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Development of a bridged structure compound from isophorone as a JP-10 alternative

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The growing environmental concerns regarding the use of fossil fuels have driven the advancement of sustainable transportation fuels. This study presents a method for synthesizing a novel jet fuel substitute from acetic acid *via* isophorone dimerization followed by reduction. The resultant compound exhibited fuel properties, such as density and energy density, that are comparable to those of JP-10. Techno-economic analysis and life-cycle assessment demonstrated its economic and environmental viability, showcasing it as a sustainable alternative to conventional jet fuels.

As environmental concerns, particularly those associated with fossil fuel usage, have increased, the development of fuels from renewable biomass feedstock has become significantly important for reducing adverse environmental effects, including greenhouse gases and other pollutants.¹ Thus, tremendous efforts have been made to develop renewable replacements for transportation fuels.² However, most studies have focused on developing upgrading strategies for biomass rather than synthesizing novel compounds that meet fuel specifications. In addition, it would be highly challenging to develop substitutes for high-density tactical fuels such as JP-10, considering their exceptional density of 0.94 g mL⁻¹.³ Fuels produced from cellulosic butanol (Biojet) featuring branched saturated hydrocarbons exhibited a density of only 0.78 g mL⁻¹.⁴ Meanwhile,

Sustainability spotlight

This work advances the field of green chemistry by developing a novel, sustainable jet fuel substitute derived from acetic acid, a renewable feedstock, with fuel properties, such as density, energy density, heat of combustion, and freezing point, comparable to those of JP-10. The work aligns with “Goal 7: Ensure access to affordable, reliable, sustainable and modern energy for all.”

saturated linear fuels obtained from syngas have a density of 0.75 g mL⁻¹.⁵ The relatively poor volumetric heating values of these fuels are attributed to their low densities. For example, the volumetric heating value of renewable Biojet is 34.3 MJ L⁻¹ compared to that of JP-10 at 39.6 MJ L⁻¹.³ Beyond linear hydrocarbons, bridged hydrocarbons have also been explored as candidates to replace JP-10. In 2021, cyclopropanated monoterpenes were synthesized and found to exhibit slightly higher energy density and heat of combustion than JP-10, although their flash points were somewhat lower.^{6,7} Likewise, bridged hydrocarbon analogues designed to mimic JP-10 also showed improvements in energy density and heat of combustion, but their freezing points were higher, and their flash points were again too low to be considered suitable for fuel application.⁸⁻¹⁰ Therefore, it is necessary to develop novel renewable compounds from biomass to replace tactical fuels.

The presence of a considerable amount of carboxylic acids, especially acetic acid and formic acid, leads to the instability and corrosiveness in the bio-oil.¹¹ Therefore, intensive efforts have been made to upgrade bio-oils by esterification of carboxylic acids with alcohols, followed by reducing esters to generate alcohols, thereby eliminating acidity and enhancing the stability of the bio-oil (Scheme 1a).^{12,13} However, the use of compounds synthesized *via* esterification and subsequent reduction methods as standalone fuels is challenging. Recently, a publication highlighted the economically viable and environmentally friendly transformation of acetone generated from acetic acid *via* ketonization into isophorone using calcium carbide (Scheme 1b).¹⁴ We anticipated that dimerizing

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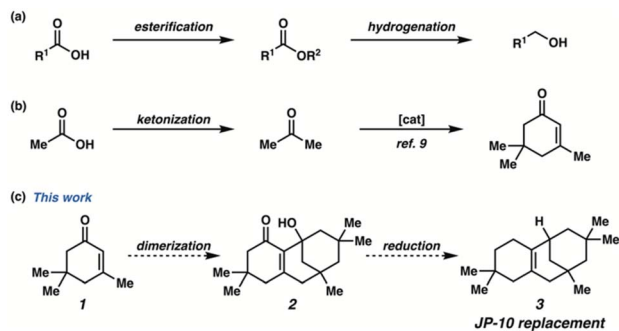
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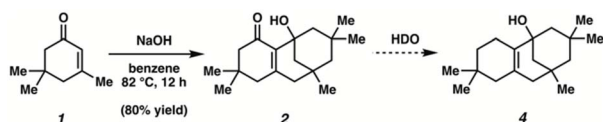


