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High-speed camera observation of cellulose fast pyrolysis under infrared irradiation

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Keywords: Cellulose Fast pyrolysis Infrared irradiation High-speed camera Selective heating Fast pyrolysis using infrared irradiation is a method for converting cellulose into valuable chemicals. In the present study, the pyrolysis behavior of cellulose sheets (Whatman cellulose) and cellulose powder (Avicel cellulose) under infrared irradiation in a nitrogen flow was investigated using a high-speed camera. In the case of cellulose sheet, pyrolysis products, including levoglucosan and carbonized materials, were locally formed in the initial stage of the reaction. Subsequently, holes with a thin layer of pyrolysis products were formed, and pyrolysis proceeded as the hole expanded. For cellulose powder, pyrolysis occurred on the surface of the cellulose exposed to infrared irradiation, and droplets of pyrolysis products were formed. The droplets moved around, collecting the liquid products, and pyrolysis products volatilized from the droplets. This pyrolysis behavior continued into the unreacted cellulose powder below, resulting in the complete pyrolysis products, liquid and or fast pyrolysis conditions. Near-infrared (NIR) spectroscopy revealed that this characteristic pyrolysis behavior was due to the more efficient absorption of infrared radiation by the pyrolysis products, liquid and products, compared to the unreacted cellulose. It was also found that the addition of solid carbonized products efficiently promoted the fast pyrolysis reactions. These findings have important implications for the fast pyrolysis of cellulose under infrared irradiation.

1. Introduction

To achieve a sustainable society and address environmental problems, it is imperative to reduce the use of finite fossil resources and transition toward alternative, renewable sources of energy and chemicals. Biomass is the only renewable carbon source that can be converted into chemicals and fuels. Cellulosic biomass is expected to be a key resource, accounting for the majority of the world's biomass reserves. Since cellulosic biomass consists of stable polymers such as cellulose, hemicellulose and lignin, it is important to develop efficient conversion technologies. Pyrolysis, the thermal de-composition of materials under oxygen-free conditions, has attracted attention as an efficient technology for the rapid decomposition of cellulosic biomass.

Pyrolysis of cellulosic biomass produces gaseous, liquid and solid products. The heating rate critically influences product selectivity. Fast pyrolysis, characterized by a rapid heating process, primarily a liquid mixture (bio-oil) from cellulosic biomass rather than solid residues [1–5]. This bio-oil serves as a versatile precursor for the synthesis of liquid fuels and a range of biochemicals. Levoglucosan (LG, 1,

6-anhydro-β-D-glucopyranose), a major component of bio-oil from cellulose, has potential as a feedstock for various chemical productions [6–8]. Additionally, since LG is easily hydrolyzed to glucose, pyrolysis-based saccharification can be proposed [9–11].

Fast pyrolysis of cellulose occurs at temperatures in the range of 400–450 °C, significantly higher than the decomposition temperatures observed for slow pyrolysis, which are around 350 °C [12–15]. During fast pyrolysis, cellulose decomposes through a transient liquid intermediate with a lifetime of less than one second [12,13,16–20]. This decomposition pathway has been captured in real time by high-speed videography [19,20]. Lédé et al. demonstrated that wood undergoes pyrolysis and transitions to a molten state at 466 °C, with the interfacial temperature between the cellulose and the liquid intermediate recorded at 443 °C [17]. Additionally, Shoji et al. inserted cellulose samples in a furnace preheated to 600 °C and monitored the resulting temperature fluctuations [15]. Notably, a transient plateau in the temperature rise was observed in the 400–450 °C range. This phenomenon is attributed to the volatilization of pyrolytic products generated during fast pyrolysis. Milosavljevic et al. calculated the heat of evaporation of these

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volatilized substances to be 538 J/g [21]. Therefore, to facilitate fast pyrolysis of cellulose, it is imperative to maintain a continuous supply of thermal energy that exceeds the endothermic demand associated with the volatilization process.

