Supplementary Information

Ultra-fast pyrolysis of lignocellulose using highly tuned microwaves: Synergistic effect of cylindrical cavity resonator and frequency-auto-tracking solid-state microwave generator

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(1) Raw rice straw samples and their thermogravimetric properties

(2) Heating properties of different cellulose samples and effect of density on reaction temperature reached using microwave irradiation.

(3) Resonance frequency shift during microwave pyrolysis.

(1) Raw rice straw samples and their thermogravimetric properties

Table S1. Composition of rice straw.[*1]

	Rice straw composition (wt%)			
	Holocellulose	α-Cellulose	Acid insoluble residue	Ash
	59.6	39.8	13.0	13.4

[*]Rice straw supplied from a local farmer in Kochi, Japan was extensively dried in air and pulverized using a blender.

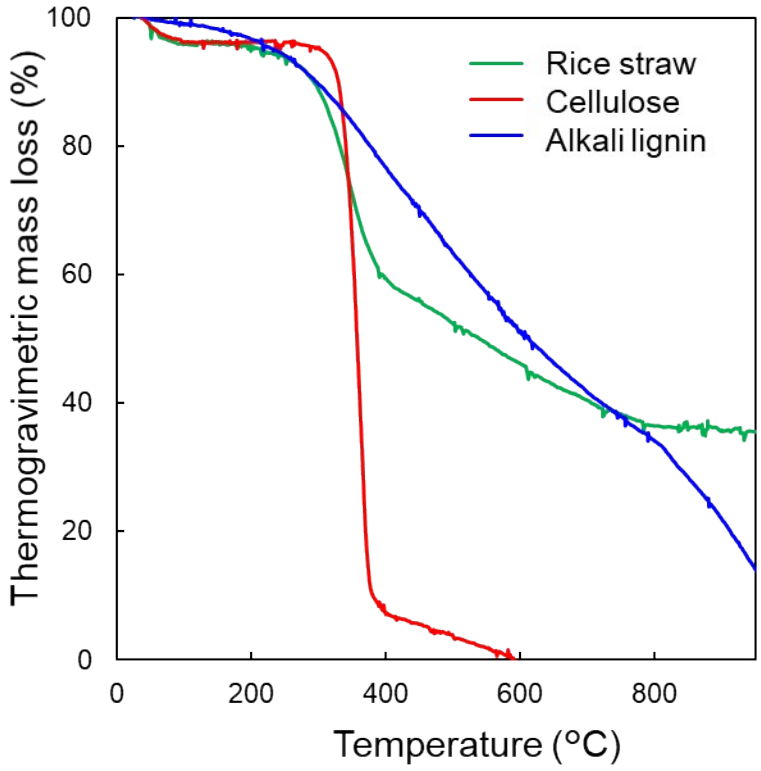

Fig. S1. Thermogravimetric curves of cellulose, alkali lignin, and rice straw.

(2) Heating properties of different cellulose samples and effect of density on the reaction temperature reached using microwave (MW) irradiation.

