The Austrian Journal of Technical
and Natural Sciences, No 5–6
ISSN 2310-5607

PREMIER
Publishing
ppublishing.org

DOI:10.29013/AJT-25-5.6-74-77

STUDY OF CATALYSTS PREPARED BY IMPREGNATION WITH AQUEOUS COBALT NITRATE SOLUTION

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Cite: *Togayev A.I. Kobilov N.S. (2025). Study of Catalysts Prepared by Impregnation With Aqueous Cobalt Nitrate Solution. Austrian Journal of Technical and Natural Sciences 2024, No 5–6.* https://doi.org/10.29013/AJT-25-5.6-74-77

Abstract

The article considers the catalyst production stage – a gradual increase in the synthesis temperature in a flow of synthesis gas – as a necessary stage of the Fischer-Tropsch synthesis, since during the development process it was determined that the formation of catalytically active centers under the influence of the catalyst was observed. It can be assumed that in this temperature range, water is released from the capillaries of the binder – aluminum oxide and micropores of H-shaped zeolites. In the presence of cationic zeolites, such a plateau is absent due to the hydrophilicity of such zeolites, in which water is retained near the cations under the influence of electrostatic forces.

Keywords: *cation exchange resin, catalyst, Fischer-Tropsch synthesis, conversion, capillary condensation, zeolites, selectivity*

Introduction

In an era of developing modern technologies, one of the main directions of the chemical industry is the creation of effective catalysts and the study of their active properties. Catalysts are of great importance in increasing the speed of chemical reactions, reducing energy consumption, and introducing environmentally friendly technologies. Today, various catalytic systems are widely used in many industries, in particular in the fields of oil and gas chemistry, pharmaceuticals, and environmental protection. In particular, cobalt-based catalysts are distinguished by their high activity and stability (Rakhmatov et al., 2023).

We will talk about the preparation of catalysts using an aqueous solution of cobalt nitrate and the analysis of their physicochemical properties. Scientific research in this area is carried out using the impregnation (i.e., wetting) method, since this method allows the active component to be evenly deposited on the surface of the base material (for example, aluminum oxide, silicon dioxide, etc.) (Togayev, 2023).

Cobalt-based catalysts are widely used in many reactions, such as oxidation, hydrogenation, and Fischer-Tropsch synthesis. Such catalysts are mainly prepared using cobalt nitrate salts. One of the most commonly used methods is the soaking method, in which the preparation process is as follows: first, a solution of cobalt(II) nitrate is prepared in water, then it is poured onto a specially selected support substance and mixed. As a result, cobalt ions are impregnated on the surface of the support. Then, through the processes of drying and calcination (i.e. heating), the final form of the catalyst is obtained (Rakhimov, 2024).

The efficiency of the catalyst depends on several factors: its surface area, cobalt content, particle size, heat resistance and chemical stability. Modern physicochemical analysis methods – for example, BET (surface characterization), XRD (X-ray diffraction), SEM (scanning electron microscopy) – allow the structure of the catalyst to be studied in depth. Studies show that correctly selected impregnation conditions (pH, concentration, time) allow obtaining catalysts with high activity.

Cobalt catalysts also play an important role in environmental protection. For example, cobalt-based catalysts are used in reactors that decompose harmful substances in automobile exhaust gases and in systems that reduce NOx oxides. In addition, they are widely used in biofuel production and exhaust gas purification.

Catalysts prepared by the impregnation method using cobalt nitrate solution are of great importance in the industrial and environmental sectors. If their physicochemical properties are thoroughly analyzed and effective reaction conditions are determined, even higher results can be achieved. In the future, this type of research will serve as an important basis for creating sustainable and energy-efficient technologies (Rakhimov, 2024).

Metod and material

In this scientific study, catalysts were prepared by the impregnation method using an aqueous cobalt(II) nitrate solution. Various support materials were used for catalyst preparation, namely zeolite-based supports (NaX, KA, CaA and H-form zeolites: HY6, HY30, HB) and granular supports without zeolite.