On the other hand, the high temperature conditions required for the fast pyrolysis of cellulose can degrade LG and complicate the product composition. Shoji et al. reported that furnace temperatures above 600 °C are necessary to achieve cellulose decomposition temperatures above 400 °C in order to maintain fast pyrolysis conditions with a small amount of cellulose in the preheating furnace [15]. Although LG (boiling point: 385 °C) volatilizes under fast pyrolysis conditions [15], it decomposes to gas products at temperatures above 600 °C in the gas phase [22]. Therefore, avoiding LG decomposition is challenging with heating methods based on conduction. In addition, since LG polymerizes at 250 °C in the liquid phase, the volatilized LG tends to polymerize and carbonize after agglomeration [22–25]. The high reactivity of molten LG can be attributed to hydrogen bonds between LG molecules, which act as acidic and basic catalysts [26,27]. These findings suggest that, to efficiently obtain LG through fast pyrolysis of cellulose, it is crucial to rapidly cool the evaporated LG while selectively heating the cellulose.

Radiant heating is one method of selectively heating cellulose [12, 13,16,18,28,29]. It efficiently heats cellulose through the adsorption of infrared (IR) [12,13,28], concentrated solar energy [29], or laser radiation [16,18]. These methods have an advantage over conduction-based pyrolysis, where biomass is heated in a hot environment or in contact with a hot metal surface, as they can selectively heat only the cellulose. Since nitrogen is not efficiently heated by infrared heating, secondary pyrolysis of volatilized LG can be suppressed by rapidly cooling in the gas phase.

Fast pyrolysis of cellulose using radiation heating has been reported. Suzuki et al. reported the formation of LG and anhydrous oligosaccharides by CO_2 laser irradiation of cellulose [30]. Kwon et al. showed that 25 % of LG was obtained from Whatman CF11 cellulose powder by heating with CO_2 laser under nitrogen flow or vacuum conditions [31]. In our previous study, up to 52.7 % of LG was obtained from cellulose under infrared irradiation in a nitrogen flow [28]. The pyrolysis mechanism was inferred from the shape of the residues after pyrolysis (char). However, the actual progress of pyrolysis remains poorly understood, and our understanding of the pyrolysis mechanism is still insufficient.

Due to the rapid progression of the reaction, direct observation of the detailed fast pyrolysis behavior of cellulose presents a significant challenge. However, the use of a high-speed camera enables direct and clear observation of the fast pyrolysis reaction of cellulose. In this study, we observed the pyrolysis behavior of cellulose using a high-speed camera and measured the near-infrared (NIR) spectra of cellulose and its pyrolysis products to investigate the fast pyrolysis behavior and degradation mechanism of cellulose under infrared irradiation.

2. Material and methods

2.1. Materials

Whatman No. 42 cotton filter paper (Whatman PLC, Maidstone, UK) and micro-crystalline cellulose powder (Avicel PH-101, Asahi Kasei Corp., Tokyo, Japan) were used to observe pyrolysis reactions. These materials were used as received, without further purification. The Whatman filter paper was cut into 1.0×4.3 cm (30 mg, dry) or 1.4×1.4 cm (18 mg, dry) pieces before use. The LG (>99.0 %, GC) was purchased from the Tokyo Chemical Industry, Tokyo, Japan, and used without purification.

2.2. Pyrolysis and high-speed camera observation

Fig. 1 shows a diagram of the experimental setup in which an IR image furnace using the near-infrared radiation of an infrared lamp (GA298, Thermo Riko Co., Ltd., Tokyo, Japan) was employed to heat the



Fig. 1. The fast pyrolysis system incorporating an infrared image furnace used in this study.

cellulose sample. This infrared heating device can irradiate infrared rays from the tip of a quartz rod (diameter: 2 cm) toward the cellulose sample. A quartz tube (inner diameter: 30 mm, length: 495 mm) was placed under the quartz rod. In the experiments with the Whatman cellulose sheets, each sample was placed on a stainless-steel mesh positioned in the center of the reactor. For the Avicel cellulose experiments, a quartz boat (length: 35 mm, width: 10 mm, height: 6 mm, AS ONE Corp., Osaka, Japan) containing the powder sample was placed on the mesh and then inserted into the reactor. A Tedlar sampling gasbag (5 L) was attached to the outlet of the reactor tube. After the nitrogen flow was adjusted to 3 L/min, the cellulose samples were heated with an infrared output of 2.0 kW. The pyrolysis behavior of cellulose samples was recorded with the HAS-U1 high-speed camera (DITECT Co., Ltd., Tokyo, Japan) at a speed of 200 frames per second.

