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Catalytic hydrogenation of quinoline in the presence of carbonaceous material obtained by pyrolysis of a cobalt complex with 1,2-diaminobenzene

Presented by Corresponding Member of the NAS of Ukraine K.M. Sukhyy

The carbonaceous material was isolated from the Co-C/SiO_2 hydrogenation catalyst, formed by pyrolysis of the Co^{II} complex with 1,2-diaminobenzene deposited on aerosil, by treatment with HCl and HF solutions. The material was studied by TEM and Raman spectroscopy. The specific surface area was determined on the results of the nitrogen adsorption measurements. It was shown that the obtained carbonaceous material consisted of amorphous carbon and nanotubes, which may be present in the original Co-C/SiO_2 catalyst or may form as a result of folding of thin carbonaceous sheets remaining after dissolution of Co and SiO_2. It was found that such carbonaceous material was an efficient catalyst for the hydrogenation of quinoline at a low residual Co content (about 0.5 %), which is a sign of the possible participation of the carbonaceous component of catalysts, obtained by pyrolysis of cobalt coordination compounds with organic ligands, in hydrogenation processes.

Keywords: *hydrogenation catalyst, pyrolysis, carbonaceous material, nanotubes, TEM, Raman spectroscopy, adsorption.*

Introduction. Hydrogenation plays an important role in organic chemistry and has been widely used for preparation of API (active pharmaceutical ingredients), active compounds for agricultural chemistry, food products (primarily oil hydrogenation), as well as for bulk production of solvents, components of fuel [1]. Currently, the catalysts based on platinum group metals (PGM) hold the leading positions in hydrogenation processes for fine organic chemistry [2]. Such systems

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are characterized by high activity, but the raise of prices for PGM is a serious obstacle to their extended use. The catalysts based on nickel, such as Ranay nickel, can be an alternative, however their application is restricted by relatively low activity and selectivity of such systems, as well as high sensitivity to oxygen [3]. Many nickel-based catalysts are pyrophoric and require special precautions during operation, and hydrogenation with nickel, especially on an industrial scale, is associated with a serious risk of accidents. Thus, the development of new efficient and safe hydrogenation catalysts that do not contain PGM is important task of modern physical chemistry.

It has recently been shown that cobalt-based systems have high potential of use in hydrogenation. This family of the catalysts was formed by pyrolysis of Co^{II} complexes with organic ligands, deposited on silica or alumina (hereinafter referred to as Co-C/SiO_2 or Co-C/Al_2O_3 systems) [4, 5]. Thermal treatment of the coordination compounds led to formation of the metallic nanoparticles and the carbonaceous material, which were localized on the surface of the inert carrier. The catalysts prepared by this route have high application potential due to their high performance, activity in hydrogenation of a wide range of organic compounds, stability on air, low cost and safety.

Up to date, there is no clear understanding of the role of the carbonaceous component in Co-based hydrogenation catalysts. It was assumed that there was some synergy between Co nanoparticles and carbon in hydrogenation [6]. On the other hand, no correlation was found between the content of carbon or its characteristics (such as parameters of Raman spectra) and the yield of hydrogenation product (1,2,3,4-tetrahydroquinoline in the process of quinoline hydrogenation) [7]. The latter observation gave rise to a supposition, that the role of carbonaceous layer may be limited to protection of Co metallic nanoparticles from oxidation on air [7]. Notably, the metal-free carbonaceous materials showed significant catalytic activity in hydrogenation processes [8, 9], and such activity could contribute to the overall catalytic performance of the Co-C/carrier system. In addition, irradiation of Co-C/SiO_2 catalyst by quick electrons (2.3 MeV) led to modification of the carbonaceous component, and a slight but systematic increase of the yields of hydrogenation products was observed when the irradiated Co-containing material was used as catalyst [10]. This discovery may serve an argument that the active sites of hydrogenation catalyst included, in some role, the carbonaceous material. Studies of the structure of the carbonaceous component in Co-C/carrier hydrogenation catalysts are important for understanding the reasons of their high performance.

