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Original Research Article

Oxidative Oligomerization of Fischer–Tropsch Internal Olefins Catalyzed by Metal Octoates of s, p and d Block Elements

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Abstract

In the present work we further expanded the scope of the study of the oxidative oligomerization process of synthetic hydrocarbon fraction obtained by Fischer–Tropsch synthesis with a total olefin content (consisting predominantly internal/branched olefins) of 10 % with the aim of producing poly-α-olefin like lubricant material. The oxidative oligomerization was carried out using commercially available metal octoates based on Co, Mn, Zn, Ca, Ba, Li, Zr, Cu and Pb and their combinations as catalyst. It was established that the yield of the oligomerization reaction depending on the active metal component used decreased in the following order: Ba > Zr > Zn > Co > Mn > Ca > Li > Cu > Pb. While at the same time, the oxidative oligomerization reaction carried out using bimetallic catalytic systems did not led to any significant increase in the product yield. The oxidative oligomerization reaction using Ba octoate as catalyst gave a yield of 30.4 % and had a kinematic viscosity at 100 °C of 3.6 cSt, Viscosity Index value of 201 and pour point of minus 10.

Keywords: Oligomerization; synthetic hydrocarbons; metal octoate; synthetic lubricant; Fischer–Tropsch synthesis products

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1. Introduction

Poly-α-olefin (PAO) based synthetic lubricants, have gained a lot of attention in the recent decades due to its excellent physico-chemical properties such as higher thermal and oxidative stability, superior viscosity index (VI) and low pour point (PP) when compared with mineral base stocks [1–3]. This in turn has led to PAO finding applications in wide range of fields, including automotive crankcase lubrication, automatic gearbox oil, hydraulic oil, compressor oil, heat transfer fluids, food grade oils, etc. [4–6]. At the same time, the PAO market is poised to expand significantly from 7.65 billion USD in 2025 to 16.55 billion USD by 2034, at a growth rate of approximately 9% for the forecasted period [7]. PAOs are primarily synthesized through the oligomerization of 1-decene; however, there are reports of using other linear alpha olefins (LAO) such as C_6, C_8, C_{12} and C_{14} as monomers/ co-monomers for the production of PAO [8,9]. There are a number catalytic routes reported in the literature for synthesizing PAOs, including the use of transition metal-based complexes [10–12], Lewis-acid co-initiator systems, such as: $AlCl_3$ [13–16] and BF_3 [17,18], metallocene and ionic liquids-based systems [19–22]. The choice of catalytic system, reaction conditions as well as the monomers used can significantly influence the

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properties of the synthesized PAOs produce with tailor made properties. For example, when using 1-decene as the starting material the product yield varied from 83 to 96% as well as the properties of the synthesized product, such as: KV^{100} (i.e., kinematic viscosity at 100 °C) in a broad range of 1.4 to 635 cSt and VI in the range of 132-250, depending on the catalyst and the type of oligomerization used for carrying out the reaction (see Table 1).

While the olefin oligomerization process itself is an integral part of the oil refineries worldwide, the target product of the oligomerization is middle distillates [23]. There are very few reports in the literature where the refinery feedstocks for the production of PAO. Sarin *et al*. [24], reported using refinery stream containing 20% LAO in the carbon number range of C_9 to C_{13}, 2% internal olefins. At the same time, the Fisher–Tropsch refineries around the world have also implemented light olefin (C_2-C_4) oligomerization to middle distillates [25]. While the higher olefins which are also present in the stream are subjected to hydrogenation. Only recently, we have shown the possibility of producing polyolefin base oil using the olefin rich gasoline (C_5-C_{10}) and diesel (C_{11}-C_{18}). Fisher–Tropsch synthesis products, which contains LAOs alongside with a significant amount of *β*- γ- and δ-olefins, also commonly referred as internal/branched olefins [26–28]. The synthesized products had a KV^{100} in the range of 1.4 - 3.4 cSt and PP varied from minus 9 to minus 50 °C (see Table 1).

