KINETICS OF NANOCARBON AND HYDROGEN EXTRACTION FROM THE PROPANE-BUTANE FRACTION

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Abstract. The kinetics of the process of obtaining nanocarbon and hydrogen from the propane-butane fraction were studied under differential reactor conditions. The experiments were conducted in a flow laboratory device operating in differential mode, consisting of a vertical stainless steel reactor with an internal diameter of 32 mm and a length of 500 mm. The initial period (≈approximately 10 min) is characterized by the reduction of Ni, Co, Fe, and Mo oxides to metal, the accumulation of free carbon, and the formation of carbon nanotube particles. The active phase of synthesis lasts 9-10 min and ensures η within 22 g/gcat. The effect of temperature on the relative yield was studied in the temperature range of 550-700 °C with a step of 10 °C. The catalyst layer thickness was ≈approximately 0.2 mm (m = 12 mg), the carbon-containing gas flow rate was 9 ± 0.1 L/h, and the process time was 12 min. According to the results of experimental studies, starting from 620°C, the process temperature did not affect the relative yield of carbon nanotubes, which was η ≈22 gs/gkat. The assessment of the influence of process parameters on the relative yield of the product and the properties of the obtained materials was conducted using the gravimetric method under isothermal conditions. The relative yield of carbon nanotubes during the process was determined using the formula η = (mmax – mkat)/mkat. Experimental studies were conducted to assess the dependence of carbon nanotube specific yield η on process time, at temperatures ranging from 550 to 700°C and hydrocarbon flow rates of 15 to 20 L/h. The initial high-silica zeolite and modified samples were studied by IR-spectroscopy, X-ray phase analysis, benzene adsorption methods before their processing, and subsequent structural characteristics, acidic properties of catalysts, and temperature-programmed desorption of ammonia.

The purpose of the work: To study the kinetic laws of the process of obtaining nanocarbon and hydrogen from the propane-butane fraction.

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Keywords: carbon-containing compounds, catalyst, temperature, contact time, nanocarbon, hydrogen.

Annotatsiya. Differensial reaktor sharoitida propan-butan fraksiyasidan nanokarbon va vodorod olish jarayonining kinetikasi o‘rganildi. Tajribalar differensial rejimda ishlaydigan, ichki diametri 32 mm va uzunligi 500 mm bo'lgan zanglamaydigan po'latdan yasalgan vertikal reaktordan iborat oqim laboratoriya qurilmasida o'tkazildi. Vaqtning boshlang'ich davri (≈10 min) Ni, Co, Fe va Mo oksidlarining metallga qaytarilishi, erkin uglerodning to'planishi va uglerod nanonaychalari zarrachalarining hosil bo'lishi bosqichidir, sintezning faol bosqichi 9-10 minut davom etadi va 22 g/gkat ichida ē ni ta'minlaydi. Haroratning nisbiy rentabellikka ta'siri 550-700 ° S harorat oralig'ida 10 ° S qadam bilan o'rganildi.Katalizator qatlami qalinligi hk≈0,2 mm (m=12 mg), uglerod o'z ichiga olgan gaz oqimi tezligi 9±0,1 l / soat, jarayon vaqti 12 min. Eksperimental tadqiqotlar natijalariga ko'ra, 620 ° C dan boshlab, jarayon harorati ug ≈22 gs / gkat bo'lgan uglerod nanotubalarining nisbiy rentabelligiga ta'sir qilmadi. Jarayon parametrlarining mahsulotning nisbiy rentabelligi va olingan materiallarning xususiyatlariga ta'sirini baholash izotermik sharoitda gravimetrik usulda amalga oshirildi. Jarayon davomida uglerod nanotubalarining nisbiy rentabelligi ē=(mmax – mkat)/mkat formulasi yordamida aniqlandi. Uglerod nanonaychalarining xos rentabelligi ēning jarayon vaqtiga bog'liqligini aniqlash uchun eksperimental tadqiqotlar 550 dan 700 °C gacha va uglevodorod oqimi tezligi 15 dan 20 L/soatgacha o'tkazildi. Dastlabki yuqori kremniyli zeolit va modifikatsiyalangan namunalar IR-spektroskopiya, rentgen fazali tahlil, ularni qayta ishlashdan oldin benzolni adsorbsiyalash usullari va keyingi strukturaviy xarakteristikalari, katalizatorlarning kislotali xossalari - ammiakning haroratga dasturlashtirilgan desorbsiyasi bilan o'rganildi.

