



Production and Characterization of Alkanes, Alkenes, and Alkadienes from Wax Through Pyrolysis for Recycling of Waste: Interpretation of Competitive Reactions

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Abstract: The widespread commercial application of wax has resulted in the generation of significant volumes of waste waxes. Pyrolysis is a highly viable method for recycling waste hydrocarbons. In this study, pyrolysis behaviors of saturated hydrocarbons with molecular weights $< 800 \text{ g mol}^{-1}$ were investigated based on molecular weight and pyrolysis temperature (423°C, 500°C, 590°C, 670°C, and 764°C). Additionally, pyrolysis products were analyzed using gas chromatography/mass spectrometry. The major pyrolysis products were alkanes, alkenes, and alkadienes. The dominant pyrolysis product—determined by the maximum peak area—depended significantly on both molecular weight and pyrolysis temperature. Alkenes were more abundant than the other products, whereas small alkadienes were rarely generated. The quantities of the pyrolysis products increased with the molecular weight of the sample. The appropriate pyrolysis temperatures for generating alkanes and alkadienes were approximately 590°C and 670°C, respectively, whereas those for generating alkenes decreased as the molecular weight of the sample increased. The dissociation energies of C–C bonds in the saturated hydrocarbon chains were calculated to discuss the experimental results. Olefins derived from pyrolysis can be used as reactants in chemical synthesis.

Keywords: saturated hydrocarbon waste, pyrolysis, gas chromatography/mass spectrometry, alkanes, alkenes, alkadienes

Introduction

Waxes usually refer to saturated hydrocarbons that crystallize when cooled and melt when heated.^{1,2} They are classified as natural or synthetic substances, and natural waxes are further classified as plant, animal, or mineral substances.^{1–4} Natural waxes produced by animals or plants are primarily wax esters, which are mixtures of fatty acids, fatty alcohols, and hydrocarbons in different proportions.^{3,4} Methanol and dichloromethane are generally used to extract wax from plant leaves, and a solvent mixture with n-hexane was used to enhance the extraction of n-alkanes.^{2,5–9} Paraffin wax is a linear or branched saturated organic compound of $\text{C}_n\text{H}_{2n+2}$.^{10–14}

Waxes are widely used in various products, including candles, crayons, textiles, paper, wood, rubber compounds, leather, protective floor and automotive polishes, pharmaceutical and food coatings, and cosmetics.^{2,4,10} In the rubber industry, waxes are used as antioxidants to form film barriers on the surfaces of the products, particularly tires, preventing ozone from interacting with the rubber surfaces.^{14–18} The

amounts of wax consumed in Europe and Asia were 9.5 and 19.8 million tons in 2011, respectively, and approximately 0.74 million tons were consumed for candles in Europe in 2018.^{19,20} Approximately 3.4% of wax around the world has been consumed in tire manufacturing.¹⁹

Via pyrolysis, large molecules decompose into small ones that can be used as fuels, solvents, or reactants in synthesis. Thus, pyrolytic techniques can be used to recycle polymeric wastes, like tire (rubber), wind turbine blade (epoxy resin), polyethylene (PE), polypropylene (PP), polystyrene (PS), and so on.^{21–32} Alkanes can be used as fuels and solvents, whereas alkenes and alkadienes can also be used as reactants in synthesis because of their carbon-carbon double bonds. Rubber articles, including tires, generally contain waxes, and thus, when rubber articles are pyrolyzed, their waxes are thermally decomposed. Waxes within plants are also pyrolyzed upon incomplete combustion.

The pyrolysis products of paraffin wax are formed via a free radical mechanism.^{33–35} Major pyrolysis products of PE are alkanes, alkenes, and alkadienes.^{36,37} The pyrolysis behavior of hydrocarbon substances at high temperatures is critical in applications such as light olefin production, conversion to liquid fuels, the depolymerization of synthetic

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polymers, and recycling.^{35,38-41} Several pyrolysis products have been used in producing novel olefins.⁴⁰ Researches on thermal cracking reactions of hydrocarbon fuel such as n-decane and n-dodecane at high pressure system as fuel heating system simulation were performed.⁴²⁻⁴⁴ Pyrolysis of polymeric saturated hydrocarbon carbons such as PE and PP has been studied.²³⁻²⁹ Unfortunately, studies on detailed pyrolysis behavior of wax with relatively lower molecular weight have been rarely performed. We investigated pyrolysis behavior of waxes under normal pressure, and simulation and experiment were combined to uncover the pyrolysis mechanism.

