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### Synthesis of jet fuel range high-density polycycloalkanes with polycarbonate waste

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This paper is dedicated to the 70<sup>th</sup> anniversary of Dalian Institute of Chemical Physics, Chinese Academy of Sciences.

Jet fuel range high-density polycycloalkanes were first synthesized with polycarbonate wastes by a two-step method which was conducted under mild conditions. In the first step, polycarbonate waste was converted to bisphenol by methanolysis. Subsequently, the bisphenol was further converted to polycycloalkanes by hydrodeoxygenation.

Due to the great social concern about the sustainable development and environment problems, the exploration of new organic carbon resources as the substitutes for fossil energies has gained tremendous attention.<sup>1</sup> Plastics are a family of organic polymers which are widely used in our life. Since 1950s, about 6.3 billion tons of plastics have been produced worldwide. However, only a small part of plastic was recycled (9%) or incinerated (12%).<sup>2</sup> Most of them were discarded after usage. Moreover, the chemical structure of most plastics renders them resistant to bio-degradation, which will lead to a lot of environmental and ecological problems.<sup>3</sup> From the point views of environment protection and full utilization of resources, it is imperative to develop some efficient catalytic technologies for the catalytic conversion valorization of waste plastics to great demanded fuels and/or bulk chemicals.<sup>4</sup> Polycycloalkanes are major components of advanced jet fuels which have been widely used for aircraft and propulsion.<sup>5</sup> Compared with conventional jet fuels, polycycloakanes have higher density (or volumetric heat value). Therefore, the utilization of polycycloalkanes can significantly increase the flight range, payload and speed of aircraft without change the volume of oil tank. To the best of our knowledge,

there is no report about the production of high-density aviation fuel with waste plastics.

Polycarbonate (PC) is a leading engineering plastic which has a high annual production capacity (~6 million tons).<sup>6</sup> Due to its excellent resistance to high temperature and impact, good ductility and optical clarity, PC has been widely used in digital storage media, electronics, construction, containers, medical, automotive industry, protective glasses, etc..7 However, the great demand of PCs also leads to plenty of PC waste. Traditional waste treatments such as landfill or incineration are not environmentally sustainable because bisphenol A, the major monomer of PC, has been considered as a xenoestrogen.8 As the result, the chemical conversion of PC has drawn a lot of attention in recent years. So far, most of reported work was concentrated on the pyrolysis,<sup>9</sup> hydrolysis,<sup>10</sup> alcoholysis,<sup>6, 11</sup> aminolysis<sup>12</sup> and glycolysis<sup>13</sup> of PC wastes. In this work, it was first reported that jet fuel range polycycloalkanes can be selectively produced by the alcoholysis of PC waste, followed by hydrodeoxygenation. The polycycloalkanes as obtained have high density. As a potential application, they can be used as high-density aviation fuels or additives to increase the volumetric heat values of current jet fuels. The strategy for this process is illustrated in Scheme 1.



Scheme 1. Strategy for the synthesis of jet fuel range  $C_{13}$ - $C_{15}$  polycycloalkanes with PC wastes.

First of all, we investigated the alcoholysis and glycolysis of PC waste. It was noticed that these reactions can even take place in the absence of catalyst. Based on our analysis (see Figure S1 in supporting information, both bisphenol A (*i.e.* **1A** in Scheme 2) and corresponding carbonates were identified as the products. The reaction pathways were proposed in

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Scheme 2. From it, we can see that the **1A** as obtained has bicyclic carbon chain structure and proper carbon chain length for jet fuel. Therefore, it can be potentially used as a precursor for high-density jet fuel. The carbonates which were generated accompanying with **1A** can be used as feedstocks for the production of many chemicals, solvents and the fuel additives to increase the octane number or oxygen content of gasoline.<sup>14</sup> Among the investigated alcohols and polyols, methanol exhibited the highest reactivity for the degradation of PC waste (see Figure 1). Under the investigated conditions (453 K, 3 h), the PC waste was completely converted, high yield (~90%) of **1A** was achieved in the absence of any catalyst. Taking into the consideration of lower price of methanol, we believe it is a promising reactant to produce **1A** from PC wastes.



Scheme 2. Reaction pathways for the alcoholysis or glycolysis of PC waste.





