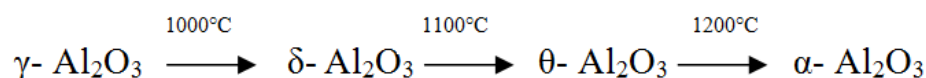
earth metals (REMs) [4, 5]. The high temperature treatment retains a sufficiently high specific surface of the carrier, which allows obtaining highly dispersed catalysts. The oxides of rare earth elements introduced into the catalyst also interact with the applied PM, which prevents sintering of the active component during prolonged overheating.

CO conversion on the Pt/ γ -Al₂O₃ catalyst has been sufficiently studied [6–8]. The kinetics of the heterogeneous CO oxidation reaction is characterized by a complex dynamic behavior associated with the occurrence of critical phenomena, the effect of temperature hysteresis.

Rhodium is more efficient in NO_x reduction and CO oxidation. Under stoichiometric conditions, Rh is less active than Pt and Pt-Rh catalyst [9], but is better at reducing NO in the presence of O₂ [10] than Pt or Pd. Rhodium is particularly good at promoting the water shift reaction [11] and is thermally stable. Special role in the reduction of NO is attributed to Rh, on which adsorption of NO occurs, followed by dissociation to atomic N and O. Besides, Rh is more selective and decomposes NO to N₂ without the formation of N₂O or NH₃ [12]. Noble metals are most often used in a combination of Pd/Rh or Pt/Rh in various ratios. Pt/Pd/Rh system is also used in three-way catalysts. The choice of the combination and metal ratio greatly depends on the commercial characteristics of the metals.

The main causes of catalyst deactivation are phase transitions at high temperature (1000°C), blocking of active centers, and substrate erosion. At 1150°C, the irreversible transition of γ -Al₂O₃ into the alpha modification occurs, which leads to a sharp decrease in the specific surface area and clogging of the pores. This can be prevented by adding rare earth oxides: CeO₂ and ZrO₂ [13, 14].

For γ -Al₂O₃, phase transitions can be presented schematically.



Phase transitions are accompanied by a decrease in the specific surface of Al₂O₃ [15]. A decrease in the specific surface of Al₂O₃ in turn leads to a decrease in the surface of the active component dispersed on the Al₂O₃ surface [16, 17]. Sintering of Al₂O₃ and reduction of the phase transition temperature are significantly affected by the presence of water vapor in the gas atmosphere, which gives an additional decrease in the specific surface by 25–50 % [18–20]. In this case, sintering proceeds through the hydroxylation stage of Al₂O₃ surface and is accelerated in the presence of water vapor [21].

Modification of Al₂O₃ is the most common and effective way to improve its thermal stability. It shifts the transformation of aluminas by more than 100°C compared to pure Al₂O₃ [22].

There is an extensive literature on the studies of the effect of modifiers on the thermal stability of Al₂O₃. In [17, 23], the mechanisms of Al₂O₃ stabilization during doping as a result of ABO₃ perovskite structure formation are outlined. The mechanisms of the effect of water vapor through surface hydroxylation (interaction of -OH groups with the Al₂O₃ surface) are described [14, 24].

It was found that the specific surface of the calcined samples at 1200°C increases with increasing ionic radius of dopant within each of the studied subgroups of alkaline earth elements and rare earth elements [25]. For a number of alkaline earth elements the specific surface increases in the series Ba²⁺ > Sr²⁺ > Ca²⁺. For some rare earth elements the specific surface increases in the series La³⁺ > Pr³⁺ > Sm³⁺ > Yb³⁺ > Ce⁴⁺.

The authors [13] propose using the CeO₂ – ZrO₂ system because cerium has a variable degree of oxidation (+3, +4) and can give extra oxygen or retain it in the oxidation reactions of exhaust gases.