Ishning maqsadi: Propan-butan fraktsiyasidan nanokarbon va vodorod olish jarayonining kinetik qonuniyatlarini o'rganish.

Kalit so'zlar: uglerodli birikmalar, katalizator, harorat, aloqa vaqti, nanokarbon, vodorod.

Аннотация. Изучена кинетика процесса получения наноуглерода и водорода из пропан-бутановой фракции в условиях дифференциального реактора. Эксперименты проводились в проточной лабораторной установке, работающей в дифференциальном режиме, состоящей из вертикального реактора из нержавеющей стали с внутренним диаметром 32 мм и длиной 500 мм. Начальный период времени (≈10 мин) является стадией восстановления оксидов Ni, Co, Fe и Mo до металла, накопления свободного углерода и образования частиц углеродных нанотрубок, активная фаза синтеза длится 9-

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10 мин и обеспечивает η в пределах 22 г/гкат. Влияние температуры на относительный выход изучалось в интервале температур 550-700 °C с шагом 10 °C. Толщина слоя катализатора составляла hk≈0,2 мм (m=12 мг), расход углеродсодержащего газа – 9±0,1 л/ч, время процесса – 12 мин. По результатам экспериментальных исследований, начиная с 620°C, температура процесса не оказывала влияния на относительный выход углеродных нанотрубок, который составил η ≈22 г/гкат. Оценку влияния параметров процесса на относительный выход продукта и свойства полученных материалов проводили весовым методом в изотермических условиях. Относительный выход углеродных нанотрубок в ходе процесса определяли по формуле η=(mmax – mkat)/mkat. Экспериментальные исследования по определению зависимости удельного выхода углеродных нанотрубок η от времени процесса проводили при температуре от 550 до 700°C и расходах углеводородов от 15 до 20 л/ч. Исходные образцы высококремнеземного цеолита и модифицированные образцы изучали методами ИК-спектроскопии, рентгенофазового анализа, адсорбции бензола до их обработки и последующей структурной характеристики, кислотные свойства катализаторов – температурно-программируемой десорбцией аммиака.

Цель работы: Изучить кинетические закономерности процесса получения наноуглерода и водорода из пропан-бутановой фракции.

Ключевые слова: углеродсодержащие соединения, катализатор, температура, время контакта, наноуглерод, водород.

Introduction

The demand for hydrogen is expected to grow dramatically shortly. The major contributors to global hydrogen demand are expected to be automotive and energy supply systems, where hydrogen serves as an energy carrier that can be stored and transported like natural gas. The growing needs of the chemical industry and energy will help to increase the production of hydrogen. For example, over the past decade, global hydrogen production has grown by 4.5% annually, and current production levels exceed 100 million tons per year. Hydrogen has no resource limit and does not generate greenhouse gases. At the same time, the use of carbon nanotubes in various fields such as the development of filters for water purification, the synthesis of multipurpose hydrogels, drug delivery, and biosensors is increasing. Multi-walled carbon nanotubes [1, 2] are promising materials with low toxicity and adsorption capacity. Multi-walled carbon nanotubes are cheaper to produce and are also preferred for the production of biosensors with high electrical conductivity [3-6]. Numerous results have increased attention to the successful use

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of carbon nanotubes in many areas of science, such as energy, construction, purification, and environmental protection [4-9].

Currently, hydrogen is a valuable chemical product and is widely used in a number of important industrial processes such as the production of ammonia [10], methanol [11, 12], hydrazine [13], and synthetic hydrocarbons [14]. Hydrogen is of great importance in the food industry, where it is utilized in the hydrogenation of vegetable oils [15, 16]. On the other hand, hydrogen is one of the most environmentally friendly sources of thermal energy [17].On an industrial scale, hydrogen is produced through the steam reforming of methane, the oxidation of heavy oil, the gasification of coal, and the electrolysis of water [18–20]. It is worth noting that biomass is also a potential source of hydrogen [21]. In general, the process can be expressed by the following equation: $C_nH_m \rightarrow nC + (m/2)H_2$ (1). This process is widely used to obtain carbon nanotubes [22]. Pure hydrogen is obtained by catalytic decomposition of carbon compounds into carbon and hydrogen [23–30].

