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Effect of Support on Hydro-metathesis of Propene: A Comparative Study of $W(CH_3)_6$ Anchored to Silica vs. Silica-Alumina

Mykyta Tretiakov, Manoj K. Samantaray*, Aya Saidi and Jean-Marie Basset*

This work is dedicated to Professor Wolfgang Hermann on the occasion of his 70th birthday

Abstract:

Hydro-metathesis of propene was carried out by using well-defined $W(CH_3)_6$ supported on silica and silica-alumina. It was observed that $W(CH_3)_6$ supported silica-alumina catalyst is much better (TON 4577) than the silica supported catalyst (TON 2104). We demonstrated that the present catalysts are much better than the previously reported (tantalum hydride/KCC-1, TON 786) catalyst. For the first time, we observed the formation of n-decane from propene, which enables us to think of using cheaper raw materials and converting them to petroleum range alkanes using hydro-metathesis reaction.

Keywords: Hydro-metathesis • Carbene • Carbyne • Organometallics • Olefin metathesis •

1. Introduction

Alkanes are the major constituents of petroleum [1]. As the global demand for petroleum products is increasing and petroleum reserve is decreasing day by day, there is a growing research interest in the transformation of low molecular weight hydrocarbon feedstock into petroleum range alkanes [2–5]. There are two main reactions for conversion of low molecular weight carbon feedstock to petroleum range alkanes: F-T process and Alkane metathesis [2,5–11]. Though F-T process has a wide industrial applications, it suffers from high reaction temperature as well as the formation of wax range alkanes, whereas alkane metathesis suffers from a lower reactivity [5,12,13].

After the discovery of alkane metathesis in 1997 by a single site Ta-H/silica catalyst, our group reported several catalytic systems for the improvement of conversion as well as TON in this reaction [2,14–16]. From the reaction mechanism we observed that the main steps for the metathesis reaction are the formation of olefins and hydrogen resulting from the C-H bond activation of the paraffin followed by olefin metathesis [16,17]. Alkane metathesis is a thermoneutral reaction, it means for the reaction to happen practically no energy is required, but modelling showed that to form an olefin which is an intermediate in alkane metathesis reaction higher amount of energy is required to overcome this kinetic energy barrier [18,19].

Based on the above concepts we believe that by using an olefin in place of an alkane with an extra amount of hydrogen we will be able to carry out the metathesis reaction which is called as “hydro-metathesis” reaction as hydrogen and olefin are used as starting materials with the catalyst. In other words, the hydro-metathesis represents the last step of alkane metathesis. However, in order to be economical, the olefin must be cheap, though propylene is more expensive than n-butene, we took it as a model molecule.

Another aspect of using olefin for hydro-metathesis reaction is, we expected that we could increase conversion as well as TON in this particular reaction as compared to alkane metathesis reaction where the reactivity of alkanes towards the catalyst is comparatively slow even if we have already considerably increased the TON of alkane metathesis [14,20].

Our group first reported the hydro-metathesis reaction in 2011 using Ta-H supported on KCC-1 [21] where we got an impressive result of the conversion of propene to higher and lower alkanes (C_2 , C_4 , C_5) with a very high TON (786) as compared to the conversion of propane using Ta-H

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on silica TON (60) [2,22]. Recently we reported the liquid phase hydro-metathesis reaction of 1-decene with various silica supported well-defined catalysts with good conversion and TONs [23]. With these exciting results we thought to use W/silica and W/silica-alumina as these new generation catalysts were proved to be very efficient in converting alkanes to their lower and higher homologues [14,20].

Herein we are reporting the hydro-metathesis of propene using well-defined $W(CH_3)_6$ supported on silica (**1**) and silica-alumina (**2**) in continuous flow reaction conditions.

Fig. 1. Well-defined silica supported W-methyl **1** and silica-alumina supported W-methyl **2**

2. Results and Discussion

2.1 Evolution of hydro-metathesis reaction

$W(CH_3)_6$ supported silica and silica-alumina catalysts and their application in alkane metathesis reaction were already reported by our group [14,20]. During the alkane metathesis reaction we observed that silica and silica-alumina supported catalysts are very efficient as compared to Ta-H supported catalyst for alkane metathesis reaction [22,24,25]. Note that W-based catalysts are better metathesis catalysts than the Ta catalysts. The literature data reported above directed us to use an oxide-supported W in hydro-metathesis reaction, as olefin metathesis is part of the hydro-metathesis. We used W/silica as well as W/silica-alumina as we observed that W/silica-alumina is more stable than W/silica and due to the expected increase in acidity on tungsten center the conversion and TON would be expected to be much better compared to W/silica and Ta/silica [2,20,22].