The pyrolysis temperature is crucial in determining the types and amounts of pyrolysis products, and the molecular weight of the sample can affect the pyrolysis behavior. In this study, we examined how pyrolysis temperature and molecular weight influence the pyrolysis product distribution of alkanes, alkenes, and alkadienes using three types of saturated hydrocarbons (waxes) with molecular weights of $< 800 \text{ g mol}^{-1}$. Paraffin wax typically has an average molecular weight range of 350 to 520 g mol^{-1} , and microcrystalline wax usually has a higher molecular weight, with a range of about 500 to 800 g mol^{-1} . Five pyrolysis temperatures of 423, 500, 590, 670, and 764°C were applied, and the temperatures required to more produce specific pyrolysis products were also examined.

The pyrolysis products were analyzed using gas chromatography/mass spectrometry (GC/MS). Difference in the three major pyrolysis products, namely alkanes, alkenes, and alkadienes, depending on the pyrolysis temperature and molecular weight was focused. We calculated the energy-minimized structures of neutral normal saturated hydrocarbon ($\text{C}_{30}\text{H}_{62}$) and radicals ($\text{CH}_3(\text{CH}_2)_n^\bullet$, $n = 0-28$) formed from $\text{C}_{30}\text{H}_{62}$ to explain the dissociation of the C-C bonds in wax. Based on their energies, the differences in the dissociation energies of the C-C bonds within the saturated hydrocarbon chains were compared based on the locations of the C-C bonds.

Experimental

Triacontane ($\text{C}_{30}\text{H}_{62}$, C30 sample) and tetracontane ($\text{C}_{40}\text{H}_{82}$, C40 sample) were purchased from Alfa Aesar (Lancashire, U.S.) and Tokyo Chemical Industry Co. (Tokyo, Japan), respectively. Polyethylene 800 (C57 sample) with M_w of 840 Da and M_n of 790 Da was purchased from Polymer Standard Service GmbH (Mainz, Germany).

The sample weight for Py-GC/MS was approximately 0.2 mg, each sample was analyzed four times. Py-GC/MS analysis was carried out using an Agilent 6890 gas chromatograph equipped with a 5973 mass spectrometer (Agilent Technology Inc., USA) equipped with a JCI-55 Curie point pyrolyzer (Japan Analytical Industry Co., Japan). Pyrofoils with Curie points of 423, 500, 590, 670, and 764°C were used. Pyrolysis was performed for 10 s using the pyrofoil under helium (He) atmosphere. A DB-5MS capillary column (30 m $\times$ 0.32 mm, 25 μm film thickness) (Agilent Technology Inc., USA) was used. The injector temperature was 250°C. The inlet temperature was 250°C, the split ratio was 1:15, and helium (1.8 mL min^{-1}) was used as the carrier gas. The GC oven temperature program was as follows: 30°C (held for 3 min) to 50°C (held for 3 min) at a rate of $10^\circ\text{C min}^{-1}$, to 180°C (held for 1 min) at a rate of $10^\circ\text{C min}^{-1}$, and then to 250°C (held for 3 min) at a rate of $10^\circ\text{C min}^{-1}$. The interface temperature of GC to MS was 250°C. The electron ionization (70 eV) was used to ionize the pyrolysis products. The MS source temperature was 230°C.

We obtained energy-minimized structures of $n\text{-C}_{30}\text{H}_{62}$ and $\text{CH}_3(\text{CH}_2)_n^\bullet$ radicals ($n = 0-28$) with a series of electronic structure calculations. A series of electronic structure calculations was carried out to obtain structures and energies of the target species. Base structures were created using Avogadro software. We performed simulated annealing with Spartan'10 to find low energy structures using molecular mechanics force field (MMFF94)^{45,46} as implemented in Spartan'10. From this, low energy conformers were obtained. All density functional theory (DFT) calculations were performed with B3LYP/6-31++G(d,p) level using the energy and frequency optimization option by Gaussian 09. From the DFT calculations, the lowest energy structure as the final best structure was obtained.

