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Catalytic Systems Based on Carbon Supports for the Low-Temperature Oxidation of Carbon Monoxide

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Abstract—Catalytic systems containing palladium, copper, and iron compounds on carbon supports—kernel activated carbon and fibrous carbon materials (Karbopon and Busofit)—for the low-temperature oxidation of CO were synthesized. The effects of the nature of the support, the concentration and composition of the active component, and the conditions of preparation on the efficiency of the catalytic system were studied. The catalytic system based on Karbopon exhibited the highest activity: the conversion of carbon monoxide was 90% at room temperature and a reaction mixture (0.03% CO in air) space velocity of 10000 h^{-1}. It was found that the metals occurred in oxidized states in the course of operation: palladium mainly occurred as Pd^{+}, whereas copper and iron occurred as Cu^{2+} and Fe^{3+}, respectively.

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INTRODUCTION

The development of low-temperature and stable catalytic systems for the oxidation of carbon monoxide is a direct way to solving the problem of the design of efficient breathing masks for protecting respiratory apparatus from carbon monoxide.

Hopcalites are well-known catalysts for CO oxidation, which are highly active at low temperatures [1, 2]. However, their considerable disadvantage consists in rapid deactivation with water vapor. Because of this, they cannot be used as a working element in breathing masks. In this context, the ability of aqueous solutions of platinum group metal chlorides in the presence of transition metal (Cu, Fe, etc.) salts to oxidize CO at room temperature is of interest [3, 4]. The heterogenization of these systems can significantly extend the range of their practical applications and, in particular, facilitate solving the above problem.

In this case, the support for the catalytic system plays an important role because the state and operational efficiency of the active component depends on the physicochemical properties of the support.

It is our opinion that carbon materials (particularly fibrous materials), which exhibit a developed contact surface and high resistance to corrosive media [5, 6], are the best suited supports for the synthesis of multicomponent metal complex catalytic systems for CO oxidation.

Published data on the low-temperature oxidation of CO in the presence of supported metal complex systems based on carbon supports mainly concerned the $PdCl_2$–$CuCl_2$ binary catalytic system [7–10]. Choi and Vannice [7] found the high catalytic activity of the catalytic composition supported on carbon in the oxidation of CO at room temperature in the presence of water vapor.

In this paper, we describe conditions for the preparation of Pd–Cu–Fe-containing catalytic systems based on carbon materials and the results of studies on the effects of the nature of the support and the composition and concentration of an active component on the activity of the synthesized samples in the oxidation of carbon monoxide.

EXPERIMENTAL

Kernel activated carbon (KAC; granule diameter, 1–2 mm; apparent density, 0.375 g/cm^3) and Karbopon and Busofit fibrous carbon materials (fiber diameter, 5–10 μm) from PO Khimvolokno (Svetlogorsk, Belarus) were used as supports for preparing catalytic systems for CO oxidation.

The support was repeatedly pretreated with a 15% aqueous HCl solution to remove mineral impurities followed by washing with distilled water to a neutral reaction. The modified forms of KAC were prepared using the following procedures:

—oxidation with a 15% aqueous H_2O_2 solution at room temperature for 4 h (KAC/H_2O_2);

—boiling in 30% nitric acid for 2 h (KAC/HNO_3);

—treatment with gaseous ammonia at 300°C for 2 h (KAC/NH_3); in this case, the support was preliminarily decarboxylated in a flow of argon at 420°C and then chlorinated with CCl_4 vapor at the specified temperature. The duration of each treatment was 1.5 h.