The oxidative polymerization of vinyl monomers was first summarized back in 1970s in a detailed review by M.M. Mogilevich [29]. Since then, various catalytic systems based on *N*-hydroxyphthalimide, (cobalt(II) phthalocyanine) and (cobalt(II) tetraphenylporphyrin pyridine) etc., are reported for carrying out oxidative oligomerization/co-polymerization of vinyl monomers, such as: styrene, α-methyl styrene, methyl methacrylate and vinyl acetate [30,31]. Metal octoate based siccatives are widely used in paint industry to accelerate the hardening of drying oils, which takes place as a result of free-radical autoxidation of the oils with air and the whole process is catalyzed by the siccative [32]. In our previous works [28], we have for the first time reported the oxidative oligomerization of olefin rich Fisher–Tropsch synthesis products using zirconium octoate as catalyst, with the reaction mechanism similar to that of free-radical autoxidation of the oils with air (see Figure 1). The aim of the present work is to further expand the scope of the study of the influence of industrially produced metal octoates based on Co, Mn, Zn, Ca, Ba, Li, Zr, Cu, Pb and their combinations on the oxidative oligomerization ability of olefin containing Fischer–Tropsch synthesis product.

2. Materials and Methods

2.1 Methods for Studying Feedstock and Oligomerization Products

The quantification of the synthetic hydrocarbon fraction obtained from the Fisher – Tropsch synthesis, was determined using capillary gas-liquid chromatography-mass spectrometry (GC-MS) on an Agilent GC 7890A gas chromatograph with an MSD 5975C mass detector (Agilent Technologies, USA) and an HP-5MS column using helium as a carrier gas. The feedstock was analyzed by introducing 1 µL of the sample diluted with the carrier gas in a ratio of 1:400. Temperature-programmed mode: initial temperature - 30 °C, hold - 5 min; heating rate - 5 °C/min to a temperature of 60 °C, hold - 1 min; heating rate - 10 °C/min to a temperature of 70 °C, hold - 1 min, heating rate 50 °C/min to a temperature of 270 °C, hold - 5 min; analysis duration - 23 min.

For determining low-temperature properties, the TPZ-LAB-12 automatic apparatus was used, designed to determine the pour point in a small sample volume in accordance with ASTM D6749-02 [33]. The determination of kinematic viscosity at temperatures of 40 and 100 °C has been carried out in accordance with ASTM D7042-16 [34].

2.2. Reagents

For the purpose of this work H_2SO_4 (98 % conc.) was purchased from EKOS-1, (Russian Federation). Monometallic siccatives were provided by LLC Himpostavshchik Don (Russian Federation) and were used as catalysts for the oxidative oligomerization process without any further purification (Table 2).

2.3. Synthesis of Hydrocarbon Fraction

To obtain synthetic hydrocarbon fraction, a bifunctional cobalt catalyst was used, prepared by extruding a mixture of 35.0 wt.% Co-Al_2O_3/SiO_2 catalyst and 30.0 wt.% HZSM-5 zeolite (SiO_2/Al_2O_3 = 40.5, Ishimbay Specialized Chemical Catalyst Plant LLC) with the addition of 1.0 wt.% Pd with a binder of 35.0 wt.% boehmite (Sasol, Pural TH 80). The binder was plasticized with an aqueous-alcoholic solution of triethylene glycol and nitric acid (the nitric acid solution was obtained by adding 1–2 mL of nitric acid with a concentration of 65 wt.% to 90–100 mL of distilled water per 100 g of mixture; triethylene glycol was added based on the volume ratio of nitric acid: triethylene glycol in the mixture equal to 1:3). The catalyst granules were formed by extrusion, dried for 24 h at room temperature, 4–6 h at 80–100 °C, 2–4 h at 100–150 °C, and calcined for 5 h at 400 °C.