Results and discussion

Figure 1 shows representative Py-GC/MS chromatograms of the saturated hydrocarbon samples at 590°C. The pyrolysis product size was marked as the number of carbon. The principal pyrolysis products are alkanes ($\text{C}_n\text{H}_{2n+2}$), alkenes (C_nH_{2n}), and alkadienes ($\text{C}_n\text{H}_{2n-2}$). The order of retention time under the analysis conditions is alkadiene $<$ alkene $<$ alkane. Of them, alkenes are the most abundant, and small alkadienes are rarely formed. The quantities of the pyrolysis products increased as the molecular weight of the sample

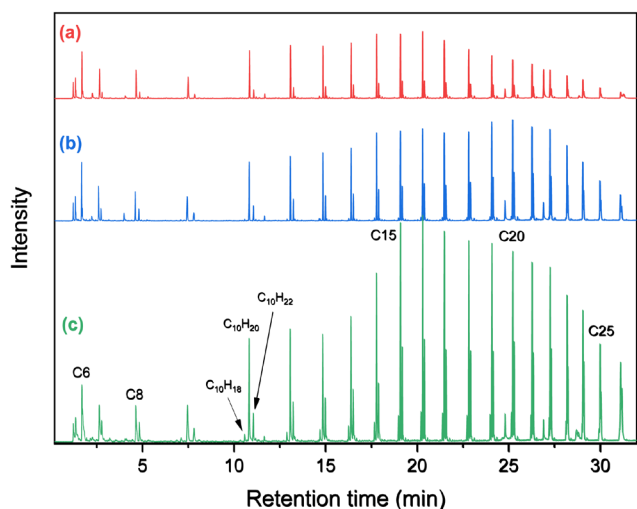


Figure 1. Py-GC/MS chromatograms at 590°C of the C30 (a), C40 (b), and C57 (c) samples.

increased.

The formation mechanisms of alkane, alkene, and alkadiene (exactly *n*-alkane, 1-alkene, and α,ω -alkadiene, respectively) via the pyrolysis of saturated hydrocarbons are shown in Scheme 1. The C-C bond is first dissociated by thermal energy, and various $\text{CH}_3(\text{CH}_2)_n^\bullet$ radicals are formed. Alkanes are formed when the $\text{CH}_3(\text{CH}_2)_n^\bullet$ radicals abstract hydrogen atoms from other species, and alkenes are generated via the rearrangement of the radicals, resulting in the loss of hydrogen atoms. If another radical is formed at the other side of an alkene via heating, an alkadiene can be produced. Alkadienes may also be generated from $^\bullet(\text{CH}_2)_n^\bullet$ biradicals following rearrangement and the loss of two hydrogen atoms. Formation of alkadienes needs more energy than those of

alkanes and alkenes because of two C-C bond cleavages.

The energy-minimized structures of $\text{C}_{30}\text{H}_{62}$ and $\text{CH}_3(\text{CH}_2)_n^\bullet$ radicals ($n = 0-28$) were calculated to explain radical formation via C-C bond dissociation, and their energies are summarized in Table 1. The energy of the radical decreases by approximately 103,246 kJ mol⁻¹ upon increasing the number of methylene groups (CH_2). The energy differences are insignificant, except that between $\text{CH}_3^\bullet$ and $\text{CH}_3\text{CH}_2^\bullet$ (-103,260 kJ mol⁻¹).