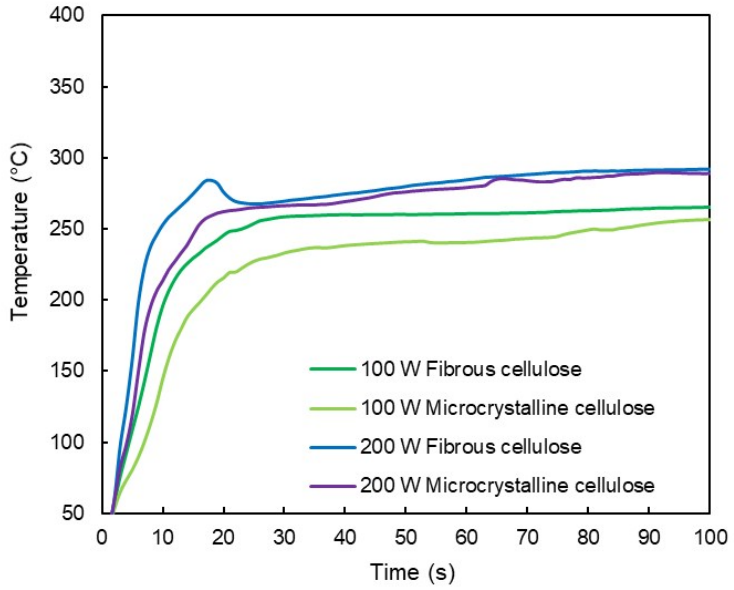

Fig. S2. Heating properties of microcrystalline and fibrous cellulose samples irradiated using 915 MHz microwaves (100 and 200 W).

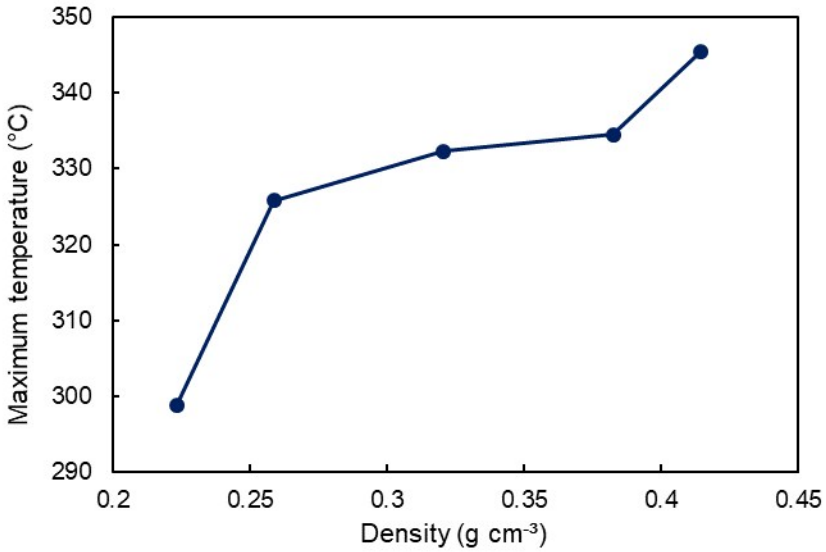

Fig. S3. Highest temperature reached during microwave (MW) pyrolysis vs density of microcrystalline cellulose (MW parameters: 915 MHz and 300 W).

(3) Pyrolysis of crystalline cellulose using MW and conventional heating methods.