The promoting of the HZSM-5 zeolite was carried out by ion exchange from a $PdCl_2$ solution for 5 h at 80 °C, after which the excess solution was removed. The wet zeolite powder was then subjected to heat treatment in the following order: 4 h at 80 °C, 4 h at 100-150 °C, and 4 h at 550 °C. The synthetic hydrocarbon fraction was obtained in a tubular reactor (inner diameter 16 mm) with a fixed catalyst bed (catalyst volume 10 cm^3, granule size 1.0-2.0 mm) at a pressure of 2.0 MPa, a temperature of 250 °C, an H_2/CO ratio at the reactor inlet of 2.0, and a gas space velocity (GSHV) of 1000 h^{-1}. Preliminary reduction of the catalyst was carried out for 1 hour in a stream of H_2 at a pressure of 0.1 MPa, a temperature of 400 °C, and a GSHV of 3000 h^{-1}. As a result of the Fischer–Tropsch synthesis, a mixture of C_{5+} hydrocarbons were obtained, from which a fraction with a boiling point from the initial boiling point to 130 °C was isolated by distillation at atmospheric pressure in order to obtain the maximum concentration of C_6-C_8 alkenes.

2.4. Oligomerization

Oxidative oligomerization was carried out in a high-pressure reactor equipped with a paddle stirrer in an air atmosphere at a pressure of 2.5 MPa with constant stirring at 600 rpm to ensure effective mass transfer and a temperature of 160 °C for 6 hours. The catalyst loading (taking into account the amount of active metal) for each experiment was 5 wt.% w.r.t the olefin content in the synthetic hydrocarbon fraction. The amount of hydrocarbon fraction sample loaded in each experiment was 80 ml. All the experiments were conducted in triplicate. After the oligomerization process, the resulting mixture was purged with gaseous ammonia to deactivate the catalyst by converting the octoates of the corresponding metals to insoluble ammonia salts followed by the vacuum filtration. In the case of Ba and Pb octoates, 5 wt.% solution of H_2SO_4 to deactivate the catalyst by converting the octoates of the corresponding metals to insoluble sulphate salts.

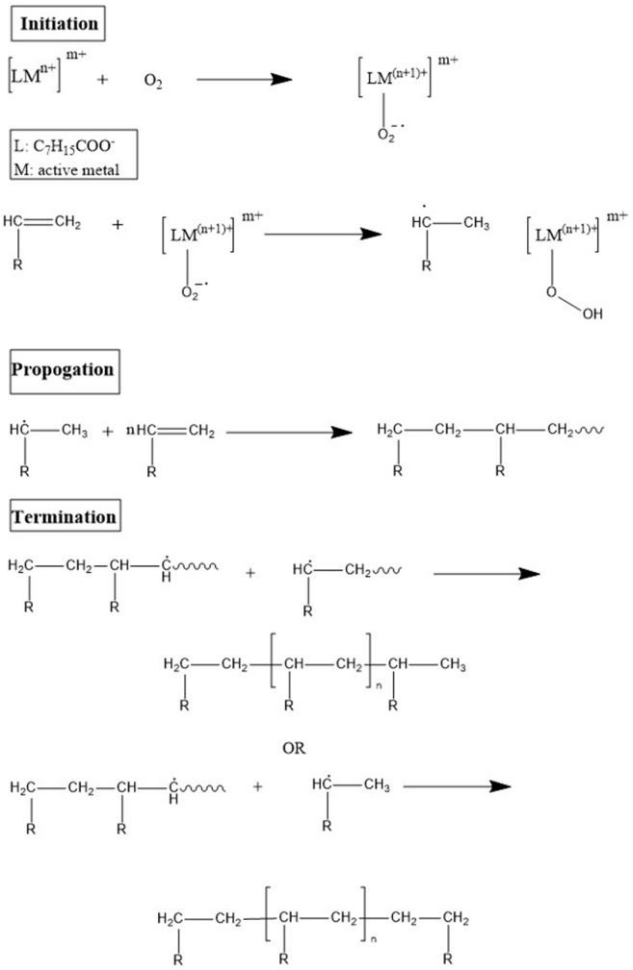

Figure 1. Proposed mechanism of oxidative oligomerization of oxidative oligomerization of olefin rich Fisher–Tropsch synthesis products.

