

Article

Catalytic Activity of Electroexplosive Cobalt Nanopowder in Hydrocarbon Synthesis by the Fischer–Tropsch Method

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Abstract

The study aims to develop a method for obtaining a high-performance catalyst for the synthesis of liquid hydrocarbons using the Fischer–Tropsch method based on ultradisperse cobalt powders obtained by the electric explosion method. To determine the catalytic activity of the obtained catalyst samples, the main process parameters, like temperature in the catalyst bed, the process pressure, the feedstock space velocity, and the ratio of reagents in the synthesis gas, were varied. It has been established that highly dispersed cobalt powder obtained by the electrical explosion method is a fairly active catalyst for the synthesis of liquid hydrocarbons via the Fischer–Tropsch process. It has been established that the overall CO conversion rate in the temperature range from 230 to 330 °C ranges from 25 to 90%. However, the formation of the main byproduct of the synthesis, carbon dioxide, is not observed below 270 °C. It was determined that for the developed catalyst sample, the optimal temperature range is from 230 to 260 °C, in which the yield of by-products of synthesis and gaseous hydrocarbons is quite low—the selectivity for methane does not exceed 20%, with the proportion of C₅₊ hydrocarbons in the liquid phase at the level of 80%. The CO conversion rate increases proportionally with growing pressure. It has been established that cobalt nanopowder exhibits high catalytic activity in reactions of liquid hydrocarbon formation with low hydrogen content in the initial synthesis gas. This fact allows us to conclude that it has potential for use in processing gases obtained during the pyrolysis of biomass or other non-traditional sources of synthesis gas, characterized by an H₂:CO ratio of 1:1 to 1.25:1. Catalysts obtained from ultradisperse cobalt powders were shown to be resistant to rapid deactivation under synthesis conditions at operating temperatures for 30 h. During long-term testing, CO conversion remained at 23.5% at 230 °C for the entire duration of the experiment.



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1. Introduction

The search for alternatives to petroleum-based liquid fuels remains one of the primary challenges in the chemical engineering area. In recent decades, environmental situations, particularly the use of secondary resources for the production of motor fuel components, have become the primary driver for addressing this challenge.

Although the Fischer–Tropsch reaction was developed in 1925, it has gained particular relevance for producing synthetic liquid fuels from natural gas (gas to liquids—GTL), coal

(coal to liquids—CTL), and biomass (biomass to liquids—BTL) in the 21st century. This is confirmed by the fact that the largest industrial-scale plant, which synthesizes liquid hydrocarbon by the Fischer–Tropsch method and produces 140,000 bbl/day of hydrocarbon fuel, was built in 2000 by Shell in Qatar [1–3].

Fischer–Tropsch synthesis underlies the liquid fuel production stage in most existing BTL technologies. For example, work [4] presents a process for producing fuel from a straw biomass source, via biomass gasification and Fischer–Tropsch synthesis using an iron-based catalyst. The yields of gasoline and diesel fuel in this process were 3.8 and 6.4 GJ/t, respectively.

In [5], an iron-based catalyst is also used in the Fischer–Tropsch synthesis stage; however, this study examines corn stover as the feedstock and two different biomass processing units: a low-temperature, oxygen-fueled fluidized bed gasifier without slag formation, and a high-temperature gasifier with in-line slag formation. In both cases, after catalytic Fischer–Tropsch synthesis and hydrotreating, liquid fractions of naphtha (gasoline blends) and distillates (diesel fuel) are obtained.

In works [6–8], the production is considered with an emphasis on the electrification of the biomass converting processes into liquid and alternative processes in the presence of a cobalt-based catalyst, with wood feedstock as biomass. The authors of [9–12] used a cobalt-based catalyst, but wood chips were used as biomass.

In [13], the BTL process for the production of liquid motor fuels based on the gasification of wood biomass in a flow and in a circulating fluidized bed was investigated. The fuel synthesis technologies included the Fischer–Tropsch synthesis, methanol synthesis followed by the methanol–gasoline process, and gasoline synthesis using the Topsoe technology. The study found that the Fischer–Tropsch synthesis appears to be the most promising technology for the commercial production of liquid fuels by gasifying wood biomass, since it provides higher efficiency and lower costs compared to other processes by 5.7–14.7% and 3.2–9.7%, respectively. Thus, the Fischer–Tropsch synthesis, with the same input data, exhibits the highest fuel energy production, output power, and mass yield of fuel.

A study [14] evaluated the production of syngas for Fischer–Tropsch processing via the gasification of lignocellulosic biomass. The process was conducted in several stages using direct electrification, indirect electrification, and combinations of both. The study found that carbon efficiency increased to a maximum of 94.5% due to a reduction in CO₂ emissions, while energy efficiency increased to a peak of 50.9%, demonstrating the efficient use of biomass.

In [15], the possibility of processing brown coal with the addition of a certain amount of waste biomass (peanut shells, pine cones, and olive seeds) was assessed through a slow pyrolysis process using a Fischer–Tropsch reactor. The results obtained in this study showed that with an increase in the percentage of biomass in the brown coal mixture, the yield of coal (biochar) decreased, while the yield of other products (tar, decomposed water, and gases) increased. It was also found that among the biomass residues used, olive pits were the most valuable compared to peanut shells and pine cones.

