

SYNTHESIS OF ALUMINIUM-CHLOROORGANIC COMPLEX BASED ON ALUMINIUM AND 1,2-DICHLOROETHANE AND ITS INVESTIGATION IN THE OLIGOMERISATION PROCESS OF ALPHA-OLEFINS

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Abstract: This article presents the synthesis of an aluminium–chloroorganic complex via the chlorination of metallic aluminium with 1,2-dichloroethane in a solvent medium, as well as the results of the oligomerisation of alpha-olefins in the presence of this catalyst. During the catalyst synthesis, a straight-run gasoline fraction with a boiling range of $\geq 85\text{--}172\text{ }^{\circ}\text{C}$ was used as the solvent. The use of this gasoline fraction accelerates the synthesis process, reducing the catalyst preparation time from 20–22 hours to 10 hours. At the same time, it enhances the fluidity of the catalyst, facilitates its introduction into the system, simplifies its application, and ensures high stable activity. Based on IR, DLS, and XPA analyses, it was determined that, in the presence of the gasoline fraction, the interaction between aluminum and 1,2-dichloroethane leads to the formation of a catalytic complex with the composition $\text{AlCl}_3\cdot\text{RAlCl}_2\cdot\text{R}_2\text{AlCl}$, consisting of nanodispersed clusters with an average size of 29.2 nm. In the oligomerization of olefins in the presence of this catalyst, the optimal conditions were established as follows: temperature – $60\text{ }^{\circ}\text{C}$ (for C_6 (1-hexene), $100\text{ }^{\circ}\text{C}$ (for C_8 (1-octene) and $\text{C}_6\text{--}\text{C}_8$ mixture); reaction time – 120 min; catalyst concentration – 1 wt%. It was determined that the obtained oligomeric products are characterized by a high viscosity index (115–130) and low pour point (-49 to $-52\text{ }^{\circ}\text{C}$).

Keywords: aluminium, 1,2-dichloroethane, straight-run gasoline, aluminium–chloroorganic complex, oligomerisation, hexene-1, octene-1

Introduction

In modern industry, the increasing demands for energy efficiency, extended equipment service life, and stability under extreme conditions have significantly intensified the need for synthetic base oils. In this regard, polyalphaolefins (PAOs) are considered one of the key components replacing mineral oils due to their superior properties, including a high viscosity index, low pour point, excellent thermo-oxidative stability, and chemical inertness [1-4].

One of the principal routes for PAO production is the oligomerization of α -olefins in the presence of cationic-type catalysts. In this process, catalysts such as AlCl_3 , BF_3 , and their complexes with alcohols or water have been traditionally widely employed. In recent years, however, intensive research has focused on ionic liquids, heterogeneous acid catalysts, and modified Lewis acids to achieve more selective and controllable processes. The cationic oligomerization mechanism is characterized by the formation of carbocationic intermediates. The reaction is initiated by the interaction of the catalyst with the olefin molecule, leading to the formation of a π -complex, followed by carbocation generation. In subsequent stages, chain propagation, isomerization, and chain termination reactions occur, which directly influence the molecular weight distribution and structural characteristics of the final product. Therefore, the nature of the catalyst and the optimization of reaction conditions play a decisive role in the synthesis of PAOs with desired properties. One of the main advantages of cationic-type catalysts is their ability to enable reactions to proceed at low temperatures and under relatively mild conditions. However, these systems also exhibit certain drawbacks, including high corrosivity, difficulties associated with catalyst regeneration, and the potential formation of undesirable by-products in the reaction mixture. For this reason, modern

research is increasingly focused on the development of more environmentally friendly, selective, and stable catalytic systems [5-9].

As a result, the cationic oligomerization process remains a relevant field of study from both fundamental and applied perspectives. The development of next-generation catalyst systems, deeper investigation of the reaction mechanism, and optimization of process parameters will enable the production of higher-quality and more economically efficient synthetic lubricants in the future.

