Cesium-Catalyzed Hydrogen Production by the Gasification of Woody Biomass for Forest Decontamination

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ABSTRACT: The large quantities of contaminated wood produced following the radioactive cesium decontamination of forests after the Fukushima Daiichi Nuclear Power Plant accident in 2011 can be used as a biomass resource for energy production via thermal treatment (e.g., gasification). To store the radioactive Cs ash produced from gasification, the immobilization of Cs in the pollucite structure is possible and requires stable Cs additives. In this study, a Cs additive (Cs$_2$CO$_3$, CsCl, CsNO$_3$, or Cs$_2$SO$_4$) was doped with timber waste sawdust (1−30 wt %). Fixed-bed downdraft-type continuous steam gasification experiments (0.7 g/min) showed that Cs$_2$CO$_3$ enhanced H$_2$ production by 157% at 800 °C. X-ray absorption fine structure analysis and scanning electron microscopy observations revealed that the form of Cs on the surface of the char was Cs$_2$CO$_3$, which provided the active sites for gasification acceleration. Thermogravimetric pyrolysis and CO$_2$ gasification experiments showed that Cs$_2$CO$_3$ lowered the activation energy and frequency factor while also enhancing the reactivity.

1. INTRODUCTION

Since the Fukushima Daiichi nuclear power plant accident in 2011, large amounts of radioactive cesium (Cs) have been released into the environment, resulting in the widespread contamination of woody plants in eastern Japan. The decontamination of residential areas (i.e., residential houses, public facilities, roads, farmland, meadows, and forests) began in April 2011 and was completed on March 19, 2018, with the exception of the difficult-to-return zones. However, the forest area is too vast to perform economically feasible decontamination, and therefore, the lower risks associated with increased distances from human settlements should be considered. Since the simulated air dose rate curve based on the assumption of natural decay agreed well with the averaged air dose rates observed at 362 locations in the forests of Fukushima Prefecture, it was apparent that radioactive Cs is still present in the forest area. For forest decontamination, various means have been considered, including woodchip soil-decontamination and sedimentary organic matter removal. However, forest decontamination results in the production of large amounts of contaminated wood that can be utilized as a biomass resource for energy production via a thermal treatment such as gasification.

During the gasification of biomass contaminated with radioactive Cs, the Cs is volatilized at high temperatures and concentrated in ash residues. For the final storage of these ashes in a landfill site, various storage methods have been considered, including cement solidification. In contrast, no optimal final storage method has been found for radioactive Cs because of its high solubility in water and its volatility at elevated temperatures. However, the immobilization of radioactive Cs in synthetic rocks, such as synthetic pollucite (CsAlSi$_2$O$_6$), has been found to be effective. In the zeolite structure of pollucite, Cs$^+$ ions can be trapped without leakage into the environment, thereby leading to more robust immobilization than that achieved using cement solidification. Previous studies have reported the synthesis of pollucite from incineration ash via hydrothermal synthesis methods. With the addition of stable Cs$_2$CO$_3$ and CsCl, Cs was immobilized in a composite product that included pollucite from alkali-activated pyrophyllite and Cs-contaminated municipal solid waste incineration fly ash. This indicates that pollucite can also be prepared from woody biomass gasification ashes. In the process of woody biomass gasification, producer gas can be generated, thereby leading to the efficient production of power, syngas, and hydrogen. The reactions taking place in biomass gasification are described as follows:

\[ \text{Biomass pyrolysis} \]
\[ \text{Biomass(solid)} \rightarrow \text{volatile(gas + tar)} + \text{char(solid residue)} \]
Char conversion

\[ C + CO_2 \rightleftharpoons 2CO \] (boudouard reaction) \hspace{1cm} (2)

\[ C + H_2O \rightleftharpoons CO + H_2 \] (water gas(WG) reaction) \hspace{1cm} (3)

Gas phase reactions

\[ CO + H_2O \rightleftharpoons CO_2 + H_2 \] (water-gas shift(WGS) reaction) \hspace{1cm} (4)

\[ C_nH_m + 2nH_2O \rightleftharpoons (2n + m/2)H_2 + nCO_2 \] (tar steam reforming) \hspace{1cm} (5)