Table 2. Composition and designation of catalysts.

Designation	Company marking	Active metal component	Mass fraction of metal, %	Appearance
Co octoate	CO-1	Cobalt	12.04	Homogeneous liquid of blue-violet color without mechanical impurities.
Mn octoate	CO-2	Manganese	10.05	Homogeneous liquid of red-brown color without mechanical impurities.
Zn octoate	CO-3	Zinc	12.00	Homogeneous transparent liquid without mechanical impurities. Colorless or with a yellowish tint.
Ca octoate	CO-4	Calcium	5.08	Homogeneous transparent liquid without mechanical impurities. Colorless or with a yellowish tint.
Ba octoate	CO-5	Barium	12.00	Homogeneous transparent liquid without mechanical impurities, colorless or yellow.
Li octoate	CO-6	Lithium	2.00	Homogeneous transparent liquid without mechanical impurities, colorless or yellow.
Zr octoate	CO-7	Zirconium	17.52	Homogeneous transparent liquid without mechanical impurities, colorless or with a yellowish tint.
Cu octoate	CO-8	Copper	8.08	Homogeneous liquid of blue-green color without mechanical impurities.
Pb octoate	CO-9	Lead	25.02	Homogeneous transparent liquid without mechanical impurities, colorless or with a yellowish tint.

The resulting mixture after oxidative oligomerization was subjected to separation by fractionation at 220 °C under the atmospheric pressure. The product yield was determined as the ratio of the mass of the bottom residue after distillation to the amount of olefins in the feedstock, as described in the works [35].

3. Results and Discussion

Synthetic lubricants based on C_6-C_{20} LAOs, are among the most valuable commercial products that meet modern world standards. Despite the development of methods for their production, the oligomerization of aliphatic internal/branched olefins in the process of synthesis of polyolefin oils remains relevant. To solve this problem, we proposed a process of oxidative oligomerization of a synthetic hydrocarbon fraction consisting of LAOs alongside with a significant amount of *β*-, γ- and δ- olefins obtained by Fischer–Tropsch synthesis. Synthesizing polyolefin base oils from synthetic hydrocarbon fractions derived from CO and H_2, by Fischer–Tropsch method instead of pure alpha-olefins such as 1-decene, expands the feedstock base and allows for the production of a value-added product from cheaper and more readily available feedstocks. We have conducted systematic studies of the effect of active metal component on the process of oxidative oligomerization of Fischer–Tropsch synthesis products to obtain polyolefin oil bases. Industrially produced metal octoates based on Co, Mn, Zn, Ca, Ba, Li, Zr, Cu and Pb as well as their combinations were used as catalysts.

3.1. Characteristics of the Feedstock Hydrocarbon Fraction

The synthetic hydrocarbons feedstock sample used in the experiments was a transparent with a slightly yellowish tint, flammable liquid with a characteristic odor like synthetic gasoline. The distribution of hydrocarbons in the feedstock was determined using GC-MS, which clearly demonstrated the predominant presence of alkanes and iso-alkanes in its composition, the content of which is ~ 90 wt.%, the rest representing mixture of small amount of LAOs and a significant amount of *β*- γ- and δ- olefins (branched olefins). The branched olefin content in the feedstock was ~ 2,7 times more than LAOs at 7.3 wt.% and the remaining 2.7 wt.% as LAOs. The feedstock contained 5-9 carbon atoms identified having a symmetrical distribution with the maximum being C_7 hydrocarbons (Figure 2).

