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Direct Synthesis of Renewable Dodecanol and Dodecane with Methyl Isobutyl Ketone over Dual-Bed Catalyst Systems

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Abstract: For the first time, we demonstrated two integrated processes for the direct synthesis of dodecanol or 2,4,8trimethylnonane (a jet fuel range C₁₂ branched alkane) with methyl isobutyl ketone (MIBK) which can be derived from lignocellulose. The reactions were carried out in two-bed continuous flow reactors. In the first bed, MIBK was selectively converted to a mixture of C12 alcohol and ketone. Over the Pd modified magnesium-aluminium hydrotalcite (Pd-MgAI-HT) catalyst, high total carbon yield (73.0%) of C12 oxygenates can be achieved under mild conditions. In the second bed, the C12 oxygenates generated in the first bed was hydrogenated to dodecanol over the Ru/C catalyst or hydrodeoxygenated to 2,4,8-trimethylnonane over the Cu/SiO₂ catalyst. The dodecanol as obtained can be used as the feedstocks in the production of sodium dodecylsulphate (SDS) and sodium dodecyl benzene sulfonate (SDBS) which are widely used as surfactants or detergents. The 2,4,8-trimethylnonane as obtained can be blended into conventional jet fuel without hydroisomerization.

With the decline of fossil energy and the increase of social concern about environmental problems, the catalytic conversion of renewable and inedible biomass to fuels^[1] and chemicals^[2] has become a very hot research topic. Lignocellulose is the major component of the agriculture wastes and forest residues. Compared with other forms of biomass, lignocellulose is much cheaper and more abundant. In the recent years, the substitution of petroleum with lignocellulose-derived platform molecules as the feedstocks in the production of liquid fuels^[3] and bulk chemicals^[4] has drawn tremendous attention.

Acetone is a by-product in the manufacture of bio-butanol and bio-ethanol by the acetone-butanol-ethanol (ABE) fermentation of lignocellulose. In a typical ABE fermentation process, butanol, acetone and ethanol are produced at a weight ratio of 6:3:1.^[5] With the large scale utilization of bio-butanol and bio-ethanol as the substitute of gasoline, the exploration of big outlets for acetone is attracting more and more attention.^[6] Methyl isobutyl ketone (MIBK) is a important chemical which has been produced in industrial scale by the one-step self-aldol condensation/ selective hydrogenation of acetone.^[7] Dodecanol is an

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intermediate in the production of sodium dodecylsulphate (SDS)^[8] or sodium dodecyl benzene sulfonate (SDBS)^[9] which are widely used as surfactants or detergents. Jet fuel is one of the most often used transportation liquid fuels. To the best of our knowledge, there is no report about the direct production of renewable dodecanol or jet fuel range hydrocarbon using MIBK as the feedstock. In this work, it was reported for the first time that dodecanol and 2,4,8-trimethylnonane (a jet fuel range C₁₂ branched alkane) can be directly synthesized in high carbon yields (~70%) by the reaction of MIBK and hydrogen over dualbed catalyst systems. In the first bed, MIBK was selectively converted to a mixture of C12 alcohol and ketone over the Pd-MgAI-HT catalyst. In the second bed, the C12 oxygenates generated from the first-bed was either hydrogenated to dodecanol over the Ru/C catalyst or hydrodeoxygenated to 2,4,8-trimethylnonane over the Cu/SiO₂ catalyst. The strategy for the two processes was illustrated in Scheme 1.



Scheme 1. Strategy for the direct production of dodecanol and 2,4,8-trimethylnonane with MIBK and hydrogen over dual-bed catalyst systems.