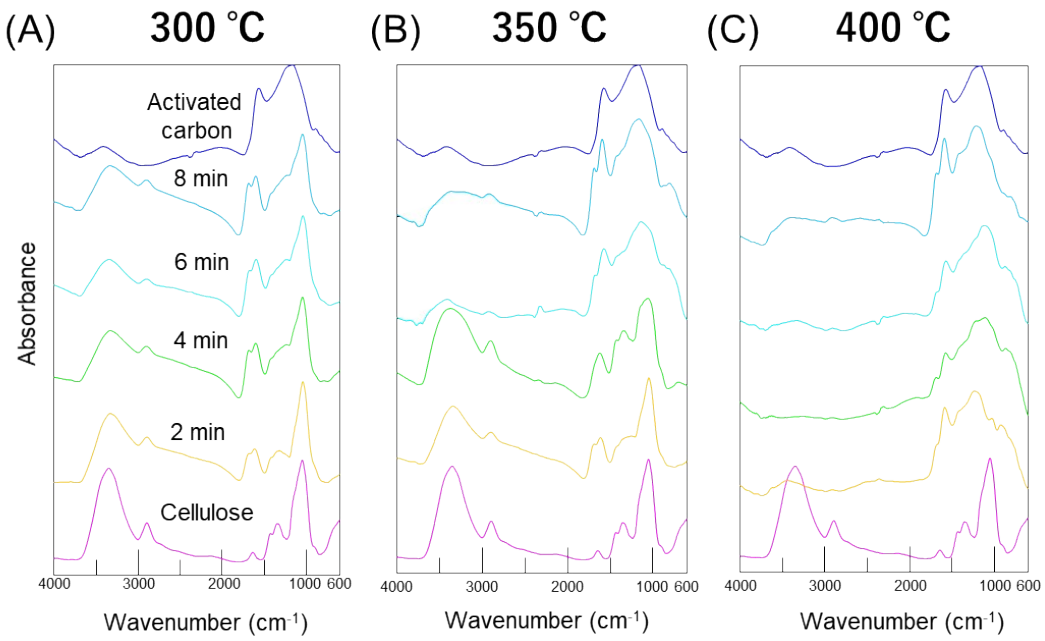

Fig. S4. Fourier-transform infrared spectra of bio-char produced from cellulose via MW pyrolysis using cylindrical cavity resonator (TM_{010} mode) at (A) 300, (B) 350, and (C) 400 °C.

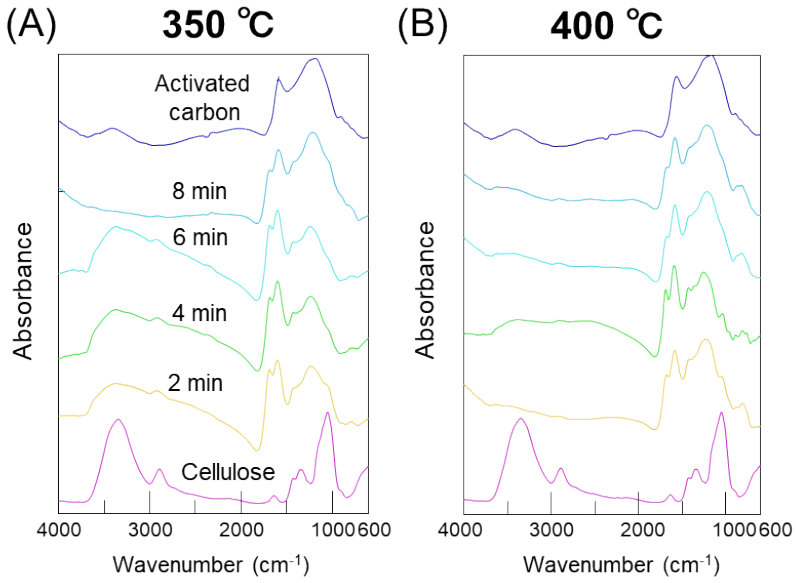

Fig. S5. Fourier-transform infrared spectra of bio-char produced from cellulose using conventional heating at (A) 350 °C and (B) 400 °C.

Table S2. Fourier-transform infrared spectra peaks of bio-char obtained from cellulose.

Wave number (cm^{-1})	Assignment
3600–3100	O–H (stretching)
3000–2800	Aliphatic C–H (stretching)
1700	C=O (stretching)
1620	C=C (stretching)
1450	Aromatic C=C (stretching)
1360–1310	C–C and C–O (skeletal vibration)
1200	C–O (stretching in pyranose ring)
1080	C–O–C (pyranose ring skeletal vibration)
1200–1000	Aromatic C–H (deformation vibration)
870, 800, 750	Aromatic out of plane bending