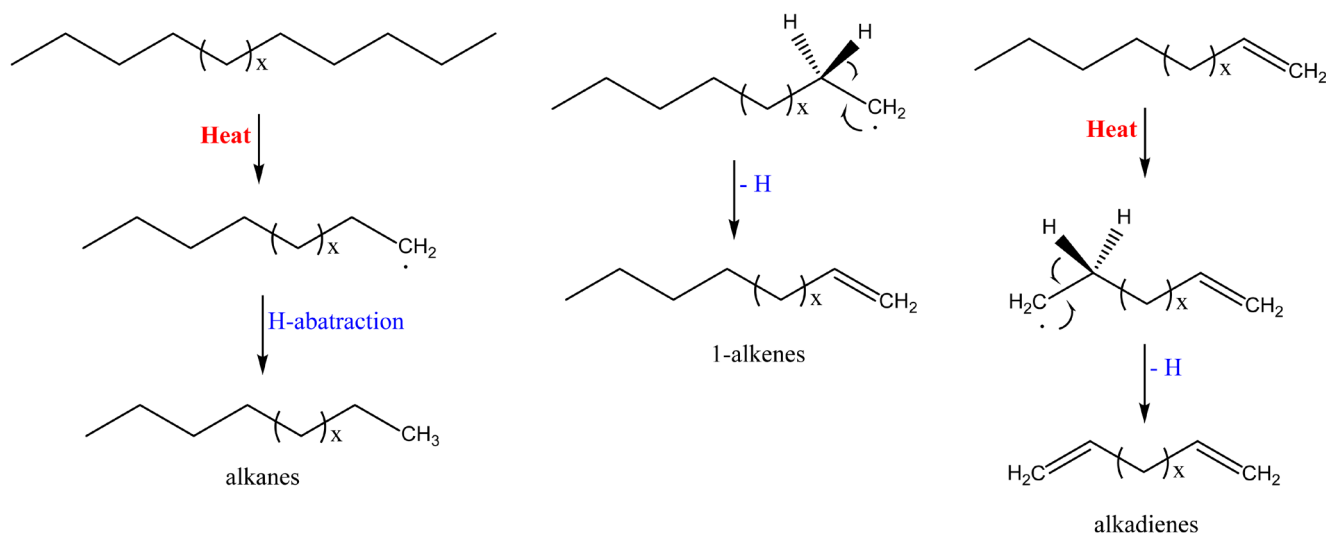
The $\text{CH}_3(\text{CH}_2)_n^\bullet$ radicals are formed from the saturated hydrocarbon chain via the dissociation of a C-C bond. The dissociation energy (E_{dis}) of the formation of the radicals from $\text{C}_{30}\text{H}_{62}$ were obtained using equations (1) and (2).

$$\text{C}_{30}\text{H}_{62} \rightarrow \text{CH}_3(\text{CH}_2)_m^\bullet + \text{CH}_3(\text{CH}_2)_{28-m}^\bullet \quad (1)$$

$$E_{\text{dis}} = E[\text{CH}_3(\text{CH}_2)_m^\bullet] + E[\text{CH}_3(\text{CH}_2)_{28-m}^\bullet] - E[\text{C}_{30}\text{H}_{62}] \quad (2)$$

The E_{dis} is approximately 355 kJ mol⁻¹ (Table 2), irrespective of the radical size. The differences in the E_{dis} values according to the radical size are insignificant, except that of the C1-C2 position (370 kJ mol⁻¹). The formation of $\text{CH}_3^\bullet$ and $\text{CH}_3(\text{CH}_2)_{28}^\bullet$ radicals is less favorable than those of the other radical species. The similar E_{dis} values indicate that the initial C-C bond dissociation occurs randomly. This is because the energy difference of the alkyl radicals by one methylene (CH_2) is very similar (103, 245-103, 248 kJ mol⁻¹), except for the case between $\text{CH}_3^\bullet$ and $\text{CH}_3\text{CH}_2^\bullet$ radicals.

The variations in the peak areas of alkanes were plotted as a function of its carbon number (Figure 2). The peak areas are the average values, and the error bars were marked.



Scheme 1. Formation mechanisms of *n*-alkanes, 1-alkenes, and α,ω -alkadienes from saturated hydrocarbon by pyrolysis.

Table 1. Energies of the energy-minimized structures of $C_{30}H_{62}$ and $CH_3(CH_2)_n$ radicals

Chemical	Energy (kJ mol ⁻¹)	Chemical	Energy (kJ mol ⁻¹)
$CH_3^\bullet$	-104,640	$CH_3(CH_2)_{15}^\bullet$	-1,653,345
$CH_3CH_2^\bullet$	-207,900	$CH_3(CH_2)_{16}^\bullet$	-1,756,592
$CH_3(CH_2)_2^\bullet$	-311,145	$CH_3(CH_2)_{17}^\bullet$	-1,859,838
$CH_3(CH_2)_3^\bullet$	-414,393	$CH_3(CH_2)_{18}^\bullet$	-1,963,084
$CH_3(CH_2)_4^\bullet$	-517,638	$CH_3(CH_2)_{19}^\bullet$	-2,066,330
$CH_3(CH_2)_5^\bullet$	-620,885	$CH_3(CH_2)_{20}^\bullet$	-2,169,576
$CH_3(CH_2)_6^\bullet$	-724,131	$CH_3(CH_2)_{21}^\bullet$	-2,272,822
$CH_3(CH_2)_7^\bullet$	-827,377	$CH_3(CH_2)_{22}^\bullet$	-2,376,067
$CH_3(CH_2)_8^\bullet$	-930,623	$CH_3(CH_2)_{23}^\bullet$	-2,479,314
$CH_3(CH_2)_9^\bullet$	-1,033,869	$CH_3(CH_2)_{24}^\bullet$	-2,582,560
$CH_3(CH_2)_{10}^\bullet$	-1,137,115	$CH_3(CH_2)_{25}^\bullet$	-2,685,806
$CH_3(CH_2)_{11}^\bullet$	-1,240,361	$CH_3(CH_2)_{26}^\bullet$	-2,789,052
$CH_3(CH_2)_{12}^\bullet$	-1,343,607	$CH_3(CH_2)_{27}^\bullet$	-2,892,297
$CH_3(CH_2)_{13}^\bullet$	-1,446,853	$CH_3(CH_2)_{28}^\bullet$	-2,995,544
$CH_3(CH_2)_{14}^\bullet$	-1,550,099	$C_{30}H_{62}$	-3,100,554