In the first part of this work, we investigated the reaction of MIBK and hydrogen over magnesium-aluminium hydrotalcite (MgAI-HT) and a series of noble metal modified magnesiumaluminium hydrotalcite (M-MgAI-HT, M = Pd, Ir, Pt, Ru) catalysts The tests were carried out by a fixed-bed continuous flow reactor (illustrated in Figure S1 of the supporting information) at 553 K and 0.6 MPa H₂. To facilitate the comparison, the atom ratios of M/AI in the M-MgAI-HT catalysts were fixed as 0.02:1. According to the analysis results of GC-MS (illustrated in Figures S2-S4 of the supporting information), 2,6,8trimethylnon-5-en-4-one (i.e. the compound 1 in Scheme 2) from the self-condensation of MIBK and some unknown heavy compounds were detected as the products over the MgAI-HT catalyst. In contrast, 2,6,8-trimethyl-4-nonanone and 2,6,8trimethyl-4-nonanol (i.e. compound 2 and 3 in Schemes 1 and 2) were identified as the two major products from the selfcondensation/hydrogenation of MIBK over the M-MgAI-HT catalysts. Besides the compounds 2 and 3, methyl-isobutyl carbinol (MIBC) was also detected in the product. This compound was generated by the hydrogenation of MIBK over the M-MgAI-HT catalysts. It is worthy mention that compound 1

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or 2,6,8-trimethylnon-5-en-4-ol (*i.e.* the compound **4** in Scheme 2), from the hydrogenation of C=O bond in compound **1** was not detected in the products over the M-MgAI-HT catalysts. This result can be rationalized because the hydrogenation of C=C bond in compound **1** is very fast and thermodynamicly more favorable than the hydrogenation of C=O bond.^[10]



Scheme 2. Reaction pathways for the generation of MIBC, compounds 1, 2 and 3 from the reaction of MIBK and hydrogen over the MgAI-HT and M-MgAI-HT catalysts.



Figure 1. MIBK conversion (black bar) and carbon yields of compound 1 (green bar), compound 2 (red bar), compound 3 (blue bar) and MIBC (white bar) over the MgAI-HT and M-MgAI-HT (M = Pd, Ir, Pt, Ru) catalysts. Reaction conditions: 1.0 g catalyst, 523 K, 0.60 MPa H₂, hydrogen flow rate: 150 mL min⁻¹.

From Figure 1, we can see that the MgAI-HT catalyst has low activity and selectivity for the production of C12 oxygenates from the condensation of MIBK. Over it, 47.0% MIBK conversion and 5.2% carbon yield of compound 1 was achieved at 523 K. After being modified with small amount of noble metals (Pd, Ir, Pt or Ru), the MIBK conversion and carbon yields of C12 oxygenates (i.e. compounds 2 and 3) over the MgAI-HT catalyst were significantly improved. Among the investigated M-MgAI-HT catalysts, Pd-MgAI-HT exhibited the highest activity and selectivity for the conversion of MIBK to compound 2 and 3 (see Figure 1). Over it, high MIBK conversion (90.6%) and good total carbon yield (72.0%) of compound 2 and 3 were achieved at 523 K and 0.6 MPa H_2 . To figure out the promotion effect of noble metals on the condensation of MIBK over the MgAI-HT catalyst, we also studied the activity of Pd-MgAI-HT for the selfcondensation of MIBK under nitrogen atmosphere. To faciliate the comparsion, the experiment was carried under the same reaction conditions as we used under hydrogen atomosphere. As we can see from Figure S5 in supporting information, low MIBK conversion (18.7%) and carbon yields of compound 1

(3.8%) were achieved over the Pd-MgAI-HT under nitrogen atmosphere. These values are even lower than those obtained over MgAI-HT under the same reaction conditions. Based on these results, we can see that the hydrogenation of compound 1 is favorable for the production of compound 2 and 3. This result can be expalined by two reason: As we know, the aldol condenation is a reversible reaction. Therefore, the hydrogenation of compound 1 from the self-aldol condensation of MIBK can decrease its concentration in the reaction system which is benefical for the generation of C₁₂ oxygenates from the pointview of reaction equilibrium. Furthermore, the hydrogenation of compound 1 can also restrain the retro-aldol condensation, which may be another reason for the promotion effect of noble metals. From Figure 1, it was also noticed that the carbon yield of unexpected MIBC (8.8%) over the Pd-MgAl-HT catalyst was evidently lower than those over the other M-MgAI-HT catalysts (>20%), which may be one reason for the excellent performance of the Pd-MgAI-HT catalyst. According to literature,^[11] the Pd catalyst is highly selective for the hydrogenation of C=C bond (instead of C=O bond) especially when it is loaded on basic supports. As we know, the hydrogenation of C=O bond in MIBK molecule will decrease its concentration in the reaction system, which is unfavorable for the self-aldol condensation of MIBK (or the generation of compound 2 and 3) from the pointview of reaction equilibrium.