EXPERIMENTAL

The experiments were conducted in a differential flow laboratory device comprising a vertical stainless steel reactor with an internal diameter of 32 mm and a length of 500 mm. To evenly heat the gas mixture discharged from the bottom of the reactor and to distribute the gas flow across the reactor cross-section, the bottom of the catalyst was filled with a nozzle (made of quartz particles) 150 mm below. A substrate dusted with the catalyst was placed at the bottom of the reactor and sealed. During the heating of the reactor, it was filled with argon.

The propane-butane mixture of gases was fed into the reaction zone, heated to operating temperature, and the reactor heating circuit was driven with an inert gas, which allowed atmospheric air to be expelled from the reaction zone. Torsion balance indicators, which record the change in the mass of the substance in the boat, were measured with a periodicity of 15 s. After the carbon nanotube formation process stopped, the hydrocarbon was expelled from the reactor with an inert gas.

The catalyst was prepared using the thermal decomposition method. The thermal decomposition method is one of the most effective methods for preparing catalysts. The catalyst powder was obtained by thermal decomposition of metal nitrates. The essence of the method is the interaction of the metal nitrates ($Ni(NO_3)_2 \cdot 6H_2O$, $Co(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_2 \cdot 9H_2O$, $Cu(NO_3)_2 \cdot 3H_2O$, $(NH_4)_2MoO_4 \cdot 24H_2O$), ammonium molybdate, and a mixture of high-silica zeolite (YKS) obtained from bentonite imported from Navbahor district, Navoi region, Republic of Uzbekistan) and organic matter (for example, a mixture of glycine and citric acid) in air at temperatures ≥500°C.At temperatures above 500°C, finely

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dispersed oxides of metals are formed. The physicochemical and textural characteristics of high-silica zeolites have been studied [32-33]. And have been used in the aromatization of natural gas, associated petroleum gases, and propane-butane fractions [34-37], as well as in the Fischer-Tropsch process and the production of olefins from dimethyl ether [38-39]. High-silica zeolites form a well-developed porous structure, with Ni, Co, Fe, Cu, and Mo particles distributed on their surface.

During the experimental studies of the synthesis process of carbon nanotubes, a propane-butane mixture (C_3H_8/C_4H_{10}=50%/50%) and an inert gas – argon (Ar) stored in a cylinder – were used as carbon-containing compounds. The removal of catalyst particles from the synthesized carbon nanotubes was achieved using the acid washing method.

Pyrolysis of a propane-butane mixture (C3H8 – 30%, C4H10 – 70%) was carried out at atmospheric pressure and temperatures of 600-650°C in laboratory and pilot reactors, with periodic operation on a fixed layer of catalyst. A filter based on zeolite was used to remove additives from gas mixtures and dry them from moisture. The waste gas was analyzed in the "Chromatek - Kristall 7000" chromatograph.

As soon as the operating temperature was reached, a mixture of gases in the required ratio was fed into the reactor. The gas-phase composition was altered by adjusting the volumetric velocities of the gas flows. The flow rate of each gas was selected based on the amount of catalyst, pyrolysis temperature, and process duration, ranging from 5 to 100 cm³/min. The pyrolysis time was varied from 1.5 hours to 4 hours.

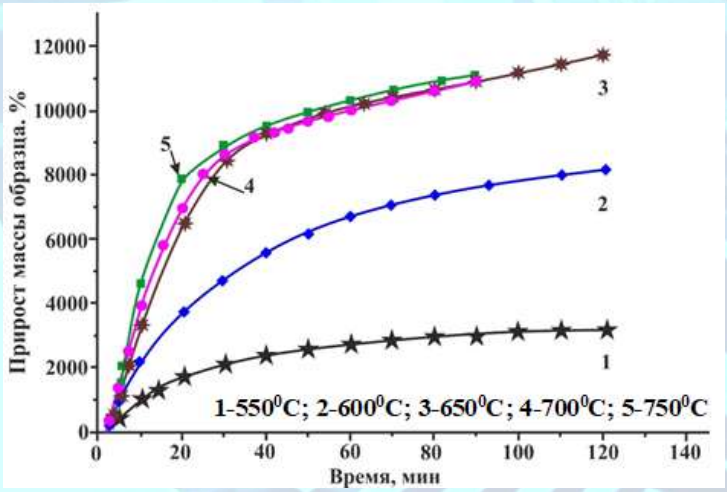


Figure 1. Kinetic curves of carbon deposition on the $(CuO)_x*(CoO)_y*(NiO)_z*(Fe_2O_3)_k*(MoO_3)m$/YKS catalyst from the C_3H_8-C_4H_{10} mixture at different temperatures

