



SYNTHESIS AND PROPERTIES STUDY OF COBALT SILICA GEL CATALYSTS

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Abstract: KSKG brand silica gel produced by "Salavat Catalyst Plant" OJSC was selected as a carrier for the catalyst. the composition and parameters of the developed porous catalyst structure corresponded to GOST 3956-76. According to the manufacturer, the pore volume of the carrier is 0.76-1.0 cm³/g, the average pore diameter is 12-16 nm, and the specific surface area is 360-370 m²/g. The nitrate form of the cobalt salt was used as a layer of the oxide form of the active component of the catalyst. The formation of active centers of cobalt catalysts occurs at all stages of preparation and depends on the chemical nature of the initial cobalt compounds, the carrier, the presence of promoters, the conditions for converting the catalyst into oxide forms, and its activation in the reaction medium. The composition of the active component, the effect of preparation, drying and calcination conditions have a significant effect.

Key words: synthesis gas, catalyst, pressure, conversion, space velocity, temperature.

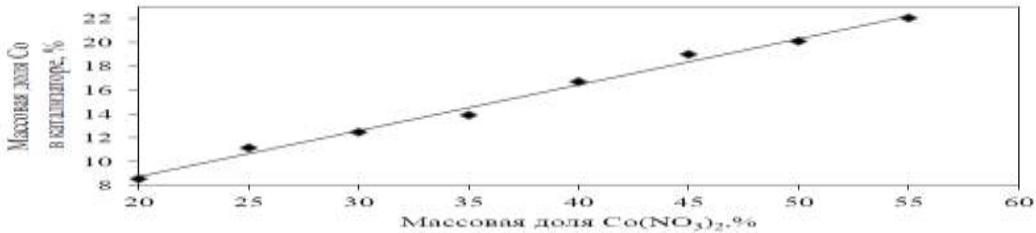
I.INTRODUCTION

Katalizator tarkibidagi kobalt tarkibining uning xossalariiga ta'sirini aniqlash uchun faol komponent miqdori og'irligi 8,5-22,0% oralig'ida katalizatorlar tayyorlandi. Kiritilgangan katalizatordagi kobalt miqdorining singdiruvchidagi kobalt miqdori konsentratsiyasiga bog'liqligi (3.1-rasm) (3.1) tenglamaga mos kelishilgan aniqlandi, korrelyatsiya koeffitsienti 0,99.

$$CCo = 0,39 \cdot CCo(NO_3)_2 + 1,06 \quad (3.1)$$

where CCO is the amount of cobalt in the catalyst, % by mass; C Co(NO₃)₂ is the mass fraction of cobalt nitrate, %.

This equation is valid in the range of 8-22% cobalt concentration in the catalyst.



Figure

3.1 - Dependence of Co in the catalyst on the concentration of cobalt nitrate in the impregnating solution.

The activity of the catalysts was determined (volume %) for the synthesis of hydrocarbons from CO and H₂ at a pressure of 0.1 MPa, OSG 100 h⁻¹ using synthesis



gas of the composition: CO - 33.3; H₂ - 66.7. The effect of cobalt content on the main catalytic properties of FT synthesis is presented in Table 3.1 and Figure 3.2.

Table 3.1 - Properties of catalysts in the process of hydrocarbon synthesis

CO content, %	Temperature, °C		Conversion rate of CO, %		Selectivity, %	
	CH ₄		C ₂ -C ₄		C ₅₊	
8,6	215	35,0	15,8	13,6	68,5	2,1
12,5	200	37,1	12,5	9,7	76,0	1,8
16,7	195	42,0	10,8	9,6	78,1	1,5
20,0	185	51,4	9,1	7,8	82,0	1,1
22,0	185	50,1	10,8	9,2	79,0	1,0

An increase in the cobalt content from 8 to 22% leads to a significant decrease in the synthesis temperature by 30 °C, while the CO conversion rate increases by 15%. As the synthesis temperature decreases, the selectivity for C₅₊ hydrocarbons naturally increases and the selectivity for C₁-C₄, CO₂ hydrocarbons decreases.