Scheme 1 (a) Removing acids from bio-oils by esterification followed by hydrogenation. (b) Synthesis of isophorone from acetic acid. (c) Design of tricyclic **3** for JP-10 replacement.

isophorone derived from acetic acid and subsequently reducing the resulting dimer to produce compound **3**, which is characterized by a significant ring strain, would enhance its heat of combustion (Scheme 1c). In this work, we established a synthetic route to obtain pure isophorone dimer **3**, which is a potential substitute for jet fuel, from acetic acid, a significant component of bio-oil.

The synthesis commenced with the preparation of isophorone dimer **2** from isophorone **1** under basic conditions (Scheme 2, see the SI for optimization of conditions). Treatment of isophorone **1** with NaOH at 82 °C produced isophorone dimer **2** in 80% yield. Hydrodeoxygenation (HDO) of ketone **2** on a scale of less than 1.0 g with PtO₂ catalysts at 1 bar afforded alcohol **4** in 47% yield (Table 1, entry 1). However, the yield of the HDO reaction decreased dramatically at the gram scale (entry 2). Therefore, we explored the HDO reaction conditions on a multi-gram scale for ketone **2** (Table 1). Screening of the solvent revealed that tertiary alcohol **4** was not observed in any solvents, except in AcOH with Adams' catalyst (entries 3 and 4). In addition, attempts to use palladium on carbon or palladium hydroxide as a catalyst were unsuccessful (entries 5–7). Increasing the hydrogen gas pressure to 70 bar not only improved the yield of tertiary alcohol **3** but also enhanced the reaction rate at 25 °C (entry 8). Trace amounts of deoxygenated tricyclic compound **3** were observed at elevated pressures. The HDO reaction at a higher temperature generated tertiary alcohol **4** in a slightly lower yield (entry 9). The desired product **4** was obtained in a lower yield with a longer reaction time (entry 10). We found that PtO₂ could be reused for the hydrodeoxygenation reaction, although the yield decreased slightly upon reuse (see the SI).

With tertiary alcohol **4** in hand, the hydrodeoxygenation of alcohol **4** was undertaken (Scheme 2). Hydrocarbon **3** was not

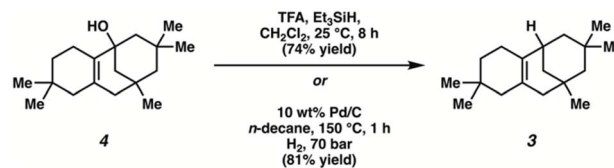


Scheme 2 Synthesis of isophorone dimer **2** from isophorone **1**, followed by the hydrodeoxygenation (HDO) reaction.

Table 1 Optimization of the hydrodeoxygenation (HDO) reaction of tertiary alcohol **3**

| Entry | Cat. (equiv) | Solvent | <i>T</i> (°C) | <i>P</i> (bar) | <i>t</i> (h) | Yield (%) |
|-------|------------------------|---------|---------------|----------------|--------------|---------------------|
| 1 | PtO ₂ (0.1) | AcOH | 25 | 1 | 72 | 47 ^{a,b} |
| 2 | PtO ₂ (0.1) | AcOH | 25 | 1 | 72 | trace ^c |
| 3 | PtO ₂ (0.1) | THF | 25 | 1 | 72 | — ^{c,d} |
| 4 | PtO ₂ (0.1) | MeOH | 25 | 1 | 72 | — ^{c,d} |
| 5 | Pd/C (1.0) | THF | 25 | 1 | 72 | — ^{c,d} |
| 6 | Pd/C (1.0) | MeOH | 25 | 1 | 72 | trace ^c |
| 7 | Pd(OH) ₂ | MeOH | 25 | 1 | 72 | — ^{c,d} |
| 8 | PtO ₂ (0.3) | AcOH | 25 | 70 | 2 | 69 ^{b,c} |
| 9 | PtO ₂ (0.1) | AcOH | 50 | 70 | 4 | 53 ^{b,c,e} |
| 10 | PtO ₂ (0.1) | AcOH | 25 | 70 | 4 | 50 ^{b,c} |