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Decomposition of a propane-butane mixture over a $(CuO)_x*(CoO)_y*(NiO)_z*(Fe_2O_3)_k*(MoO_3)m/YKS$ catalyst. Figure 1 shows the kinetic curves of carbon evolution from a propane-butane mixture over a $(CuO)_x*(CoO)_y*(NiO)_z*(Fe_2O_3)_k*(MoO_3)m/YKS$ catalyst at various temperatures. Figure 15 shows that the most favorable temperature for this reaction is 6500 °C-7500 °C. The carbon evolution did not change with increasing temperature, and was 110-120 g/g.

In the $(CuO)_x*(CoO)_y*(NiO)_z*(Fe_2O_3)_k*(MoO_3)m/YKS$ catalyst, the propane-butane mixture decomposes in the previous layer, at the reactor entrance, to form carbon nanofibers and hydrogen. In the subsequent zones of the reactor, a hydrogen-hydrocarbon mixture is formed, and we observed how hydrogen affects carbon formation. We diluted the C_3H_8-C_4H_{10} mixture with hydrogen, and we saw that the growth rate of the initial carbon nanofibers decreased. However, as a result, the carbon yield increased by 250-260 g/g.

Decomposition of a propane-butane mixture without a catalyst

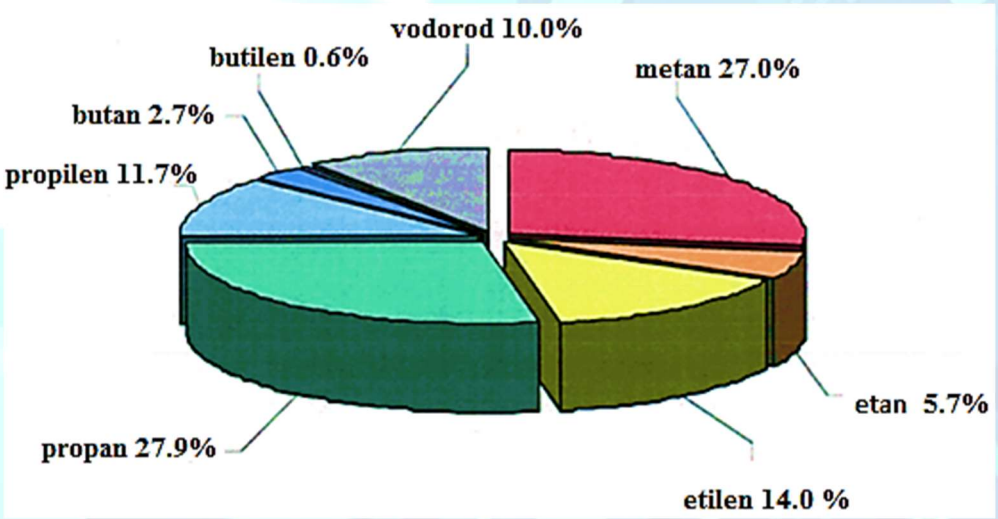


Figure 2. Ratio of hydrocarbons leaving the reactor after pyrolysis of a propane-butane mixture without a catalyst, T=700

The fact that the pyrolysis process in the propane-butane mixture in an empty reactor was quite active is evident from the reaction results. As a result of pyrolysis, olefins, ethylene, propylene, and butylenes are also formed. They are also more active in forming nanofibers than saturated hydrocarbons. When the reaction temperature was increased to 700 °C, the decomposition of the propane-butane mixture increased. The volume of hydrogen at the exit from the reactor was 71-72 mol%, and methane was 25-26 mol%. The catalyst worked moderately for 13-14 hours. The yield of carbon nanofibers after 24 hours was 320 g/g.