In addition to cerium, other rare earth elements with variable oxidation degree can be used. As an alternative, we suggest europium compounds, which are characterized by oxidation degrees +2 and +3. The results of research into the systems of such composition are not presented in open scientific sources. Therefore, the study of physicochemical characteristics and catalytic activity of oxide systems of this composition is of great interest.

The purpose of this work is to synthesize, study physicochemical properties and evaluate the catalytic activity of the new composition Pt,Eu/Al₂O₃ in the conversion of gas mixture simulating exhaust system of a gasoline-powered vehicle.

Experimental part

Synthesis

Initial materials for sample preparation were γ -alumina, a solution of hexahydroxoplatinic acid in monoethanolamine provided by OOO "Ecoalliance" (the town of Novouralsk), and a solution of europium nitrate. The synthesis of the catalyst samples included several stages. A suspension of γ -Al₂O₃ was prepared, followed by grinding in a bead mill to a particle size according to the parameter $D_{90} = 7 \mu\text{m}$. The resulting suspension was divided into two equal parts, into one of which a solution of europium nitrate was added. Then a solution containing platinum ions was added to both suspensions. The Pt content in both samples was 0.3 wt%. The Eu₂O₃ content in Pt,Eu/Al₂O₃ sample was 4.94 wt %. The obtained suspensions were used for application onto a cordierite honeycomb carrier (25.4 mm in diameter and 101.6 mm in length, 63cell/cm²) by the aspiration method [26]. The solid content per block was 120 g/dm³, which corresponds to a PM loading of 10 g/ft³. The model block catalysts (MBC) obtained were then dried at 150⁰C and calcined at 500⁰C in air.

The suspensions were also used to prepare the coatings by forming a thin layer on the polymer substrate, followed by drying at 150⁰C for 4 h. The obtained coatings were scraped and ground in a mortar to obtain powder samples of

the catalysts for evaluating the parameters of surface, porosity and structure.

Characterization

The surface and porosity parameters of the powder samples were determined by the low-temperature nitrogen adsorption/desorption method. The measurement was carried out on the ASAP - 2400 device from Micrometrics (USA). Sample preparation included degassing in dynamic vacuum when heated to 350⁰C, followed by cooling to room temperature and filling the measuring cell with helium to atmospheric pressure. Measurement of isotherms was performed by volumetric method by periodic dosing of gaseous nitrogen into the cell with the sample. The specific surface area (S_{specific} , m²/g) was calculated by the BET method. Total volume and average pore diameter were derived from the amount of gas adsorbed at a relative pressure close to unity, assuming that the pores at this time are filled with liquid adsorbate.

The powder samples were studied by X-ray phase analysis with a diffractometer URD-6 in CuK α -radiation ($\lambda=1.5418 \text{ \AA}$). Imaging was carried out by scanning by points in the interval of angles 2θ from 20 to 800 with scanning step $\tau=0.050$ and accumulation time 10s at each point. X-ray diffraction patterns were processed using Origin software. The determination of crystal lattice parameters (CLP) and the size of the coherent scattering region (CSR) were calculated by the Williamson-Hall method using the Scherer formula.

Catalytic activity

Using a motorless gas analyzer Horiba CTSJ.2003.12, the temperature at the start of the operation of model block catalyst samples (temperature of 50 % conversion or "ignition temperature" – "Light-off" test) and maximum conversion in a pulsating mode at a temperature of 400⁰C ("Perturbation" test) were determined. The composition of the working gas mixture is given in Table 1.

The gas mixture flow rate was 60 dm³/min when a 101.6 mm long model block catalyst was examined. Heating was performed from 100 to 400⁰C at a rate of 20⁰C/min, followed by exposure at 400⁰C for 3 min.