Avicel cellulose was pyrolyzed under similar conditions to obtain liquid pyrolysis products. The heating was stopped at 5 s, leaving unreacted cellulose and the pyrolysis products before volatilization in the quartz boat. The remaining products in the quartz boat were extracted with 25 mL of methanol. The liquid products adhering to the quartz tube and sample bag were also extracted with 50 mL of methanol.

In the process of infrared heating, the radiation intensity increases over time, eventually reaching a plateau. As depicted in the left-hand image (1) of Fig. 2, an illumination area was visible at the tip of the quartz rod. The size of this illuminated region stabilized after a certain period (as shown in Fig. 2 (2)). The moment when this area achieved constancy is designated as 0 ms in this study.

2.3. UV-Vis-NIR spectroscopy

To investigate the infrared absorption characteristics of cellulose and its pyrolysis products, the UV-Vis-NIR spectra of each sample were analyzed using a UV-Vis-NIR spectrometer (UV-3600i Plus, Shimadzu Corp., Tokyo, Japan). Whatman cellulose, Avicel cellulose, LG, and activated carbon (charcoal activated, powder (treated with HCl), Nacalai Tesque, Inc., Kyoto, Japan) were used as samples. Activated carbon was used as a model for solid carbonized products from cellulose. In addition, the liquid products from Avicel cellulose were also analyzed.



Fig. 2. Definition of 0 ms in this study: (1) Image and time notation in high-speed camera footage (2) Area profile of the illuminated region.

3. Results and discussion

3.1. Observation of cellulose fast pyrolysis under infrared irradiation

Fig. 3 shows the pyrolysis behavior of Whatman cellulose under infrared irradiation. A dark-colored pyrolysis product (indicated by a red circle) formed at 1500 ms, after which a hole emerged in its vicinity at 1595 ms. The pyrolysis process then progressed in concentric circles around this hole (2000 ms and 3500 ms). A volatile, colorless mist was also emitted from this region. These findings suggest a decomposition behavior under infrared irradiation, where pyrolysis products are selectively and locally exposed to high temperatures, and fast pyrolysis occurs only in the immediate vicinity (refer to Fig. 4). In contrast, unreacted cellulose remains unheated due to its reflective properties toward infrared radiation.

High-speed camera observation of Avicel cellulose revealed that, similar to Whatman cellulose, dark-colored pyrolysis products formed during the initial phase of irradiation, as indicated by the red circles in Fig. 5 (1495 ms). This was followed by the formation of spherical droplets (1700 ms). Similar to the Whatman cellulose, colorless mist products were observed to evaporate from these droplets. As these droplets rolled and moved, they grew larger by accumulating other pyrolysis products. The growth of the droplets was offset by a decrease due to volatilization of the product, resulting in a constant droplet size (Fig. 6). Interestingly, colorless unreacted cellulose was observed under the pyrolysis products, indicating that such reactions occurred only on the surface exposed to infrared radiation. This phenomenon keeps the yield of LG constant regardless of the amount of sample introduced [28].

3.2. UV-Vis-NIR spectroscopic analysis of cellulose and its pyrolysis products

Fig. 7 shows the UV-Vis-NIR spectra of cellulose samples (Whatman and Avicel) and activated carbon, which serves as a model material for solid carbonized products, in relation to the irradiance of the infrared lamp used in this study. The left vertical axis of the figure denotes reflectance, with lower values indicating greater light absorption at each wavelength. The irradiance distribution of the infrared heater peaks at approximately 800 nm, indicating that both Whatman and Avicel cellulose exhibit negligible infrared absorption and are therefore not effectively heated. In contrast, activated carbon demonstrates significantly higher infrared absorption compared to cellulose, with an absorption rate approaching 100 %. This observation suggests that solid carbonized products absorb infrared irradiation efficiently, resulting in localized heating during pyrolysis.