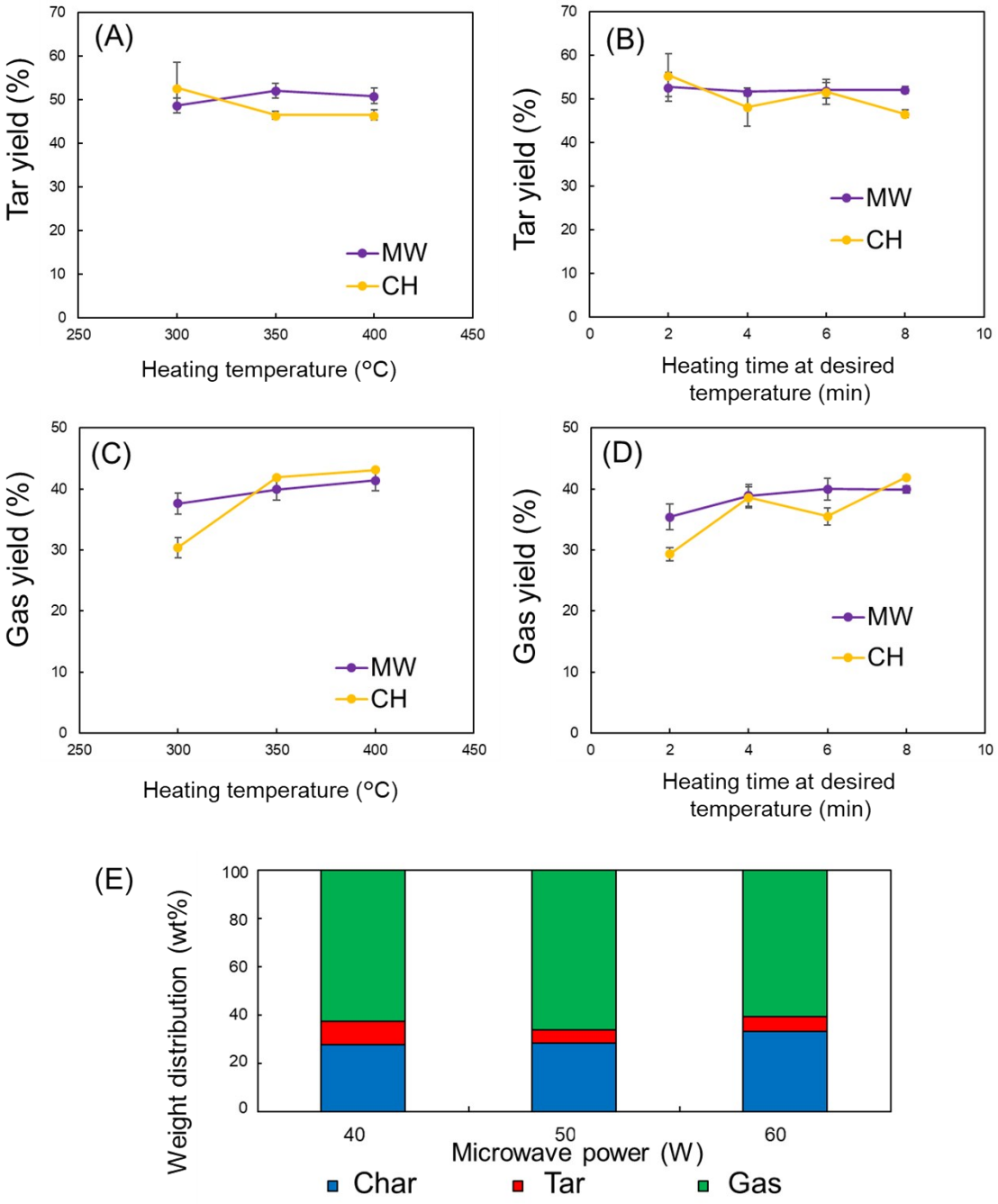

Fig. S6. Distribution of microwave (MW) heating and conventional heating (CH) pyrolysis products of crystalline cellulose and rice straw.