Table 1. Composition of the working gas mixture in the test to determine the characteristics of catalytic activity

Component	Concentration, volume fractions
O ₂	1.05 %
O _{2p} (pulsating flow)	1.15%
CO	1.6 %
H ₂	0.4%
NO	0.1 %
C ₃ H ₆	0.025%
C ₃ H ₈	0.025%
CO ₂	14.0 %
H ₂ O	10.0 %
N ₂	Remainder

Results and discussion

The surface and porosity parameters

Before measuring the surface and porosity parameters, the powder samples of the catalysts were calcined at 1000 and 1100°C for 4 h. The results of surface and porosity parameter measurements are shown in Table 2.

In the state after calcination at 1000°C for 4 h, a great difference in surface and porosity parameters between the samples was not observed. However, after 1100°C, a sharp decrease in the specific surface and pore volume for the initial γ -Al₂O₃ to 18.1 m²/g was observed. The pore diameter increased significantly, indicating active sintering of the material. The introduction of platinum reduced the degree of surface degradation and porosity, which was also evidenced by a less pronounced increase in the value of the average pore diameter.

It was found that the modification of alumina-based Pt-containing catalyst with europium oxide contributes to maintaining high values of specific surface and porosity. It is note-

worthy that the pore diameter practically does not change, which indicates high resistance of the catalytic system to sintering processes.

X-ray diffraction

The results of the structure analysis by the X-ray phase analysis are presented in Table 3.

It was shown that after calcination at 1000°C in the samples only alumina θ -phase was detected. The CSR size for the sample modified with europium was the smallest. With increasing calcination temperature up to 1100°C for the initial alumina and catalytic Pt/Al₂O₃ system, the formation of α -Al₂O₃ was observed, which was accompanied by its active sintering, as evidenced by the high CSR value of this phase. Besides, as shown earlier, this was accompanied by pore collapse and a sharp decrease in the value of specific surface. The modification with europium prevents the rapid formation of α -Al₂O₃ at this calcination temperature, which is probably due to the formation of the EuAlO₃ phase, which undoubtedly contributes to maintaining a high specific surface value and, as a consequence, the catalyst resistance to thermal deactivation.

Catalytic activity

The catalytic activity was measured for freshly prepared samples of model block catalysts and hydrothermal aging in moist nitrogen atmosphere (10% H₂O + 90% N₂) at 1050°C.

Figure 1 shows the temperature dependencies of the degree of CO, C_xH_y and NO_x transformation for the samples of model block catalysts in a freshly prepared state.

The behavior of the samples in the fresh state in all three processes is different.

Table 2. Results of surface and porosity parameter measurements after calcination at 1000°C and 1100°C during 4 h

Sample	1000°C/4h			1100°C/4h		
	S _{specif, cm²/g}	V _{por, cm³/g}	D _{por, Å}	S _{specif, cm²/g}	V _{por, cm³/g}	D _{por, Å}
γ -Al ₂ O ₃	113	0.434	110	18.1	0.208	302
Pt/ γ -Al ₂ O ₃	118	0.458	111	45.0	0.280	252
Eu,Pt/Al ₂ O ₃	114	0.433	109	83.0	0.361	121

Table 3. Structure parameters of powder catalyst samples

Sample	1000°C/4h		1100°C/4h	
	CSR size, Å		CSR size, Å	
	θ -Al ₂ O ₃	α -Al ₂ O ₃	θ -Al ₂ O ₃	α -Al ₂ O ₃
γ -Al ₂ O ₃	37	–	88	427
Pt/ γ -Al ₂ O ₃	30	–	70	220
Pt, Eu/Al ₂ O ₃	27	–	58	–

The sample modified with europium is characterized by higher activity as evidenced by the shift of the curve in the area of lower temperature values.

Table 4 summarizes the key catalytic activity parameters obtained from the temperature-dependent conversion degree curves for the samples in the fresh state and after hydrothermal aging.