Table 1. Physicochemical properties of carbon supports

Support	S_{sp}, m^2/g	Water sorption, $g_l/g_{support}$	Weight loss in the temperature range 120–430°C, mg	Elemental composition, wt %				
				C	H	N	Cl	O
KAC	950	0.722	3.8	89.1	1.83	–	–	9.10
KAC/H_2O_2	820	0.904	8.3	84.1	1.86	–	–	14.04
KAC/HNO_3	480	0.808	21.3	65.5	2.75	0.55	0.59	30.20
KAC/NH_3	609	0.455	4.8	78.8	2.32	2.67	9.20	5.10
Karbopon	780	0.640	14.7	76.7	4.60	0.60	–	18.10
Busofit	670	0.170	8.8	89.6	1.80	0.50	–	8.60

After modification, the oxidized samples were washed with distilled water to a neutral reaction and dried at 120°C. The sample modified with gaseous ammonia was washed with a 10% aqueous HCl solution to remove excess ammonia and then with distilled water. Table 1 summarizes the characteristics of the carbon materials.

The catalytic systems were prepared by impregnating the supports with aqueous solutions of palladium, copper, and trivalent iron salts (chlorides, acetates, and bromides) with an initial Pd concentration of 2.5 mg/cm^3 in the impregnating solution. To prepare samples with high oxidant concentrations in the catalysts, impregnating solutions with Cu/Pd and Fe/Pd weight ratios of 15.0 and 0.8, respectively, were used. To prepare samples with low oxidant concentrations, impregnating solutions with Cu/Pd and Fe/Pd weight ratios of 5.0 and 0.4, respectively, were used. The minimum volume of an impregnating solution was 1 or 2.5 cm^3 per gram of air-dry KAC or carbon fiber, respectively; the pH of impregnating solutions was varied from 3 to 4.5. The support was kept in an impregnating solution for 0.5 h at room temperature; then, it was kept in a flow of warm air at 40°C until the removal of excess water. After impregnation, the catalyst was dried in air at 25°C for 16 h.

Some samples were prepared by supporting the active component onto a support in two stages. After the first stage of impregnation, the catalyst was treated with a reaction mixture (0.5% CO in air) at a flow rate of 125 cm^3/min at 25°C for 2 h. Then, the second portion of the active component was supported onto the sample.

The thermogravimetric analysis of the supports was performed on an MOM thermoanalytical system (Hungary) over the temperature range 20–450°C at a heating rate of 5 K/min. The sample weight was 100 mg.

The XPS data were obtained on a Kratos Axis His instrument (UK) with the use of a 169-W source of AlK_α X-ray radiation (1486.6 eV) with a 40-eV transmitted energy analyzer. The spectra were obtained at normal emission with the use of a charge neutralizer.

The sorption of water (g) was determined as follows: A weighed portion of a carbon support (~0.5 g) was placed in an excess of distilled water and allowed to stand for 3 h. Then, the samples were centrifuged in a laboratory centrifuge (4000 rpm) for 15 min. After weighing, the samples were dried at 100°C to constant weight. The sorption of water ($g_l/g_{support}$) was calculated as the weight ratio between the adsorbed liquid and the dry sample

$$g = \frac{m_1 - m_2}{m_2},$$

where m_1 is the sample weight after centrifugation and m_2 is the sample weight after drying at 100°C.

The activity of the catalytic systems in the oxidation of CO at room temperature (20°C) was determined in a glass flow reactor with a system for the fixation of the bed height of fibrous catalysts (the loaded fibrous sample volume was constant, and it amounted to 12 cm^3).

The concentrations of CO at the reactor inlet and outlet were determined by gas chromatography on a 2.5-m column packed with AG-2 activated carbon at a carrier gas (air) flow rate of 100 cm^3/min. The conversion of CO was calculated from the difference between CO concentrations in gas samples of equal volumes taken upstream and downstream of the reactor.

The activity of the catalysts was also characterized by the reactor capacity per unit weight (1 g) of palladium and calculated from the equation

$$A = V_{CO}\alpha/m_{Pd}\ \text{cm}^3\ (\text{g Pd})^{-1}\ \text{s}^{-1},$$

where V_{CO} is the flow rate of CO (cm^3/s) and α is the conversion of CO (volume fraction).