In [16], the production of liquid fuel from waste tires and water hyacinth was studied. A comparative study of co-pyrolysis and co-gasification with the integration of the Fischer–Tropsch process showed that with increasing reactor temperature, the yield of gasoline increased and the yield of diesel fuel decreased, resulting in the maximum yield of liquid fuel—8817.15 gallons per day—being obtained at a temperature of 230 °C.

In the article [17], the authors also used waste tires as a feedstock in the Fischer–Tropsch pyrolysis and gasification processes, but for the production of aviation fuel. The authors of the article concluded that, from an economic standpoint, both methods are more

advantageous compared to other routes to producing aviation fuel, such as processing alcohol into jet gas or hydroprocessing esters and fatty acids. Thus, the production of aviation fuel through the pyrolysis process is more expensive (USD 0.78 per liter) than through the Fischer–Tropsch process (USD 0.66 per liter), but is cheaper than other methods. It was also found that, from an environmental standpoint, pyrolysis emits less CO compared to the Fischer–Tropsch synthesis, but has a higher potential environmental impact in several categories.

The authors of [18] evaluated the production of fuel using the Fischer–Tropsch method by using recovered waste and landfill gas. They found that using such feedstocks makes it possible to reduce specific greenhouse gas emissions to 72.56 kg (in CO₂ equivalent) per unit of energy produced.

It should be noted that waste gasification products are most often characterized by a low hydrogen content, which imposes strict requirements on the activity of GTL process catalysts when processing synthesis gases with a composition far from the stoichiometric ratio of H₂:CO components equal to 2:1.

As can be seen, a wide variety of feedstock sources, including recycled materials and waste, can be used to produce syngas for the Fischer–Tropsch reaction. Furthermore, a variety of catalytic systems are used to synthesize liquid hydrocarbons in the Fischer–Tropsch method. The particular interest is catalysts based on ultradisperse metal powders.

The authors of [19] investigated an industrial ultradisperse iron catalyst promoted with manganese for slurry-phase Fischer–Tropsch synthesis. The catalyst demonstrated high CO conversion and provided a high yield of C₂–C₄ olefins. Catalyst activity remained relatively stable over 577 h of operation, indicating relatively slow deactivation over time.

Another study [20] investigated the catalytic properties of ultradispersed iron particles promoted by manganese for liquid-phase Fischer–Tropsch synthesis. The catalysts were prepared by physically mixing ultradispersed iron particles and manganese oxide, as well as by liquid-phase deposition of manganese on iron particles. The resulting catalysts exhibit high selectivity for olefin production, particularly high selectivity for lower olefins. Furthermore, it was found that these catalysts provide high yields of lower olefins.

In [21], similarly, the effect of preliminary oxidative treatment of ultradisperse iron particles on their catalytic properties was investigated in a liquid-phase Fischer–Tropsch synthesis process. Despite the similar composition of the products obtained using a catalyst with and without preliminary oxidation, the catalytic activity of the pre-oxidized catalysts obtained by keeping ultrafine iron particles in a nitrogen atmosphere containing a given amount of oxygen was higher than that of the untreated ones. The authors of [22] investigated liquid-phase Fischer–Tropsch synthesis using a slurry bed reactor. It was shown that the catalytic activity of ultradisperse iron particles was higher than that of a precipitated iron catalyst.

In [23], fundamental studies of a catalyst based on ultradisperse cobalt particles were conducted to develop catalysts for the Fischer–Tropsch synthesis using a slurry bed reactor. The resulting catalyst was more active in this reaction than the precipitated catalyst due to its non-porous structure. It was also found that the addition of Cr, Ti, Mn, Mo, or Zr to the resulting catalyst was effective in increasing the catalytic activity. Among the listed catalysts, Co–Cr exhibited the highest activity. The yield of hydrocarbons over time on the Co–Cr catalyst obtained in a mixed solution of H₂O and CH₃OH was 14.9 g cat^{−1} h^{−1}. The increase in activity was explained by a decrease in the Co particle size. The distribution of the hydrocarbons obtained also depended on the particle size. The selectivity for CH₄ decreased, while for C₅₊ hydrocarbons it increased due to a decrease in the particle size.

In [24], the activity of catalysts based on ultradispersed Co particles for liquid-phase Fischer–Tropsch synthesis at a temperature of 270 °C and a pressure of 30 atm was studied.

It was found that Cr, Ti, and Mn turned out to be effective additives for increasing the catalytic activity of this catalyst. Among the catalysts considered, the Co-Cr-based catalyst showed the highest activity. Such an indicator of the productivity of chemical processes as the spatio-temporal yield of hydrocarbons (the amount of hydrocarbon product manufactured per unit of time, normalized to the volume of the reactor or catalytic loading) for this catalyst was approximately two times higher than for the cobalt catalyst. The activity of the Co-Cr catalyst intensified with increasing Cr content and reached a maximum with the addition of 2 wt.% of Cr. The distribution of the obtained hydrocarbons also depended on the Cr content. The cobalt catalyst containing 2 wt.% of Cr demonstrated the lowest selectivity for methane formation and the highest selectivity for liquid hydrocarbon fuel formation.

In [25], the authors considered the synthesis of highly dispersed iron-based Fischer-Tropsch catalysts using an organic solvent based on an MCF support. In the case of the Fe/MCF-E catalyst, obtained by impregnation using ethanol, which forms a highly dispersed ϵ -iron phase deposited on supports during the corresponding industrial FTS process, excellent stability and activity were demonstrated for 100 h. The Fe/MCF-E catalyst demonstrated high overall olefin selectivity and separate selectivity for C_2 – C_4 olefins.