Table 2. Dissociation energies of normal saturated hydrocarbon of $C_{30}H_{62}$ depending on the carbon site

Carbon site	Dissociation energy (kJ mol ⁻¹)
C1-C2	370
C2-C3	356
C3-C4	357
C4-C5	355
C5-C6	356
C6-C7	355
C7-C8	356
C8-C9	355
C9-C10	355
C10-C11	355
C11-C12	355
C12-C13	355
C13-C14	355
C14-C15	355
C15-C16	355

The C6 (C_6H_{14}) and C7 (C_7H_{16}) products were not detected at 423°C. Using the C30 sample, the peak area generally increased as the alkane size increased, except at 423 and 500°C. The variations at these temperatures indicated that the most abundant alkane was the ~C20 product. Using the C40 sample, the peak areas increased as the pyrolysis temperature increased from 423 to 590°C and then decreased as the pyrolysis temperature increased further. At 423°C, the peak

intensities were insignificant. Except at 423 and 500°C, the most abundant alkane was the ~C20 product. The variations when using the C57 sample showed trends similar to those observed using the C40 sample. The abundances of alkanes at 590°C exceeded those at the other temperatures, and thus, a pyrolysis temperature of 590°C is appropriate for use in producing alkanes.

The variations in the peak areas of alkenes showed different trends from those of alkanes (Figure 3). The C6 (C_6H_{12}) and C7 (C_7H_{14}) products were not observed at 423°C. The peak intensities of alkenes at 423°C were significant. Using the C30 sample, the peak areas increased as the pyrolysis temperature increased from 423 to 764°C. The size of the most abundant alkene decreased as the pyrolysis temperature increased, and the most abundant alkenes at pyrolysis temperatures of 500, 590, 670, and 764°C were C17, C16, C10, and C10 species, respectively. Using the C40 sample, the peak areas generally increased as the pyrolysis temperature increased, except at 764°C. The C10–C20 alkenes were more abundant than the others. Using the C57 sample, similar trends were observed compared to those observed using the C40 sample. The peak areas generally increased as the pyrolysis temperature increased, except at 764°C. The C10–C22 alkenes were more abundant than the others. The temperature required to produce the most abundant alkene varied depending on the sample size, and it decreased from 764 to 590°C as the sample size increased from C30 to C57. This implies that the