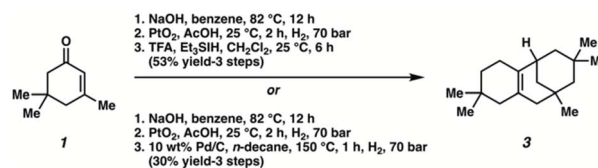
^a Results on a small scale (<1 g). ^b Isolated yield of tertiary alcohol **4**. ^c Results on a multigram scale. ^d Not observed. ^e Deoxygenated tricyclic **3** was obtained in 3% yield.



Scheme 3 Synthesis of deoxygenated tricyclic **3**.

detected using Lewis acids (*e.g.*, InCl₃) with hydride sources (see the SI for the optimization of conditions). To our delight, the tertiary alcohol was successfully removed with Et₃SiH and TFA, providing deoxygenated tricyclic **3** in 74% yield (Scheme 3). Additionally, hydrocarbon **3** was obtained *via* hydrodeoxygenation of tertiary alcohol **4** using 10 wt% Pd/C at 150 °C and 70 bar (Scheme 3).

To enhance synthesis efficiency and cost-effectiveness, a one-pot approach was employed to produce hydrocarbon **3** directly from isophorone **1** without intermediate purification. Although hydrocarbon **3** was obtained in higher yield from alcohol **4** under hydrodeoxygenation with Pd/C compared to the Et₃SiH and TFA reduction (Scheme 3), the Et₃SiH and TFA reduction protocol provided a higher overall yield of hydrocarbon **3** when



Scheme 4 One-pot synthesis of tricyclic **3**.



Table 2 Selected specifications of compound 3, JP-10, and JP-8

| | 3 | JP-10 | JP-8 |
|--|-------|----------------|----------------|
| Density ^a (g mL ⁻¹) | 0.91 | 0.94 | 0.78 |
| Energy density (MJ L ⁻¹) | 38.2 | 39.6 | 33.6 |
| Heat of combustion ^b (MJ kg ⁻¹) | 42.0 | 42.1 | 43.1 |
| Freezing point (K) | < 183 | < 194 | < 226 |
| Viscosity at 40 °C (mm ² s ⁻¹) | 7.61 | — ^c | — ^c |
| Flash point (°C) | 137.6 | 54.4 | 38 |
| Sulfur content (mg kg ⁻¹) | 3.25 | — ^c | — ^c |

^a Measured by the ASTM D4052 method. ^b Measured by the ASTM D4809 method. ^c Not available.

used as the final step in a one-pot synthesis starting from isophorone 1 (Scheme 4).

Subsequently, the specifications of compound 3, including its heat of combustion, density, energy density, freezing point,

viscosity, flash point and sulfur content were analyzed and compared with those of JP-10 and JP-8 to determine whether deoxygenated tricycle 3 could be a suitable replacement for jet fuel (Table 2). The density of 3 at 15 °C was significantly higher than that of JP-8, but slightly lower than that of JP-10. Energy density, which is crucial for volume-constrained fuel applications, was assessed. The energy density for compound 3 was 38.2 MJ L⁻¹, which is significantly higher than that of JP-8, but slightly lower than that of JP-10. The net heat of combustion (specific energy), which is important in weight-limited applications, was also measured. The net heat of combustion of compound 3 was 42.0 MJ kg⁻¹, which is almost the same as that of JP-10. Jet fuel needs to have a freezing point below 233 K to accommodate the high-altitude and low-temperature conditions experienced by aircraft during operation. The viscosity of compound 3 at 40 °C was measured to be 7.61 mm² s⁻¹. The flash point, an important indicator of fire safety during storage