Ultradispersed metal powders are also used in other processes. For example, the authors of [26] integrated ultradispersed CuO nanoparticles with two-dimensional metal-organic frameworks for improved electrochemical reduction of CO_2 to ethylene. The resulting catalyst exhibited good electrocatalytic properties. Ultradispersed diamond is used in CO hydrodechlorination and oxidation reactions as an excellent support for catalysts based on Pd and Au nanoparticles. A palladium-containing catalyst exhibits high selectivity in the hydrodechlorination of 1,3,5-trichlorobenzene, significantly exceeding the efficiency of Pd supported on activated carbon and commercial 5% Pd/C samples. The authors of [27] controlled the content of ultradisperse Pt metal particles deposited on a CeO_2 catalyst for the water vapor reforming reaction. It was shown that the molar activity of such catalysts in the water vapor reforming reaction strongly depends on the platinum content, increasing by 2.5 times with a change in concentration.

Metal nanopowder obtained by the electrical explosion of conductors [28,29] exhibits high reactivity in various chemical reactions, making it promising for use in the chemical synthesis of various functional materials, including catalysts. At the same time, the study of the catalytic activity of the nanopowder itself is of interest; in particular, iron nanopowder is known to be active in Fischer-Tropsch synthesis processes [30–32]. Research into the catalytic activity of electroexplosive cobalt nanopowder is also promising, since the catalytic activity of cobalt in Fischer-Tropsch synthesis processes is known and has been studied in sufficient detail in [33–35].

An analysis of literary sources shows that despite the existence of a variety of catalytic systems for the implementation of the Fischer-Tropsch reaction, most of them do not provide a high yield of liquid products that can be used as components of motor fuels.

Thus, the aim of this study is to develop a method for obtaining and testing in a laboratory unit a high-performance catalyst for the synthesis of liquid hydrocarbons using the Fischer-Tropsch method based on ultradisperse cobalt powders obtained by the electric explosion method.

2. Results and Discussion

Before conducting experimental studies on the catalytic unit, the specific surface area of the catalyst powder samples was measured before and after pressing with a binder. The

surface area, determined using the BET method, was 12 m², a low value for a catalyst sample. After granulation, the surface area decreased slightly to 11 m².

Determination of the powder particle size distribution revealed the presence of particles ranging from 60 nm to 300 nm, with 90% of the particles being no larger than 147 nm, and 50% being no larger than 95 nm. The particle size results are presented in Figure 1.

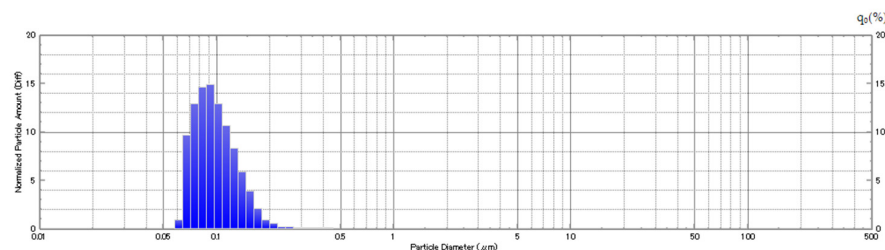


Figure 1. Ultradisperse cobalt powder particle size distribution.

Industrial catalysts obtained by the traditional method of impregnating a porous support are characterized by particle sizes ranging from 20 to 200 nm [36]. The electric explosion method produces proportionate particles, and the resulting powders are characterized by high surface energy.

Particle images obtained using scanning electron microscopy are shown in Figure 2. In this work, spherical nanosized cobalt particles suitable for producing active catalytic systems for Fischer–Tropsch synthesis were obtained using the electric explosion method. A distinctive feature of this method for producing cobalt nanopowder is the transfer of a large amount of energy to the metal in a very short period of time—the process duration ranges from 10^{−7} to 10^{−5} s, and the current density is 105 A/mm². The materials obtained in this way are characterized by a large number of crystal lattice defects and high surface energy.

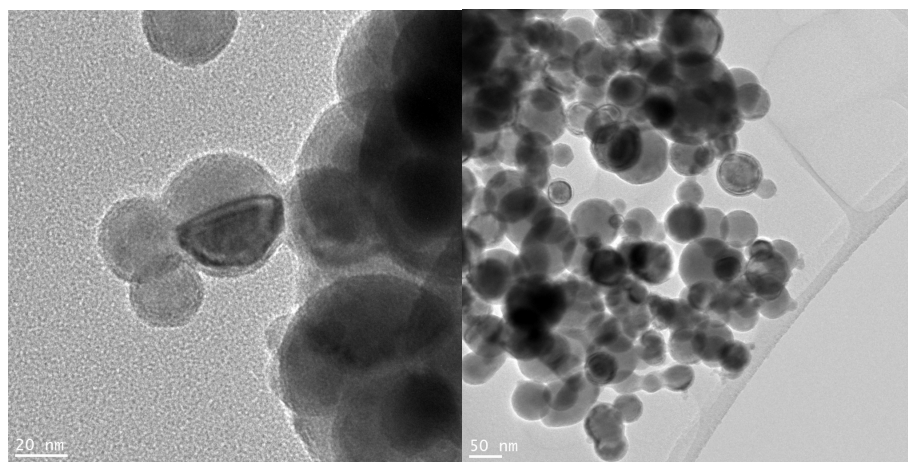


Figure 2. Micrographs of the obtained cobalt particles.