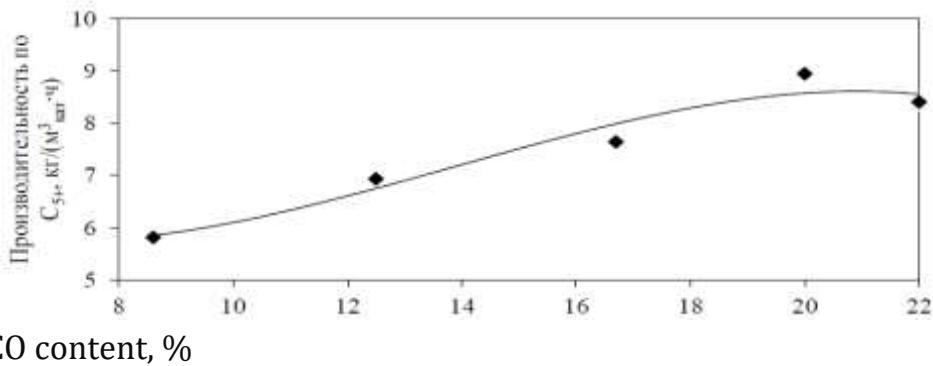


Figure 3.2 - Catalyst performance for C₅₊ hydrocarbons as a function of cobalt content in the catalyst

Catalyst performance for C₅₊ hydrocarbons increases and passes through a maximum at 20% cobalt. A further increase in the amount of cobalt in the catalyst leads to a decrease in selectivity and productivity for C₅₊ hydrocarbons, which is apparently related to a change in the dispersion of the active component [154, 155].

Thus, it was determined that the optimal amount of the active component in the cobalt-silica gel catalyst on the KSKG carrier is 17-20% by weight.

3.1.2 Effects of heat treatment regimes

To determine the effect of the heat treatment modes of the catalysts, the samples were calcined at the following temperatures: 200, 250, 300, 400, 500 °C; the corresponding catalysts are designated as Co/SiO₂(t).

The resulting catalyst samples were characterized by XPA, BET, thermally programmed reduction (TPR) and thermally programmed CO desorption (CO TPD). All samples of the catalyst are characterized by a very close amount of cobalt, which is 20% by mass.

The results of the study of samples by the TPV method are presented in Figure 3.3 and Table 3.2.





For Co/SiO₂ catalyst samples with final calcination temperatures of 200, 250, and 300 °C, weak peaks between 150–200 °C are observed, which may be attributed to the reductive decomposition of residual cobalt nitrate. Co/SiO₂ (400 °C) and Co / SiO₂ (500 °C) samples do not have such peaks, which indicates almost complete decomposition of cobalt nitrate at final processing temperatures of 400 and 500 °C. In addition to the defined low temperature peak, in the region of 280-650 °C, two intense recovery peaks are observed with a maximum of 300-330 °C and 370-470 °C, which are then designated as signal 1 and signal 2., respectively (Table 3.2).

These hydrogen acceptance peaks can be attributed to the gradual reduction of the Co₃O₄ cobalt oxide phase according to equations 3.1-3.2 [156, 157].