The effects of reaction conditions on the conversion of PC waste and **1A** yield were studied. From Figure 2, it was noticed that the PC waste conversion and **1A** yield increased with the increment of reaction temperature then stabilized when the reaction temperature was higher than 453 K. This result means that high reaction temperature is favorable for the methanolysis of PC waste. With the increase of initial substrate dosage from 1 g to 7 g, no evident decrease of PC waste conversion or **1A** yield was observed (see Figure 3). This is advantageous in real application. However, the further





**Figure 2.** Conversion of PC waste and the yield of **1A** in methanol as the function of reaction temperature. Reaction conditions: 3 h; 1 g PC waste and 40 mL methanol were used for each test.



**Figure 3.** Conversion of PC waste and the yield of **1A** in methanol as the function of initial substrate dosage. Reaction conditions: 453 K, 3 h; 40 mL methanol were used for each test.

Subsequently, we explored the synthesis of polycycloalkane by the HDO of 1A. First, we compared the performances of activated carbon loaded noble metal catalysts for this reaction. From Figure 4, it was noticed that Pt/C has significantly higher activity than those of Pd/C and Ru/C for the hydrogenation of 1A. According to the characterization results illustrated in Table S1 in supporting information, no clear relationship was observed between the activities of catalysts and their specific surface areas, average metal particle sizes or metal dispersions. Therefore, we think that the excellent performance of Pt/C should be attributed to its higher activity for the hydrogenation of bezene ring in aromatic compounds.<sup>15</sup> Based on our analysis (see Figures S2-S4 in supporting information), and 4,4'-(propane-2,2-diyl)dicyclohexanol 4-(2cyclohexylpropan-2-yl)cyclohexanol (i.e. 1B and 1C in Scheme 3) from the total hydrogenation and partial HDO of 1A were identified as the major products. No propane-2,2diyldicyclohexane (i.e. 1D in Scheme 3) from the complete

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HDO of **1A** was detected in the product even after we increased the reaction temperature from 373 K to 453 K (see Figure 5).



Scheme 3. Reaction pathways for the hydrodeoxygenation (HDO) of 1A. Reaction conditions: 373-453 K, 3 MPa  $H_2$ , 1 h; 5 mmol 1A, 40 mL cyclohexane, 0.04 g Pt/C catalyst and 0.5 g (or 0 g) zeolite were used for each test.



**Figure 4.** Conversions of **1A** and the yields of products over the M/C (M = Pt, Pd, Ru) catalysts. Reaction conditions: 373 K, 3 MPa H<sub>2</sub>, 1 h; 5 mmol **1A**, 40 mL cyclohexane, 0.04 g catalyst were used for each test.



**Figure 5.** Conversion of **1A** and the yield of products over the Pt/C as the function of reaction temperature. Reaction conditions: 3 MPa  $H_2$ , 1 h; 5 mmol **1A**, 40 mL cyclohexane, 0.04 g Pt/C catalyst were used for each test.

To overcome this problem, some acidic zeolites (such as H-MOR, H-ZSM-5 and H- $\beta$ ) were introduced as the co-catalysts for the HDO of **1A**. As we expected, the presence of acidic zeolites significantly increased the carbon yield of **1D** from the complete HDO of **1A** (see Figures S5-S9 in supporting information). This can be explained because the presence of acid sites promotes the dehydration of **1B** and **1C**. In the

previous work of Huber et al.,<sup>16</sup> the dehydration, followed by hydrogenation has been found to be the major pathway for the HDO of biomass derived oxygenates. Among the investigated zeolites (see Figure 6), the H- $\beta$  exhibited the best promotion effect. This can be rationalized by two reasons: 1) Pore size. As we can see from the structures of different zeolite supports illustrated in Figure S10 in supporting information, H-ZSM-5 zeolite only has 10-membered pore. In contrast, the H- $\beta$  zeolite has 12 members pores in three dimensions whereas H-MOR has 12 member pores only in one dimension (8 members in another dimension).<sup>17</sup> The bigger pore size of H- $\beta$  is beneficial for mass transfer that is very important for the liquid-phase reactions at low temperature. 2) Acidity. Based on the NH<sub>3</sub>-TPD and NH<sub>3</sub> chemisorption results shown in Figure 7 and Table 1, the H-β zeolite has higher acid strength and acid amount than those of H-MOR. This may be another reason why the H- $\beta$  zeolite is better than the H-MOR for the HDO of 1A. Besides 1D, some C7-C8 cycloalkanes were also obtained from the HDO of 1A. According to literature,18 the C7-C8 cycloalkanes may generated from the hydrocracking of 1D which is easier to occur at the highly branched carbon (this is because the alkyl group has electron donation effect which can increase the stability of carbonium ion). As a potential application, these cycloalkanes can be blended into the current gasoline or jet fuel.