In a typical experiment, 106 mg of silica supported W catalyst **1** (15 micromoles W) was loaded in a

continuous flow reactor and a mixture of propene 6 mL/min and hydrogen 6 mL/min in presence of argon was passed through the catalytic bed at 150 °C for 24 hours. The conversion of the reactant was observed every 36 minutes by analyzing the products through gas chromatography (GC).

Initially, we choose catalyst **1** and we observed that the catalyst is stable for 24 hours with the maximum conversion being observed during initial 6.5 hours of the reaction. The overall conversion of 51 % and a TON of 3532 were achieved at our contact time. In order to eliminate the percentage of hydrogenation of propene to propane, which is competing with the hydro-metathesis, we subtracted the propane concentration (propane formation is a logical competing reaction) and the olefins concentration (coming from olefin metathesis). We got, for propene hydro-metathesis, 25 % conversion and 1635 TON (only higher and lower alkanes) Fig. 2 and 3.

Fig. 2. **2A** and **2C** represent total conversion (Olefins + Alkanes, red line) and corresponding TON (blue line), **2B** and **2D** represent conversion of propene to only alkanes except propane (red line) and TON (blue line) in hydro-metathesis reaction of propene (using propene to hydrogen ratio 1:1 for **2A** and **2B** and 1:2 ratio for **2C** and **2D**) in a continuous flow reactor at 150 °C in presence of catalyst precursor **1** (106 mg), hydrogen and argon.

During the reaction, along with alkanes we also observed a considerable amount of mainly C₂, C₄ and C₅ olefins (Fig. 3A). We envision that we can only reduce the formation of olefin if we could increase the partial pressure of hydrogen. To prove our point we thought to move from propene to hydrogen ratio of 1:1 to 1:2 and eventually 1:3 and observe the effect of hydrogen.

With this concept, we used 1:2 ratio of propene to hydrogen. To our expectation, we observed the increase of overall TON to 4328 and TON of only alkanes to 2104 with the decrease of formation of olefins (Fig. 3C and 3D). Furthermore, we thought to move again forward to increase propene to hydrogen ratio to 1:3. At the end of the reaction, we observed that the TON is almost equal to that of 1:2 ratio with the formation of hydrogenolysis products along with other alkanes (SI Fig. S1-S4) which suggests that 1:2 ratio of propene to hydrogen is suitable for our catalyst for hydro-metathesis reaction in our conditions.

Fig. 3. 3A and 3C represent total distribution of products [olefins (orange), alkanes (blue), and propane (green)] when propene to hydrogen ratio 1:1 for 3A and 1:2 for 3C. 3B and 3D represent only alkanes (blue) except propane (green) when propene to hydrogen 1:1 for 3B

and 1:2 for 3D in a continuous flow reactor for hydro-metathesis reaction catalyzed by catalyst precursor 1.

We previously showed that W(CH₃)₆ grafted on silica-alumina catalyst is a much better catalyst for alkane metathesis reaction than the silica counterpart [20], we thought to move from W(CH₃)₆ supported silica catalyst to silica-alumina catalyst. As our above results showed that propene

to hydrogen 1:2 ratio is better we choose to use 1:1 ratio along with 1:2 ratio of propene to hydrogen for hydro-metathesis reaction.

Fig. 4. 4A and 4C represent total conversion (Olefins + Alkanes, red line) and corresponding TON (blue line), 4B and 4D represent conversion of propene to only alkanes except propane (red line) and TON (blue line) in hydro-metathesis reaction of propene (using propene to hydrogen ratio 1:1 for 4A and 4B and 1:2 ratio for 4C and 4D) in a continuous flow reactor at 150 °C in presence of catalyst precursor 2 (75 mg), hydrogen and argon.

In a typical reaction 75 mg of W/silica-alumina (15 micromoles W) catalyst 2 were loaded in a continuous flow reactor, propene 6 mL/min and hydrogen 6 mL/min in the presence of argon were passed on the catalyst bed at 150 °C for 24 hours. The overall conversion of 57 % with a TON of 6402 was obtained. After removing the propane and olefins we got around 29 % conversion and 3279 TON (only higher and lower alkanes).

Additionally, 1:2 propene to hydrogen ratio also tested and as expected we observed a higher overall TON of 10736 (which contain olefins (except propene), propane and other alkanes) and a TON of 4577 was obtained for only alkanes after excluding olefins and propane from the products.

It is worth noting that the results obtained using W/silica-alumina are much better than the results obtained using W/silica and Ta/KCC-1. While comparing the product distribution in both W/silica and W/silica-alumina we observed that in both cases we could obtain alkanes from C₁ to C₁₀ with different quantities.