Catalytic complexes based on aluminum metal and chlorinated compounds have been synthesized for olefin oligomerization processes [10]. In the known method, aluminum metal is chlorinated with carbon tetrachloride at temperatures of 40–80 °C. The resulting catalytic complex allows the processes of polyisobutylene and butyl rubber production to be carried out at relatively low temperatures (–97 to –85 °C) with a 2–4-fold reduced catalyst consumption. However, during catalyst synthesis, by-products such as C₂Cl₆, C₂Cl₄, and C₂Cl₂ are formed, and their removal from the system requires washing with carbon tetrachloride. In addition, due to the exothermic nature of the reaction, the use of excess CCl₄ to regulate the process leads to increased economic costs.

A method for preparing a catalyst based on aluminum metal and 1,2-dichloroethane for olefin conversion is known [11-13]. Using this method, a catalytic complex capable of ensuring high olefin conversion in oligomerization, alkylation, and oligo-alkylation reactions has been obtained. The main drawback of this catalytic complex is the formation of a viscous, resin-like mass alongside the active catalytic centers during synthesis, which complicates its practical application. At the same time, the use of individual C₇–C₁₄ saturated hydrocarbons as solvents in the catalyst synthesis is not considered economically favorable.

Therefore, research efforts have been directed toward improving the properties of aluminum-based catalytic complexes and enhancing the economic efficiency of the synthesis process by replacing individual saturated hydrocarbons with straight-run gasoline (SRG) as a solvent. The present study reports the synthesis of an aluminum–chloroorganic complex (ACC) based on aluminum and 1,2-dichloroethane (DCE) using SRG as a solvent, as well as the results of oligomerization processes of hexene-1 (C₆), octene-1 (C₈) α -olefins and their mixtures in the presence of this catalyst.

Experimental part

The equipment required to carry out the synthesis of the catalyst based on aluminum and 1,2-dichloroethane, as well as the oligomerization of α -olefins under laboratory conditions, includes a mechanical stirrer, thermometer, reflux condenser, a three-neck round-bottom flask equipped with a dropping funnel, and a heating device.

The SRG used as a solvent in the catalyst synthesis was obtained from the primary oil refining process at the Heydar Aliyev Oil Refinery.

The composition of the SRG fraction and the composition of hydrocarbons evolved in the gaseous state during the interaction of Al with 1,2-DCE in the SRG medium were analyzed using an AutoSystem XL gas chromatograph (PerkinElmer) equipped with a flame ionization detector. For the analysis of organic compounds, a capillary column (100 m $\times$ 250 μ m $\times$ 0.5 μ m) with a 5% methylphenylpolysiloxane stationary phase was used. The injector temperature was 250°C, and the detector temperature was 280°C. Helium was used as the carrier gas at a flow rate of 1 mL/min.

The hydrocarbon composition of SRG was determined by chromatographic analysis, and the distribution of the hydrocarbon groups is summarized in Table 1.

Table 1. Group composition of hydrocarbons in SRG

Components	Total mass%
n-Paraffins	36.55
i-paraffins	44.70
Naphthenes	15.40
Aromatics	3.35

The particle size distribution of the catalyst was studied at 25 °C using the dynamic light scattering (DLS) method on a Horiba LB-550 instrument. The IR spectrum of the catalyst was recorded on an ALPHA Fourier spectrometer in the range of 400–4000 cm^{-1} , while the phase composition was analyzed using a TD-3500 diffractometer.

The pour point of the oligomerization product was determined according to the ASTM D2386 standard test method. Kinematic viscosity and viscosity index were determined by the viscometric method in accordance with GOST 25371-2018.

Results and discussion

To enhance the efficiency of the catalyst synthesis based on aluminum and 1,2-dichloroethane, an SRG fraction with a boiling range of ≥ 85 –172 °C was used as a solvent. In the synthesis of the ACC, the initial step involves removing the oxide layer from the aluminum surface. For this purpose, 0.2–0.3 wt.% of AlCl_3 , calculated based on the amount of aluminum metal, is used. The effects of temperature, component ratios, and reaction time on the ACC synthesis process were systematically investigated. The process was carried out within a temperature range of 30–80 °C and a duration of 1–15 hours, allowing the determination of optimal conditions. Fig. 1 illustrates the dependence of aluminum conversion on reaction time at different temperatures.