The impregnation method was carried out as follows: an appropriate amount of cobalt nitrate solution was used for each support. In the preparation of the solution, analytically pure $Co(NO_3)_2 \cdot 6H_2O$ was dissolved in distilled water and then poured onto a zeolite or other granular support and uniformly impregnated. During the impregnation process, the mixture was continuously stirred at room temperature for 3–4 hours. After that, the wet samples were dried at 100 °C and calcined in a muffle furnace at 450 °C for 4 hours. During the calcination stage, cobalt salts decompose and turn into active metal or oxide forms.

Results

The effect of synthesis temperature

It is known that the catalyst production stage – a gradual increase in the synthesis temperature in a stream of synthesis gas – is a necessary stage of the Fischer-Tropsch synthesis, since during the development process catalytically active centers are formed under the influence of. Studies by the authors (Rakhmatov, 2023) showed that during the development of a cobalt catalyst, a change in the state of cobalt occurs: under the influence of the reaction medium, part of CoO is converted to $Co^{\delta+}$. This leads to a change in the nature of CO adsorption. Thus, the active surface of the catalyst is formed during the production process.

In the course of development, the dependence of the main synthesis parameters on temperature was obtained for each catalyst.

Figure 1 shows the dependence of CO conversion on the synthesis temperature in the presence of impregnation catalysts. With increasing temperature, the conversion of CO increased in the presence of all catalysts. The highest CO conversion – 80% – was obtained in the presence of a catalyst with HB zeolite. It is interesting that in the dependence obtained with the presence of Co/HB, Co/HY6 Co/HY30 and without a zeolite catalyst, a "step" was observed when the synthesis temperature increased from 190 to 210 °C, probably due to capillary condensation of the synthesis products. It can be assumed that in this temperature range, water is released from the capillaries of the binder – aluminum oxide and micropores of H-shaped zeolites. In the presence of cationic zeolites, such a plateau is absent due to the hydrophilicity of such zeolites, in which water is retained near the cations under the influence of electrostatic forces. The lowest CO conversion was obtained in the presence of a KA zeolite-based absorption catalyst – no more than 20% over the entire temperature range.

Figure 2 shows the dependence of the selectivity for the formation of C_{5+} hydrocarbons on the synthesis temperature. In the presence of all catalysts, this indicator decreased with increasing temperature. In the presence of catalysts without zeolites and catalysts containing HB and HY30 zeolites, the selectivity for the formation of C_{5+} hydrocarbons decreased by 1.1–1.2 times in the temperature range 190–210 °C. In the presence of catalysts based on Co/CaA, Co/NaX cationic zeolites, a decrease of this indicator by 1.5 and 1.3 times, respectively, was observed when increasing from 210 to 220 °C. In addition, in the temperature range 210–240 °C, the selectivity for the formation of C_{5+} hydrocarbons was practically independent of the synthesis temperature in the presence of all catalysts except Co/CaA and Co/NaX.

Figure 1. *Dependence of CO conversion on FTS temperature*

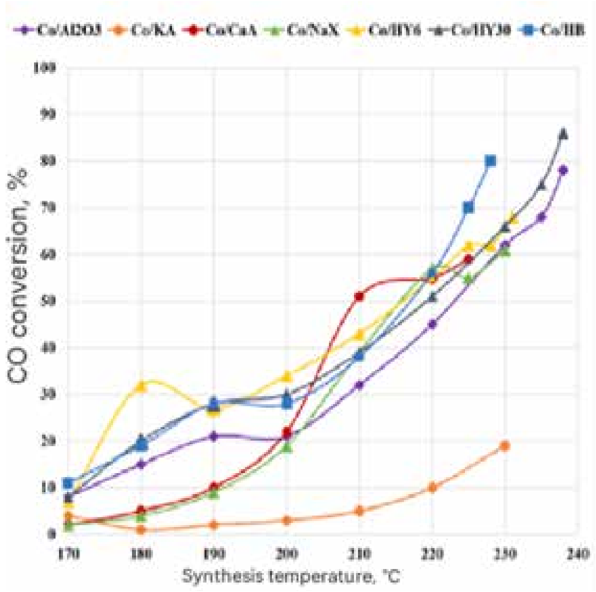

Figure 2. *Dependence of the selectivity of the formation of C_{5+} hydrocarbons on the FTS temperature*