The first step in determining the catalytic activity of the obtained samples was to determine the yield of the main products (gaseous and liquid) at a constant reactor pressure of 0.9 MPa, a reactant ratio of 2:1 (H₂:CO), and a feedstock space velocity of 7.5 min^{−1}, with temperature varied from 230 to 330 °C in 10 °C increments. All measurements were performed after the process had reached steady state. The main results of this experiment series are presented in Table A1 and Figures 3–5.

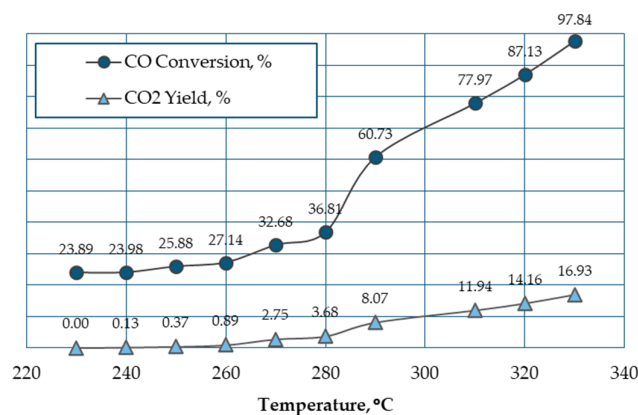


Figure 3. Dependence of the CO conversion degree and CO₂ yield on the process temperature.

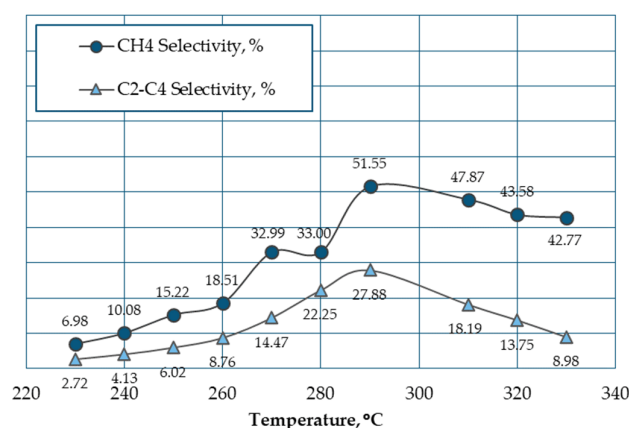


Figure 4. Dependence of the gaseous hydrocarbon formation selectivity on the process temperature.

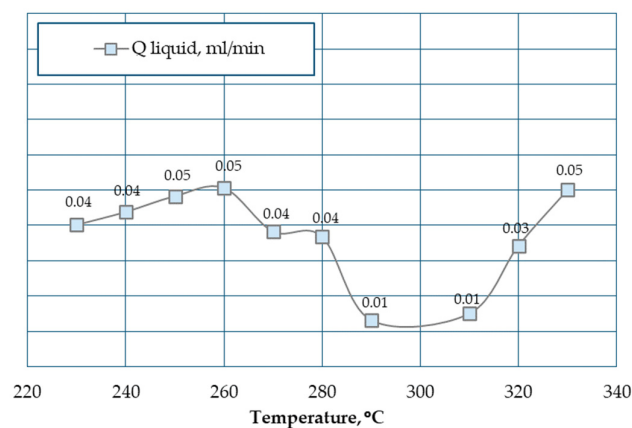


Figure 5. Dependence of the liquid product yield on the process temperature.

The first series of experiments investigated the effect of process temperature on the yield and qualitative composition of liquid and gaseous synthesis products. The maximum CO conversion of 97.84% was achieved at 330 °C. The activity temperature range of the highly dispersed cobalt powder obtained by electrically exploding a conductor corresponds to the activity temperature ranges of “classical” massive or supported cobalt-based catalysts. The dependence of the CO conversion (round markers) and CO₂ yield (triangular markers) on process temperature is shown in Figure 3; the selectivity for gaseous synthesis products and the yield of liquid products (Q) are shown in Figures 4 and 5.

The curves in Figure 3 show that at low temperatures, practically no CO₂ formation occurs, with a conversion rate of approximately 23–25%, despite the large amount of liquid

product being formed. Upon reaching a synthesis temperature of 270 °C, the CO₂ yield in the gaseous synthesis products sharply increases to 2.745%, with a simultaneous increase in CO conversion. A subsequent increase in temperature leads to a significant increase in CO conversion and an increase in the CO₂ content in the synthesis products, suggesting that the Reaction (1) is activated:



It is known that cobalt-containing Fischer–Tropsch synthesis catalysts are less prone to CO₂ formation in the gaseous synthesis products. The high CO₂ yield in this case may be due not only to the presence of impurities, primarily Fe, in the starting powder, but also to the high surface energy of the resulting powders. The temperature in the explosion zone reaches 20,000 K or more, and the particle velocity ranges from 2 to 10 km/s. Under these conditions, powders with a large number of crystal lattice defects and high surface energy are formed.

The previous conclusions are confirmed by the dependences in Figure 4: in the temperature range of 270–300 °C, selectivity for gaseous hydrocarbon formation increases, reaching a maximum at 290 °C and subsequently declining. The yield of liquid products, in contrast, reaches a minimum and then increases explosively; however, at temperatures above 300 °C, water predominates in the liquid synthesis products.