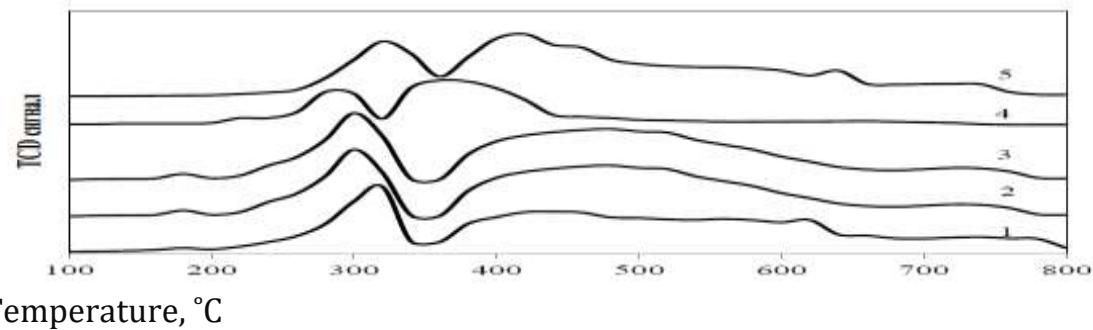


Figure 3.3 - TPR spectra of catalysts: 1 - Co/SiO₂ (200); 2 - Co/SiO₂ (250); 3- Co/SiO₂(300); 4 - Co/SiO₂ (400); 5 - Co/SiO₂(500)

With an increase in the calcination temperature of the catalysts (Table 3.2), the ratio S₂/S₁ of the signal areas of 2 and 1 increases, approaching a value equal to 3, the theoretically expected value from stoichiometry (equations 3.1-3.2). reduction of cobalt oxides. The lower values of S₂/S₁ ratio for samples with final calcination temperature lower than 400 °C can undoubtedly be attributed to the complete oxidation of cobalt at low calcination temperatures.

Table 3.2 - TPR spectra of catalyst samples depending on calcination temperature

Ignition temperature, °C		Signal 1		Signal 2		S ₂ /S ₁	
maksimal, °C		area S ₁ , %		maximum, °C		area S ₂ , %	
200	303	28		474	72	2.5	
250	317	30		440	70	2.4	
300	318	27		430	73	2.7	
400	295	26		370	74	2.9	
500	328	26		416	74	2.9	

The X-ray image for all samples of catalysts is the same (Fig. 3.4). SiO₂ is X-ray amorphous, cobalt catalysts contain the crystalline phase of Co₃O₄ oxide (000-09-0418). The calculated unit cell parameters of Co₃O₄ are about 8.08 Å.



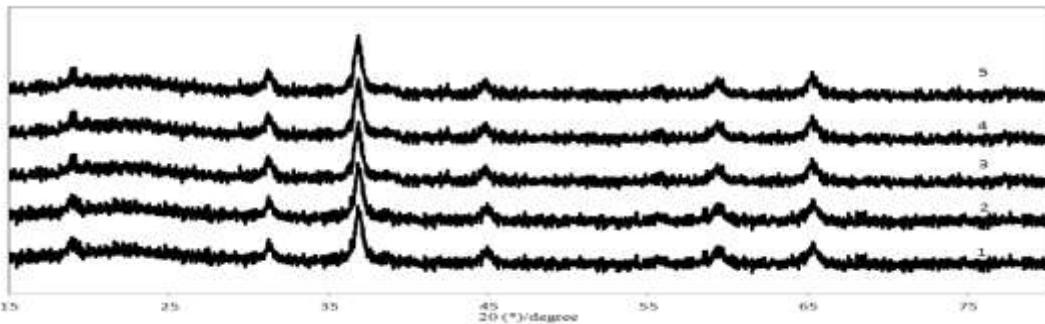


Figure 3.4 – Diffractograms of catalysts: 1 - Co/SiO₂(200); 2 - Co/SiO₂(250); 3- Co/SiO₂(300); 4 - Co/SiO₂(400); 5 - Co/SiO₂(500)

The catalytic activity of all samples was studied in the temperature range of 160–195 °C (synthesis temperature increased to the values of CO conversion level of about 50%), OSG 100 h-1 and pressure of 0.1 MPa. The obtained results are presented in Table 3.3. Table 3.3 - Catalytic properties of Co/SiO₂ catalyst samples with different final calcination temperatures

Ignition temperature, °C		Synthesis temperature, °C		Conversion rate of CO, %		Selectivity, %	
CH4	C2-C4			C5+		CO2	
200	190	48,8		14,5	14,5	69,5	1,5
250	195	45,8		16,8	18,0	63,6	1,6
300	180	52,1		7,5	7,1	83,8	1,6
400	180	52,2		5,2	5,6	88,7	1,5
500	180	52,5		5,4	5,4	87,0	2,2