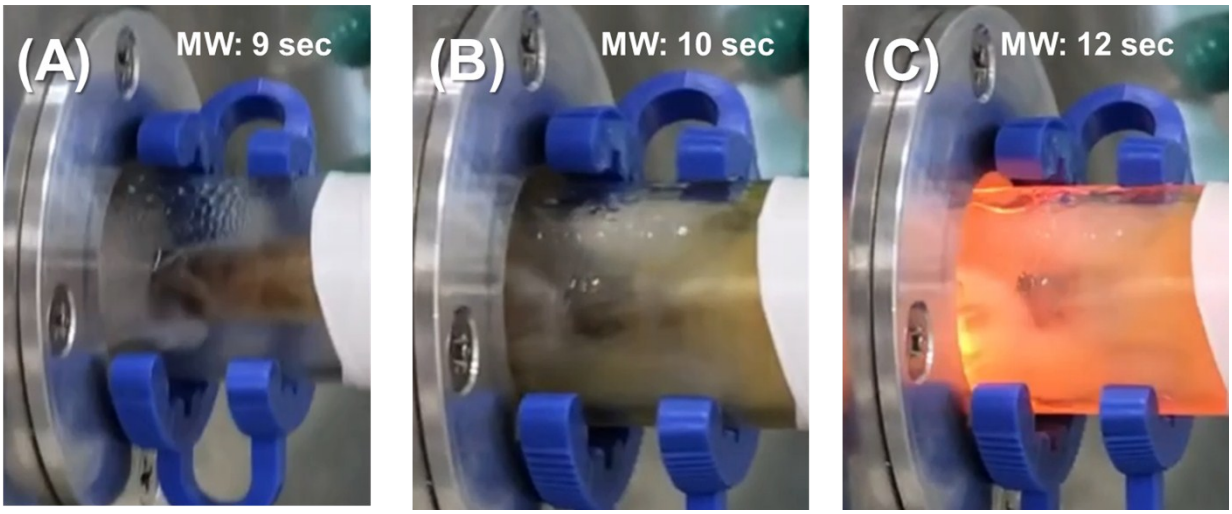

Fig. S7. Images of microwave (MW) pyrolysis of rice straw at 915 MHz. (A) 9, (B) 10, and (C) 12 s after beginning of MW irradiation at 200 W.

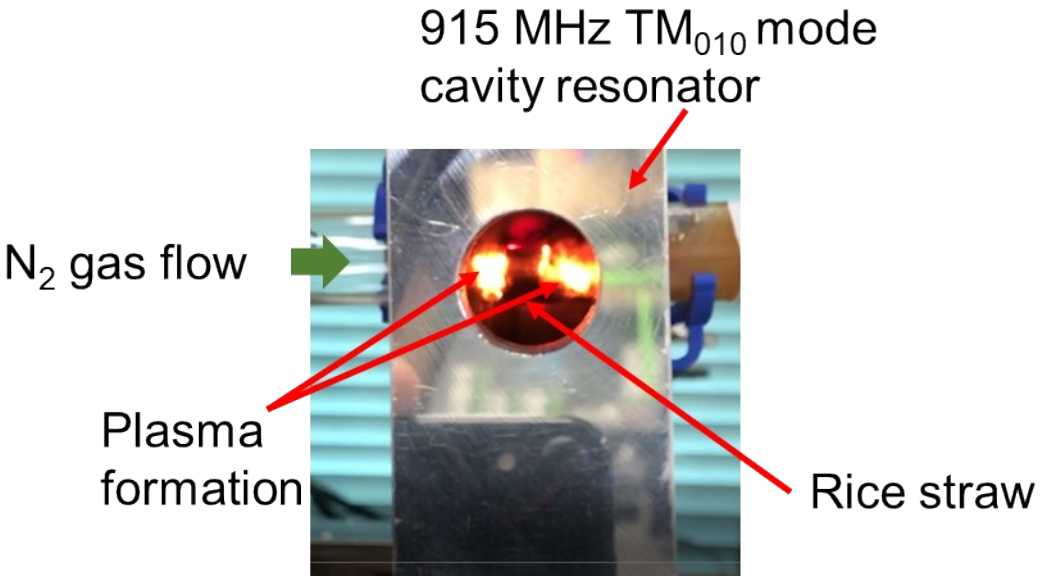

Fig. S8. Image of plasma formation during pyrolysis of rice straw under 915 MHz microwaves irradiation.

(3) Resonance-frequency shift of lignocellulose during MW pyrolysis.

Table S3. S_{21} parameters of cellulose and activated carbon (AC) mixtures of different ratios (center frequency of 2.45 GHz).*

S_{21} parameter	100% Cellulose	80% Cellulose + 20% AC	60% Cellulose + 40% AC	40% Cellulose + 60% AC	20% Cellulose + 80% AC	100% AC
Resonance frequency (MHz)	2.474	2.473	2.473	2.471	2.469	2.465
Insertion loss (dB)	53.8	58.1	58.2	66.3	69.7	72.3
Quality factor	1557	782	792	283	192	103

*The amount of 50 mg cellulose and AC mixture was added to a 6 × 4 × 30 mm alumina boat that was inserted into a quartz tube placed at the center of the cylindrical cavity resonator (TM_{010} mode).