The obtained dependencies are consistent with generally accepted concepts regarding the activity of cobalt catalysts in the synthesis of liquid hydrocarbons using the Fischer–Tropsch method; specifically, the temperature and pressure ranges of activity are confirmed. The relatively low specific surface area of cobalt powders obtained by electric explosion results in their low productivity, which is offset by the ease of production, low cost, and resistance to sintering, which allows them to be used for processing synthesis gases with a high content of various impurities.

The next series of experiments was conducted at a constant temperature of 270 °C, a stoichiometric reactant ratio of 2:1, FSV of 7.5 min^{−1}, and varying synthesis pressure from 0.3 to 1.2 MPa. The results are presented in Figure 6. The choice of process temperature was determined by the results obtained in the previous stage.

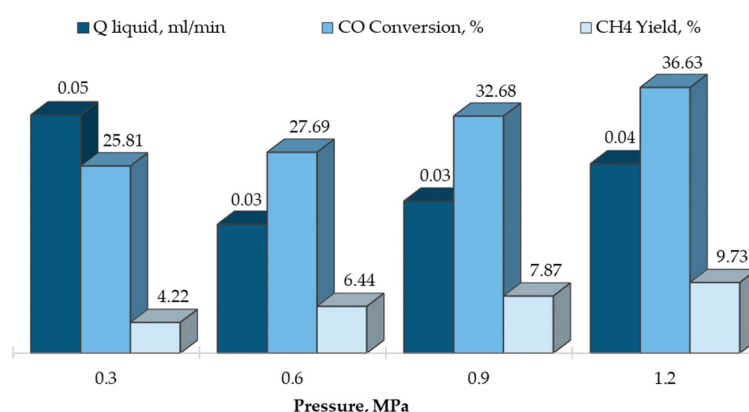


Figure 6. Dependence of the yield and composition of products on the process pressure.

Increasing pressure has a positive effect on the key process parameters: CO conversion increases significantly (from 25 to 35%), liquid product yield increases, and the hydrocarbon content increases, consistent with basic concepts of the synthesis of liquid hydrocarbons from CO and H₂. At low pressures (approximately 0.3 MPa), the high liquid yield is due to the predominance of the water-vapor shift Reaction (1), which leads to the formation of water and suppression of the target hydrocarbon synthesis reactions. Further increases in

pressure above 1.2 MPa do not lead to an increase in the conversion degree or an increase in the yield of the target products.

The next series of experiments aimed to identify the effect of FSV on the yield and composition of the synthesis products. The results of this experiment series are presented in Figure 7.

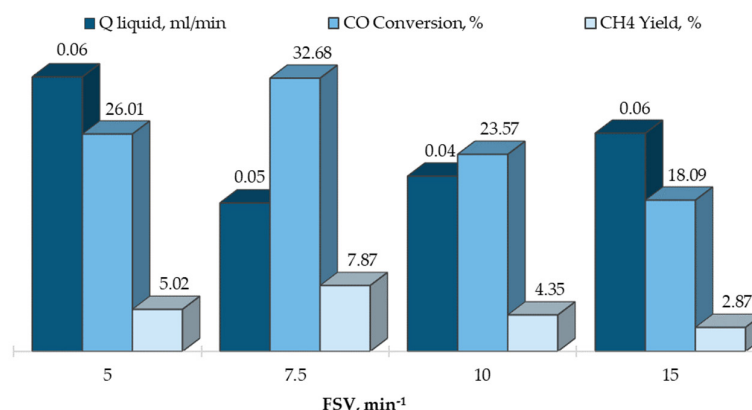


Figure 7. Dependence of the yield and composition of products on the FSV.

The maximum CO conversion is achieved at an FSV of approximately 7.5 min⁻¹. This relatively low value is due to the properties of the resulting cobalt powder: the high degree of dispersion and small particle size prevent high specific surface areas from being achieved without the use of a carrier. Increasing the FSV leads to a decrease in the conversion rate and the yield of gaseous products. The increase in liquid yield is explained by the increased water content, as Reaction (1) proceeds much more simply, without spatial restrictions.

The subsequent series of experiments aimed to identify the effect of the H₂:CO reactant ratio on the process. At a temperature of 270 °C, a pressure of 0.9 MPa, and an FSV of 7.5 min⁻¹, the hydrogen content in the syngas was reduced to a 1:1 ratio. The experimental results are presented in Figure 8.

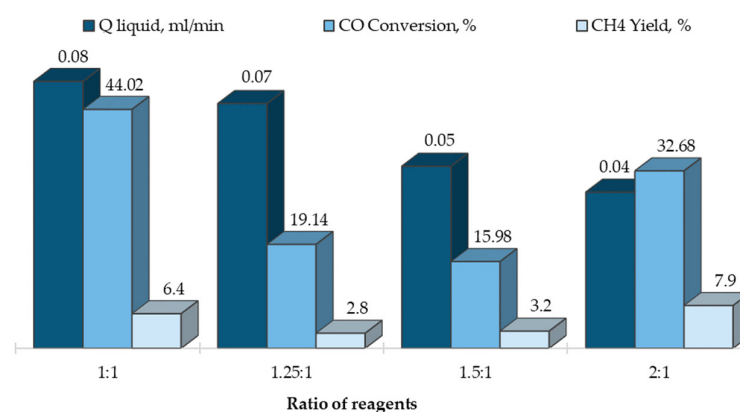


Figure 8. Dependence of the yield and composition of products on the ratio of reactants in synthesis gas.