RESULTS AND DISCUSSION

Effect of the Nature of the Support

The data given in Table 1 indicate that the specific surface areas of KAC and its modified forms varied over the range 950–480 m^2/g; the carbon fibers were characterized by the values of 780 and 670 m^2/g for Karbopon and Busofit, respectively. After oxidation treatments, the water sorption capacity of KAC increased. At the same time, the sorption of water by carbon considerably decreased (by a factor of ~1.5)

after its treatment with CCl_4 and NH_3. The Karbopon carbon fiber and KAC exhibited similar adsorption parameters with respect to water. The Busofit carbon fiber was characterized by the lowest sorption of water.

The oxidative treatment of KAC increased the oxygen contents of the samples. For example, after treatment with 15% H_2O_2 and 30% HNO_3, the oxygen content of the sample increased by a factor of ~1.5 and more than 3, respectively. At the same time, after the treatment of KAC with CCl_4 vapor and NH_3 gas, the oxygen content of KAC decreased. The increased concentration of chlorine in KAC/NH_3 suggests a rather high degree of chlorination of the support surface upon its treatment with CCl_4 vapor, whereas it is likely that the appearance of nitrogen (2.7%) in the sample was due to the formation of amino groups on the support surface on the treatment of chlorinated KAC with gaseous ammonia [11]. As for carbon fibers, note that the oxygen content of Karbopon was higher (by a factor of 2) than that of Busofit.

The greatest weight losses over the range 120–430°C were observed in KAC/HNO_3 and Karbopon samples (21.3 and 14.7%, respectively), which were characterized by the highest oxygen contents. Obviously, the weight loss in the specified temperature range was mainly due to the decomposition of carboxyl groups [6]. The parent KAC and KAC/NH_3, which exhibited weight losses of 3.8 and 4.8%, respectively, were found to be most stable on heating in air. The samples of KAC/H_2O_2 and Busofit occupied an intermediate position.

Figure 1 shows the dependence of CO conversion on the duration of the experiment for catalyst samples containing 0.35% Pd, 5.25% Cu, and 0.28% Fe on KAC and its modified forms. At a 0.5 vol % concentration of CO in air and a reaction mixture space velocity of 1120 h^{-1} in the presence of a catalyst based on KAC, 98% conversion was reached after 30 min. The surface modification of KAC with functional groups decreased the activity of the catalytic system. This occurred to the greatest extent on KAC/HNO_3. In the presence of a catalyst based on this support, only 21% conversion of CO was reached after a 30-min experiment, whereas the conversion of CO was 76 or 45% on KAC/H_2O_2 or KAC/NH_3, respectively. It is likely that the activity of the catalytic system based on KAC/HNO_3 was low because of a high concentration of oxygen-containing groups, in particular, carboxyl groups, in the sample. The low initial activity of the catalytic system based on KAC/NH_3 may be explained by a considerable concentration of amino groups in the sample and a low water sorption by the support.

It was found that the activity of catalysts based on carbon supports decreased at high reaction mixture space velocities (9000–10000 h^{-1}) and a low concentration of CO (0.03 vol %) in air to be purified (respiratory conditions).