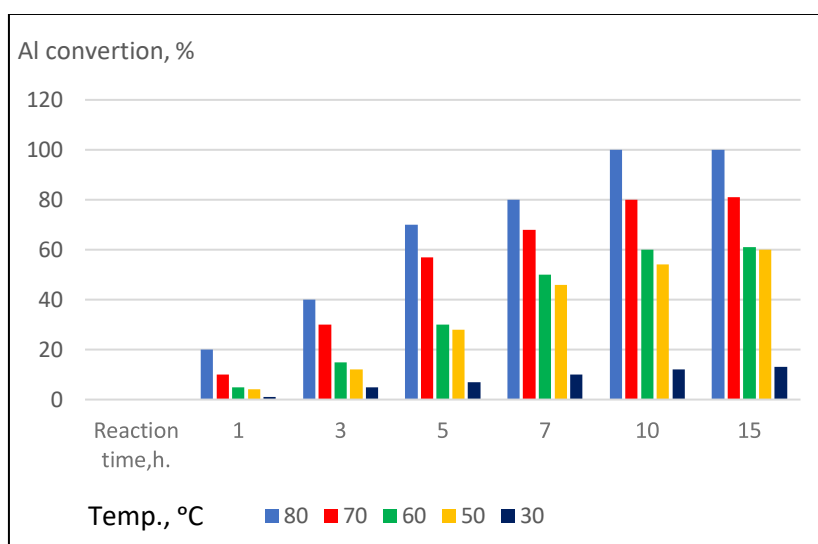


Fig. 1. Dependence of aluminum conversion on reaction time at 30°C, 50°C, 60°C, 70°C, and 80°C

As seen from Fig. 1, the activation and conversion of aluminum are very low in the temperature range of 30–70 °C. Specifically, at 30 °C, the aluminum consumption in the reaction over 15 hours is 13 wt.%, whereas at 70 °C it reaches 81 wt.%. Complete consumption of aluminum in the SRG medium is observed at 80 °C within 10 hours. In contrast, when individual straight-chain saturated hydrocarbons are used as solvents, this process is completed within 20–22 hours [14].

In the synthesis of the ACC, the molar ratio of Al/DCE also affects aluminum conversion. The following figure presents the dependence of aluminum conversion on the Al/DCE molar ratio. As seen from Fig. 2, the complete reaction of Al is observed at an Al/DCE molar ratio of 0.3. At Al/DCE molar ratios above 0.3, the amount of DCE is insufficient for the complete reaction of aluminum. As seen from the graph, when the Al/DCE molar ratio is reduced from 2 to 0.3, the Al conversion increases from 10% wt. to 100% wt.

Experiments show that the amount of solvent affects the flowability of the resulting catalyst. Thus, when the solvent is used in insufficient quantities, the catalyst is obtained in a thicker form, making its handling more difficult. DCE : SRG mass ratio of 1:2 is typically used. Considering that

tertiary C–H bonds in branched hydrocarbons are weaker than those in linear hydrocarbons [15-17], the iso-structured hydrocarbons present in SRG can be expected to act more readily as hydrogen donors. This facilitates stabilization of the carbocations formed in the reaction medium and substantially suppresses their participation in exothermic side reactions, such as isomerization, resinification, and related transformations [18, 19]. As a consequence, SRG not only accelerates catalyst formation but also improves temperature control during the synthesis process, while directly contributing to the formation of a stable catalytically active alkylaluminum chloride complex.