Samples calcined at low final temperatures of 200 and 250 °C showed low activity in FT synthesis. Thus, the rate of conversion to CO at 190 °S in the Co/SiO₂(200 °S) catalyst was 48.8%, and only 45.8% at 195 °S for Co/SiO₂ (250 °S). An increase in gas formation (S1-S4) is observed with an increase in the synthesis temperature, and as a result, the selectivity for S5+ hydrocarbons decreases. Calcination of catalyst samples in the range of 300-500 °C increases their catalytic activity: even at 180 °C, the conversion rate of CO in such samples exceeds 50%. The selectivity for C5+ hydrocarbons is also high in catalysts calcined at a temperature of 400–500 °C. This indicates that more polymerization sites are formed on the surface of the catalytic systems. The calcination temperature has a significant effect on the physico-chemical and catalytic properties of the supported catalysts, and it should be in the range of 400-500 °C for the developed catalyst.

3.1.3 Effect of Al₂O₃ promoter addition on properties of cobalt catalysts

Promotion plays an important role in the preparation of efficient catalysts for FT synthesis [158–163]. The introduction of promoters helps to increase the activity of the catalysts and the selectivity towards the target fractions of hydrocarbons. Information on the use of aluminum as a hydrocarbon synthesis catalyst promoter in the scientific and patent literature is very limited. The use of aluminum additives in the formation of a catalyst on a silicate support is described in separate works, but they





are included at the stage of its preparation; to change the support [164-166]. Therefore, we studied the effect of Al₂O₃ stimulation on the fineness of the active component, the composition and properties of the Co-Al₂O₃ / SiO₂ system. Aluminum oxide in its nitrate form is introduced in the step of impregnating the catalyst with cobalt nitrate. The content of aluminum oxide varied in the range of 0-10% mass fractions. The obtained samples corresponded to the general formula Co-xAl₂O₃/SiO₂. Mass fractions x = 0,4, 1, 2, 3, 4, 6 and 10% in catalysts doped with Al₂O₃. XRD, BET, TEM, SEM, TPR and CO TPD methods were used to characterize calcined and reduced Co/SiO₂ and Co-xAl₂O₃/SiO₂ catalysts. Comparison of the data on the composition of calcined catalysts (Table 3.4) and the parameters of the pore structure (Table 3.5) made it possible to determine the significant influence of the composition of aluminum additives on the properties of the Co-xAl₂O₃/SiO₂ system. Catalyst Co/SiO₂ and catalysts - the amount of cobalt in Co-Al₂O₃/SiO₂ with 0,4-3% Al₂O₃ is in the range of 20,9-22,8%. The samples are very close in specific surface area - about 230-240 m² / g.

Table 3.4 - The content of cobalt in the catalyst and the size of its crystallites

Catalyst		Mass fraction of Co, %		Crystallite size, nm
RFA			PEM	
Co3O4		Co		Co
Co/SiO ₂	21,9	20	15,0	12,4
Co-0,4Al ₂ O ₃ /SiO ₂	20,9	21	15,8	8,0
Co-1Al ₂ O ₃ /SiO ₂	22,8	20	15,0	8,0
Co-2Al ₂ O ₃ /SiO ₂	21,1	19	14,3	10,7
Co-3Al ₂ O ₃ /SiO ₂	21,1	20	15,0	-
Co-4Al ₂ O ₃ /SiO ₂	19,9	20	15,0	-
Co-6Al ₂ O ₃ /SiO ₂	19,7	19	14,3	-
Co-10Al ₂ O ₃ /SiO ₂	15,4	20	15,0	-

Increasing the content of Al₂O₃ to 4-6 and 10%, especially with a solution saturated with active components, leads to a change in the nature of the carrier impregnation process. The amount of cobalt in the catalysts decreases to 19,7-19,9 and 15,4%, the value of the specific surface of the catalysts changes. For catalysts without additives and containing 0,4-3% Al₂O₃, the specific surface area does not exceed 239 m² / g, with an amount of 4-6% Al₂O₃ - it increases significantly. Co-10Al₂O₃/SiO₂ catalyst has the largest specific surface area of 297 m²/g and the smallest pore radius of 8,2 nm. In this case, the decrease in the pore size of the support due to the supported active component can be compensated by the appearance of additional porosity due to the accumulation of a mixture of cobalt and aluminum oxides on the surface of the catalyst [167].