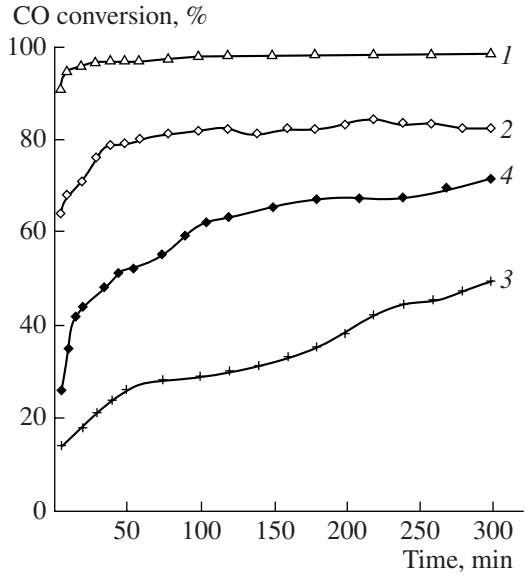

Fig. 1. Dependence of CO conversion on the duration of the experiment for catalysts based on (*1*) KAC, (*2*) KAC/H_2O_2, (*3*) KAC/HNO_3, and (*4*) KAC/NH_3. Metal contents of the catalysts (wt %): Pd, 0.35; Cu, 5.25; and Fe, 0.28. Impregnating solution, pH 4.1. Experimental conditions: 0.5 vol % CO in air; v_0 = 1120 h^{-1}; catalyst loading, 2.5 g.

A comparison between the catalytic systems based on Karbopon and Busofit samples demonstrated that, at equal concentrations of the active component in the samples, the activity of the catalyst prepared based on Busofit was lower. At a reaction mixture space velocity of 10000 h^{-1} and a CO concentration of 0.03 vol % in the mixture, a 28% conversion of CO was reached on this catalyst (Fig. 2, curve *4*). The maximum degree of CO conversion into CO_2 on a sample prepared based on Karbopon under the same conditions was 57% (curve *1*). As noted above, the sorption of water on Karbopon was much greater than that on Busofit (by a factor of more than 3.5). To demonstrate the crucial role of water in CO oxidation on Pd–Cu–Fe supported metal systems, we performed a special experiment with predrying the reaction mixture before supplying it to the catalyst by passing through series columns packed with KOH, $CaCl_2$, and SiO_2. We found that keeping the catalyst containing 1.25% Pd, 6.25% Cu, and 0.50% Fe on Karbopon in a dry reaction mixture resulted in a dramatic decrease in CO conversion (from 97 to 9%), whereas the subsequent treatment of the catalyst with a moistened reaction mixture completely restored its initial activity (Fig. 3). The experimental results suggest the participation of water in redox reactions that occur on the catalyst.

According to Rakitskaya et al. [12], the role of water molecules in redox reactions that occur with the participation of metal complexes supported onto a solid surface consists in the formation of a particular surface

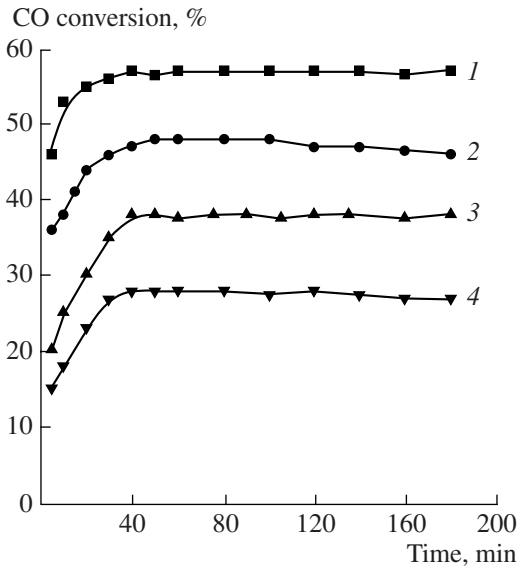

Fig. 2. Dependence of CO conversion on the duration of the experiment for catalysts based on (*1*–*3*) Karbopon and (*4*) Busofit. Metal contents of the catalysts (wt %): (*1*, *2*, and *4*) Pd, 1.25; Cu, 6.25; and Fe, 0.50 or (*3*) Pd, 1.25; Cu, 18.75; and Fe, 1.0. Impregnating solution, pH (*1*, *3*, and *4*) 3.7 or (*2*) 4.1. Experimental conditions: 0.03 vol % CO in air; v_0 = 10000 h^{-1}; catalyst loading, 1 g.