The effects of Pd content, hydrogen pressure and reaction temperature on the catalytic performance of Pd–MgAI-HT were also studied. As we can see from Figures S6 in supporting information, the activity of Pd–MgAI-HT catalyst increases with the increment of Pd content, reaches the maximum when the atomic ratio of Pd/AI is about 0.02, and decreases with the further increment of the Pd/AI atomic ratio in Pd–MgAI-HT catalyst. Analogously, we also noticed that there is also an optimal hydrogen pressure (or reaction temperature) for the total carbon yield of C₁₂ oxygenates over the Pd_{0.02}-MgAI-HT catalyst, respectively. According to the results shown in Figures S7 and S8, the highest total carbon yield of C₁₂ oxygenates (73.0%) was achieved over the Pd_{0.02}-MgAI-HT catalyst when the reaction was conducted at 553 K and 0.6 MPa H₂.



Figure 2. Conversion of feedstock (black bar) and carbon yields of ketone dimer (red bar) and alcohol dimer (blue bar) over the $Pd_{0.02}$ -MgAl-HT catalyst. Reaction conditions: 1.0 g catalyst, 553 K, 0.60 MPa H₂, hydrogen flow rate: 150 mL min⁻¹, ketone (or MIBC) flow rate: 0.05 mL min⁻¹.

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Subsequently, we also explored the applicability of Pd-MgAl-HT for the self-condensation of other ketones which can be derived from lignocellulose. Among them, mesityl oxide can be produced from the self-aldol condensation of acetone.^[12] The 2pentanone and 2-heptanone can be obtained by alkylation reaction of the acetone and ethanol (or butanol).^[3f, 13] Moreover, 2-pentanone can also be obtained a by-product in the selective hydrogenation of furfural to 2-methylfuran,^[14] a feedstock in the production of diesel and jet fuel range branched alkanes.^[15] According to Figure 3, the Pd-MgAI-HT is active for the condensation of all the investigated ketones. Over the Pd_{0.02}-MgAI-HT catalyst, high carbon yields of C10-C14 oxygenates (59.7-76.2%) were obtained under the same reaction conditions as we used for MIBK. Compare with MIBK, slightly lower carbon yield of C12 oxygenates (68.5% vs. 73.0%) was obtained when we used mesityl oxide as the feedstock. This can be explained because mesityl oxide is less reactive than MIBK (due to its conjugated structure). As the result, mesityl oxide must be hydrogenated to MIBK then form C₁₂ oxygenates by self-aldol condensation. It is very interesting that high carbon yield of C12 oxygenates was achieved when we used MIBC as the feedstock. Compared with MIBK, MIBC is less reactive. This can be comprehended because MIBC must be dehydrogenated to MIBK before it is converted to compounds 2 and 3.



Figure 3. Carbon yields of dodecanol (\blacktriangle) and MIBC (\blacktriangledown) from the reaction of MIBK and hydrogen over the dual-bed catalyst system of Pd_{0.02}-MgAI-HT and 5wt.% Ru/C. Reaction conditions: 0.6 MPa H₂, 1.0 g Pd_{0.02}-MgAI-HT for the first bed (553 K) and 1.0 g 5wt.% Ru/C for the second bed (373 K), hydrogen flow rate: 150 mL min⁻¹, MIBK flow rate: 0.05 mL min⁻¹.