Table 3.5 - BET characteristics of Co-xAl₂O₃/SiO₂ catalysts



Catalyst	Surface area, m ² /g	Hole diameter, nm	Pore volume, cm ³ /g
Co/SiO ₂	236	11,2	0,66
Co-0,4Al ₂ O ₃ /SiO ₂	232	10,9	0,63
Co-1Al ₂ O ₃ /SiO ₂	239	11,0	0,66
Co-2Al ₂ O ₃ /SiO ₂	227	11,2	0,64
Co-3Al ₂ O ₃ /SiO ₂	227	10,4	0,59
Co-4Al ₂ O ₃ /SiO ₂	245	10,5	0,64
Co-6Al ₂ O ₃ /SiO ₂	255	10,7	0,68
Co-10Al ₂ O ₃ /SiO ₂	297	8,2	0,61

For all samples - without additives and with different composition of Al₂O₃ calcined at a temperature of 350 ° C, the radiographic pattern is the same (Fig. 3.5).

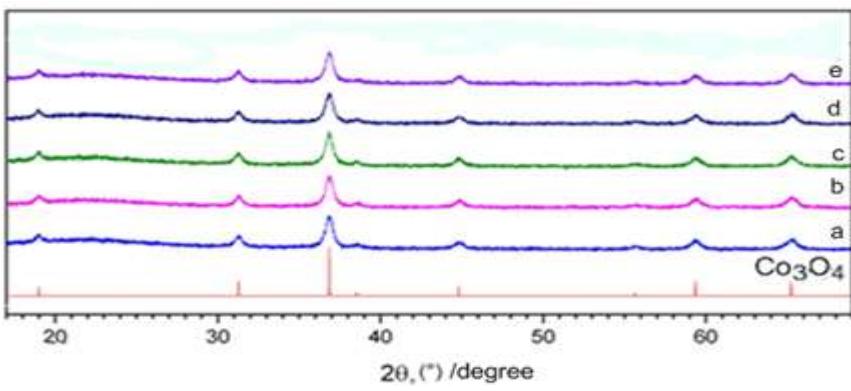
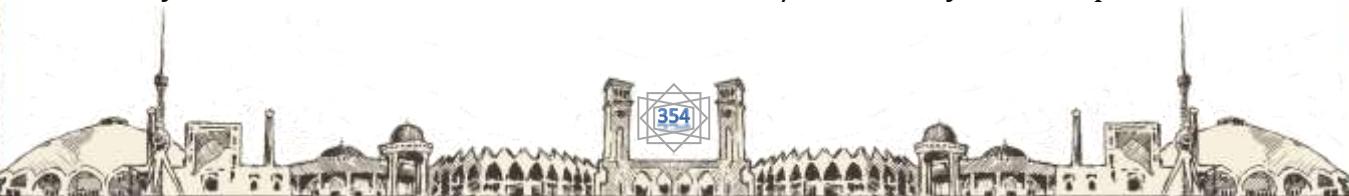


Figure 3.5 - Diffractograms of catalysts: - Co/SiO₂, b - Co- 0.4Al₂O₃/SiO₂, c - Co-1Al₂O₃/SiO₂, d - Co-2Al₂O₃/SiO₂, e - Co-3Al₂O₃/SiO₂

SiO₂ is X-ray amorphous, cobalt catalysts contain a series of lines of the Co₃O₄ oxide crystal phase (000-09-0418). The calculated unit cell parameters of Co₃O₄ are about 8.08 Å. The size of Co₃O₄ crystallites varies in the range of 19-21 nm (Table 3.4), there are no significant deviations due to changes in the composition of the components in the catalyst.