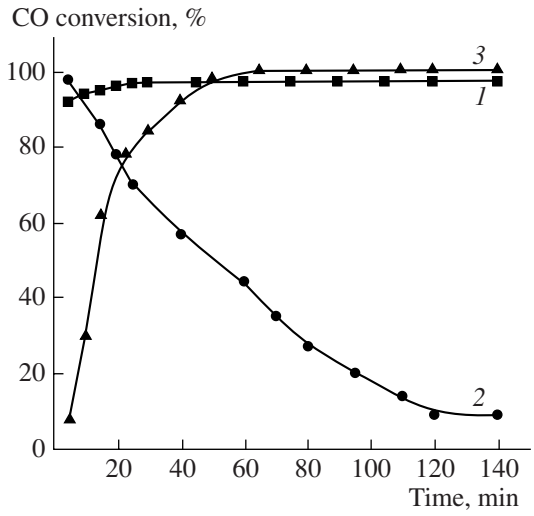

Fig. 3. Dependence of CO conversion on the duration of the experiment on the catalyst containing 1.25% Pd, 6.25% Cu, and 0.50% Fe on Karbopon (impregnating solution, pH 3.7) pretreated with (*1*) a reaction mixture with a relative air humidity of 40%, (*2*) a reaction mixture dried through series columns packed with KOH, $CaCl_2$, and SiO_2, and (*3*) a reaction mixture with a relative air humidity of 100%. Experimental conditions: 0.5 vol % CO in air; v_0 = 625 h^{-1}; catalyst loading, 1 g.

complex and a hydrate film, which is necessary for electron transfer.

However, Choi and Vannice [7] found that water acts not only as a solvent of metal-containing complexes but also as an oxidizing agent along with Pd^+.

The rate-limiting step of the low-temperature oxidation of CO in the presence of $PdCl_2$ supported on carbon is the formation of the PdClCO complex

$$2PdCl_{2(aq)} + CO + H_2O_{(ad)} = 2PdCl_{(aq)} + CO_2 + 2HCl_{(ad)};$$

$$PdCl_{(aq)} + CO \longleftrightarrow PdClCO_{(aq)}.$$

In the presence of water vapor in a flow, an additional reaction occurs with the formation of zerovalent palladium. In this case, water serves as an agent responsible for electron transfer from the CO molecule to Pd^+:

$$PdClCO_{(aq)} + H_2O_{(ad)} \longleftrightarrow Pd(0) + CO_2 + HCl_{(ad)} + 1/2H_{2(ad)}.$$

The subsequent redox processes with the participation of Cu^{2+} and Fe^{3+} salts can be represented by the following reaction schemes:

$$Pd(0) + CuCl_{2(aq)} \longleftrightarrow PdCl_{(aq)} + CuCl_{(aq)};$$

$$Pd(0) + FeCl_{3(aq)} = PdCl_{aq} + FeCl_{2(aq)};$$

$$PdCl_{(aq)} + FeCl_{3(aq)} = PdCl_{2(aq)} + FeCl_{2(aq)}.$$

The main role of oxygen is to oxidize the reduced forms of the oxidizing agents:

$$2CuCl_{(aq)} + 1/2O_2 + 2HC1 = 2CuCl_{2(aq)} + H_2O;$$

$$2FeCl_{2(aq)} + 1/2O_2 + 2HC1 = 2FeCl_{3(aq)} + H_2O.$$

XPS data indicate that, in catalysts based on KAC and Karbopon exposed to a reaction atmosphere under experimental conditions, iron and copper occurred in oxidized states: copper occurred as Cu^{2+} (E_b(Cu $2p_{3/2}$) = 934.4 eV), and iron occurred as Fe^{3+} (E_b(Fe $2p_{3/2}$) = 711.0 eV). The binding energy of Pd $3d_{5/2}$ was 337.6 eV, which occupied an intermediate position between E_b for the Pd^{2+} and Pd^+ states in the ammonia complexes of palladium $[Pd(NH_3)_4]^{2+}$ on zeolite [13]. The acetate ions (CH_3COO^-) occurring in the catalytic system can stabilize palladium in the state Pd^+; we believe that Pd^+ was present in the tested samples.