It can be seen that in the freshly prepared state, the sample modified with europium is characterized by higher catalytic activity, as evidenced by lower 50% conversion temperatures and higher degree of conversion at 400°C. However, after the hydrothermal aging (HTA) procedure, the T_{50} values of CO and hydrocar-

bon oxidation as well as the maximum CO conversion for the europium-modified sample were lower but the difference was insignificant. The observed decrease in the activity in the oxidation processes may be related to the peculiarities of the interaction of europium with the carrier phase and the active component during aging in moist nitrogen atmosphere. It is noteworthy that the maximum conversion of hydrocarbons and NO_x is higher with a significant difference in the reduction of nitrogen oxides.

For a more detailed analysis of the behavior of the samples during the conversion of nitrogen oxides, Figure 2 shows the dependence of the degree of transformation on the temperature in the section from 200 to 380°C.

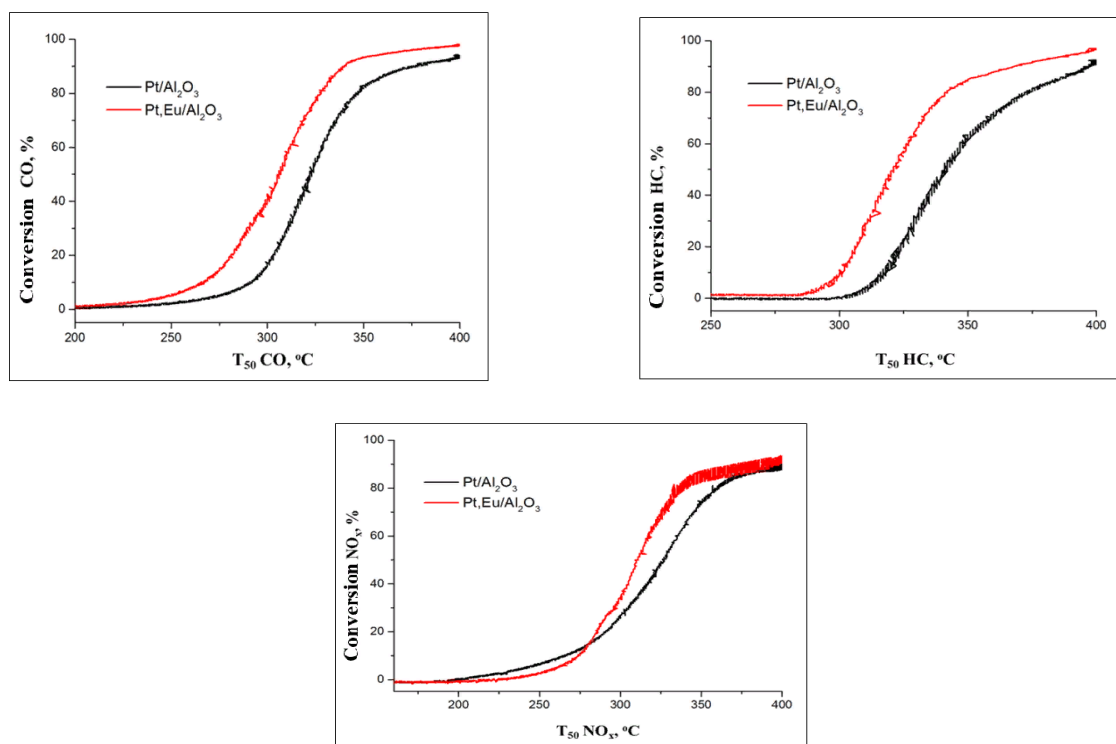


Fig. 1. Dependence of conversion degree on temperature for the samples of model block catalysts $\text{Pt}/\text{Al}_2\text{O}_3$ and $\text{Pt,Eu}/\text{Al}_2\text{O}_3$ in the fresh state in the reactions: a) CO oxidation; b) hydrocarbon reduction; c) NO_x reduction.