The aim of this study was to isolate the carbonaceous component from the hydrogenation catalyst obtained by pyrolysis of Co^{II} complex with 1,2-diaminobenzene (DAB), deposited on Aerosil, as well as to determine the size of the pieces of the carbonaceous material, their spectral properties and to estimate its catalytic performance.

We expected that such carbonaceous pieces would have the shape of 2D layers, and planned to analyze their thickness by observation Moiré pattern by transmission electron microscopy, taking in mind that Moiré pattern is a reliable proof of formation of thin layers with periodic structure [11]. Surprisingly, we found that the carbonaceous material had the shape of the nanotubes. We suppose that such nanotubes formed upon rolling up the thin layers of carbon, which formed on the surface of the larger Aerosil particles.

Results and discussion. The Co-containing hydrogenation catalyst was prepared by thermal decomposition of a complex formed *in situ* by deposition of cobalt(II) acetate $Co(CH_3CO_2)_2$ and DAB in 1 : 5 ratio on the surface of Aerosil (fumed SiO_2), followed by pyrolysis in argon at 800 °C (Fig. 1), as previously reported [10]. An excess of DAB compared to stoichiometric ratio (1 : 3) was used, since some quantity of DAB could be lost due to evaporation during heating. The black

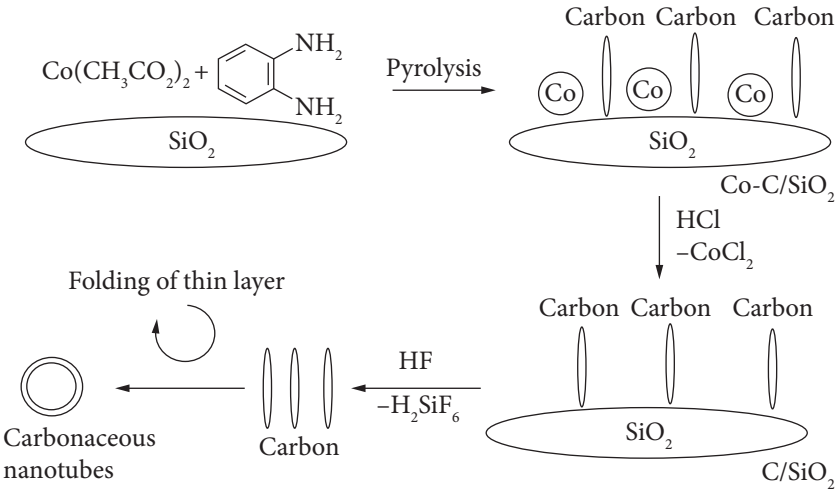

Fig. 1. Scheme of the carbonaceous material formation and isolation

powder obtained after pyrolysis (sample Co-C/SiO_2) contained 9.8 % (by weight) of metallic Co and ca. 15 % of carbon.

The composites prepared by this and similar methods, have demonstrated good catalytic characteristics in the hydrogenation of a number of organic compounds. In particular, the hydrogenation of quinoline in presence of such Co-containing catalysts at $P(H_2)$ = 50 bar, T = 100 °C, 3 mol. % Co loading in methanol during 24 h led to formation of 1,2,3,4-tetrahydroquinoline with 75 % yield. Increase in the catalyst loading to 4 mol. % under the same conditions led to the growth of 1,2,3,4-tetrahydroquinoline yield to 90 % [7]. However, the hydrogenation of substituted quinolines at 50 bar of H_2 at 100 °C did not provide satisfactory yields of the products, but almost quantitative hydrogenation of quinoline and a series of substituted quinolines was achieved upon pressure increase to 100 bar, at 3 mol. % content of Co [7]. Thus, studies of the catalytic performance of the materialsreported in this paper were carried out at $P(H_2)$ = 100 bar, T = 100 °C in methanol, with a reaction time of 24 hours. Under these conditions, quinoline was hydrogenated to 1,2,3,4-tetrahydroquinoline in presence of Co-C/SiO_2 (0.018 g per 0.129 g of quinoline, i. e. 0.03 mmol of Co per 1 mmol of quinoline, 5 mL of methanol) with 98 % yield. The catalytic performance of this catalyst was consistent with previously reported typical representatives of this family of Co-containing species [6, 7].