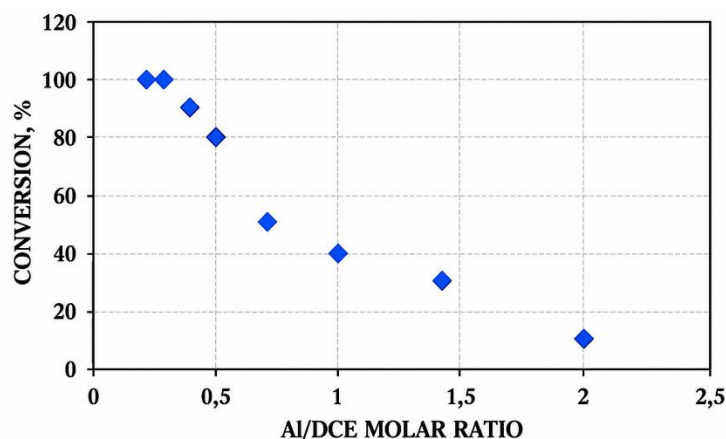


Fig. 2. Dependence of aluminum conversion on the Al/DCE molar ratio (reaction temperature – 80 °C; reaction time – 10 h)

The methods of Dynamic Light Scattering Spectroscopy (DLS), Infrared Spectroscopy (IR), and X-ray Phase Analysis (XPA) were used to determine the particle sizes, chemical composition, and complex formation of the obtained catalytic complex in the liquid phase.

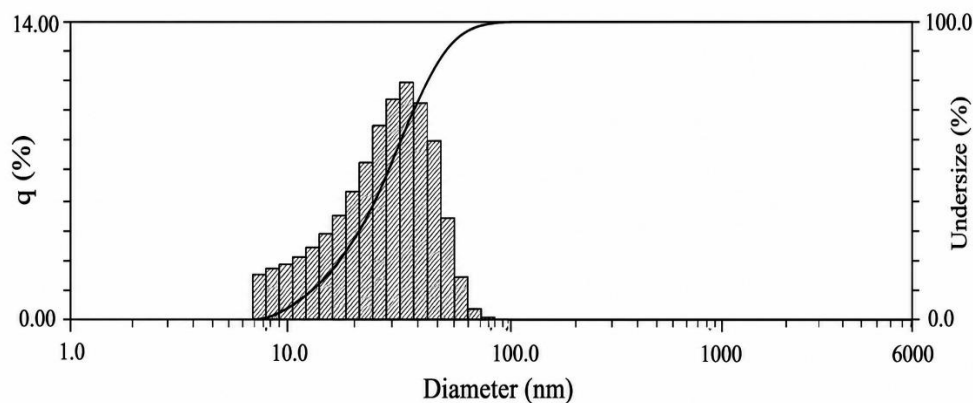


Fig. 3. Size distribution diagram of ACC particles

Based on the results of the DLS analysis (Fig. 3), it was determined that the catalytic complex consists of nanodispersed clusters with sizes ranging from 8.7 to 66.8 nm, with an average size of 29.2 nm.

In the IR spectra of ACC (Fig. 4), the ν and δ vibrations of the C–H bonds in the CH_3 group in the 2800-3000 cm^{-1} and 1300-1400 cm^{-1} regions, respectively, are observed. In the 1400-1500 cm^{-1} regions, absorption bands corresponding to the deformation vibrations (δ) of the C–H bonds of the CH_2 group are observed. The absorption bands recorded in the 1270-1280 cm^{-1} region are attributed to C–C bonds. According to the available literature data, the absorption bands observed in the IR spectrum in the 406.44–484.47 cm^{-1} region are assigned to Al–Cl and Al–Cl–Al vibrations, while those appearing in the 653.10–705.80 cm^{-1} region are attributed to Al–C vibrations [20-22]. The presence of Al–Cl, Al–C, and methyl and methylene groups in the IR spectrum suggests that the

reaction between aluminum metal and 1,2-DCE results in the formation of complexes composed of Al_2Cl_6 , AlRCl_2 , and AlR_2Cl species.