Table 4. Parameters of catalytic activity in the model gas mixture of MBC samples in the fresh state and after hydrothermal aging at 1050°C for 4h

Sample	Aging	Temperature of the 50% conversion, °C			Conversion at 400°C, %		
		CH_x	CO	NO_x	CH_x	CO	NO_x
$\text{Pt}/\text{Al}_2\text{O}_3$	Freshly prepared	341	322	326	91.61	93.54	90.34
$\text{Pt,Eu}/\text{Al}_2\text{O}_3$		321	306	311	96.54	98.01	93.03
$\text{Pt}/\text{Al}_2\text{O}_3$	2% O_2 +10% H_2O +88% N_2 at 1050°C for 4h	394	384	396	71.55	82.82	62.23
$\text{Pt,Eu}/\text{Al}_2\text{O}_3$		395	391	396	74.19	80.57	70.18

Composition of the working gas mixture:
 O_2 – 1.05±0.10%, CO – 1.6%, H_2 – 0.4%, NO – 0.1%, C_3H_6 – 0.025%,

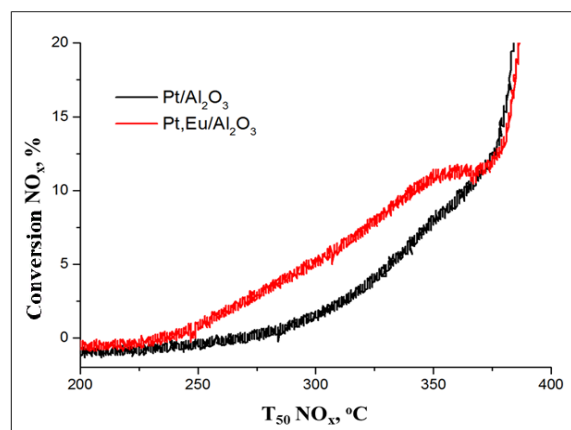


Fig. 2. Temperature dependence of NO_x transformation rate for the samples after HTA at 1050°C for 4 h.

The europium-modified sample is characterized by higher conversion values, which further indicates the positive effect of europium in the deNO_x process.

For the initial carrier material, the formation of alumina alpha phase was observed after calcination at 1100°C , which was accompanied by pore collapse and a sharp decrease in the specific surface value. For the sample containing platinum, the processes of recrystallization and textural degradation were less pronounced. The modification of the catalytic system with europium prevents the formation and active sintering of alumina alpha phase, probably due to the formation of europium aluminate phase. This in turn contributes to retaining a high value of the specific surface area and pore volume.

In the freshly prepared state, the samples of model block catalysts containing europium showed the greatest activity in all three studied processes. After hydrothermal aging in moist nitrogen atmosphere, the sample modified with europium appeared to be slightly worse in the CO and hydrocarbons oxidation but significantly better in the processes of nitrogen reduction.

Thus, the modification of the Pt-containing alumina-based catalyst with europium enhances the stability of the structure as well as surface parameters and porosity of the catalytic system to high temperatures up to 1100°C . Also the activity of model block catalysts in the fresh state increases significantly and there occurs a more effective conversion of

nitrogen oxides after hydrothermal aging at 1050°C for 4 h in moist nitrogen atmosphere.

In subsequent works, it would be interesting to study the role of Eu in NO_x conversion processes in three-way catalysts.

Conclusions

Suspensions containing $\gamma\text{-Al}_2\text{O}_3$, europium and platinum salts were applied to the cordierite carrier using the aspiration method, and then dried at 150°C and calcined at 500, 1000, 1100°C . At 1100°C , a sharp decrease in specific surface, pore volume and an increase in pore diameter were observed for the initial $\gamma\text{-Al}_2\text{O}_3$. The samples containing europium and platinum retained high values of specific surface and porosity up to 1100°C , which indicates high resistance of the catalytic system to sintering processes.