In order to remove Co the Co-C/SiO_2 catalyst was treated by aqueous HCl (see Fig. 1, details are provided in Experimental section). The resulting material (hereinafter referred to as C/SiO_2) contained carbonaceous material on Aerosil, the remaining Co content was evaluated by EDX analysis and it was found to be 0.1 % by weight. Finally, SiO_2 was dissolved by treatment with aqueous HF, and black powder formed, which mass was *ca*. 15 % of the mass of the initial Co-C/SiO_2 composite. The weight of the carbonaceous material corresponds well to C content in the Co-containing starting material. The remaining Co content in this material was about 0.5 % by weight.

The structure of the carbonaceous material was examined by transmission electron microscopy. Surprisingly, instead of the sheets of carbon, the sample predominantly consisted of nanotubes, and only a few flat pieces of amorphous carbon could be distinguished (Fig. 2). The diameter of the nanotubes was about 10—15 nm, and their length was in the range of 20—100 nm, but there were several nanotubes with alength about 300 nm and more. In addition, several very large tubes

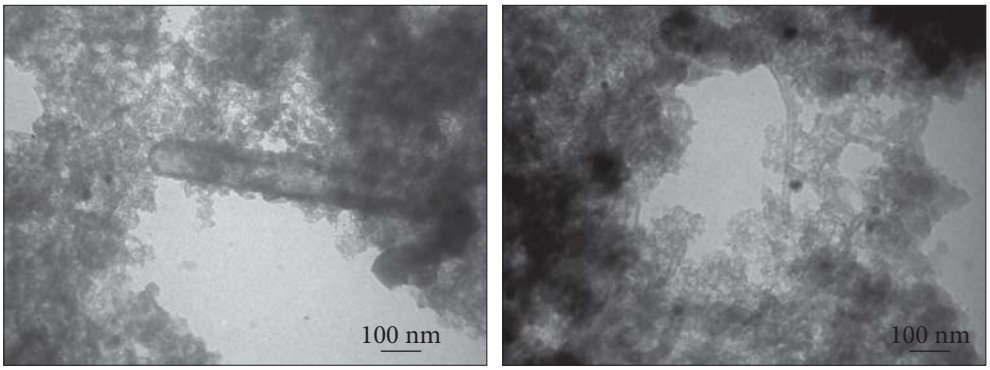

Fig. 2. Two representative TEM images of the carbonaceous material, obtained by dissolving Co and SiO_2 components from Co-C/SiO_2 catalysts

with diameter *ca*. 100 nm were found. Such nanotubes could be present in the initial Co-C/SiO_2 composite, or they could have formed after separation of the carbonaceous material from the Aerosil support. In the latter case, the formation of nanotubes can be explained by rolling of the thin carbonaceous sheets; such sheets were hold on the surface of significantly larger Aerosil particles (which have size of hundreds nm), but probably rolled up as soon as their support dissolved. Anyhow, the formation of nanotubes is a strong argument in favor the supposition that the carbonaceous material was formed during pyrolysis as thin layers in contrast to massive particles. Considering that the nanotube with a diameter 10 nm can be formed by rolling of the flat carbonaceous layer of minimal width equal to ca. 30—35 nm, the size of the carbonaceous layers existing in the starting material (Co-C/SiO_2 catalyst) was about 30×30 nm and more.

It should be noted that although only several large (100 nm in diameter) nanotubes were found on the TEM images, their weight fraction may be about ten percent, since their size exceeds the size of "small tubes" by an order of magnitude or even more.