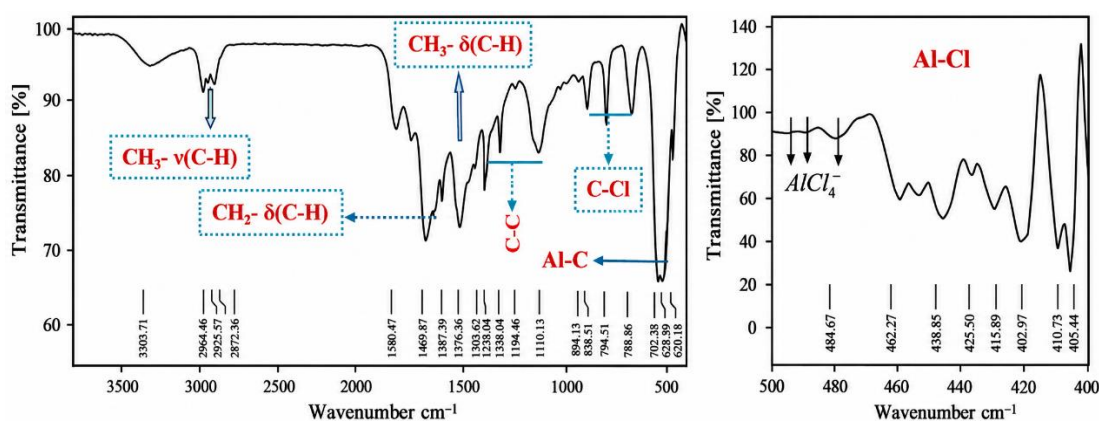


Fig. 4. IR spectrum of the aluminum–chloroorganic complex

To confirm the formation of an AlCl_3 -based complex within the ACC, the X-ray phase analysis was employed. For this purpose, the X-ray diffraction (XRD) pattern of a pure AlCl_3 sample, which had been isolated by sublimation after heating ACC to 150 °C, was recorded. According to the diffractogram shown in Fig. 5, the peaks observed in the spectrum at $2\theta = 27.6^\circ$, 15.8° , 23.3° , 24.5° , 26.5° , 28.0° , 35.1° , 39.2° , 41.5° , 44.2° , 46.8° and 52.3° are consistent with the literature data reported for chloroaluminate [23]. The appearance of characteristic peaks corresponding to the $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ crystalline hydrate in the spectrum is attributed to the interaction of active AlCl_3 with atmospheric moisture.