When the reaction temperature was increased to 750 oC, the hydrogen content at the exit of the reactor increased by 81-82 mol %, while the methane content decreased by 16-17 mol %, but the

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$(CuO)_x*(CoO)_y*(NiO)_z*(Fe_2O_3)_k*(MoO_3)_m$/YKS catalyst was reduced to 5-5.5 hours. The volume flow rate of the propane-butane mixture was 12,000 h-1. After 8 hours of reaction, the yield of carbon nanofibers was 50 g/g. From this, the following conclusion follows: the optimal temperature for the formation of hydrogen and carbon nanofibers from propane-butane mixtures under the influence of a $(CuO)_x*(CoO)_y*(NiO)_z*(Fe_2O_3)_k*(MoO_3)$m/YKS catalyst in a continuous reactor is 700-725 °C. Under optimal conditions of this reaction, 700-750 liters of hydrogen and 320-350 g of carbon nanowires can be obtained from 1 g of $(CuO)_x*(CoO)_y*(NiO)_z*(Fe_2O_3)$k*$(MoO_3)$m/YKS catalyst from that propane-butane mixture.

Figure 3 shows the results of chromatographic analysis of the gas composition formed as a result of the reaction of a propane-butane mixture at 750 °C for 200 minutes.

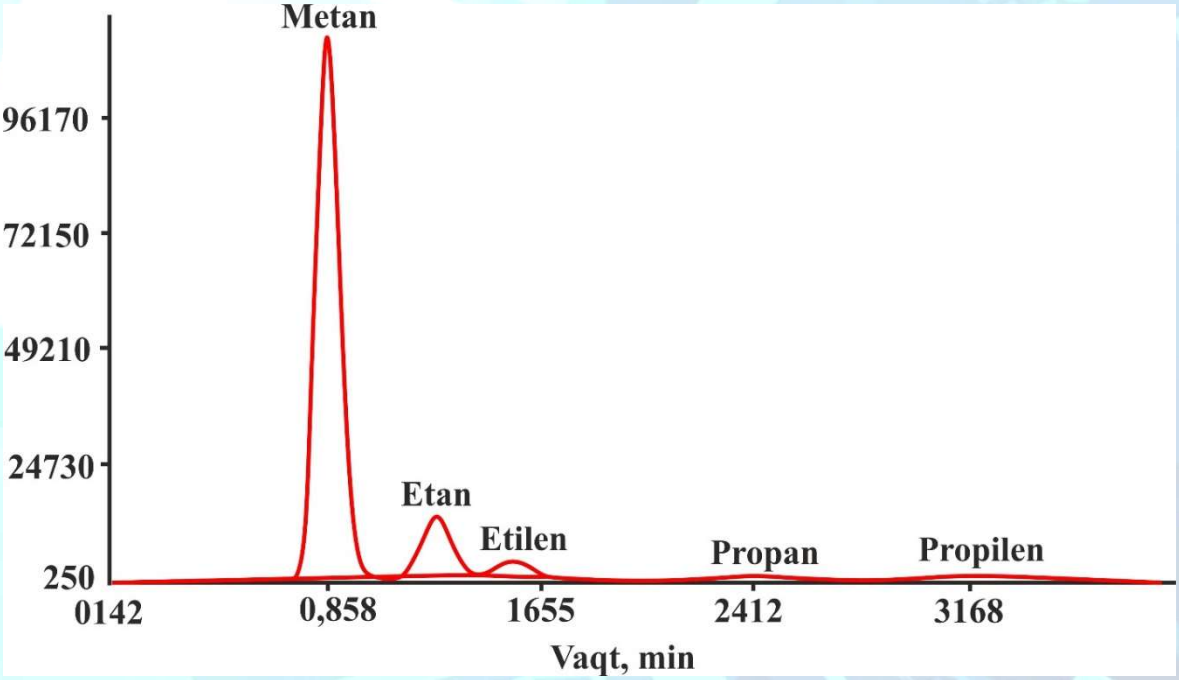


Figure 3. Chromatogram of hydrocarbon products formed during the decomposition of a C_3H_8-C_4H_{10} mixture: Treak.=7500C (τreak=200 min) Catalyst - (CuO)x*(CoO)y*(NiO)z*(Fe_2O_3)k*(MoO_3)m/YKS

A scanning electron microscopy micrograph of nanocarbon formed by the decomposition of a propane-butane mixture over a catalyst created at 700 °C is presented in Figure 4.

