Supplementary Information Reactive Boiling of Cellulose for Integrated Catalysis Through a Liquid Intermediate

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The experiments and calculations considered in the manuscript are described in

the supplementary information to provide more detail.

S0. Overview of Supplementary Files

The supplementary information consists of the following files.

- Supplementary Information Methods and Figures (this document)
- Supplementary Video 1
- Supplementary Video 2
- Supplementary Video 3
- Supplementary Video 4
- Supplementary Video 5

The following is a brief description of each video file.

Supplementary Video 1

This video shows a single microcrystalline cellulose particle thermally decomposing on an 80 ppi RhCe/ α -Al₂O₃ foam at 700 °C observed at ~45° from the surface normal. Each frame represents one millisecond of real time, and the dimension of the particle before conversion is about 300 µm. Frames from this video were used to produce Figure 1a. (Quicktime; 5.4 MB)

Supplementary Video 2

This video shows two microcrystalline cellulose particles thermally decomposing on an 80 ppi RhCe/ α -Al₂O₃ foam at 700 °C observed at ~45° from the surface normal. Each frame represents one millisecond of real time, and the dimension of both particles before

conversion is about 300 μ m. Clearly visible within the video is the combination of the two liquid intermediate droplets to a single, larger liquid particle. Frames from this video were used to produce Figure 4. (Quicktime, 9.8 MB)

Supplementary Video 3

This video shows a single cellulose particle about 300 μ m in size decomposing on a flat RhCe/ α -Al₂O₃ surface at 700 °C observed at ~80°.from the surface normal. Each frame represents one millisecond of real time. Frames from this video were used to produce Figure 1b. (Quicktime, 7.5 MB)

Supplementary Video 4

This video shows a single particle of sucrose about 400 μ m in size melting and reacting on a flat RhCe/ α -Al₂O₃ surface at 700 °C observed at ~80° from the surface normal. Each frame represents one millisecond of real time. (Quicktime, 6.5 MB)

Supplementary Video 5

This video shows the impact and volatilization of a droplet of glycerol (~800 μ m) on a hot, autothermal catalytic foam. The liquid droplet initially breaks up leaving a large spherical droplet which forms a gaseous film between itself and the catalytic foam. (Quicktime, 9.3 MB)

S1. Method for Small Cellulose Particles on Catalytic Foams

Visualization of microcrystalline cellulose particles on a 80 ppi α -Al₂O₃ foam coated with Rh-Ce (2.5 wt% each) occurred in a 20 mm I.D. quartz reactor tube shown in Figure S1. Three foams (10 mm high, 18 mm wide) were stacked on top of each other with a fourth blank 80 ppi foam on the bottom, wrapped in ceramic paper (for friction fit), and slid into the reactor tube. A type K thermocouple was inserted 10 mm from the leading surface. A 1 cm pyrex tube combined with a size 20 pyrex reactor tube end cap provided the connection between the reactor and a cellulose hopper necessary for solid particle delivery. A quartz light pipe was inserted through the end cap and contacted the leading surface providing a light sample to an optical pyrometer. The cellulose particles (avg. 315 µm) stored in an acrylic tube hopper (~10 cm diameter) were pushed into a feed tube (0.25 inch I.D.) using a 0.25 inch wood auger driven in

reverse with a servo motor permitting variable feed rates. Air was supplied by a gas cylinder and metered by a flow control valve calibrated with a bubble column.

The reactor in Figure S1 was wrapped in a resistive heater controlled by a variac and wrapped in insulation. Autothermal operation was initiated by heating the reactor externally with air flow to ~400 °C, at which point particles were delivered to the surface. Steady operation was obtained within ~5 minutes, and the heater was turned off. Varying surface temperatures were obtained by varying the air flow rate or cellulose flow rate.

Digital video was obtained by focusing a Photron Fastcam Ultima APX with color image through the curved quartz reactor. The four optical lenses placed in series to obtain the presented magnification were: 1) a Micro-NIKKOR 105 mm lens by Nikon of Japan, 2) a Nikon PN-11 extension tube by Nikon of Japan, 3) a Kenko extension tube for Nikon/AF 36 mm by Kenko of Japan, and 4) a Kenko 2x Teleplus MC7 telephoto extension tube by Kenko of Japan. Light was provided to the catalyst necessary for high speed imaging by a Solarc Light LB-50 by Welch Allyn, Inc. of New York, U.S.A.