It is important to note that the quantity of small carbonaceous particles, with linear size less than 20 nm, was very low on TEM images. Thus, most of the carbon material formed relatively large sheets, which could be fold into nanotubes, as opposed to formation of discrete small pieces of graphitized carbon.

The Raman spectrum of the carbonaceous material contains two bands with positions of maxima as 1345 and 1587 cm^{-1}, which can be assigned to the D and G bands, respectively (Fig. 3). This spectrum is typical for multiwall carbon nanotubes [12] or graphene with a high content of defects [13, 14]. The G-band arises from the stretching of carbon-carbon bonds, while the D-band is associated with the defects. The I_D/I_G parameter can be considered as the measure of the structural disorder and defects in carbonaceous materials [13, 14]. The defect-free graphene is characterized by zero I_D/I_G [13, 14].

The I_D/I_G ratio for the Raman spectrum of the carbonaceous material, considered herein, is equal to 0.96. This value falls in the range previously reported for the Co-C/SiO_2 hydrogenation catalysts [7, 10]. However, this ratio has increased compared to the parent Co-C/SiO_2 system (I_D/I_G was 0.6 [10]), such an increase may be caused by the formation of defects due to acidic treatment and probable hydrolytic cleavage of epoxy-groups, formed upon pyrolysis, as well as hydration of some of C=C double bonds.

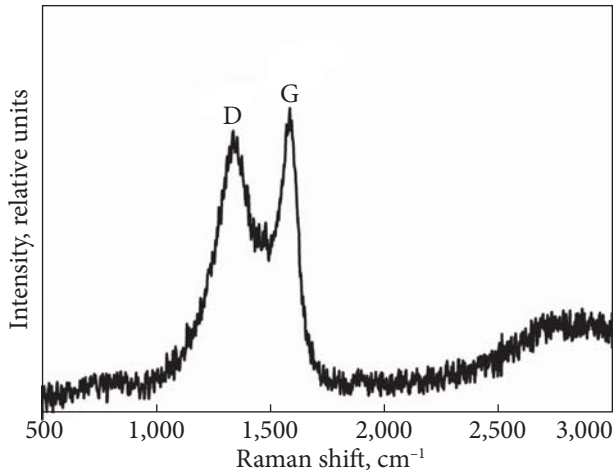

Fig. 3. Raman spectrum of the carbonaceous material, isolated from the Co-C/SiO_2 hydrogenation catalyst

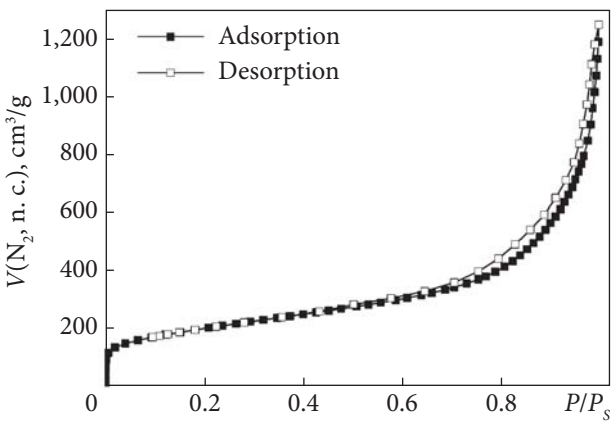

Fig. 4. Isotherms of nitrogen adsorption (filled boxes) and desorption (empty boxes) on the carbonaceous material, isolated from the Co-C/SiO_2 hydrogenation catalyst

The isotherm of nitrogen sorption on the carbonaceous material (Fig. 4) was also typical for the carbon nanotubes [12]. The material contained a significant volume of micropores (*t*-Plot micropore volume is 0.12 cm^3/g, Dubinin—Radushkevich micropore volume is 0.25 cm^3/g), while BJH most frequent pore diameter (*dV*/*dD*), calculated from the desorption branch, was 1.7 nm. Total pore volume (calculated by adsorption branch) at P/P_S = 0.99 was 1.65 cm^3/g, S_{BET} was 720 m^2/g. Notably, S_{BET} for the carbonaceous material considered herein exceed the values, reported for single wall carbon nanotubes (597 m^2/g) and for reduced graphene oxide (512 m^2/g) [15]. These high adsorption characteristics of the carbonaceous material are consistent with formation of dispersed species, mostly consisting of nanotubes and thin carbonaceous sheets.