The infrared absorption properties of liquid products obtained from the fast pyrolysis of cellulose were investigated. Fig. 8 shows the UV-Vis-NIR spectra of the volatile products and the extract of the residue (such as droplets shown in Fig. 5). Both were obtained by extracting the condensates of the volatile products recovered in the sample bag and the residues containing liquid products (droplets) remaining inside the sample boat with methanol, followed by evaporation of the methanol. The samples were collected after irradiating Avicel cellulose for 5 s, which was insufficient for the complete thermal degradation of cellulose.



Fig. 3. Pyrolysis behavior of Whatman cellulose under infrared irradiation with nitrogen flow (2 kW, 3 L/min).



Fig. 4. Mechanism of pyrolysis progression in sheet-type cellulose under infrared irradiation.



Fig. 5. Pyrolysis behavior of Avicel cellulose under infrared irradiation with nitrogen flow (2 kW, 3 L/min nitrogen flow).



Fig. 6. Mechanism of pyrolysis progression of powder-type cellulose under infrared irradiation.



Fig. 7. UV-Vis-NIR spectra of Whatman cellulose, Avicel cellulose and activated carbon, and radiant luminance of infrared irradiation.



Fig. 8. UV-Vis-NIR spectra of volatile products and residue extract from Avicel cellulose pyrolyzed by infrared irradiation (2 kW, 5 s, nitrogen flow: 3 L/min).

Both spectra display almost identical signals, with enhanced absorption in the 300–1200 nm region compared to the unpyrolyzed cellulose. This suggests that the droplets generated from cellulose and their volatiles are composed of similar components. It should be noted that the peak irradiance of the infrared lamp (850 nm) falls within this range, indicating that components with absorptivity in this region may act as sensitizers for irradiation absorption. Levoglucosan, glycolaldehyde and other minor volatile components were identified [28]; however, these compounds are colorless and cannot account for the absorption observed in the 300–1200 nm range. Other colored substances, including intermediates of char formation, may contribute to the absorption in this range, although their exact nature remains unclear at present.

To confirm this hypothesis, volatile products, pure LG, and pure water were added to a small region of Whatman cellulose, and their pyrolysis behaviors were compared to that of untreated cellulose. A methanol solution (5 μ L) of the volatile products obtained from Avicel pyrolysis was added to an area (5 mm in diameter) near the edge of the irradiation quartz rod of the IR lamp, where pyrolysis was expected to be less effective than at the center due to the influence of the unirradiated part of the cellulose sheet. Therefore, unless the additives have a positive effect on IR heating, the thermal degradation of cellulose is expected to occur at the center of the irradiation rod. After removing the methanol in an oven at 105 °C for 5 min, the sample was subjected to irradiation under a nitrogen flow. The experiment with pure LG was performed in a similar way, using a LG-in-methanol solution at a concentration of 11 g/ L, which was similar to the LG content in the solution of volatile products. As for pure water, immediately after the addition of 5 µL of water, the sample was subjected to irradiation.

Their observed pyrolysis behaviors are shown in Fig. 9. The area designated by red circles indicates the region where the additives were applied. When pure LG was added, thermal degradation of cellulose occurred from the center, whereas thermal degradation initiated from the area containing volatile products. These results suggest that the volatile products other than LG absorb irradiation more efficiently, leading to the selective heating that initiates pyrolysis. This suggestion is



Fig. 10. UV-Vis-NIR spectra of LG and water, and radiant luminance of infrared irradiation.

supported by the observation that LG does not absorb infrared irradiation in the range of 700–1400 nm (Fig. 10). Interestingly, a similar tendency was observed when pure water was used, suggesting that pure water also acts as a sensitizer for irradiation. In the spectrum of pure water (Fig. 10), a relatively strong absorption signal at 980 nm is observed, which may contribute to the efficient heating of cellulose in this area. Since water evaporates at 100 °C, this effect is limited to temperatures around 100 °C (the boiling point of water); however, it may assist the subsequent heating process of cellulose to temperatures sufficient for pyrolysis.