Additional videos are shown as Figures S2 and S3, and Figure 1 from the manuscript is repeated as Figure S4. Figure S2 shows the behaviour of two particles impacting and coming to rest near each other on a the catalytic foam within ~20 ms. After converting to an intermediate liquid, the two particles combine in about 3 ms to form a larger liquid particle which ultimately volatilizes at ~180 ms. Gaseous evolution is clearly visible from 100 - 137 ms. The frames illustrate the particles' tendency to form spherical liquid droplets. White streaks in frames 100 and 123 ms are small particles passing through the frame before contacting the surface and volatilizing. Figure S3 shows a lone, irregularly shaped particle coming to rest on an alumina strut, converting to a liquid, and volatilizing to gases in ~123 milliseconds. The particle forms an

intermediate liquid overhanging the narrower strut, but surface tension pulls the liquid into a more spherical conformation.

S2. Method for Small Particle Cellulose Feedstock

Microcrystalline cellulose obtained from FMC biopolymer (Lattice NT-200) was sieved to average size ~315 μ m. As shown in Figure S5, the particles are not perfectly spherical, but rather have an aspect ratio (defined as length/width) of 1.39 ± 0.05 (95% confidence interval) when considering only particles large enough for examination with high speed photography (>20 μ m). Examination of cellulose particles by light scattering reveals in the size distribution (vol %) that most of the mass of the cellulose exists as large particles (>200 μ m). However, most of the particles are small (~1 μ m). Figure S6 includes scanning electron micrographs of microcrystalline particles coated in Pt. The particles clearly exhibit high porosity with large void spaces (10-40 microns) within the particle.

S3. Method for Small Particle Conversion on Catalytic Foams

Three video files with $T_{surface} = 700$, 825, and 900 °C were obtained by varying the cellulose and air flow rates as listed in Figure S7. At each of the listed conditions, approximately 50 particles were tracked from resting impact (not moving on the surface) to the point in time when no visual trace of the particle was evident. At each of the three conditions, the total conversion time was directly proportional to the square of the initial particle radius (measured at the point of resting impact). The difference between the measured conversion times and a linear fit appears to increase with increasing particle dimensions. As shown in the first three panels of Figure S7, the proportionality constant decreased with increasing surface temperature. In other words, a hotter surface resulted in faster heat transfer approximately consistent with $q_{conduction}=h_{surface}(\Delta T)$. The fourth panel shows a frame of video captured through the reactor wall. The

thermocouple is clearly visible in the upper left corner for size comparison (~300 μ m). The limitation of the depth of field of the camera only permits the measurement of particles that fall on the foam located in the center of the video frame.

Particles landing on struts of the catalytic foam within the depth of field of the camera were clearly visible and measureable during their conversion lifetime as shown in Figure 1. The width of four additional particles of varying size are included in Figure S8 to show that the behaviour of the particle in Figure 1 was representative of other particles. All observable particles exhibited nearly constant (or only slightly decreasing) width for most of their lifetime through the conversion from solid to liquid. Near the end of the particle conversion, fast linear decrease in particle width was observed.

S4. Method for Small Particle Conversion on Catalytic Flat Surfaces

Particles of cellulose and sucrose pyrolyzing on a flat catalytic wafer (Rh-Ce/ α -Al₂O₃, 2.5 wt% each) were also recorded using a high speed camera using the experimental setup depicted in Figure S9. A quartz reactor was held in place 1-2 cm above the catalytic wafer by metal clamps. Air was supplied by a high pressure gas cylinder through a needle valve, and particles of solid material were supplied from a side tube sealed with a pushrod. A quartz fiber light pipe delivering light to an optical pyrometer was attached through the quartz reactor such that it contacted the catalytic surface. A butane torch was clamped directly below the catalytic wafer such that its distance was adjustable to control the surface temperature. The high speed camera was placed at a low angle relative to the flat surface (~10°) obtaining light through a flat pyrex protector plate. Light was provided from behind the particles.