As the final aim of this work, we explored the possibility for the direct synthesis of dodecanol and jet fuel range branched alkane with MIBK and hydrogen over dual-bed catalyst systems. In the first bed, MIBK was condensed to C12 oxygenates over the Pd_{0.02}-MgAI-HT catalyst under the optimal reaction conditions (553 K and 0.6 MPa H₂). In the second bed, the C₁₂ oxygenates generated in the first bed was further hydrogenateded to dodecanol when 5wt.% Ru/C was used as the catalyst (see Figure S9 in supporting information). The carbon yield of dodecanol from such a dual-bed catalyst system is about 70% which is very close to the carbon yield C_{12} oxygenates (73.0%) in the first bed. As potential applications, the dodecanol as obtained can either be sulphated to produce dodecylsulphate (SDS)^[8] or be dehydrated to dodecene wich is used as feedstock in the production of sodium dodecyl benzene sulfonate (SDBS).^[9] The by-product MIBC can be recycled and used as feedstock to increase the carbon yield of dodecanol.



Figure 4. Carbon yields of 2,4,8-trimethylnonane (▲) and 2-methylpentane (▼) from the reaction of MIBK and hydrogen over the dual-bed catalyst system of Pd_{0.02}-MgAI-HT and 5wt.%Cu/SiO₂. Reaction conditions: 553 K, 0.60 MPa H₂; 1.0 g Pd_{0.02}-MgAI-HT catalyst, 1.5 g 5wt.%Cu/SiO₂ catalyst, hydrogen flow rate: 150 mL min⁻¹, MIBK flow rate: 0.05 mL min⁻¹.

As another option, the C_{12} oxygenates generated in the first bed can also be hydrodeoxygenated to 2,4,8-trimethylnonane and 2-methylpentane when 5wt.% Cu/SiO₂ was used as the catalyst in the second bed (see Figures S9-S11 in supporting information). High carbon yield (~70%) of 2,4,8-trimethylnonane was achieved over such a dual-bed catalyst system. The 2,4,8trimethylnonane as obtained has the carbon chain length of jet fuel range and branched chemical structure. As a potential apllication, it can be blended into conventional jet fuel without hydroisomerization. The 2-methylpentane which is obtained as the by-product from this dual-bed catalyst system can be used as renewable gasoline.

The stabilities of both dual bed catalyst systems were studied. During the 24 h continuous tests, no evident deactivation was observed, which means the catalysts are stable under the investigated conditions. In the future research, it is also possible to synthesize renewable dodecanol and jet fuel range alkanes with hexanol or butanol which can be selectively obtained from the hydrogenolysis of cellulose or 1,4-anhydroerythritol.^[16]

A new route was developed for the direct synthesis of dodecanol and jet fuel ranged branched alkane with MIBK which can be obtained from lignocellulose. The reactions were conducted by dual-bed catalyst systems. In the first bed, the MIBK was condensed into a mixture of C12 ketone and C12 alcohol (C12 oxygenates). Among the investigated catalysts, the Pd_{0.02}-MgAI-HT catalyst demonstrated the best performance. Over it, 73.0% carbon yield of C₁₂ oxygenates was achieved at 553 K and 0.6 MPa. In the second bed, the condensation products generated in the first bed were further hydrogenated to dodecanol over the Ru/C catalyst or hydrodeoxygenated to 2,4,8-trimethylnonane (a jet fuel range C₁₂ branched alkanes) over the Cu/SiO₂ catalyst. High carbon yields (~70%) of dodecanol and 2,4,8-trimethylnonane were achieved by the dual-bed catalyst systems. The dual-bed catalyst systems were stable under invesigated conditions. No evident deactivation was

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observed during the 24 h continuous test. This work paves a new way for the synthesis of renewable surfactants and jet fuel range branched alkane with lignocellulosic platform compounds.

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For the first time, dodecanol and jet fuel range C_{12} branched alkane were directly synthesized in high carbon yield (~70%) by dual-bed catalyst systems with hydrogen and methyl isobutyl ketone which can be derived from lignocellulose.

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Direct Synthesis of Renewable Dodecanol and Dodecane with Methyl Isobutyl Ketone over Dual-Bed Catalyst Systems

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