Effect of Impregnation Conditions

As follows from Fig. 4, under respiratory conditions, an increase in the pH of an impregnating solution from 3.7 to 4.1 resulted in an increase in the activity of the catalyst prepared based on the parent KAC (curves *1*, *2*). The decrease in the concentration of oxidizing agents (copper and iron compounds) did not noticeably change the activity of the catalytic system (curve *3*). At the same time, the catalyst based on Karbopon exhibited higher activity if it was prepared at pH 3.7, whereas a decrease in the concentration of oxidizing agents in the catalyst considerably increased the activity of the catalytic system (Fig. 2, curves *1*–*3*).

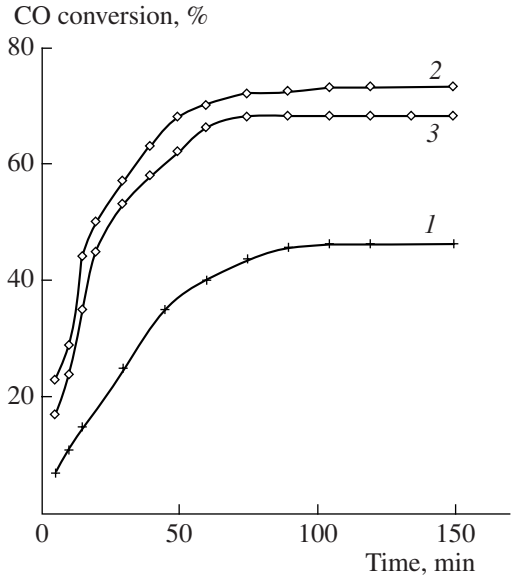

Fig. 4. Dependence of CO conversion on the duration of the experiment for catalysts based on KAC. Metal contents of the catalysts (wt %): (*1*, *2*) Pd, 0.35; Cu, 5.25; and Fe, 0.28 or (*3*) Pd, 0.35; Cu, 1.75; and Fe, 0.14. Impregnating solution, pH (*1*) 3.7 or (*2*, *3*) 4.1. Experimental conditions: 0.03 vol % CO in air; $v_0 = 9000\ h^{-1}$; catalyst loading, 5 g.

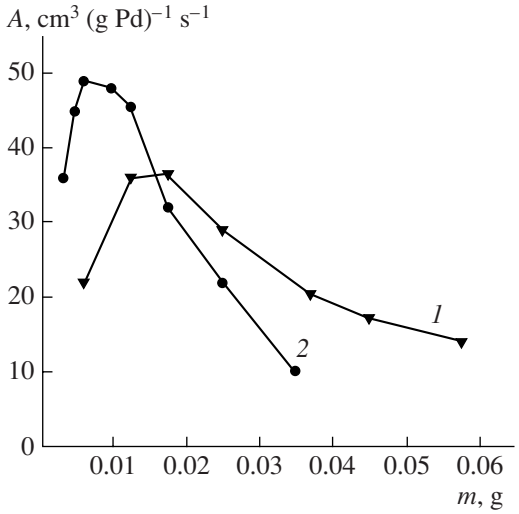

Fig. 5. Dependence of the reactor capacity (*A*) on the palladium content (*m*) of the reactor for catalysts prepared based on (*1*) KAC (pH 4.1) and (*2*) Karbopon (pH 3.7) (Cu/Pd and Fe/Pd weight ratios, 5.0 and 0.4, respectively). Experimental conditions: 0.03 vol % CO in air; $v_0 = 10000\ h^{-1}$; experiment time, 2 h; KAC- and Karbopon-based catalyst loadings, 4.5 and 1 g, respectively.