3.2. Monometallic Catalysts

In order to study the possibility of using common commercially available siccatives as catalysts for oxidative oligomerization the preliminary studies were carried out using a single metal octoate as a catalyst. The choice of the metal octoate as a catalyst significantly influenced the yield of the final product, as it can be seen from Figure 3, when using Ba octoate as a catalyst highest product yield (30.4 %) was obtained while on the other hand, a product yield of only 6.0 % was obtained when Pb octoate was used as a catalyst. The yield of oligomerization products, depending on the active metal, changed in the following order: Ba > Zr > Zn > Co > Mn > Ca > Li > Cu > Pb. The high product yield when using Ba octoate as a catalyst was probably due to the fact that Ba octoate has the lowest oxidizing ability and therefore do not cause oligomer destruction [36]. All metal octoates, in addition to the role of catalyst, are strong oxidizers and therefore, oxidative destruction reactions occur in parallel with the polymerization reaction with Pb octoate having the highest oxidizing ability.

Previously, we studied the process of oxidative oligomerization of Fischer–Tropsch

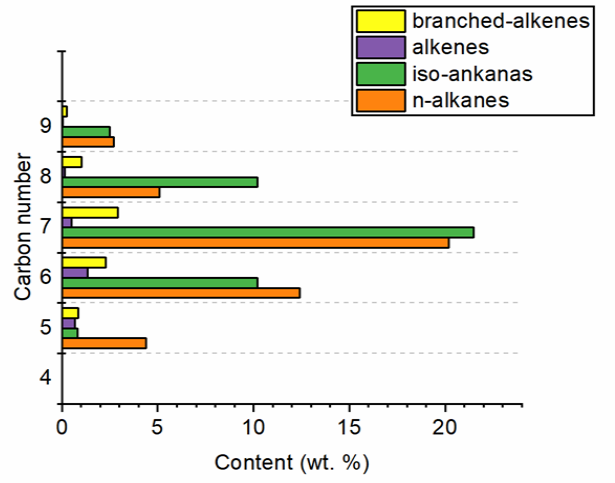

Figure 2. Molecular weight distribution of feedstock.

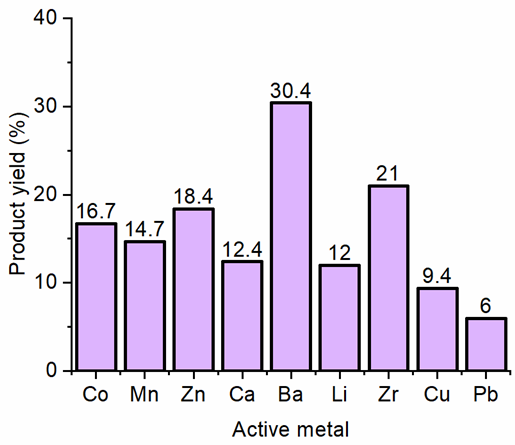

Figure 3. Influence of active metal component on the product yield of oxidative oligomerization.

synthesis products – fractions of C_{10}-C_{15} hydrocarbons with a total alkene content of 64.7 wt. % using zirconium octoate as a catalyst under similar conditions [28]. It was determined that at a temperature of 160 °C, a catalyst content of 5.0 wt. %, a process duration of 6 h and a pressure (air) of 2.5 MPa, the yield of the target fraction (oil fraction of hydrocarbons) was 52.7 %. In the present work however, the product yield does not exceed 21.0 %, which can be explained by the low initial concentration of olefins in the feedstock (Figure 2).

Table 1. Influence of catalytic systems and monomer choice on the synthesized product ([a]KV100 – kinematic viscosity at 100 °C).