Figures S10 and S11 depict the conversion of two very different shaped cellulose particles at 700 °C. Both particles generate a liquid layer in contact with the surface followed by gas evolution as small bubbles. The liquid layer progresses up the

particle finally generating a fully liquid droplet. The liquid droplet, hemispherical in shape, decreases rapidly in size leaving a clean surface behind.

Calculations in Figure 2 were completed with the following measurements in Figure S12. The height (x) and width (2y) were measured directly from the video frames by relating the number of pixels to objects of known dimension such as the optical fiber. The radius of curvature, R_c , defining the shape of the liquid droplet was calculated permitting a calculation of the liquid droplet volume, V. As shown in Figure S13 and S14, the radius of curvature and droplet volume exhibited constant linear change with time.

Figures S15 and S16 depict the conversion of two particles of sucrose (mp = 190 °C) on the flat catalytic surface at 700 °C. The solid crystalline particle appears very different (transparent with flat surfaces) than the microcrystalline (semi-crystalline) cellulose particle at the moment of impact. Both sucrose particles rapidly melt to a nonvolatile liquid droplet which initiates reactive gas and volatile organic evolution. The liquid droplets rapidly decrease in volume leaving a clean, flat surface.

S5. Glycerol Droplets on Catalytic Foams and Wafers

The paper refers to glycerol droplets converting by reactive flash volatilization to volatile species. Glycerol boils under atmospheric pressure at ~290 °C permitting a liquid particle to form a gaseous film between itself and a hot surface. Figure S17 shows the impact dynamics (impact and breakup) of a large particle (~800 µm) of glycerol on a catalytic foam at 650 °C. Due to its boiling point restriction, the temperature difference between the surface and the particle must be approximately 350 °C. The key observation of the frames (particularly 27-47 ms) is the absence of the nucleating gaseous bubbles.

S6. Method for Cellulose Rod Experimental Setup

Cellulose rods were prepared by pulping (grinding at high speed in the presence of distilled water) prepared sheets of cellulose-rich biopolymer. Analysis of the cellulose-rich sheets showed that some hemicellulose was still mixed with cellulose (79.8% glucan, 19.5% xylan, 0.3% lignin, 0.4% ash). The liquid pulp was poured into a mold with a mesh bottom (50 cm x 50 cm x 1 cm deep), compressed with a board, and dried for two days. The semi-dry slab was removed from the mold, and air-dried in the presence of a fan for three additional days. The board was then cut using a table saw in the following manner. All four edges were lightly trimmed (1-2 mm) creating a square slab. Strips of cellulose were cut with a saw to 7 mm width. The strips were then trimmed with a razor blade to 7 mm cross width, creating a cellulose rod 7mm x 7mm x 500mm.

Processing of the cellulose rods was performed in the setup outlined in Figure S18. Three catalytic foams and one blank heat shield were wrapped in ceramic paper and held in place inside a quartz reactor by friction fit. A thermocouple was placed 10 mm down from the leading edge of the catalytic bed. The reactor was wrapped in insulation. Attached to the top of the reactor was a size 20 pyrex reactor endcap fused to a 1 cm I.D. pyrex tube extending ~1 m from the top of the reactor. Gases (N₂, O₂, and CH₄) flowed into the cap through the outside of the annulus to the catalyst. A quartz light pipe was inserted into the outside of the annulus and down to the leading edge of the catalyst. The long pyrex tube was graduated ($\Delta x=1$ cm) along its entire length to measure the movement of the rod with time. Metal weights (60, 120, 180 g) were inserted through the top of the tube, and an end cap assured a reactor seal. The catalytic reactor was attached to a secondary fixed bed reactor with 9 cm long by 18

mm diameter of Rh-Ce/ α -Al₂O₃ spheres. The secondary bed assured that all organics were broken down to non-condensable carbon species (CO, CO₂, CH₄) for measurement with a gas chromatograph. The secondary reactor bed was wrapped in a resistive heater and then in 1 inch thick ceramic insulation and the bed temperature was maintained at 700 °C. Effluent gases were sampled during intial rod experiments to validate carbon flow rates measured visually.