Fig. 5. **5A** and **5C** represent total distribution of products [olefins (), alkanes () and propane ()] when propene to hydrogen ratio 1:1 for **5A** and 1:2 for **5C**. **5B** and **5D** represent only alkanes () except propane () when propene to hydrogen 1:1 for **5B** and 1:2 for **5D** in a continuous flow reactor for hydro-metathesis reaction catalyzed by catalyst precursor **2**.

In the case of W/silica-alumina the conversion of propene to higher and lower alkanes is more, and hence the TON is higher than in the case of W/silica. Both catalysts form almost similar product distribution with the formation of ethane is higher followed by n-butane, pentane, hexane, heptane, octane, nonane, and decane. Formation of methane, ethane, butane, and pentane is

understandable and it comes from the direct addition of hydrogen to olefin metathesis products (Fig. 6). The case of methane is just the positive effect of hydrogen on a W-methylidene moiety.

2.2. Mechanistic outlook

Interestingly, along with C₁ to C₅ alkanes, we also observe C₆ to C₁₀ alkanes as minor products. Whereas C₁ to C₅ alkanes come from the direct metathesis of the starting olefin followed by reduction of the newly formed olefins, higher alkanes are believed to be formed by the competitive cross-metathesis reaction between the newly formed higher olefins resulting in the formation of the higher alkanes after reduction.

Looking at the mechanism (Fig. 6) we could observe that there is a very little difference between alkane metathesis and hydro-metathesis reaction [6]. In alkane metathesis, the metal-alkyl bond is formed by C-H bond activation whereas in hydro-metathesis reaction metal-alkyl bond is formed by insertion of the olefin to metal hydride bond followed by formation of a metalla-carbene hydride (by α -H elimination).

Besides, the mechanism conveys us that substituents in 1,2-position in the metallacyclobutane intermediate (of the Chauvin mechanism) [26] is less favored than the substitution in 1,3-position due to steric reason, which clearly explains why we could observe more ethane and n-butane as compared to methane and other alkanes [26]. Furthermore, in the case of branched alkanes according to the stereo chemical rules of olefin metathesis [27] we should observe isobutane rather than isopentane (1,3-di-substituted metallacyclobutane is favored over 1,2-di-substituted metallacyclobutane). We are exactly observing that trend in our product distribution curve (Intermediates 4 favored over 5 whereas intermediate 6 favored over 7) and intermediate 8 is more favored over 9.

Fig. 6. The proposed mechanism of propene hydro-metathesis using W catalyst.

3. Conclusions

We employed two well-defined W catalysts for the first time for hydro-metathesis reaction. Our target was to convey a message where lower olefins can be converted to transportation fuel range alkanes. As literature reports conveyed W-based catalysts are the better catalysts for olefin metathesis reaction than that of Ta-based catalyst, we employed W-based catalysts for hydro-metathesis reaction, as olefin metathesis reaction is one of the main paths of the hydro-metathesis reaction. To our expectation, we observed higher range alkanes with very high TON 4577 which is not possible while using propane as a reactant. The unprecedented formation of n-decane from propene was observed and opens the way of converting butenes to petroleum range alkanes using hydro-metathesis reaction. We believe this

study has many perspectives in future for an efficient hydro-metathesis reaction.

4. Experimental

4.1. General

All experiments were carried out by using standard Schlenk and glove box techniques under an inert nitrogen atmosphere. The syntheses and the treatments of the surface species were carried out using high vacuum lines ($< 10^{-5}$ mbar) and glove-box techniques. Pentane was distilled from a Na/K alloy under N_2 and dichloromethane from CaH_2 . Both solvents were degassed through freeze-pump-thaw cycles. $SiO_2-Al_2O_{3-500}$ was prepared by calcination at $400^\circ C$ in presence of air followed by dehydroxylation at $500^\circ C$ under high vacuum ($< 10^{-5}$ mbar) for 24 h containing $0.80-0.90$ OH/nm 2 . SiO_2-700 prepared from Aerosil silica from Degussa (specific area of 200 m 2 /g), which