According to X-ray phase analysis data, after calcination at 1000°C in catalytic system $\text{Pt}/\text{Al}_2\text{O}_3$, the formation of $\alpha\text{-Al}_2\text{O}_3$ is observed, which is accompanied by its active sintering. The modification with europium prevents the formation of $\alpha\text{-Al}_2\text{O}_3$ at this calcination temperature, which contributes to retaining high values of the specific surface and, as a consequence, the catalyst resistance to thermal deactivation.

The activity of model block catalysts containing europium ($\text{Pt,Eu}/\text{Al}_2\text{O}_3$) in the fresh state significantly increases and a more efficient conversion of nitrogen oxides after hydrothermal aging at 1050°C compared to the NO_x conversion in the $\text{Pt}/\text{Al}_2\text{O}_3$ system occurs.

In the freshly prepared state, the sample modified with europium was characterized by

higher catalytic activity, as evidenced by lower 50% conversion temperatures and higher degree of conversion at 400°C. It was found that the europium-modified sample has a higher maximum conversion of hydrocarbons and NO_x than the Pt/Al₂O₃ sample, with a significant difference in the reduction of nitrogen oxides. After hydrothermal aging, the maximum CO conversion for the Pt,Eu/Al₂O₃ sample was slightly lower than that for the Pt/Al₂O₃ sample (80.57 and 82.82%, respectively), but this difference is insignificant.

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**TƏRKİBİNDƏ AVROPIUM (III) OKSİDİ OLAN ÜÇTƏRƏFLİ KATALİZATOR
MODİFİKATORUNUN SİNTEZİ, FİZİKİ-KİMYƏVİ XASSƏLƏRİNİN ÖYRƏNİLMƏSİ VƏ
KATALİTİK AKTİVLİYİNİN QIYMƏTLƏNDİRİLMƏSİ**

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Tətbiq olunan Pt tərkibli katalizatorların nadir torpaq metalları ilə modifikasiyası onların aktivliyini və termal deaktivasiyaya qarşı müqavimətini artırmaq üçün effektiv üsullardan biridir ki, bu da həm daşıyıcı materialın strukturunun və fakturasının istilik dayanıqlığının artması, həm də yüksək temperaturlara məruz qaldıqda tətbiq olunan platin metallarının (PM) dispersiya və sinterləşməyə qarşı müqavimət dərəcəsinin artması ilə əlaqələndirilir. Bir-birinə zidd olan məlumatlar, funksiyası CO, C_xH_y və NO_x-i eyni vaxtda çevirmək olan benzinlə işləyən avtomobillərin işlənmiş qazlarının təmizlənməsi sistemində üçtərəfli katalizatorun bir hissəsi kimi istifadə üçün uyğun modifikatorun seçilməsini çətinləşdirir. Bu işdə europiumla modifikasiya olunmuş Pt tərkibli γ -Al₂O₃ əsaslı katalizatorun onun fiziki-kimyəvi xassələrinə və CO, C_xH_y və NO_x reduksiya proseslərinin oksidləşməsində aktivliyinə təsiri qiymətləndirilmişdir. Zəhərli komponentlərin nisbətən toksik olmayan CO₂, N₂ və H₂O-ya 100⁰C ilə 400⁰C arasında olan temperatur aralığında çevirmə dərəcəsinin eyni vaxtda ölçülməsi ilə silindrik pətək blokunun səthinə tətbiq olunan katalizatorun benzinlə işləyən avtomobilin işlənmiş qazını simulyasiya edən qaz qarışığının keçirilməsi ilə katalitik aktivlik qiymətləndirilmişdir. Pt/ γ -Al₂O₃ katalitik sisteminin europium ilə modifikasiyası tədqiq edilmiş hər üç reaksiyada aktivliyin arttığı göstərildi ki, bu da üçtərəfli katalizatorun modifikatoru kimi istifadə üçün böyük potensialın olduğunu göstərir.

Açar sözlər: üçtərəfli katalizator, alüminium oksidi, avropium oksidi, platin, konversiya, katalitik aktivlik.