No.	Oligomerization type	Initiator/ catalyst	Monomer	Reaction conditions	Properties of the obtained product	Ref.
1	Cationic	$AlCl_3$ + alcohol	1-decene	Catalyst – $AlCl_3$ (1 wt.% w.r.t 1-decene) + 1-propanol (0.18 wt.% w.r.t 1-decene) Pressure – not reported, Temperature – 25 °C, reaction time – 4 h, solvent – n-heptane	Yield – 96.0 % [a]KV^{100}– 53.6 cSt, VI – 158 PP – not reported	[16]
2	Cationic	BF_3 + alcohol alkoxylates [(RO-CHR'-CHR"-O)$_n$-H	1-decene	Catalyst – BF_3 + Ethonic 610-3 (C_6 - C_{10} mixture of alcohol ethoxylate,1.0 mole% w.r.t 1-decene), Pressure –10 psig, Temperature – 32 °C, reaction time – 2 h	Yield – 96.7 % [a]KV^{100}– 1.4 cSt, VI – not reported PP – < minus 65°C	[17]
3	Ion-coordination	$AlCl_3$ + $Ti(Obu)_4$	1-decene	Catalyst – $AlCl_3$ (2 wt.% w.r.t 1-decene) + Ti(Obu)4 (0.05 wt.% w.r.t 1-decene), Al/Ti ratio equal to 21, Pressure – not reported, Temperature – 100 °C, reaction time – 2 h	Yield – not reported [a]KV^{100} – 12.3 cSt, VI – 132 PP – minus 30 °C	[19]
4	Ion-coordination	Ph_2C(Cp-9-Flu)$ZrCl_2$ + MAO	1-decene	Catalyst – Ph_2C(Cp-9-Flu)$ZrCl_2$ (0.001 wt.% w.r.t 1-decene), MAO: Ph_2C(Cp-9-Flu)$ZrCl_2$ equal to 1000:1 Pressure – 200 psig, Temperature – 70 °C, reaction time – 0.5 h	Yield –82.8 % [a]KV^{100}– 635 cSt, VI – 282 PP – not reported	[20]
5	Ion-coordination	$(Me_2Cp)_2ZrCl_2$ + MAO	1-decene	Catalyst – $(Me_2Cp)_2ZrCl_2$ (1 wt.% w.r.t 1-decene), MAO: $(Me_2Cp)_2ZrCl_2$ equal to 2000:1 Pressure – not reported, Temperature – 20-25 °C, Reaction time – 16 h Solvent – toluene	Yield –92 % *KV^{100}– 312 cSt, VI – 250 PP – not reported	[21]
6	Radical initiated	AIBN	Synthetic gasoline fraction(C_5-C_{10}), alkene content 76.0 wt.%	Catalyst – AIBN (0.5 wt.% w.r.t alkene content) Temperature – 200 °C, Reaction time – 12 h	Yield –23.4 % [a]KV^{100}– 1.4 cSt, VI – not reported PP – minus 46 °C	[26]
7	Radical initiated	AIBN	Synthetic gasoline fraction(C_5-C_{10}), alkene content 79.3 wt.%	Catalyst – AIBN (0.5 wt.% w.r.t alkene content) Temperature – 200 °C, Reaction time – 12 h.	Yield –24.5 % [a]KV^{100}– 1.6 cSt, VI – not reported PP – minus 50 °C	[26]
8	Radical initiated	AIBN	Synthetic hydrocarbon (C_{11}-C_{18}), alkene content 31.8 wt.%	Catalyst – AIBN (0.5 wt.% w.r.t alkene content) Temperature – 200 °C, Reaction time – 12 h solvent – acetone	Yield –39.5 % [a]KV^{100}– 3.4 cSt, VI – 146 PP – minus 9 °C	[27]
9	Oxidative	Zirconium octoate	Synthetic hydrocarbon fraction (C_{10}-C_{15}), alkene content 64.7 wt. %	Catalyst – Zirconium octoate (5 wt.% w.r.t alkene content), Pressure – 2.5 MPa, Temperature – 160 °C, Reaction time – 6 h Medium – air	Yield –52.7 % [a]KV^{100}– 10.9 cSt, VI – 100 PP – minus 31 °C	[28]

3.3. Bimetallic Catalytic Systems

In order to improve the product yield as well as to study the synergistic effect of different metal octoates, the oxidative oligomerization process was carried out using various bimetallic catalytic systems (Figure 4). The criteria for selecting the combination of metals in bimetallic catalytic systems was as follows: a) recommended by the manufacturer Ba + Co (K1) and Zr + Ca (K2), used for accelerating the drying of paint, b) metal octoates that gave the highest product yield Ba + Zr (K3) and Zr + Zn (K4). At the same time, the total catalyst content w.r.t olefins was left unchanged to 5.0 wt. %, while the ratio of the active metals in the bimetallic catalyst was 1:1.