Figure 6. Conversion of 1A and the yields of products under the co-catalysis of Pt/C and acidic zeolites. Reaction conditions: 453 K, 3 MPa  $H_2$ , 1 h; 5 mmol 1A, 40 mL cyclohexane, 0.04 g Pt/C and 0.5 g zeolite were used for each test.

Table 1. Specific surface areas ( $S_{BET}$ ), acid mounts and pore sizes of the zeolites used in this work.

Zeolite	S <sub>BET</sub>	Acid amount	Pore size	Reference	
	(m² g-1)a	(mmol g <sup>-1</sup> ) <sup>b</sup>	(nm) <sup><i>c</i></sup>		
H-MOR	413	0.79	0.65×0.7	17	
			0.26×0.57		
H-ZSM-5	311	0.99	0.51×0.55	17, 19	
			0.53×0.56		
Η-β	482	0.68	0.66×0.67	19	
			0.56×0.56		

<sup>a</sup> Measured by N<sub>2</sub>-physisorption. <sup>b</sup> Measured by chemisorption. <sup>c</sup> The values reported in literature.

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Figure 7.  $NH_3\text{-}TPD$  profiles of the H-MOR (a), H-ZSM-5 (b) and H- $\beta$  (c) zeolites.

The influences of reaction temperature and reaction time on the catalytic performance of Pt/C + H- $\beta$  catalyst were investigated as well (see Figures 8 and 9). Under the optimized conditions, high yield (87%) of **1D** was achieved. According to literature,<sup>20</sup> this compound has high density (0.92 g mL<sup>-1</sup>) at room temperature. Therefore, it can be potentially used as high-density aviation fuel or additive to improve the volumetric heat values of current jet fuels.



**Figure 8.** Conversion of **1A** and the yields of products over the Pt/C + H- $\beta$  catalyst as the function of reaction temperature. Reaction conditions: 3 MPa H<sub>2</sub>, 1 h; 5 mmol **1A**, 40 mL cyclohexane, 0.04 g Pt/C and 0.5 g H- $\beta$  were used for each test.



**Figure 9.** Conversion of **1A** and the yields of products over the Pt/C + H- $\beta$  catalyst as the function of reaction time. Reaction conditions: 413 K, 3 MPa H<sub>2</sub>; 5 mmol **1A**, 40 mL cyclohexane, 0.04 g Pt/C and 0.5 g H- $\beta$  zeolite were used for each test.

As we know, **1A** is produced by the condensation of acetone and phenol. Acetone can be obtained by the acetone-butanolethanol fermentation of lignocellulose.<sup>21</sup> Phenol can be derived from lignin.<sup>22</sup> Therefore, this work provides a new method for the production of renewable high-density aviation fuel with lignocellulose. As the analogues of 1A, 4,4'-(ethane-1,1-diyl)diphenol and 4,4'-methylenediphenol (i.e. 2A and 3A in Figure 10) are two bisphenols which be produced by the condensation of phenol with acetaldehyde and formaldehyde<sup>23</sup> from the partial oxidation of ethanol and methane from fermentation of lignocellulose. In real application, 3A has been widely used as a substitute for 1A.8 As an extension of this work, we also explored the HDO of 2A and **3A** over the Pt/C + H- $\beta$  catalyst (see Figure 10). To facilitate the comparison, the experiments were carried out under the optimized conditions for the HDO of 1A. As we expected, 2A and 3A were totally converted under the investigated conditions. High yields of jet fuel range polycycloalkanes were achieved (see Figure 10). It is very interesting that tricycloalkanes were also obtained accompanying with dicycloalkanes when we used 2A and 3A as the feedstocks (see Figures S11-S16 in supporting information). The tricycloalkane yield increases with the decrease of methyl group number in the substrates. This result means that the presence of alkyl group is unfavorable for the generation of tricycloalkanes. When 3A was used as substrate, the tricycloalkane **3F** was obtained as major product. This is advantageous in real application because tricycloalkanes have higher densities than those of dicycloalkanes (see Table 2).

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**Figure 10.** Results for the HDO of bisphenols over the Pt/C + H- $\beta$  catalyst. Reaction conditions: 413 K, 3 MPa H<sub>2</sub>, 4 h; 5 mmol bisphenol, 40 mL cyclohexane, 0.04 g Pt/C and 0.5 g H- $\beta$  zeolite were used for each test.