The reaction was initiated by flowing CH_4 , O_2 , and N_2 over the catalyst and supplying heat externally from a butane torch. Methane was selected as the control fuel due to its inability to exhibit homogeneous chemistry at the considered temperatures upstream of the catalyst where the virgin cellulose rod rested. The methane catalytic partial oxidation (CPO) reaction lit off at ~350 °C generating sufficient heat to initiate an orange glow at the leading edge of the catalyst. The rod was supported on a sealed pull-pin and dropped on the catalyst after light off. Initiation of the methane CPO reaction generates sufficient heat to initiate cellulose decomposition. In general, 5-10 cm of rod was permitted to process before data was collected (reported in Figure 3) to allow steady-state processing conditions. The first catalyst was replaced every three rods.

Figure S19 shows the surface temperature measured with the optical pyrometer attached to the quartz light pipe on the surface of the reaction catalyst. Without the presence of a cellulose rod, methane CPO exhibited $T_{surface}$ behavior that was satisfactorily fit with a third degree polynomial as a function of the dilution parameter (N₂ mol%). The methane temperature served as a measure of the bulk surface temperature. Naturally, the interaction of the rod with the catalyst foam will alter the temperature immediately at the interface in a complex manner requiring future study.

Figure S20 shows that rod height as a function of time for three rods with the 60 gram weight as a function of the bulk surface temperature to demonstrate the behavior of continuous, steady state cellulose processing. Each data point represents the recorded time at which the top edge of the cellulose rod passed a length marker on the reactor tube. These experiments generated a large number of measurements for each experimental parameter combination. By examining multiple applied pressures and dilution parameters, Table S1 (data used in Figure 3) was constructed showing cellulose processing rates. The 95% confidence interval listed in the Figure 3 caption was calculated by the method of Table S2.



Figure S1. Cellulose particle conversion was studied on RhCe-coated alumina foams in air in a visibly transparent 20 mm I.D. quartz tube surrounded by a resistive heater wrapped in ceramic insulation. Particles were delivered to the surface using an acrylic tube hopper through which passed a 0.25 inch wood auger operating in reverse. Cellulose flow rate was set by controlling the rotational speed of the wood auger with a servo motor. Air was supplied with a high pressure gas cylinder and metered using a gas-flow needle valve. A thermocouple was inserted into the catalytic bed 10 mm below the leading surface. A quartz fiber light pipe was inserted through the feed end-cap and impinged on the leading catalytic surface perpendicularly. Effluent was sent to an incinerator.



Figure S2. Microcrystalline cellulose particles (~300 μ m) reacting to volatile species in air on a 700 °C Rh-Ce/ α -Al₂O₃ surface have been visualized with high-speed photography on an 80 ppi Al₂O₃ foam support at C/O=1.15 at 45° from the surface normal with temporal resolution of one millisecond. Two particles (impacting at 0 and 17 ms) convert to a liquid and combine (82-85 ms) to a single particle which reactively volatilizes completely (180 ms).



Figure S3. Conversion of ~300 μ m microcrystalline cellulose particle to volatile species on a 700 °C RhCe/ α -Al₂O₃ surface. Cellulose passes through a molten intermediate state before volatilizing completely.



Figure S4. Conversion of ~300 μ m microcrystalline cellulose particle to volatile species on a 700 °C RhCe/ α -Al₂O₃ surface. Cellulose passes through a molten intermediate state before volatilizing completely.



Figure S5. Above: Particles of microcrystalline cellulose exhibited a distribution of aspect ratios defined as length/width with average aspect ratio 1.39 ± 0.05 . **Below:** Sieved microcrystalline cellulose considered in the small particle experiments exists mostly as ~300 µm particles, but most of the particles are small (~1 µm). Particle size distributions were collected by light scattering.



Figure S6. Microcrystalline cellulose particles sieved to 300 μ m average particle size (by volume) are shown in panel A. Particles exist as porous structures of crystalline cellulose rods 1-20 microns in width (Panels B-E). The surface of a crystalline micro-rod (panel E).