Effect of Component Concentrations

Figure 5 shows the dependence of the reactor capacity per gram of Pd on the palladium content of the reactor for catalysts based on KAC and Karbopon (Cu/Pd and Fe/Pd weight ratios of 5.0 and 0.4, respectively). As can be seen, the maximum capacity was 49 cm^3 (g Pd)$^{-1}$ s^{-1} for the catalyst based on Karbopon, and it was reached at a palladium content of 0.00625 g in the reactor, whereas this value was ~36 cm^3 (g Pd)$^{-1}$ s^{-1} for the catalyst based on KAC, and it was reached with a more than twofold increase in the active component content of the reactor.

Effect of Preparation Conditions

Figure 6 shows the activity of catalyst samples with 0.55% Pd, 2.75% Cu, and 0.22% Fe on Karbopon prepared by the single-step and two-step supporting of the active component. As can be seen, supporting the active component onto Karbopon in two steps with intermediate treatment in a reaction atmosphere resulted in a noticeable increase in the activity and stability of the catalyst. With a catalyst loading of 2 g, 90% CO conversion was reached and the reactor capacity was 81 cm^3 (g Pd)$^{-1}$ s^{-1}. The catalyst retained its activity after remaining in air for a long time (24 months).

The experimental results indicate that a change in the surface complexes of the active component occurred in the course of the catalytic experiment in the presence of CO and O_2 to improve the catalytic properties of these complexes.

According to XPS data (Table 2), the Cu/Pd and Fe/Pd weight ratios in the near-surface layer of the freshly prepared catalyst based on KAC were 17.8 and 4.3, respectively, which are much higher than the weight ratios between these metals in the catalyst, as calculated from their concentrations in the impregnating solution (5.0 and 0.4 for Cu/Pd and Fe/Pd, respec-

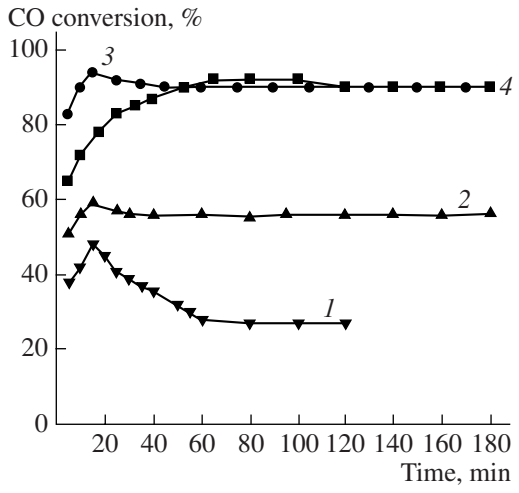

Fig. 6. Dependence of CO conversion on the duration of the experiment for catalysts containing 0.55% Pd, 2.75% Cu, and 0.22% Fe on Karbopon upon (*1*) single and (*2–4*) double supporting (impregnating solution, pH 3.7): (*1–3*) freshly prepared samples and (*4*) a catalyst kept in air for 24 months. Experimental conditions: 0.03 vol % CO in air; $v_0 = 10000\ h^{-1}$; catalyst loading, (*1*, *2*) 1 or (*3*, *4*) 2 g.

Table 2. Elemental composition of near-surface layers of catalysts containing (I) 0.3 wt % Pd, 1.5 wt % Cu, and 0.12 wt % Fe on KAC and (II) 0.6 wt % Pd, 3.0 wt % Cu, and 0.24 wt % Fe on Karbopon

Sample	Element concentration, wt %				Weight ratio		Atomic ratio		
	Pd 3*d*	Cu 2*p*	Fe 2*p*	O 1*s*	Cu/Pd	Fe/Pd	Pd/O	Cu/O	Fe/O
I freshly prepared	0.273	4.873	1.190	16.26	17.8	4.30	0.0025	0.075	0.021
I after treatment with the reaction mixture	0.383	5.183	1.290	10.87	13.3	3.32	0.0054	0.120	0.039
II freshly prepared	0.177	26.980	5.860	19.04	152	33.0	0.0014	0.360	0.088
II after treatment with the reaction mixture	0.496	16.360	10.511	22.31	33.0	21.0	0.0033	0.180	0.135