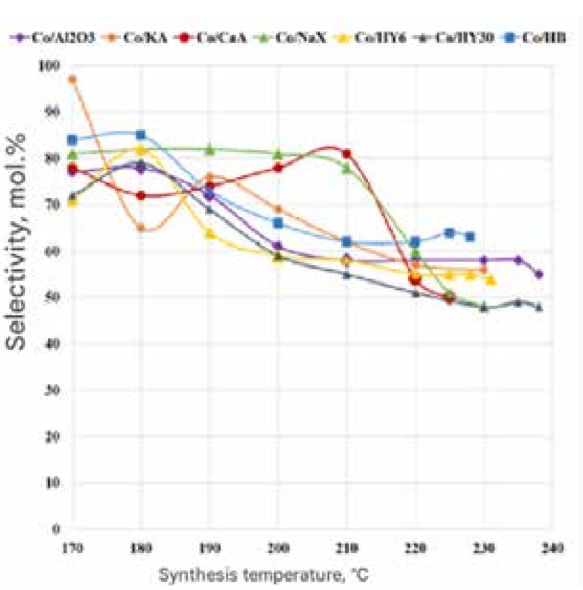

Figure 3 shows the dependence of the yield of C_{5+} hydrocarbons on the synthesis temperature. In the presence of all catalysts, the yield of liquid hydrocarbons increased with increasing synthesis temperature. In the presence of catalysts based on H-forms of zeolites and catalysts without a zeolite, a step was observed at 180–200 °C. The highest yield of C_{5+} hydrocarbons – more than 97 g/m^3 – – was obtained in the presence of a catalyst based on HB zeolite. In the presence of catalysts based on zeolites cationized with alkali and alkaline earth metals, the yield of C_{5+} hydrocarbons was significantly lower and amounted to 58 g/m^3 in the presence of Co/NaX and Co/CaA, and 21 g/m_3 in the presence of Co/KA.

The selectivity of methane formation increases with increasing synthesis temperature in the presence of all catalysts. However, the nature of this dependence is somewhat different. In the presence of a zeolite-free catalyst and catalysts based on H-form zeolites, this dependence reaches a maximum at 210–230 °C, which corresponds to 25–30%. In the presence of catalysts based on zeolites cationized with Na and Ca, the selectivity of methane formation increases sharply – by 2.3– –3.6 times – with an increase in temperature from 210 to 220 °C. In the presence of Co/CA, this indicator increases from 1 to 20%, in parallel with an increase in the Fischer-Tropsch synthesis temperature.

Thus, despite the literature data on the sharp increase in methane production when using zeolites as carriers compared to conventional Fischer-Tropsch synthesis catalysts, the selectivity of the catalyst with HB zeolite is lower than that of the catalyst without zeolite over the entire temperature range.

Conclusion

The results of this study showed that the composition of cobalt catalysts prepared by the impregnation method, especially with or without zeolite, significantly affects their activity and selectivity. This confirms that the formation of catalytic active sites and the process of CO adsorption on the reaction surface depend on the nature of the support material.

Also, as a result of a gradual increase in the synthesis temperature, a partial conversion of CoO to the Co^δ+ form was observed, which is an important factor in the formation of the active surface of the catalyst. Although zeolites have a high surface area and ion exchange capacity, they do not always guarantee high selectivity. In particular, the low selectivity of HB-zeolite indicates that the optimal location of the active sites and metal dispersion may not be sufficiently ensured.

The results indicate that in the development of cobalt-based catalysts, a thorough analysis of the properties of the support material, optimization of synthesis conditions, and consideration of the effect of the reaction medium are important in increasing efficiency.

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submitted 17.04.2025;
accepted for publication 01.05.2025;
published 29.05.2025

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