The highest product yield was obtained using bimetallic catalysts based on K3 and K4 equaling to 25.7 % and 22.3 % respectively. While at the same time, using bimetallic catalysts based on K1 and K2 resulted in lower product yield of 15 % and 17 %, respectively. In the case of K1 bimetallic catalysts the unexpected drop in the product yield can be associated with the inhibitory effect of the active metal ions in reaction mixture. While for the K1, K2, and K3 bimetallic catalysts there is no significant synergetic effect observed between the Ba and Co octoates, Zr and Ca octoates and the Ba and Zr octoates, with the product yield being arithmetic mean between the values obtained on monometallic catalysts. The obtained results indicated that the bimetallic catalytic systems did not improve the product yield of the oxidative oligomerization process.

3.4. Properties of the Oligomerization Product

The obtained results showed that the highest product yield was obtained on monometallic Ba octoate catalyst (30.4 %) and was therefore selected for further studies. One of the important criteria for classifying synthetic lubricants is the KV^{100} value and viscosity index (VI). The synthesized product had a KV^{100} of 3.6 cSt, which was similar to the values obtained through radical initiated oligomerization of synthetic hydrocarbons, while at the same time having a very high VI of 201 (see Table 2, No. 8) [27]. At the same time however, the PP of the synthesized product is only minus 10, which when compared with the product of oxidative oligomerization with Zr octoate [28] is three times higher (see Table 2, No. 9). This can be due to the presence of a long linear chains in the Ba octoate catalyzed products as a branch or main chain is pivotal for higher VI of synthetic oils, however, it is at the same time, detrimental to low pour point. Thus, depending on the choice of the metal octoates the properties of the oxidative oligomerization product can differ significantly.

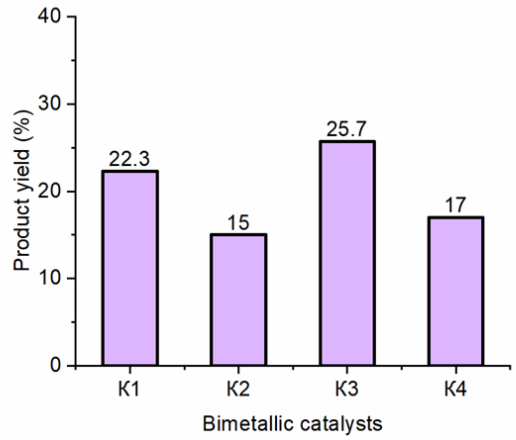

Figure 4. Influence of bimetallic catalysts on the product yield of oxidative oligomerization.

4. Conclusions

In this work, studies were conducted to examine the effect of each individual metal octoate on product yield and attempts were made to combine metal octoates in order to intensify the process. Under the reaction conditions: Pressure 2.5 MPa air, catalyst content of 5.0 wt. % w.r.t to olefin in feedstock, temperature of 160 °C and reaction time of 6 hours, the yield of oligomerization products, depending on the active metal component was: Ba > Zr > Zn > Co > Mn > Ca > Li > Cu > Pb. In order to increase the yield of the oligomerization product, 4 bimetallic catalytic systems, based on the combinations of metal octoates were studied. Under similar conditions, however, the bimetallic catalytic systems did not give any significant increase in the product yield. The oligomerization product based on the Ba octoate catalyst had a kinematic viscosity at 100 °C of 3.6 cSt, Viscosity Index value of 201 and pour point of minus 10. Commercially available synthetic lubricants formulations contain 80 to 98% of base stock and a combination of various additives. The obtained product can be used as basis for obtaining lubricants having a wide range of applications. The prospects of this study may be related to the search of additives for synthesized polyolefin base oils in order to reduce their pour point while maintaining the achieved excellent viscosity index.

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CRediT Author Statement

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