The catalytic performances of the C/SiO_2 material and the carbonaceous silica-free material in hydrogenation of quinoline were tested under the same conditions, as those used for testing the catalytic properties of the initial Co-C/SiO_2 composite (i.e. T = 100 °C, $P(H_2)$ = 100 bar, 24 h, solvent — methanol). It was found that in presence of both these materials, 1,2,3,4-tetrahydroquinoline (THQ) is formed. In the case of C/SiO_2, the THQ yield was 80 % at catalyst loading 0.06 g per 1 mmol of quinoline (0.06 g of the catalyst contained $6 \cdot 10^{-5}$ g of Co; in the reaction mixture Co content was 0.1 mol. % per mole of quinoline). In the case of silica-free carbonaceous

material, the quantitative yield of THQ was achieved at catalyst loading at the level of 0.06 g per 1 mmol of quinoline (Co content in 0.06 g of the carbonaceous catalyst was $3 \cdot 10^{-4}$ g; in the reaction mixture Co content was 0.5 mol. % per 1 mole of quinoline). If catalyst loading was reduced to 40 mg per 1 mole of quinoline (corresponding to 0.3 mol. % of Co per 1 mole of quinoline), the yield of THQ was 90 %. Thus, treatment of the Co-C/SiO_2 catalyst with HCl and HF resulted in increase of its catalytic performance in hydrogenation of quinoline, measured as the yield of THQ per 1 mole of Co loaded in the reaction mixture. These findings can be explained by significant contribution of the carbonaceous material to the overall catalytic performance of the Co-C/SiO_2 composite. The increase in catalytic performance can be explained by the growth of accessible surface of the carbonaceous component, including easier access of the reagents to monoatomic Co sites, which can form in the layers of doped carbon [16].

It can be concluded that not only metallic Co nanoparticles but also carbonaceous material and/or Co monoatomic sites embedded in carbonaceous material participate in the catalytic hydrogenation of quinoline.

Experimental part. Aerosil A300 was purchased from UkrReaChim Ltd. (Kyiv, Ukraine). All other materials were provided by Enamine Ltd. (Kyiv, Ukraine) and UkrOrgSintez Ltd. (Kyiv, Ukraine). The solvents were purified according to the standard procedures. Co-C/SiO_2 catalyst was prepared as described in [10].

For elimination of Co from Co-C/SiO_2, 3 g of Co-C/SiO_2 was placed in 100 mL of concentrated HCl and the mixture was stirred in a round-bottom flask equipped with a reflux condenser at the temperature of 35 °C for two days to dissolve the cobalt. The resulting solid was filtered, washed with concentrated HCl (2 × 30 mL), followed by distilled water (3 × 50 mL) and ethanol (2 × 50 mL). The solid was subsequently dried using a rotary evaporator, affording 2.7 g of the C/SiO_2 material. Residual cobalt content was 0.1 % by weight (according to EDX analysis).

In order to eliminate Aerosil and isolate carbonaceous material from C/SiO_2, the material obtained in the previous step (2.7 g) was treated with an aqueous HF. The material was placed in a plastic beaker and 40 mL of aqueous HF (40 wt %) was added to dissolve SiO_2. The suspension was sonicated for 15 minutes in an ultrasonic bath, then centrifuged at 6000 rpm for 3 minutes and decanted. This procedure was repeated three times. The hydrofluoric acid was then replaced with a 50/50 (v/v) H_2O/EtOH mixture (40 mL), and the same sequence of operations was repeated three times more. The resulting solid was filtered through a Schott glass filter, washed with ethanol (2 × 30 mL), dried on a rotary evaporator, yielding 0.45 g of the carbonaceous material. Residual cobalt content was about 0.51 % by weight (by EDX analysis).