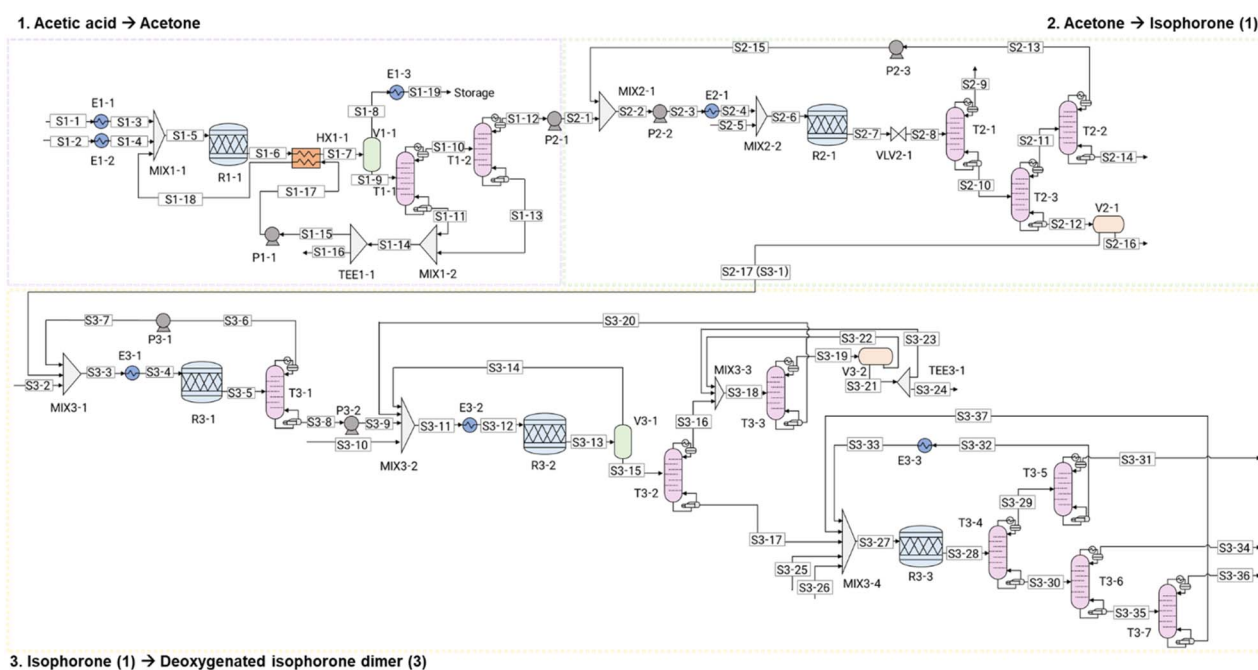


Fig. 1 Overall process for the production of deoxygenated isophorone dimer 3 with bio-acetic acid: (1) acetone synthesis from bio-acetic acid, (2) isophorone 1 synthesis from acetone, and (3) deoxygenated isophorone dimer 3 synthesis from isophorone 1.