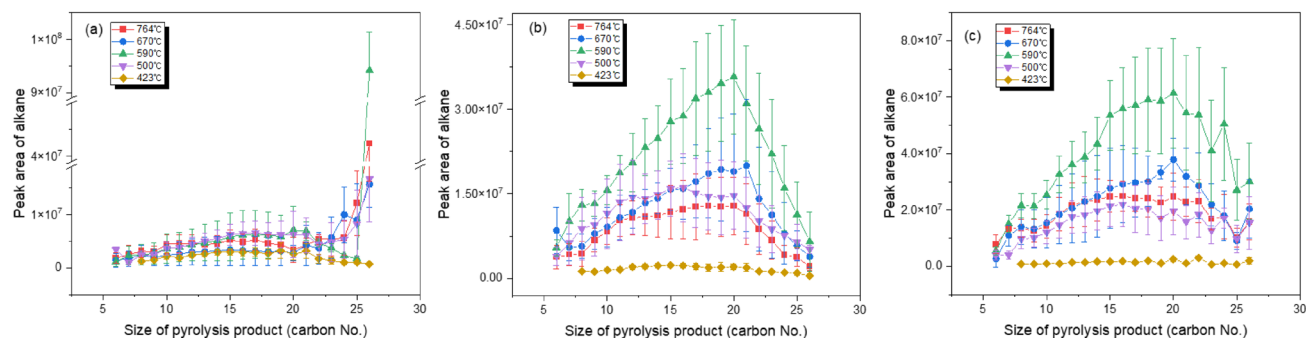


Figure 2. Variations in the peak areas of normal alkanes (C_nH_{2n+2}) produced by pyrolysis with the pyrolysis product size (Carbon No.) of the C30 (a), C40 (b), and C57 (c) samples.

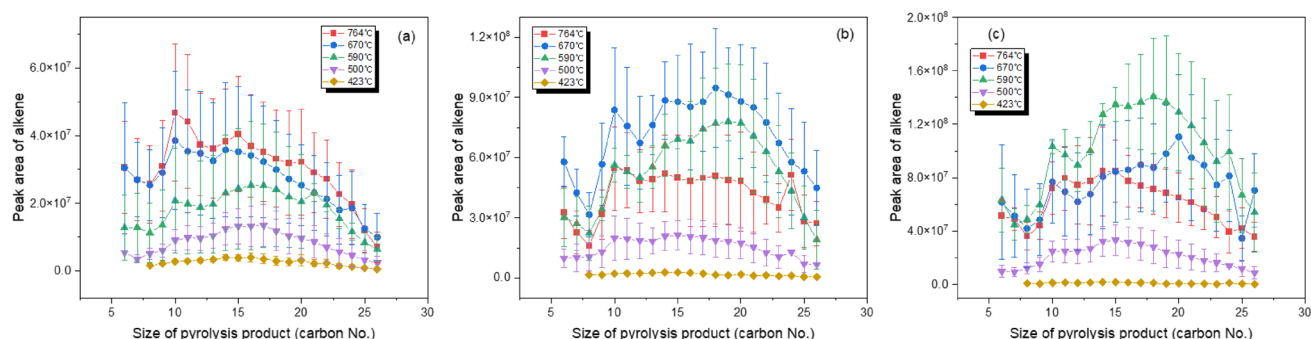


Figure 3. Variations in the peak areas of alkenes (C_nH_{2n}) produced by pyrolysis with the pyrolysis product size (Carbon No.) of the C30 (a), C40 (b), and C57 (c) samples.

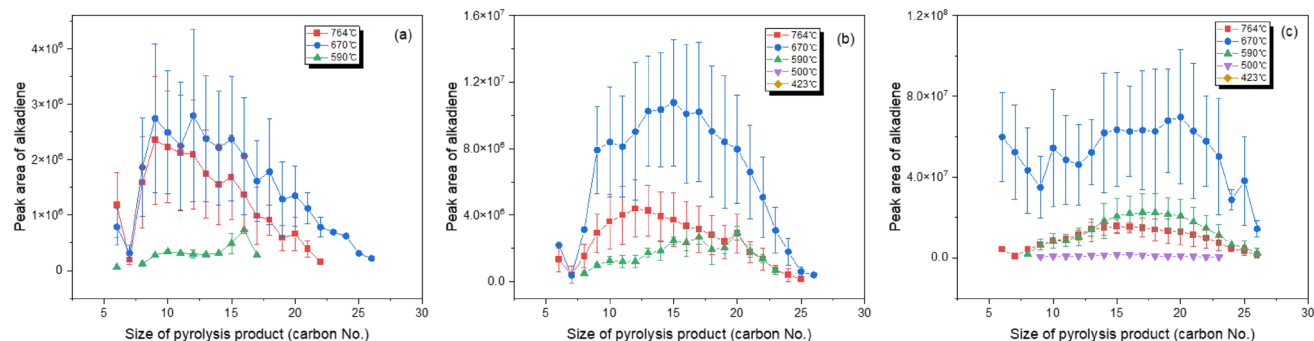


Figure 4. Variations in the peak areas of alkadienes (C_nH_{2n-2}) produced by pyrolysis with the pyrolysis product size (Carbon No.) of the C30 (a), C40 (b), and C57 (c) samples.

optimal pyrolysis temperature to produce alkenes decreases by increasing the molecular weight of the sample.