As shown in Fig. 11, these findings explain the heterogeneous progression of the pyrolysis of cellulose sheet from the edge of the hole, where pyrolysis products such as char, liquid substances, and water accumulate and absorb infrared radiation more efficiently. This leads to a rapid temperature increase in this area, quickly heat the surrounding cellulose to promote its degradation in a fast pyrolysis manor. Under such conditions, the coexisting LG can also evaporate efficiently.



Fig. 9. Fast pyrolysis behavior of Whatman cellulose when methanol extract of pyrolysis products from cellulose, LG or water were added in the upper right corner (red circle).



Fig. 11. Fast pyrolysis mechanism of cellulose under infrared irradiation.

3.3. Acceleration of pyrolysis using carbon

The results of this study indicate that infrared heating causes fast pyrolysis around pyrolysis products that absorb infrared irradiation and are efficiently heated. Therefore, we tried to promote the fast pyrolysis of cellulose by adding carbon, which efficiently absorbs infrared irradiation. The pyrolysis behavior of Whatman cellulose was observed when carbon was added as 1–9 points or applied to the entire surface of the cellulose with a pencil. The carbon-covered area ratio relative to the entire surface of the cellulose sheet was 0.5 %, 1.5 %, 2.3 %, and 100 % for 1 point, 4 points, 9 points, and the entire surface, respectively. Fig. 12 shows the high-speed camera observation results, while Fig. 13 depicts the amount of cellulose sample over time (ms) based on its area. In Fig. 13, the rate of decrease in the sample amount slows down when the sample volume falls below 20 %. At a heating time of 3000 ms for (1) original, (2) a single point, and (3) 4 points in Fig. 12, unreacted cellulose remains on the upstream side of the nitrogen flow (left side of the picture). This occurs because the cooling effect of the nitrogen is more pronounced on the upstream side.

The original (without carbon) showed little change up to 2000 ms. This is because the original cellulose reflected the infrared radiation.



Fig. 13. Relationship between the area of cellulose sample with (1) no carbon added, and carbon added at (2) a single point, and (3) 4 points, (4) 9 points, and (5) to the entire surface, and infrared irradiation time.

After 2000 ms, pyrolysis progressed, and most of the cellulose was pyrolyzed by 4000 ms. In contrast, when carbon was added at a single point, decomposition progressed from 0 to 1000 ms, and pyrolysis was observed to initiate from that point. In the case of 4 or 9 points of carbon addition, the pyrolysis process was accelerated compared to the single-point addition. However, there was little difference between the 4- and 9- point case, likely due to interference between neighboring circles during concentric pyrolysis process. In the case of full surface coating, pyrolysis began even before the maximum infrared power was reached, and the pyrolysis advanced rapidly. This is because the entire sample was uniformly and rapidly heated by the addition of carbon. These results suggest that fast pyrolysis can be accelerated by adding a substance with high infrared absorption, such as carbon.

4. Conclusions

The pyrolysis behavior of cellulose under infrared irradiation was observed using a high-speed camera. The following conclusions were drawn:



* Percentage of the sample area covered by carbon, total sample area base

Fig. 12. Fast pyrolysis behavior of Whatman cellulose when (1) no carbon added, and carbon added at (2) a single point, and (3) 4 points, (4) 9 points, and (5) to the entire surface (*percentage of the sample area covered by carbon, total sample area base).

- 1. Localized pyrolysis products were generated in the sheet cellulose sample, and pyrolysis progressed in the vicinity of these products.
- 2. In the powdered cellulose sample, droplets of pyrolysis products were formed only on the irradiated surface.
- 3. UV-Vis-NIR analysis revealed that liquid and solid products absorb infrared irradiation more efficiently than cellulose. A mechanism of localized pyrolysis was proposed, whereby these pyrolysis products absorb infrared radiation more efficiently and reach higher temperatures.
- 4. Pyrolysis was accelerated by adding carbon, which readily absorbs infrared radiation.
- 5. The finding of this study has important implications for improving the efficiency of fast pyrolysis of cellulose using infrared heating.

CRediT authorship contribution statement

Kawamoto Haruo: Writing – review & editing, Supervision, Funding acquisition. **Minami Eiji:** Writing – review & editing, Data curation. **Nomura Takashi:** Writing – original draft, Investigation, Formal analysis, Data curation, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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