Table 3 Reactions and operating conditions in the overall synthesis process

| Process | Component in Fig. 1 | Reaction | Temperature (°C) | Pressure (bar) | Conversion rate (%) | Ref. |
|----------------------------|---------------------|--|------------------|----------------|---------------------|------------|
| 1 (Acetic acid to acetone) | R1-1 | $2\text{C}_2\text{H}_4\text{O}_2 \rightarrow \text{C}_3\text{H}_6\text{O} + \text{H}_2\text{O} + \text{CO}_2$ | 180 | 27.6 | 50.0 | 14 |
| 2 (Acetone to 1) | R2-1 | $2\text{C}_3\text{H}_6\text{O} \rightarrow \text{C}_6\text{H}_{10}\text{O} + \text{H}_2\text{O}$ | 280 | 80 | 55.0 | 15 |
| | | $\text{C}_6\text{H}_{10}\text{O} + \text{C}_3\text{H}_6\text{O} \rightarrow \text{C}_9\text{H}_{14}\text{O} + \text{H}_2\text{O}$ | | | 95.0 | |
| | | $\text{C}_9\text{H}_{14}\text{O} + \text{C}_3\text{H}_6\text{O} \rightarrow \text{C}_{12}\text{H}_{18}\text{O} + \text{H}_2\text{O}$ | | | 50.0 | |
| 3 (1 to 4) | T2-3 | $\text{C}_{12}\text{H}_{18}\text{O} + \text{H}_2\text{O} \rightarrow \text{C}_9\text{H}_{14}\text{O} + \text{C}_3\text{H}_6\text{O}$ | 220 | 30 | 100 | |
| | R3-1 | $\text{C}_9\text{H}_{14}\text{O} + \text{C}_9\text{H}_{14}\text{O} \rightarrow \text{C}_{18}\text{H}_{28}\text{O}_2$ | 82 | 1 | 80.5 | This study |
| | R3-2 | $\text{C}_{18}\text{H}_{28}\text{O}_2 + 2\text{H}_2 \rightarrow \text{C}_{18}\text{H}_{30}\text{O} + \text{H}_2\text{O}$ | 350 | 1 | 69.0 | |
| | R3-3 | $\text{C}_{18}\text{H}_{28}\text{O}_2 + \text{H}_2 \rightarrow \text{C}_{18}\text{H}_{28}\text{O} + \text{H}_2\text{O}$ | 350 | 1 | 31.0 | |
| | R3-3 | $\text{C}_{18}\text{H}_{30}\text{O} + \text{H}_2 \rightarrow \text{C}_{18}\text{H}_{30} + \text{H}_2\text{O}$ | 150 | 1 | 81.0 | |



and handling, was found to be relatively high (137.6 °C). In addition, hydrocarbon 3 exhibited low sulfur content, a critical property for jet-oil applications.

To evaluate the feasibility of using deoxygenated tricycle 3 as a JP-10 replacement, a commercial-scale process for producing tricycle 3 from bio-acetic acid was simulated using Aspen PLUS V14.0. As shown in Fig. 1, the process comprises three stages: (1) synthesis of acetone from bio-acetic acid, (2) synthesis of isophorone 1 from acetone, and (3) synthesis of deoxygenated tricycle 3 from isophorone 1. The first two stages involve the production of isophorone from bio-acetic acid, for which reaction data can be obtained from the literature.^{15,16} The operating conditions and yields for the reaction producing hydrocarbon 3 from isophorone 1 are provided in the experimental work of this study. The reactions and operating conditions required for the process design are listed in Table 3. The solvents, acetic acid used in R3-2, and decane used in R3-3, are assumed to be recycled with additional makeup solvent to compensate for process losses.

Using the mass balance obtained from the simulation, techno-economic analysis (TEA) and life cycle assessment (LCA) were performed. Detailed explanations and simulation results of the process are provided in the SI.

Fig. 2 illustrates the TEA results for the overall process. As shown in Fig. 2a, with the previously reported price of bio-acetic acid (USD 677 per ton),¹⁶ the minimum selling price (MSP) of compound 3 is USD 6470 per ton, which is 8.8% lower than that of JP-10 (USD 7091 per ton). A reduction in the cost of bio-acetic acid feed has a significant impact on the economic viability of compound 3 as a replacement for JP-10. For instance, a bio-acetic acid supply of USD 450 per ton would result in an MSP of USD 5010 per ton, and free bio-acetic acid would result in an MSP of USD 2111 per ton, indicating that the compound 3 production could be even more beneficial under optimistic assumptions regarding the utilization of biomass resources.

As alcohol 4 is the direct precursor of the final product, improving the conversion rate of R3-2 to increase the yield of alcohol 4 decreases the MSP of hydrocarbon 3. Thus, the effect of the conversion rate of R3-2 on the MSP of hydrocarbon 3 was analyzed, as shown in Fig. 2b. The MSP of hydrocarbon 3 varies significantly with changes in not only the cost of bio-acetic acid but also the conversion rate of R3-2. Even with a conversion rate of 50% in R3-2, the price of compound 3 was USD 7090 per ton when bio-acetic acid was supplied at USD 480 per ton, implying that the hydrodeoxygenation reaction in R3-2 could be a key factor in designing an economically feasible jet fuel production process.