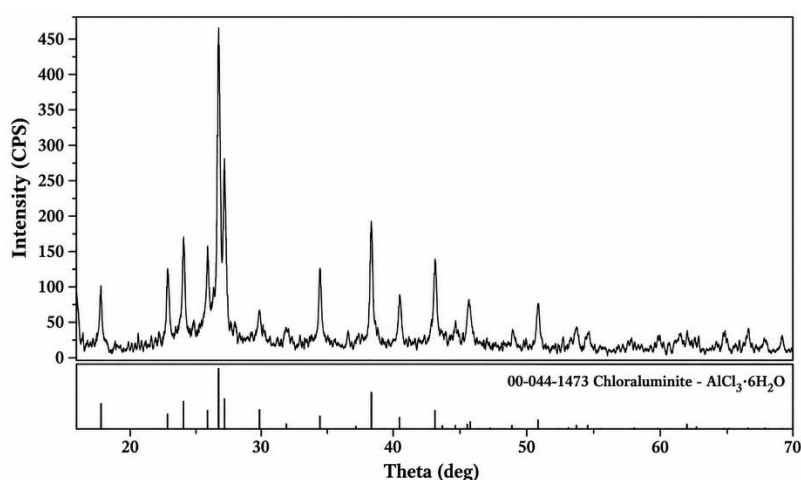


Fig. 5. Diffractogram of the aluminum–chloroorganic complex

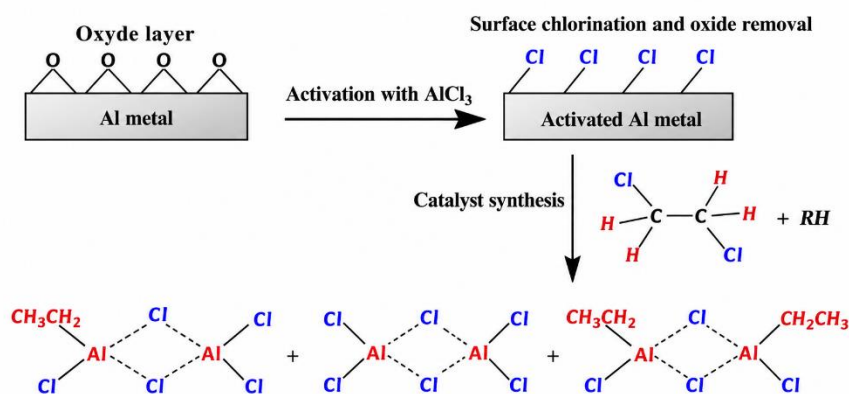
In general, although the interaction reactions of Al with alkyl halides have been investigated by various researchers for many years, the mechanism of the process has still not been fully elucidated. During the reaction of metallic Al with alkyl chlorides, a mixture of several organoaluminum chloride species is formed, accompanied by the evolution of gaseous hydrocarbons [24, 25]. Table 2 presents the composition of the gaseous hydrocarbons evolved during the interaction of Al with 1,2-DCE in the SRG medium.

Table 2. Composition of the gaseous hydrocarbons evolved during the interaction of Al with 1,2-DCE in the SRG medium

Component name	Amount, %
Ethylene	2.07
Propylene	4.57

i-Butane	32.84
Butene-1	0.11
n-Butane	2.16
3-Methyl-butene-1	29.97
i-Pentane	8.88
n-Pentane	0.52
2,2-Dimethylbutane	0.33
2,3-Dimethylbutane	1.90
2-Methylpentane	0.57
3-Methylpentane	0.20
2,4-Dimethylpentane	15.88
Total	100

Based on the results of IR and XRD spectroscopic analyses, the formation of the aluminum-chloroorganic complexe through the interaction of Al with 1,2-DCE can be described in a simplified manner, as illustrated in Scheme 1. RH : the $\geq 85\div 172^{\circ}\text{C}$ fraction of SRG.



Scheme 1. Simplified scheme for the formation of an aluminum–chloroorganic complex based on Al and 1,2-dichloroethane in a solvent medium

The oligomerization of C₆, C₈ α -olefins and their mixture in a 1:1 mass ratio was carried out using 0.3–1.0 wt.% of the synthesized catalyst within a temperature range of 40–120 °C for 10–130 minutes. The obtained results are presented in Figs 6–8.

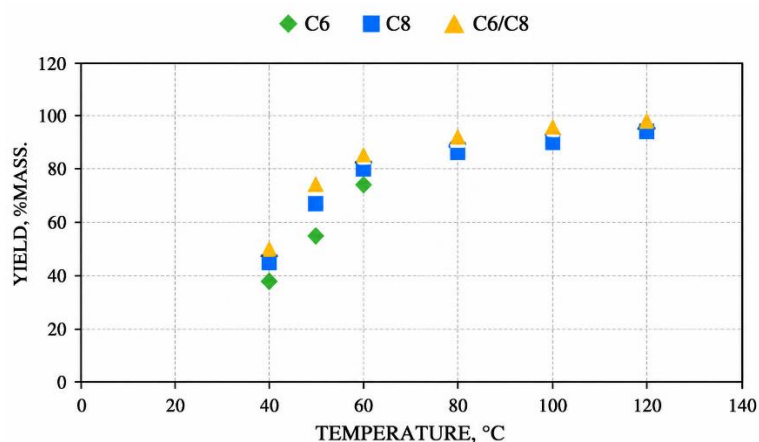


Fig. 6. Dependence of oligomer yield on temperature (catalyst concentration – 1 wt.%; reaction time – 120 min)