Experiment	Α	В	С
Support	Foam	Foam	Foam
Catalyst	Rh-Ce	Rh-Ce	Rh-Ce
Surface T / °C	700	825	900
Air Flow / SLPM	1.52	2.21	1.98
Cellulose Flow / g hr ⁻¹	53.1	37.2	30.6
N ₂ /O ₂ Ratio	3.76	3.76	3.76
C/O Ratio	1.15	0.55	0.51

Figure S7. The conversion of microcrystalline cellulose particles on Rh-Ce/ α -Al₂O₃ foam supports (80 ppi) exhibited conversion time in milliseconds (impact to disappearance) linearly dependent on the square of the particle radius. Panel A is the data shown in Figure 2A with a different independent axis. Hotter surfaces decreased conversion time by increasing heat transfer rates to the particle. The lower-right panel shows a frame captured by high-speed photography for the conversion of particles with a 700 °C surface (thermocouple in the top-left corner is 300 μ m in width). Experimental conditions for the three sets of data in panels A, B, and C are located in the table.



Figure S8. The conversion multiple microcrystalline cellulose particles on Rh-Ce/ α -Al₂O₃ foam supports (80 ppi) on a 700 °C surface exhibited a constant particle diameter (width measured parallel to surface) before rapid, linear decrease.



Figure S9. Cellulose particle conversion was studied on RhCe-coated alumina wafer in air heated from below by a butane torch. Particles were fed to the surface by addition to the side of the reactor by a sealed push rod. Air was supplied by a compressed cylinder through a needle valve to control the flow. A quartz fiber light pipe was inserted through a side arm and impinged on the leading catalytic surface perpendicularly.



Figure S10. Cellulose particle conversion was studied on RhCe-coated alumina wafer in air heated from below by a butane torch at 700 °C.



Figure S11. Cellulose particle conversion was studied on RhCe-coated alumina wafer in air heated from below by a butane torch at 700 °C.



$$R_{C} = \frac{X^{2} + Y^{2}}{2X}$$
$$V = \pi \left(RX^{2} - \frac{1}{3} X^{3} \right)$$

Figure S12. Cellulose particles completely converted to liquid were measured for height, x, and width, 2y, as a function of time. The radius of curvature, R_c , and volume, V, of the droplet were then calculated by the listed equations.



Figure S13. Cellulose particles completely converted to liquid exhibit approximately linear change in volume as a function of time. Twelve particles are shown decreasing in volume starting from arbitrary time corresponding to region C of Figure 3.



Figure S14. Cellulose particles completely converted to liquid exhibit approximately linear change in radius of curvature as a function of time. Twelve particles are shown decreasing in radius of curvature starting from arbitrary time corresponding to region C of Figure 3.



Figure S15. Crystalline sucrose particle conversion was studied on RhCe-coated alumina wafer in air heated from below by a butane torch at 700 °C.



Figure S16. Crystalline sucrose particle conversion was studied on RhCe-coated alumina wafer in air heated from below by a butane torch at 700 °C.



Figure S17. Impact of a large droplet (~800 μ m) of room temperature glycerol on a Rh-Ce (2.5 wt% each) on 80 ppi alpha alumina (99%) foam at C/O=1.5 and a surface temperature of 650 deg C measured with an optical parameter. Large droplet impact (2 ms) results in two smaller particles with the back particle remaining in the frame until completely disappearing in 177 ms. Original image collected at 1000 frames per second. Frames have been contrast adjusted to enhance viewing. Liquid glycerol droplets do not exhibit development of bubbles but rather appear to move around on the hot surface under a film of gas consistent with the Leidenfrost phenomenon.



Figure S18. Conversion of cellulose rods to synthesis gas was examined using a RhCe-coated 80 ppi alumina foam catalyst in a 20 mm I.D. quartz reactor wrapped in ceramic insulation. A size 20 pyrex tube end-cap was fused with a graduated 1 cm O.D. pyrex tube extending ~1 m above the reactor and 1 cm past the interface between the end-cap and the reactor. Cellulose rods (7 mm by 7 mm) placed in the pyrex tube were pressed against the foam catalysts by cylindrical steel weights placed on top of the cellulose rod. A thermocouple was placed in the catalytic bed 10 mm from the catalyst/cellulose interface. A quartz light pipe fiber was inserted in the feed end-cap and contacted the catalyst perpendicularly. Effluent gases then passed over a 9 cm RhCe bed of spheres heated by an external resistive heater to 700 °C to ensure complete breakdown to combustion products and synthesis gas. Products were sampled with a syringe, and the effluent was passed to an incinerator.