Reducing the hydrogen content in the syngas has a negative impact on the conversion rate and yield of target products, which are significantly reduced. However, at an H₂:CO ratio of 1:1, the conversion rate increases sharply, due to the catalyst's surface structure and high surface energy. Liquid yield also increases, rising by 20% at this ratio. These properties make the developed catalyst attractive for converting low-hydrogen syngases into liquid products.

Liquid synthesis products obtained under various process parameters exhibit a wide fractional composition, with high isoparaffin and aromatic hydrocarbon content (up to 43

and 31 wt.%, respectively) and low paraffin hydrocarbon content (no more than 13% wt.%). Increasing pressure leads to an overall heavier fractional composition of the resulting products and an increased yield of the kerosene fraction (180–260 °C), while at low pressure (up to 0.6 MPa), the gasoline fraction (80–180 °C) predominates.

Industrial catalysts of Fischer–Tropsch synthesis are characterized by a high specific surface area, as they are produced using supports with a high specific surface area, approximately 500 m²/g. In this case, the resulting catalysts exhibit performance comparable to industrial catalysts, as their preparation method using a binder solution prevents particle sintering during the synthesis process. Furthermore, their high surface energy allows for significant productivity.

To confirm the stability of the resulting catalysts and the feasibility of their industrial implementation, after identifying the optimal process conditions, long-term tests were conducted at the previously established optimal parameters. Each test lasted 30 h, beginning when the unit reached steady-state operation. The CO conversion rate remained constant throughout the test, averaging 23.685% at 230 °C and 27.110% at 260 °C. The test results are presented in Table A2. These results allow us to rule out rapid catalyst deactivation in the initial stages of the process.

After unloading the catalyst from the reactor, it experiences significant heating, even sparking, when exposed to moisture vapor and atmospheric oxygen. This significantly complicates catalyst studies following catalytic activity tests. In addition to cobalt, the spent catalyst also contains cobalt and iron oxides formed during interaction with moisture vapor and atmospheric oxygen. Figure 9 shows micrographs of the catalyst surface after the experiments.

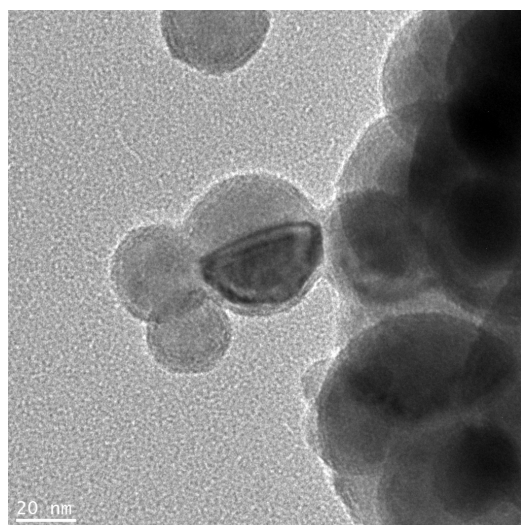


Figure 9. Micrographs of the cobalt particles after the experiments.

1. It has been established that highly dispersed cobalt powder obtained by the electrical explosion method is a fairly active catalyst for the synthesis of liquid hydrocarbons via the Fischer–Tropsch process. The overall CO conversion rate in the temperature range from 230 to 330 °C ranges from 25 to 90%. However, the formation of the main byproduct of the synthesis, carbon dioxide, is not observed below 270 °C. In this temperature range, the CO conversion rate is around 30%, while the proportion of C₅₊ hydrocarbons does not fall below 78%. The relatively low CO conversion rate is due to the low specific surface area of the powder, necessitating its application to a support with a high specific surface area.

2. It was determined that for the developed catalyst sample, the optimal temperature range is from 230 to 260 °C, in which the yield of by-products of synthesis and gaseous hydrocarbons is quite low—the selectivity for methane does not exceed 20% with the proportion of C₅₊ hydrocarbons in the liquid phase at the level of 80%.
3. It has been shown that growing process pressure increases the overall CO conversion rate, but requires high costs for industrial implementation. The CO conversion rate increases proportionally with growing pressure.
4. It has been established that cobalt nanopowder exhibits high catalytic activity in reactions of liquid hydrocarbon formation with low hydrogen content in the initial synthesis gas. This fact allows us to conclude that it has potential for use in processing gases obtained during the pyrolysis of biomass or other non-traditional sources of synthesis gas, characterized by an H₂:CO ratio of 1:1 to 1.25:1.
5. Catalysts obtained from ultradisperse cobalt powders were shown to be resistant to rapid deactivation under synthesis conditions at operating temperatures for 30 h. During long-term testing, CO conversion remained at 23.5% at 230 °C for the entire duration of the experiment.

3. Materials and Methods

Highly dispersed cobalt powder was obtained by the electrical explosion of a conductor in an inert environment method, the essence of which is to pass a pulsed current of high density and power through a conductor, in this case a cobalt wire (Table 1 shows the composition) with a diameter of 1 mm, according to [37]. The pulse duration was 10^{−5} s, and the current density was 105 A·mm^{−2}. A detailed description of the technique is presented in [33].

Table 1. Chemical composition of using cobalt wire, wt.%.

Co	C	Si	Mn	Fe	Ni	S	P	Al
99.9500	0.0040	0.0030	0.0001	0.0030	0.0200	0.0003	0.0030	0.0002

The specific surface area of the obtained cobalt powder was measured using a Sorbi-M unit using the multi-point BET adsorption method (ZAO META, Novosibirsk, Russia).