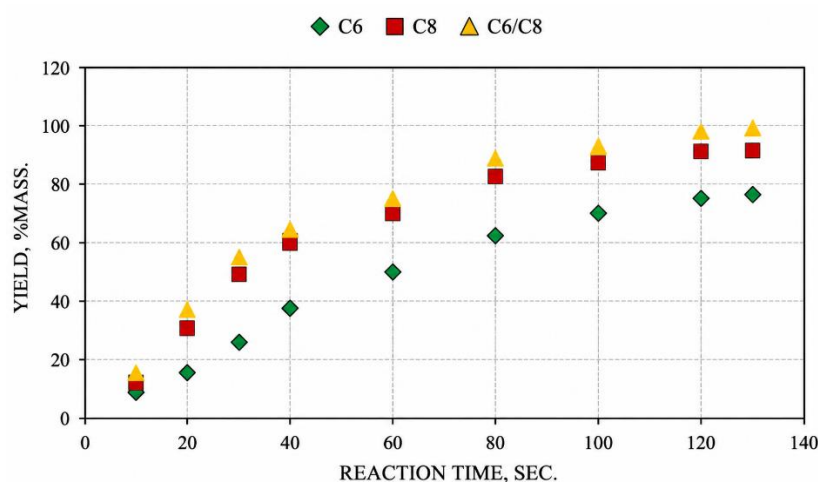


Fig. 7. Dependence of oligomer yield on reaction time (catalyst concentration – 1 wt.%; temperature – 60 °C for C₆; 100 °C for C₈ and the C₆/C₈ (1:1) mixture)

When 1 wt.% of ACC is introduced into the system, the oligomerization of α -olefins is accompanied by a temperature rise from 20 °C to 100 °C. However, experimental results show that the further course of the reaction and the yield of the obtained oligomer depend on the temperature applied to the system (Fig. 6). Thus, during the oligomerization of C₆ at 40 °C for 120 minutes, the oligomer yield is 38 wt.%, whereas at 60 °C this value increases to 75 wt.%. Due to the intensive boiling of C₆ (boiling point of C₆ is 63 °C), oligomerization was not carried out at temperatures above 60 °C. In the oligomerization of C₈ and the C₆/C₈ (1:1) mixture in the presence of ACC, increasing the temperature from 40 °C to 100 °C leads to an increase in oligomer yield from 45 wt.% to 90.9 wt.% and from 50.8 wt.% to 98.2 wt.%, respectively. Raising the temperature above 100 °C has only a minor effect on the oligomer yield.

As seen from Fig. 7, increasing the reaction time up to a certain limit has a positive effect on the oligomer yield. Specifically, the maximum yield of oligomers is observed at a reaction time of 120 minutes. Further increase in reaction time does not result in a significant change in oligomer yield.

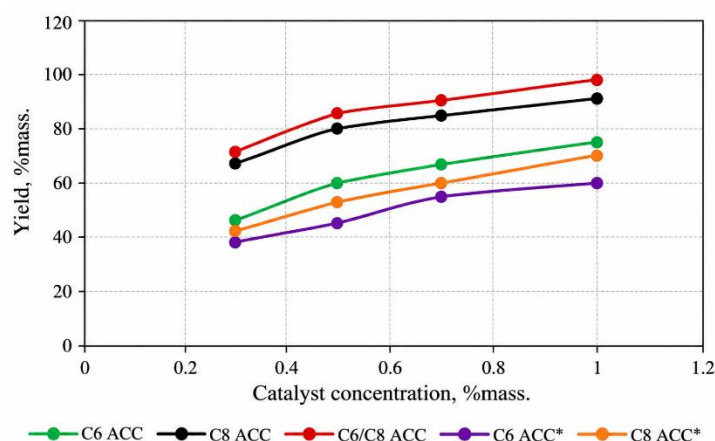


Fig. 8. Dependence of oligomer yield on catalyst concentration (reaction time – 120 min; temperature – 60 °C for C₆; 100 °C for C₈ and the C₆/C₈ (1:1) mixture).