Table S4. Dielectric and conductivity parameters of cellulose and activated carbon (AC) mixtures of different ratios.

Parameter	100% Cellulose	80% Cellulose + 20% AC	60% Cellulose + 40% AC	40% Cellulose + 60% AC	20% Cellulose + 80% AC*	100%AC*
Relative permittivity	1.42	2.53	4.56	8.52	-	-
Dielectric loss	0.044	0.167	0.746	6.169	-	-
Tan δ	0.031	0.066	0.164	0.724	-	-
Conductivity (S/cm)	0	0	0.508	1.45	3.70	6.14

* Unsuitable for complex dielectric properties measurements using the cavity perturbation method owing to their high electrical conductivity.

Table S5. S_{21} parameters of activated carbon (AC) at different loading amounts (center frequency of 2.45 GHz).

S_{21} parameter	Loading amount of AC (mg)				
	10	20	30	40	50
Resonance frequency (GHz)	2.484	2.481	2.480	2.478	2.476
Insertion loss (dB)	52.5	54.7	56.3	57.7	59.2
Quality factor	1825	1454	1188	1057	891.6

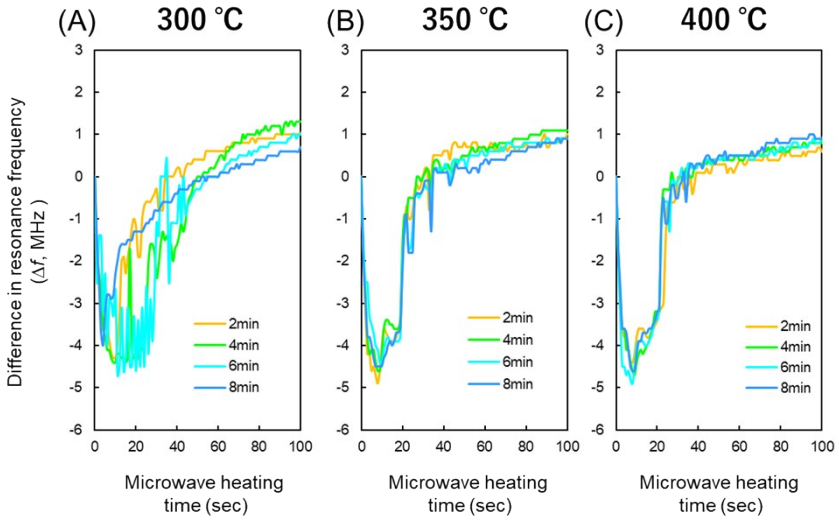

Fig. S9. Changes in resonance frequency during microwave (MW) pyrolysis of cellulose using cylindrical cavity resonator (TM_{010} mode) at 2.45 GHz. (A) 300, (B) 350, and (C) 400 °C.

Table S6. S_{21} parameters of rice straw and activated carbon (AC) mixtures of different ratios (center frequency of 915 MHz).*

S_{21} parameters	100% Rice straw	80% Rice straw + 20% AC	60% Rice straw + 40% AC	40% Rice straw + 60% AC	20% Rice straw + 80% AC	100% AC
Resonance frequency (MHz)	924.2	922.8	917.4	911.2	898.4	898.3
Insertion loss (dB)	68.4	68.5	75.5	80.6	82.8	82.0
Quality factor	318.0	322.9	153.6	93.3	101.7	82.0

*The amount of 1 g of rice straw and AC mixture was loaded in a quartz tube (Fig. 9C) and placed at the center of the cylindrical cavity resonator (TM_{010} mode).

References

1 S. Tsubaki, K. Oono and A. Onda, *Heliyon*, 2019, **5**, e01887.