Particle size distribution analysis was performed using a Shimadzu SALD-7101 laser diffraction particle size analyzer (Nakagyo-ku, Japan).

Particle size determination and microscopy were performed using a JEOL JEM-2100F microscope (Akishima, Japan).

The resulting highly dispersed cobalt powder was pressed using an oil press in press dies with an internal diameter of 13 mm into tablets no more than 5 mm high, followed by crushing and collection of a 0.5–1.0 mm fraction for loading into the catalytic unit.

Chromatographic analysis of the gas mixtures was performed on a Chromatec-Crystal 5000.1 chromatograph (Yoshkar-Ola, Russia). Gas samples were analyzed using the following chromatographic columns with a column temperature of 80 °C:

- A 4 m packed column with NaX (60/80) sorbent for detecting and calculating the concentrations of hydrogen, oxygen, nitrogen, methane, and carbon monoxide (carrier gas flow rate (argon)—30 mL/min);
- A 1.5 m packed column with Carbosieve S-II (60/80) sorbent for detecting and calculating carbon dioxide concentrations (carrier gas flow rate (helium)—30 mL/min);
- A 25 m Agilent HP-AL-S capillary column for detecting and calculating C₁–C₆ hydrocarbon concentrations.

The concentrations of the gas mixture components were calculated using the Chromatec Analyst 2.5 absolute calibration method.

4. Experiment

The study of the catalyst activity was carried out on the laboratory unit “Katakon”. Figure 10 shows the simplified diagram.

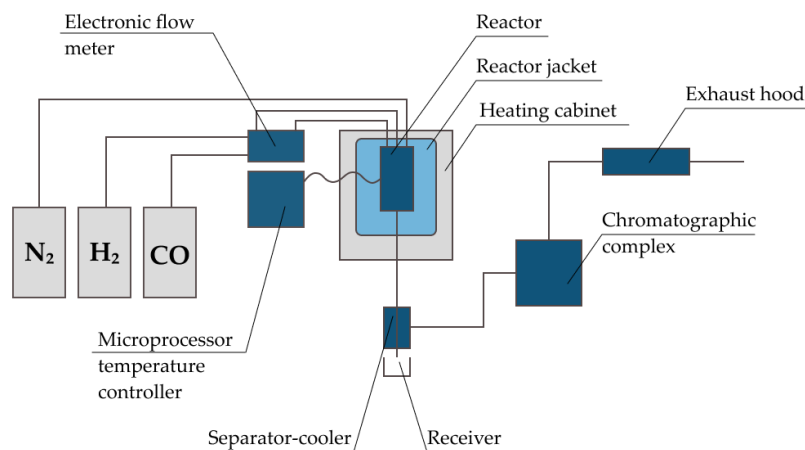


Figure 10. Laboratory unit diagram.

The reactor is a plug-flow, fixed-bed reactor. Granulated catalyst is loaded into the reactor through a top inlet, after the top flange has been removed. Temperature control is achieved using two thermocouples. The top of the evaporator serves as a reactant mixer, while the bottom prevents condensation of the liquid products immediately at the reactor outlet. Using electric current, the furnace transfers heat through a copper bandage to the reaction space, which is enclosed in housing along with the evaporator. The reactor is enclosed in a layer of thermal insulation. The grate serves to retain the catalyst granules within the reactor (Figure 11).

Carbon monoxide and hydrogen, passing through an electronic flowmeter, which serves as a dosing unit, mix and enter the reactor, located in a thermo-cabinet. The thermo-cabinet maintains a high temperature to prevent the products exiting the reactor from clogging the tube if their mixture has a sufficiently high viscosity at room temperature. The gas-product mixture exits the reactor, is cooled in a tubular cooler, and is sent for separation to the separator cooler. The gas from the separator is fed to a gas chromatograph for analysis, and the liquid portion is collected for analysis in a receiver. The temperature inside the catalyst bed, reactor jacket, and the air in the thermo-cabinet are controlled by the microprocessor temperature controller.

To determine the catalytic activity of the obtained catalyst samples, the main process parameters were varied, namely, the temperature in the catalyst bed (T), the process pressure (P), the feedstock space velocity (FSV) (by changing the amount of synthesis gas supplied to the reaction zone), and the ratio of reagents in the synthesis gas. The variation limits were T from 230 to 330 °C, P from 0.3 to 1.2 MPa, the FSV from 5 to 15 min^{−1}, and the ratio of reagents H₂:CO from 1:1 to 2:1.

Each experiment designation begins with the catalyst identification (Co), followed by the process temperature, pressure, and feedstock space velocity. Each individual experiment was conducted for 1 h only after the setup's operating conditions had been established. No preliminary catalyst reduction was performed, but drying was performed in a nitrogen stream at 140 °C.