Figure 3.6 shows TEM images and size distribution diagrams of cobalt crystallites for catalysts with different Al₂O₃ doped contents.

Particles with a size of 5-20 nm, mostly 9-15 nm, were recorded on the surface of the Co/SiO₂ catalyst. The most important number of particles in the size range of 10-13 nm. Samples with Co-2Al₂O₃/SiO₂ catalyst and high additive content are characterized by a wide distribution of cobalt crystallite sizes. Among the 6-12 nm particles in the Co-2Al₂O₃/SiO₂ catalyst, most are 8-14 nm, mainly 9-12 nm particles. The maximum number of particles with a size of 9 nm. The most active synthesis catalysts supplied with 0.4-1% Al₂O₃ have 5-12 nm particles on their surface. The distribution diagrams (Fig. 3.6 b, c) have a clear maximum corresponding to the cobalt crystallite size of 8 nm. Among catalyst particles containing 0.4 Al₂O₃, the share of crystallites with a size of 9-11 nm is slightly higher. The maximum size distribution of cobalt crystallites on the surface of the Co-1Al₂O₃/SiO₂ catalyst corresponds to 8 nm.





The results of TEM studies do not completely match the results of XRF due to the peculiarities of the method, but they are in good agreement with the materials presented in reviews [168, 169] and a number of other works [170, 171].

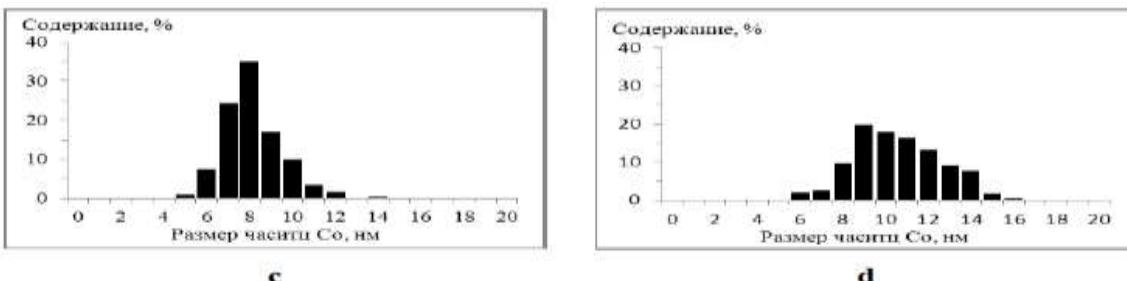


Figure 3.6 - TEM micrograph of reduced catalysts and histogram of cobalt particle size distribution: a- Co/SiO₂, b - Co - 0.4Al₂O₃/SiO₂, c - Co - 1Al₂O₃/SiO₂, d - Co - 2Al₂O₃ / SiO₂

Summary

In the thesis, a series of studies aimed at the development of technology for the synthesis of cobalt catalyst and higher hydrocarbons C35+ from CO and H₂ were carried out. The developed catalyst is characterized by increased (1.8 times) selectivity for high C35+ hydrocarbons (ceresine). It was found that alumina can be used as a cobalt-silicate gel catalyst promoter, which allows for increased selectivity and productivity for C5+ hydrocarbons.

A systematic study of the synthesis of C35+ higher hydrocarbons using the developed catalyst made it possible to determine the optimal technological parameters (temperature, pressure, space velocity and synthesis gas composition). It has been shown that nitrogen-diluted synthesis gas can be used in high C35+ hydrocarbon technology.

For a pilot industrial reactor for the synthesis of hydrocarbons, its stable operating range is determined. In order to increase the heat exchange in tubular reactors, it is proved theoretically and experimentally that it is necessary to carry out the synthesis in the flow-circulation mode. Thus, the gas load for a flow-through pilot industrial reactor can be increased 10 times while maintaining the quasi-isothermal mode of its operation.

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