Table 3. Composition and activity of catalytic systems in CO oxidation

Catalyst no.	Catalyst composition		Catalyst activity	
	support	active component concentration (wt %)	$w \times 10^6$ mol CO (g Cat)$^{-1}$ s^{-1}	$w_{ACA} \times 10^3$ CO molecule (Pd atom)$^{-1}$ s^{-1}
1	KAC	0.30 Pd, 4.50 Cu, 0.24 Fe	1.237	42.31
2	KAC	0.30 Pd, 1.50 Cu, 0.12 Fe	0.774	25.10
3	Karbopon	0.60 Pd, 9.00 Cu, 0.48 Fe	3.255	62.10
4*	Karbopon	0.55 Pd, 2.75 Cu, 0.22 Fe	3.124	60.22
5**	Carbon	0.2 Pd, 11.5 Cu	0.446	22.70

Note: Experimental conditions: 3.5 vol % CO in air; flow rate, 60 cm^3/min; catalyst weight, 0.3 g.
* The sample was prepared by two-stage supporting.
** Activity values taken from [7].

tively). The palladium content of the near-surface layer was close to the total palladium content of the catalyst. This fact suggests that, in the course of catalyst preparation based on KAC, palladium was almost uniformly distributed over the entire volume of the granule, whereas copper and iron were concentrated in the near-surface layer (in this case, iron was concentrated to a much greater extent). The treatment of the catalyst with a reaction mixture resulted in an enrichment of the surface layer with palladium and a noticeable increase in the metal/oxygen atomic ratio. In the catalyst based on Karbopon, palladium mainly concentrated on the inner fiber surface, whereas the fraction of surface copper and iron atoms in Karbopon was much higher than that in KAC. The treatment of the catalyst with a reaction mixture also resulted in a redistribution of metals in the bulk of the fiber with a much more intense migration of palladium to the surface than that in KAC. Note that, in this case, the ratio between Pd/O and Fe/O in the near-surface layers of a freshly prepared catalyst and a catalyst treated with a reaction mixture remained almost the same for KAC and Karbopon.

Thus, the treatment of catalysts with a reaction mixture resulted in an increase in the palladium content of the near-surface layer and in the formation of a complex with an optimum composition on the surface of the carbon support. The migration processes of metals, particularly palladium, occurred more intensively on Karbopon.

In order to compare the experimental results on the activity of catalysts based on carbon supports in CO oxidation with published data, we performed special experiments under conditions analogous to those described by Choi and Vannice [7]. A catalyst (0.3 g) was loaded in the reactor, and experiments were performed at a reaction mixture (3.5 vol % CO in air) flow rate of 60 cm^3/min. Table 3 summarizes the results.

In Table 3, it can be seen that the atomic catalytic activity (the number of reacted CO molecules per palladium atom) of catalyst no. 1 was higher than the analogous characteristic of a catalyst described by Choi and Vannice [7] by a factor of ~2.

A decrease in the concentration of oxidizing agents (copper and iron salts) as active constituents supported on KAC resulted in a decrease in the conversion of CO molecules on a palladium atom by ~60%, whereas this effect was insignificant in samples based on Karbopon.

The atomic catalytic activity of the catalyst prepared by two-stage supporting of 0.55% Pd, 2.75% Cu, and 0.22% Fe on Karbopon was 60.2×10^{-3} CO molecule (Pd atom)$^{-1}$ s^{-1}, which is higher than the activity of palladium in the best catalyst on KAC or in the palladium–copper-containing catalyst based on carbon by a factor of 1.5 or 2.7, respectively.

Thus, the experimental results suggest that low-temperature catalytic systems for CO oxidation that met the requirements imposed on respiratory devices can be developed with the use of a fibrous carbon material (Karbopon) as a support and the solutions of palladium, copper, and iron salts as active components.

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