For comparison, the oligomerization process of C₆ and C₈ olefins was carried out at various concentrations of both ACC synthesized in a SRG environment and ACC* synthesized in a heptane environment, and the results are shown in Fig. 8. As seen, increasing the catalyst concentration from 0.3 wt.% to 1.0 wt.% in the presence of ACC leads to an increase in oligomer yield from 46 wt.% to 75 wt.% for C₆, from 67.8 wt.% to 90.9 wt.% for C₈, and from 71.2 wt.% to 98.2 wt.% for the C₆/C₈

(1:1) mixture. In contrast, in the presence of ACC*, the yields of oligomerization products increase from 38 to 60 wt.% for C₆ and from 41.9 to 70.4 wt.% for C₈. Due to the influence of iso-structured hydrocarbons present in the SRG used during catalyst synthesis, more structurally stable active catalytic centers are formed; as a result, ACC exhibits higher activity compared to ACC*.

The viscosity–temperature properties and pour point of the oligomer products obtained in the presence of ACC and ACC* are presented in Table 3.

Table 3. Physical properties of oligomerization products of C₆ and C₈ olefins obtained in the presence of ACC and ACC*.

Samples	Kinematic viscosity, (mm ² /s)		Viscosity index	Pour point, °C
	At 100°C	At 40°C		
C ₆ (ACC)	4.90	25.75	115	-52
C ₆ (ACC*)	3.10	12.95	99	-60
C ₈ (ACC)	6.40	37.95	120	-49
C ₈ (ACC*)	4.30	21.20	110	-50
C ₆ /C ₈ (1/1) (ACC)	9,8	66.55	130	-51

As seen from Table 3, the oligomer products obtained from the oligomerization of C₆ and C₈ olefins, particularly their mixture, in the presence of ACC are characterized by a high viscosity index (115–130) and low pour point (–49 to –52 °C).

Thus, the conducted experiments have shown that iso-structured hydrocarbons present in the SRG used as a solvent during catalyst synthesis act more readily as hydrogen donors compared to normal hydrocarbons. As a result, they directly participate in the formation of a fluid-viscous catalytic complex composed of aluminum alkyl chlorides with high stable activity, thereby enhancing the efficiency of the oligomerization process.

Conclusion

An aluminum–chloroorganic complex was synthesized based on aluminum metal, 1,2-dichloroethane, and a straight-run gasoline fraction boiling in the range of ≥85–172 °C as a solvent. The effects of temperature, solvent amount, and Al/DCE molar ratio on the synthesis process were investigated, and the optimal conditions were determined as follows: reaction time – 10 h; temperature – 80 °C; Al/DCE molar ratio – 0.3; DCE/SRG mass ratio – 1:2.

Based on IR, DLS, and XRF analyses, it was established that the interaction of Al and DCE in the presence of SRG leads to the formation of an aluminum–chloroorganic complex with the composition AlCl₃·RAlCl₂·R₂AlCl, consisting of nanodispersed clusters with an average size of 29.2 nm.

The oligomerization of C₆, C₈ α-olefins and their mixture (1:1 mass ratio) was carried out using 0.3–1.0 wt.% of the synthesized catalyst within a temperature range of 40–120 °C for 10–130 minutes, and the optimal conditions were determined as follows: temperature – 60 °C (for C₆), 100 °C (for C₈ and C₆–C₈ mixture); reaction time – 120 min; catalyst concentration – 1 wt.%.

It was found that, compared to ACC* synthesized in a heptane medium, ACC synthesized in an SRG medium exhibits higher stable activity, allowing the oligomer yield of C₆ and C₈ olefins to increase from 60 wt.% to 75 wt.% and from 70.4 wt.% to 90.9 wt.%, respectively, under the specified optimal conditions.

The oligomer products obtained from the oligomerization of C₆ and C₈ olefins, especially their mixture, in the presence of ACC are characterized by a high viscosity index (115–130) and low pour point (–49 to –52 °C).

Conflicts of Interest

The authors declare no conflict of interest.

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