Alkadienes were not produced at 423°C for any of the samples, and they were not generated at 500°C for the C30 and C40 samples (Figure 4). The abundances of the alkadienes formed from the C57 sample at 500°C were not significant. The low level of alkadiene production indicates that the formation of alkadiene requires more energy than those of alkane and alkene. The peak areas of the alkadienes at 670°C exceeded those at 764°C. Using the C30 sample,

alkadienes larger than C17 and C22 were not observed at 590 and 764°C, respectively. The peak areas at 670 and 764°C generally decreased compared to that of the C9 species as the alkadiene size increased. Using the C40 sample, alkadienes larger than C23 were not observed at 590°C, and the C9–C20 alkadienes were major species at 670 and 764°C. Using the C57 sample, the peak areas at 764°C were even lower than those at 590°C, and the C10–C23 alkadienes were major species at 590, 670, and 764°C. The abundances of the alkadienes at 670°C were higher than those at the other

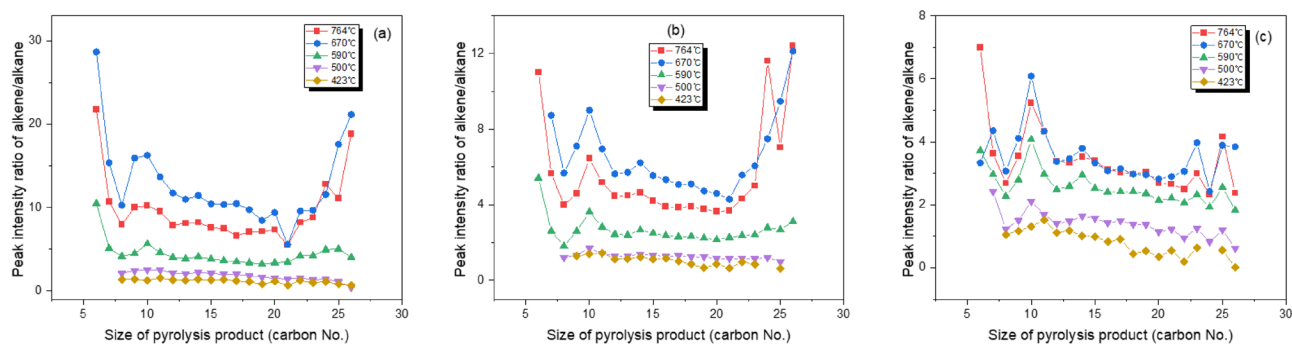


Figure 5. Variations in the peak intensity ratios of alkene/alkane with the pyrolysis product size (Carbon No.) of the C30 (a), C40 (b), and C57 (c) samples.

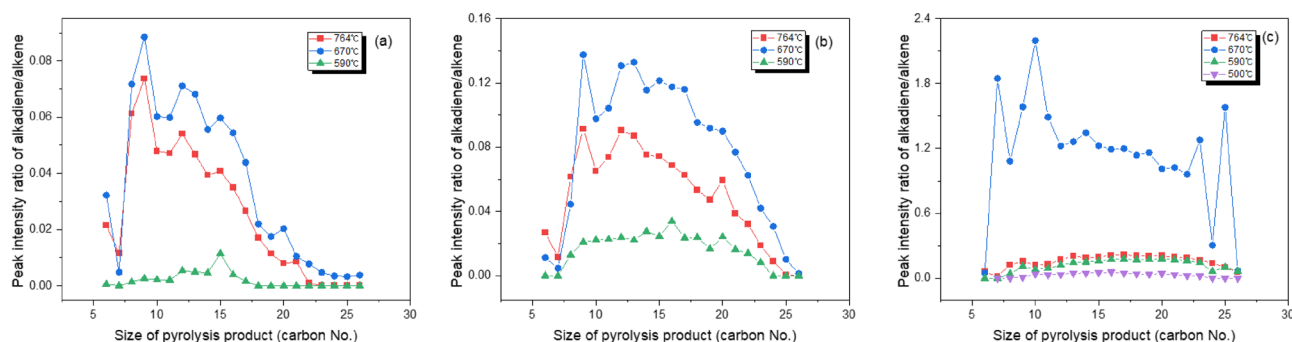


Figure 6. Variations in the peak intensity ratios of alkadiene/alkene with the pyrolysis product size (Carbon No.) of the C30 (a), C40 (b), and C57 (c) samples.

temperatures, and thus, a pyrolysis temperature of 670°C is more appropriate for use in producing alkadienes than the other temperatures.