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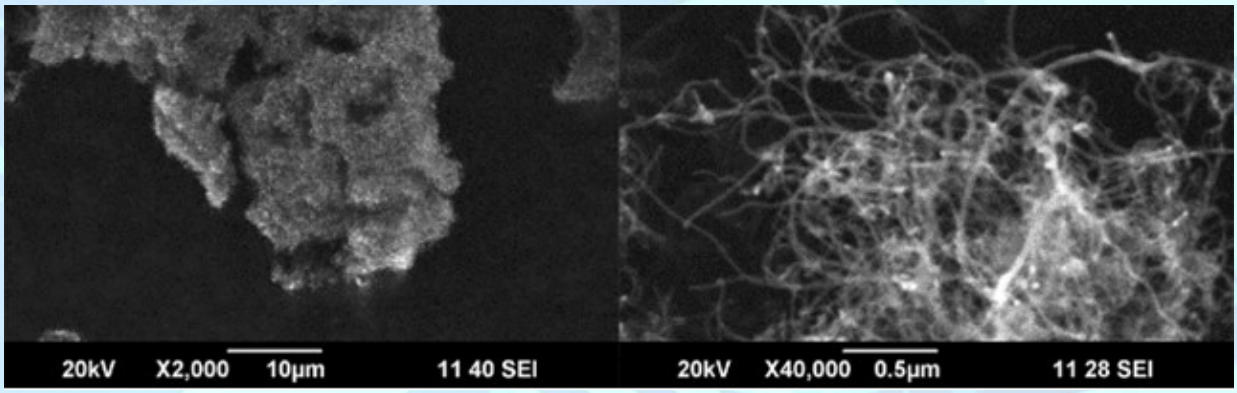


Figure 4. Scanning electron microscopy microphotograph of nanocarbon produced by decomposition of propane-butane mixture at 700 °C, catalyst (CuO)x*(CoO)y*(NiO)z*(Fe_2O_3)k*(MoO_3)m/YKS

Determining the amount of carbon in a carbonized sample is an important task. For this purpose, we conducted studies on the amount of carbon in the synthesized sorbents.

To do this, we subjected the sorbent to thermal treatment at 700-800°C for two hours.

As is known, the main indicator characterizing the efficiency of sorbents is their sorption capacity. In order to determine the sorption capacity of sorbents, we determined the absorption of oil and gasoline by sorbents.

Adsorption was studied in a specially designed apparatus consisting of a glass tube 25 cm long and 5 mm in internal diameter, and 20 ml vials closed with magnetic caps. The vial was equipped with inlet and outlet ports. The data obtained for determining the sorption capacity of the sorbents are presented in Table 1.

Table 1

Sorption capacity of sorbents

Temperature of thermal treatment of sorbents	Sorption time, hours	Sorption capacity, g/g	
		About oil	According to gasoline
750°C	1	0,58	0,28
	2	0,64	0,24
	24 (day)	0,92	0,29
800°C	1	0,46	0,22
	2	0,48	0,28
	24 (day)	0,86	0,32

CONCLUSION

The process of obtaining nanocarbon and hydrogen from propane-butane fraction was studied using a 15% Ni*5 % Co*5 % Fe*5 % Cu*2 % Mo/YKS catalyst with high catalytic activity and selectivity. Experimental studies to

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determine the dependence of the relative yield of carbon nanotubes on the process time were carried out at temperatures from 550 to 700°C and a hydrocarbon flow rate of 15 to 20 l/h.The initial period of time (≈10 min) is the stage of reduction of Ni, Co, Fe, and Mo oxides to metal, accumulation of free carbon, and formation of carbon nanotube particles. The active phase of synthesis lasts 9-10 min and ensures that η is within 22 g/gkat. The effect of temperature on the yield was studied in the temperature range of 550÷700°C with a step of 10°C. The catalyst layer thickness was ≈0.2 mm (m=12 mg), the carbon-containing gas flow rate was 9±0.1 l/h, and the process time was 12 min. According to the results of experimental studies, starting from 620°C, the process temperature does not affect the yield of carbon nanotubes, which was η ≈22 gs/gcat.

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