Figure S19. The bulk surface temperature was measured using an optical pyrometer collecting light through a quartz light pipe touching the surface of an Rh-Ce/ α -Al₂O₃ 80 ppi foam operating with CH₄, O₂ and N₂. The total gas flow rate was maintained at 5.0 SLPM, and the C/O ratio was maintained at 0.8. The dilution (N₂ / (N₂ + O₂ + CH₄)) was varied between 0.5 and 0.8 to vary the surface temperature on the catalytic foam.



Figure S20. Rods of cellulose (7mm x 7 mm x 500 mm) were pressed against an operating Rh-Ce/ α -Al₂O₃ 80 ppi foam catalyst operating with CH₄, N₂ and O₂. The rate of rod pyrolysis was measured as rod height as a function of time.

Table S1. The conversion of rods of cellulose (7 mm X 7 mm X 500 mm) on Rh-Ce/Al₂O₃ 80 ppi foams was examined to determine conversion rate (flux through the catalyst) as a function of the dilution parameter and accompanying bulk surface temperature.

		Char		Transition			Pyrolysis		
Experiment #	6	3	5	7a	18	1	0	4	2
Weight (grams)	60	60	60	60	60	60	60	60	60
Flux (kg hr ⁻¹ m ⁻²)	28	37	39	80	91	98	111	135	154
ΔR (95%)	4	6	6	12	10	9	22	19	12
D, Dilution Parameter	0.77	0.75	0.725	0.713	0.697	0.68	0.65	0.59	0.53
T _{Surface} (°C)	688	716	745	757	772	785	806	838	867
Experiment #	16	17	15	14b	14a	12	10	9	11
Weight (grams)	120	120	120	120	120	120	120	120	120
Flux (kg hr ⁻¹ m ⁻²)	39	50	54	79	108	123	161	199	250
ΔR (95%)	5	4	1	15	11	26	21	24	36
D, Dilution Parameter	0.77	0.75	0.725	0.713	0.697	0.68	0.65	0.59	0.53
T _{Surface} (°C)	688	716	745	757	772	785	806	838	867
Experiment #	24	23	25	21	20	19	22		
Weight (grams)	180	180	180	180	180	180	180		
Flux (kg hr ⁻¹ m ⁻²)	48	70	69	102	136	199	327		
ΔR (95%)	9	15	11	19	24	40	106		
D, Dilution Parameter	0.77	0.75	0.725	0.713	0.697	0.68	0.65		
T _{Surface} (°C)	688	716	745	757	772	785	806		
Experiment #					27	26			
Weight (grams)					150	90			
Flux (kg hr ⁻¹ m ⁻²)					208.61	118.55			
ΔR (95%)					30.31	13.27			
D, Dilution Parameter					0.65	0.65			
T _{Surface} (°C)					806	806			

Table S2. The experimental data in Figure 3 exhibited a 95% confidence interval equivalent to ~21 kg hr⁻¹ m⁻². Variance from all experiments was pooled and the degrees of freedom was calculated as 553.

Experiment #	St Dev	# of Measurements	Variance
18	1.464	36	2.14
1	1.29	35	1.66
0	1	13	1.14
4	2.57	30	6.60
2	1.66	32	2.76
14a	1.11	16	1.24
12	3.78	33	14.26
10	3.03	32	9.17
9	2.76	22	7.61
11	4.02	26	16.19
20	3.19	29	10.17
19	5.72	32	32.67
22	4.97	11	24.71
7a	0.6	13	0.36
14b	1.451	15	2.11
21	1.575	11	2.48
5	0.79	28	0.62
3	0.72	25	0.52
6	1.12	25	1.25
15	2.037	25	4.15
17	1.995	25	3.98
16	1.542	32	2.38
25	0.703	6	0.49
23	1.602	18	2.57
24	0.656	9	0.43
			. – .
		Pooled Variance	151.7
		PV^0.5	12.32
		N - 25 - 1	553
		Interval (<u>+</u> g/hr)	1.03
		Conversion to Flux	20.94