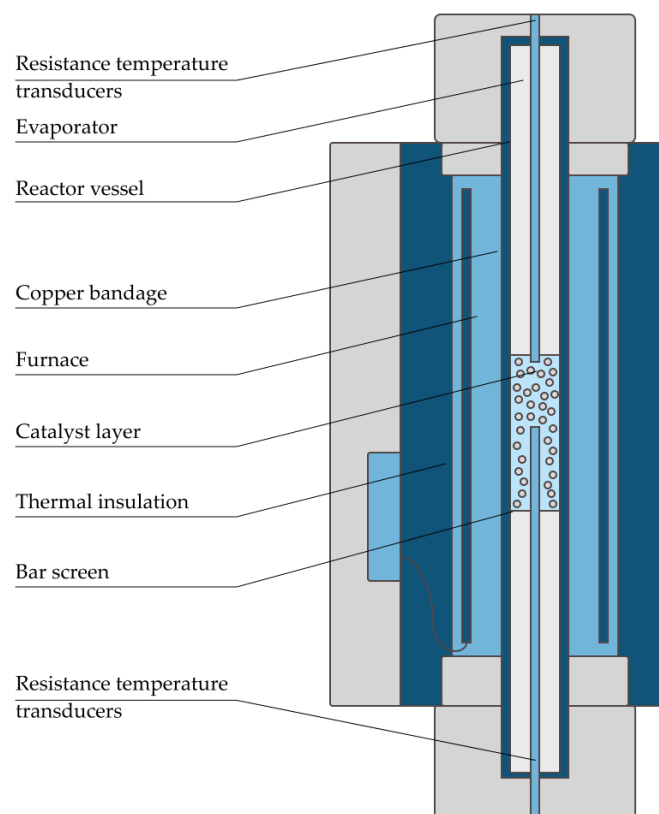


Figure 11. Reactor configuration.

The efficiency of the catalyst in the Fischer–Tropsch synthesis was assessed by the following parameters:

CO Conversion (CCO), %—the proportion of reacted CO from that passed through the reactor:

$$CCO = ((MCO_{in} - MCO_{out}) \cdot 100\%) / MCO_{in} \quad (2)$$

where MCO_{in} and MCO_{out} —the amount of CO in grams fed to the reactor and the amount of CO contained in the exhaust gas, respectively.

Catalyst capacity (C_{C5+}), $kg/(m^3 \cdot h)$ —the amount (kg) of liquid hydrocarbons formed from the passed gas (M_{C5+}) per time unit (h) on volume of catalyst unit (m^3) ($V_{kat \cdot h}$), $kg_{C5+}/m^3 kat/h$:

$$C_{C5+} = M_{C5+} / V_{kat \cdot h} \quad (3)$$

C_{5+} hydrocarbon formation selectivity (S_{C5+}), %—the number of carbon atoms in the reacted CO ($CCO_{in} - CCO_{out}$), used to form C_{5+} hydrocarbons:

$$S_{C5+} = C_{C5+} 100\% / (CCO_{in} - CCO_{out}) \quad (4)$$

Selectivity of methane formation (S_{CH4}), %—the number of carbon atoms in the reacted CO ($MCO_{in} - MCO_{out}$), that went into the formation of methane:

$$S_{CH4} = M_{CH4} 100\% / (MCO_{in} - MCO_{out}) \quad (5)$$

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Appendix A

Table A1. Results of the experiment.

No.	T	P	FSV	H ₂ :CO	CO Conversion	CO ₂ Yield	CH ₄ Yield	CH ₄ Selectivity	C ₂ –C ₄ Selectivity	Proportion of C ₅₊ in Liquid Phase
	°C	MPa	min ^{−1}					%		
1	230	9	7.5	2:1	23.890	0.000	0.653	6.982	2.716	86.235
2	240	9	7.5	2:1	23.980	0.125	1.152	10.083	4.131	85.766
3	250	9	7.5	2:1	25.879	0.373	2.280	15.225	6.023	84.761
4	260	9	7.5	2:1	27.136	0.886	3.482	18.506	8.764	83.987
5	270	9	7.5	2:1	32.683	2.745	7.870	32.988	14.469	78.744
6	280	9	7.5	2:1	36.814	3.680	9.886	32.999	22.251	75.329
7	290	9	7.5	2:1	60.730	8.068	23.225	51.554	27.878	22.394
9	310	9	7.5	2:1	77.971	11.941	32.747	47.866	18.186	19.622
10	320	9	7.5	2:1	87.130	14.164	38.479	43.576	13.755	61.146
11	330	9	7.5	2:1	97.840	16.932	50.614	42.774	8.984	70.916
12	270	3	7.5	2:1	25.810	1.074	4.224	33.568	14.058	89.034
13		6	7.5	2:1	27.688	2.044	6.442	33.906	15.411	74.908
14		12	7.5	2:1	36.631	3.630	9.734	22.648	9.114	72.509
15		9	5.0	2:1	26.005	1.376	5.023	16.261	5.578	89.109
16	270	9	10	2:1	23.572	1.065	4.350	22.229	6.904	74.007
17		9	15	2:1	18.094	0.444	2.867	20.955	6.366	80.625
18		9	7.5	1:1	44.024	1.823	6.367	22.754	12.304	82.919
19	270	9	7.5	1.25:1	19.142	0.348	2.762	16.273	4.631	88.286
20		9	7.5	1.5:1	15.975	0.499	3.153	20.250	5.716	86.349

Table A2. Results of the experiment under optimal technological conditions for 30 h.

No.	T	P	FSV	H ₂ :CO	CO Conversion	CO ₂ Yield	CH ₄ Yield	CH ₄ Selectivity	C ₂ –C ₄ Selectivity	Proportion of C ₅₊ in Liquid Phase
	°C	MPa	min ^{−1}					%		
1	230	9	7.5	2:1	23.685	0.000	0.663	6.994	2.875	86.135
2	240	9	7.5	2:1	23.612	0.098	1.163	10.113	4.236	85.625
3	250	9	7.5	2:1	25.723	0.353	2.310	15.845	6.125	84.658
4	260	9	7.5	2:1	27.100	0.844	3.511	18.941	8.963	83.873

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