were partly dehydroxylated at 700 °C under high vacuum ($< 10^{-5}$ mbar) for 24 h to give a white solid having a specific surface area of 190 m²/g and containing around 0.5-0.7 OH/nm². Propylene (99.99 %), Hydrogen (99.9999 %), Argon (99.999 %) and calibration mixture (methane, ethane, ethylene, propane, propylene, iso-butane, n-butane, trans-2-butene, 1-butene, iso-butene, cis-2-butene – each component 5000 ppm) gases were purchased from Specialty Gases Center of Abdullah Hashim Industrial Gases & Equipment Co. Ltd. (Saudi Arabia). Propylene cylinder was connected successively to Agilent Gas Clean Moisture and Agilent Gas Clean Oxygen filters prior the mass flow controller of the reactor. Argon and Hydrogen cylinders were connected to Thermo Scientific Triple filters (Oxygen, Moisture, Hydrocarbons) prior the mass flow controllers of the reactor. W(CH₃)₆ (SI Fig. S5 and S7) and supported pre-catalyst **1** and **2** were prepared according to literature procedures (SI Fig. S8 and S9) [14, 20]. IR spectra were recorded on a Nicolet 6700 FT-IR spectrometer by using a DRIFT cell equipped with CaF₂ windows (SI Fig. S8 and S9). The IR samples were prepared under argon within a glove box. Typically, 64 scans were accumulated for each spectrum (resolution 4 cm⁻¹). Elemental analyses were performed at Mikroanalytisches Labor Pascher (Germany) and Core Lab ACL KAUST.

4.2. GC Analysis

The gas products were analysed every 36 min by on-line gas chromatography using a Varian 450-GC system with a flame-ionisation detector (FID) for analysis of hydrocarbons. Column: GS-GASPRO; 30 m length, 0.32 mm diameter (Agilent J&W GC Columns). Method: Front injector setpoint 220 °C, oven: 40 °C hold for 2 minutes followed by 10 °C/min rate up to 220 °C hold for 10 minutes. TCD heater set point 175 °C, filament temperature 225 °C. FID set point 250 °C. Split ratio 20. GC was calibrated and quantification of the products was performed using calibration mixture. Retention times for each component of the calibration mixture was established based on at least 3 independent runs and used for calibration curve. The analysis of higher molecular weight products (from n-pentane to n-decane) was conformed using GC-MS.

4.3. GC-MS Analysis

For the GC-MS analysis, gaseous products of the reaction were collected to LabPure FEP Gas Bag 6x6 Septum connected to the vent of the reactor and analysed at KAUST Core Lab.

4.4. Hydro-metathesis reaction

Catalytic propylene hydro-metathesis was performed on a micropilot (PID Eng&Tech) equipped with a stainless steel reactor at atmospheric pressure. The catalyst powder was placed in the reactor supported by preliminary dried quartz wool. Conversions were calculated based on carbon mass balance.

4.4.1 Reaction A (Silica-supported catalyst, 1:1 ratio of reactants):

[W(Me)₆]/SiO₂₋₇₀₀ (106 mg, loading 2.60 % of W based on ICP-OES, 15 micromol W) was charged in a glove-box, into a 0.5 inch diameter stainless steel reactor fitted with a four-channels isolating valve. After connection to the rack, all the tubing were purged bypassing the reactor by a mixture of helium (80 ml/min), argon (30 mL/min) and hydrogen (20 mL/min) flow for 30 minutes, with argon and hydrogen flow for 20 minutes and with the reaction mixture – 6 mL/min of propylene, 6 mL/min of hydrogen and argon (10 vol %) for additional 10 minutes. Then the four-channel valve was switched on and the reactor was heated at 150 °C and 1 atm. The gas products were analysed on line with a GC.

4.4.2 Reaction B (Silica-supported catalyst, 1:2 ratio of reactants):

Reaction B (1:2 ratio of reactants): [W(Me)₆]/SiO₂₋₇₀₀ (106 mg, loading 2.60 % of W based on ICP-OES, 15 micromol W) was charged in a glove-box, into a 0.5 inch diameter stainless steel reactor fitted with a four-channels isolating valve. After connection to the rack, all the tubing were purged bypassing the reactor by a mixture of helium (80 ml/min), argon (30 mL/min) and hydrogen (20 mL/min) flow for 30 minutes, with argon and hydrogen flow for 20 minutes and with the reaction mixture – 6 mL/min of propylene, 12 mL/min of hydrogen and argon (10 vol%) for additional 10 minutes. Then the four-channel valve was switched on and the reactor was heated at 150 °C and 1 atm. The gas products were analysed on line with a GC.

4.4.3 Reactions C and D (Silica-Alumina-supported catalyst):

Reaction C (1:1 ratio of reactants) and D (1:2 ratio of reactants): $[\text{W}(\text{Me})_6]/\text{SiO}_2\text{-Al}_2\text{O}_{3.500}$ have been done with the same procedure with 75 mg of catalyst (loading 3.65 % of W based on ICP-OES) having the same amount micromoles of W catalyst in the reactor.

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Highlights

- Highest TON in hydro-metathesis has been achieved till date
- Silica-alumina supported $W(Me)_6$ proved to be more active than silica-supported catalyst
- Proposed mechanism is in good agreement with experimental results
- For the First time we observed n-decane from the hydro-metathesis reaction of propene