To consider the unexpected reduction in the conversion rate of R3-3, we varied it and observed its impact on the MSP of tricycle 3 (Fig. 2c). Even with a reduced conversion rate of R3-3, recycling unreacted alcohol 4 ensured that the lower conversion rate in R3-3 did not significantly affect the MSP of compound 3. Further discussion of the TEA results, including the economic impact of parameters such as catalyst replacement period, catalyst cost, solvent recovery rate, and solvent consumption, can be found in the SI.



Fig. 2 TEA results of deoxygenated tricycle 3 production: (a) MSP of compound 3 depending on the cost of bio-acetic acid, (b) MSP of compound 3 depending on bio-acetic acid cost and conversion rate of R3-2, and (c) MSP of compound 3 depending on bio-acetic acid cost and conversion rate of R3-3. The solid line indicates the current price of JP-10.

Carbon emissions *via* cradle-to-gate LCA for hydrocarbon 3 were estimated to confirm its potential to replace JP-10 as a sustainable fuel, based on the system boundary shown in Fig. S1. The LCA followed ISO 14040 and 14044 guidelines, considering all inputs and outputs. Environmental impacts were assessed using the Ecoinvent V3 database and the ReCiPe



2016 v1.1 Midpoint (H) method. Global warming potential (GWP) was estimated in units of CO₂-equivalent weight per unit value of low heating value; the detailed assumptions, relevant parameters and calculation results can be found in the SI.

As shown in Fig. S4, 74% of the total emissions originated from isophorone production, and an additional 12% and 5% were attributed to the utilities required for recovering the solvent decane used in the R3-3 process. These results indicate that the environmental sustainability of hydrocarbon **3** is mainly determined by the carbon footprint of the feedstock, the yield of the target product, and the amount of solvent used.

LCA results in Fig. S6 further highlight the importance of these factors. A decrease in the conversion rate of R3-2 to produce alcohol **4** has a major impact on the GWP, as it reduces the total amount of the product. Furthermore, reducing decane consumption can significantly lower the GWP of hydrocarbon **3** to 76 g CO₂ eq MJ⁻¹, which is below that of JP-8 (80 g CO₂ eq MJ⁻¹). Despite the strict conditions in designing the process, such as 99.9% purity of the final product and no recycling of the byproduct from R3-2, hydrocarbon **3** had a GWP comparable to that of JP-8, while showing a higher energy density than JP-8. Assuming an optimistic outlook for the HDO reaction presented in this study, hydrocarbon **3** is expected to be an economical and sustainable alternative to JP-10.

Conclusions

In conclusion, a novel bridged-structure hydrocarbon **3** was successfully designed and synthesized from acetic acid. We demonstrated that hydrocarbon **3** could be efficiently produced using a one-pot strategy without the need for intermediate purification. Hydrocarbon **3** exhibited fuel properties comparable to those of JP-10, highlighting its potential as a viable alternative to conventional JP-10. Furthermore, the TEA and LCA revealed that production at scale can offer a lower MSP and carbon emissions than existing jet fuels, highlighting its economic and environmental feasibility. These findings establish hydrocarbon **3** as a sustainable and economically viable alternative to traditional jet fuels and contribute to the advancement of next-generation aviation energy solutions. This study demonstrates a proof-of-concept for alternative fuel synthesis using acetic acid-derived isophorone from biomass feedstocks, with ongoing efforts to extend its applicability to real bio-oil.

Author contributions

Han Byeol Kim: data curation, investigation, methodology, and writing – original draft. Hyerim Kim: data curation, investigation, methodology, and writing – original draft. Hwayeon Jeon: data curation. Pratip Kumar Dutta: data curation, investigation, methodology. Do Hee Oh: data curation. Kyeongsu Kim: conceptualization, supervision. Jeong-Myeong Ha: funding acquisition, supervision. Seo-Jung Han: conceptualization, formal analysis, funding acquisition, investigation, methodology, project administration, resources, supervision, writing – original draft, and writing – review and editing.

Conflicts of interest

The authors declare no competing interests.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: experimental procedures, optimization table, characterization data, NMR spectra, techno-economic analysis, and life cycle assessment. See DOI: <https://doi.org/10.1039/d5su00243e>.

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