The TEM measurements were performed using a PEM-125K transmission electron microscope (Selmi Ltd., Ukraine) operating at a 100 kV acceleration voltage. The sample was prepared for TEM as reported in [10]. In particular, the suspension of the sample in methanol was prepared by ultrasonic treatment for 1 minute. A drop of suspension was applied to a Cu grid (300 mesh), covered by a film of amorphous carbon, and dried on air. Raman spectrum of the material was measured using a Renishaw inVia Raman microscope (Renishaw, UK), equipped with $\lambda = 514$ nm laser; the solid was slightly compressed for measurement. N_2 sorption studies were performed using Micro300C-Analysis Station2 (AltaMira Instruments, USA) at 77 K. Prior to the measurements the sample was dehydrated and degassed at 300 °C in 10^{-4} Torr vacuum for 4 h. The Co content in the samples was determined by energy-dispersive X-ray spectroscopy (EDX) using Apollo XL SDD EDAX instrument (EDAX Inc, USA).

The experiments on hydrogenation of quinoline were performed, as described previously [7].

Conclusions. It was shown that the carbonaceous material formed during pyrolysis of Co^{II} complex with 1,2-diaminobenzene deposited on Aerosil, consisted of the flat pieces of carbon and carbon nanotubes. Such carbonaceous material can be described as amorphous carbon or graphene with high a defect content. The nanotubes could form upon pyrolysis of Co^{II} complex in the first stage of catalyst preparation or as a result of rolling of the carbon sheets after dissolving of Co and the silica carrier. No dense graphite-like particles were detected by TEM. High adsorption characteristics of the carbonaceous material were also consistent with the presence of only fine carbonaceous species. It was shown that the carbonaceous component had significant catalytic performance in hydrogenation of quinoline, despite the low content of residual Co (*ca*. 0.5 %), implying that the carbonaceous material made considerable contribution to the catalytic properties of the Co-based catalyst. The catalytic activity of the carbonaceous material may be associated with the intrinsic properties of the carbonaceous material or may be caused by the presence of single-atom Co sites, incorporated in such material. These results are important for understanding the structure of active Co-based hydrogenation catalysts, formed by pyrolysis of Co^{II} complex on Aerosil.

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КАТАЛІТИЧНЕ ГІДРУВАННЯ ХІНОЛІНУ
В ПРИСУТНОСТІ ВУГЛЕЦЕВОГО МАТЕРІАЛУ, ОДЕРЖАНОГО ПІРОЛІЗОМ
КОМПЛЕКСУ КОБАЛЬТУ З 1,2-ДІАМІНОБЕНЗОЛОМ

З каталізатора гідрування Co-C/SiO_2, утвореного піролізом нанесеного на аеросил комплексу Co^{II} з 1,2-діамінобензолом, шляхом оброблення розчинами HCl та HF виділено вуглецевий матеріал, який досліджено методами ТЕМ та раманівської спектроскопії. За результатами дослідження адсорбції азоту визначено питому площу поверхні. Показано, що одержаний вуглецевий матеріал складається з аморфного вуглецю та нанотрубок, які можуть бути присутніми у вихідному каталізаторі Co-C/SiO_2 або утворюватися в результаті скручування тонких вуглецевих листів, що залишаються після розчинення Co та SiO_2. Встановлено, що такий вуглецевий матеріал є ефективним каталізатором гідрування хіноліну за низького вмісту залишкового Co (близько 0,5 %), що є ознакою можливої участі вуглецевої складової каталізаторів, одержаних піролізом координаційних сполук кобальту з органічними лігандами, в процесах гідрування.

Ключові слова: *каталізатор гідрування, піроліз, вуглецевий матеріал, нанотрубки, ТЕМ, раманівська спектроскопія, адсорбція.*