**Table 2.** Densities and the freezing points of polycycloalkane products.

Polycycloalkane	Density (g mL <sup>-+</sup> )	Freezing point (K)	Reference
	0.92	288.6	20
	0.89	252.1	20
	0.96	270.0	24
$\bigcirc \bigcirc \bigcirc$	0.88	254.3	20
	0.96	258.0	25

To get deeper insight into the cyclization during the HDO of **1A**, we investigated the influence of H- $\beta$  dosage on the catalytic performance of Pt/C + H- $\beta$  (see Figures 11). In the absence of H- $\beta$  zeolite, dodecahydro-1*H*-fluoren-3-ol (*i.e.* **3F** in Scheme 4) was obtained as the main product from the hydrogenation of **3A** over the Pt/C catalyst (see Figure 11 and Figures S17-S18 in supporting information). This result means that the cyclization can take place even in the absence of H- $\beta$ . With the increase of H- $\beta$  dosage, the **3E** yield increased which can be explained by the HDO of **3E** under the co-catalysis of Pt/C + H- $\beta$ . The **3E** yield reached the maximum when 0.5 g H- $\beta$ was used together with Pt/C. With the further increment of H- $\beta$  dosage, the **3E** yield increased. This can be explained by the further hydrocracking of **3E** to **3D** under the co-catalysis of Pt/C + H- $\beta$ .



Scheme 4. Reaction pathways for the generation of different products from the HDO of 3A over the Pt/C + H- $\beta$  catalyst.



**Figure 11.** Conversion of **3A** and the yield of products over Pt/C + H- $\beta$  as the function of H- $\beta$  dosage. Reaction conditions: 413 K, 3 MPa H<sub>2</sub>, 4 h; 5 mmol **3A**, 40 mL cyclohexane, 0.04 g Pt/C and H- $\beta$  zeolite were used for each test.

The effect of reaction time on the catalytic performance of Pt/C + H $\beta$  was studied as well. Based on the results illustrated in previous literature<sup>25, 26</sup> and Figure 12, the reaction pathways for the generation of different products from the HDO of **3A** over Pt/C + H- $\beta$  catalyst was proposed as Scheme 4. As the first step, one benzene ring of **3A** is hydrogenated, which leads to the generation of **3G**. **3G** is converted to **3H** over the acid sites which may be generated by the hydrogen spillover of Pt/C. Subsequently, **3H** is hydrogenated to **3F**. These three steps are very fast because neither **3G** nor **3H** is identified in the HDO product. Then, the **3F** is further hydrodeoxygenated to **3E** under the co-catalysis of Pt/C and H- $\beta$ . With the extension of reaction time, **3E** may be hydrocraked to **3D** and/or its isomers. As another possibility, **3D** may also be directly produced by the HDO of **3A**.



**Figure 12.** Conversion of **3A** and the yields of products over the Pt/C + H- $\beta$  catalyst as the function of reaction time. Reaction conditions: 413 K, 3 MPa H<sub>2</sub>; 5 mmol **3A**, 40 mL cyclohexane, 0.04 g Pt/C and 0.5 g H- $\beta$  zeolite were used for each test.

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#### Conclusions

Jet fuel range polycycloalkanes were selectively synthesized at high overall yield (~80%) by the methanolysis of PC waste, followed by the HDO. Methanol was found to be highly reactive for the alcoholysis of PC waste. In the absence of catalyst, high yield (~90%) of bisphenol A was achieved from the methanolysis of PC waste after the reaction was carried out at 453 K for 3 h. Subsequently, the bisphenol A was further hydrodeoxygenated to jet fuel range C<sub>15</sub> bicycloalkanes under the co-catalysis of activated carbon loaded noble metal and acidic zeolites. Among the investigated catalyst systems, the combination of Pt/C and H- $\beta$  (denotes as Pt/C + H- $\beta$ ) exhibited the best performance, which can be rationalized by the high activity of Pt/C for hydrogenation of benzene ring, the high pore size and acidity of H- $\beta$  zeolite. The Pt/C + H- $\beta$  catalyst is also applicable for the synthesis of C13-C15 polycycloalkanes with the bisphenols which can be derived from lignocellulose. The  $C_{13}$ - $C_{15}$  polycycloalkanes as obtained have higher densities. As a potential application, they can be used as advanced aviation fuels or fuel additives to improve the volumetric heat value of current jet fuels.

#### **Conflicts of interest**

There are no conflicts to declare.

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## Synthesis of jet fuel range high-density polycycloalkanes with polycarbonate waste

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Jet fuel range high-density  $C_{13}$ - $C_{15}$  cycloalkanes were first synthesized at high overall yield (~80%) with polycarbonate wastes.