The C-C bond dissociation occurs randomly. Thus, longer alkyl radicals are more formed as the sample size increases, leading to production of larger pyrolysis products. As shown in Figures 3 and 4, the distributions of alkenes and alkadienes with larger size increased as the sample size increased.

The abundances of the three major pyrolysis products were compared using their ratios of alkene/alkane and alkadiene/alkene. The alkene/alkane peak intensity ratios were > 1.0 , even at low pyrolysis temperatures (Figure 5), which means that the formation of alkenes is more favorable than that of alkanes. The formation of alkanes is the intermolecular reaction between an alkyl radical and a gas phase neutral species. The probability of the alkyl radical to react with a neutral molecule may not be high enough at any moment to generate an alkane. On the contrary, the formation of alkene is an intramolecular reaction. When an alkyl radical has excess energy, the rearrangement to form an alkene by the loss of hydrogen atom can occur easily. The alkene/alkane

peak intensity ratios increased as the pyrolysis temperature increased and then decreased at 764°C, and the ratios generally decreased as the molecular weight of the sample increased. Using the C30 sample, the peak intensity ratios (C7–C25 range) were 0.7–1.5, 1.1–2.5, 2.6–5.6, 8.4–17.6, and 7.1–12.8 at pyrolysis temperatures of 423, 500, 590, 670, and 764°C, respectively. Using the C40 sample at the same temperatures, the respective peak intensity ratios were 0.6–1.5, 1.0–1.7, 1.8–3.6, 4.3–8.7, and 3.6–11.6, respectively, while those observed using the C57 sample were 0.3–1.5, 0.8–2.4, 1.8–4.1, 2.4–6.1, and 2.35.2, respectively. The alkene/alkane peak intensity ratios generally decreased as the sample size increased, which indicates that the formation of alkenes becomes less favorable than that of alkanes as the sample size increases.

The alkadiene/alkene peak intensity ratios were < 0.2 , except when using the C57 sample at 764°C (Figure 6), and thus, the formation of alkenes is more thermodynamically favorable than that of alkadienes. Using the C30 sample, the peak intensity ratios were < 0.10 . The peak intensity ratio increased as the pyrolysis temperature increased, and then

it decreased at 764°C. The C9 pyrolysis product exhibited the maximum peak intensity ratios at 670 and 764°C. Using the C40 sample, the peak intensity ratios were < 0.15 . The peak intensity ratio increased as the pyrolysis temperature increased, and then it decreased at 764°C. The C9 pyrolysis product again displayed the maximum peak intensity ratios at 670 and 764°C. Using the C57 sample, the peak intensity ratios at 670°C were > 1.0 , whereas those at the other pyrolysis temperatures were < 0.2 . Using the C30 and C40 samples at 670 and 764°C, the peak intensity ratios decreased as the size of the pyrolysis product increased beyond that of the C9 product, indicating that the formation of large alkadienes is not favorable.

Conclusions

The principal pyrolysis products of the saturated hydrocarbons were alkanes (C_nH_{2n+2}), alkenes (C_nH_{2n}), and alkadienes (C_nH_{2n-2}). The abundances of pyrolysis products varied depending on the molecular weight of the sample and pyrolysis temperature as well as the pyrolysis product size. The most abundant pyrolysis product also varied depending on the molecular weight and pyrolysis temperature. Alkenes were more abundant than the others, whereas small alkadienes were rarely formed. The quantities of the pyrolysis products increased as the molecular weight of the sample increased. The energy of the radical decreases by approximately 103,246 kJ mol⁻¹ by increasing one methylene group. The pyrolysis products were formed via C-C bond dissociation, and the E_{dis} values of the bonds were approximately 355 kJ mol⁻¹, irrespective of their locations. The appropriate pyrolysis temperatures for use in producing alkanes and alkadienes were 590 and 670°C, respectively. The most favorable temperature to produce alkenes shifted from 764 to 590°C as the sample size increased, and the optimal pyrolysis temperature decreased as the molecular weight of the sample increased. The formation of alkenes becomes more favorable than that of alkanes as the sample size increases. If it is possible to fractionate alkenes and alkadienes from the pyrolysis products, they can be used as reactants in synthesis.

Conflict of Interest: The authors declare that there is no conflict of interest.

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