As described in an earlier study by Kapteijn et al., for the CO₂ gasification of activated carbon, alkali metal carbonates act as reactivity-activating catalysts.\(^1\)\(^5\) Another previous study investigated the alkali–metal–salt-catalyzed gasification of sawdust where the reactivity of CO₂ gasification was accelerated.\(^1\)\(^6\) They compared the reaction rates of the different alkali metals (i.e., K₂CO₃, Na₂CO₃, NaOH, and NaCl) by modeling the reactivity, dX/dt, based on the random pore model (RPM). However, there are some cases in which the RPM does not agree with the experimental reactivity, even though the modified RPM (MRPM) can be estimated due to the dominant catalytic activity after 40% conversion.\(^1\)\(^6\),\(^1\)\(^7\) From the perspective of the reaction kinetics, it was revealed that K₂CO₃ addition to char resulted in the highest dX/dt value among the various systems examined, thereby indicating that K₂CO₃ exhibited a high catalytic activity.

Furthermore, in the Gothenburg biomass gasification (GoBiGas) demonstration plant for biomethane production from woody biomass (including bark, wood pellets, wood chips, and recovered wood), which comprises a 32-MWth dual fluidized bed gasifier, tar clogging occurred at a gas cooler downstream of the gasifier, and the tar yield was controlled by feeding a K₂CO₃ solution into the process to activate the olivine bed material.\(^1\)\(^8\) It was therefore considered that the loading of Cs salts into the feedstock may also increase the activity of the steam gasification process, including pyrolysis and gasification with steam or CO₂. The Cs present in ash could therefore be utilized for pollucite synthesis while also being immobilized in the pollucite structure. The added Cs performs the role of promoting Cs nucleation to initiate formation of the pollucite zeolite structure.

Thus, we herein report the determination of the optimal Cs salt to enhance the gasification, in addition to the elucidation of the effect of the Cs salt loading on gaseous product formation and carbon conversion to gas, the effect of the steam amount on Cs-loaded gasification, identification of the metal species present in the solid product char, the role of Cs in steam gasification, and the reactivity of the pyrolysis and CO₂ gasification processes upon the addition of Cs to woody biomass feedstock.

2. MATERIALS AND METHODS

2.1. Feedstock Preparation. Timber waste (TW) sawdust of Japanese cedar and cypress trees (radioactive concentration: 20–40 Bq/kg) was procured from a local sawmill and pulverized to particle sizes of 2.0–2.8 mm (for continuous gasification tests) or 75–250 μm (for thermogravimetric analysis (TGA)). After drying in an oven overnight at 105 °C, the pulverized materials were dehydrated in a desiccator under reduced pressure. The properties of the sawdust are presented in Tables 1 and 2, based on proximate, ultimate, and chemical component analyses. The proximate
analysis was based on the JIS M8812 standard, while the ultimate analysis was based on the JIS M8813 standard for C, H, and N and the combustion–ion chromatography method for S. The chemical composition was analyzed by inductively coupled plasma–mass spectrometry for K, Ca, Mg, Al, Fe, and Mn, by atomic absorption spectroscopy for Na, and by absorption photometry for Si and P. Aqueous solutions of the cesium salts (Cs₂CO₃, CsCl, CsNO₃, or Cs₂SO₄) or other metallic salts (K₂CO₃ or Na₂CO₃) were sprayed onto the dried material surfaces to give a salt weight of 1–30 wt % relative to the total sprayed sawdust. The sprayed sawdust was dried in an oven overnight at 105 °C and then dehydrated in a desiccator under reduced pressure at 25 °C. One portion of the sprayed dried material with a size of 2.0–2.8 mm was used for the continuous gasification tests, while the other portion was carbonized in a SUS316 reactor (25.4 mm OD, 21.8 mm ID) with heating to 800 °C at a rate of 10 °C/min to obtain the char feedstock of CO₂ gasification for thermogravimetric (TG) observations. The sprayed dried material measuring 75–250 μm was used as the pyrolysis feedstock, as detailed in the next section.

The analytical results for the various materials (Table 1) were obtained using samples measuring <1 mm, where samples of different sizes were employed for the continuous gasification and TGAs. In general, the degree of chemical composition is dependent on the size of the feedstock. However, although the feedstocks employed were of different sizes, it was found that this did not affect the gasification and TGAs because the catalytic effects were clarified by comparing the kinetics and feedstock products with different salt loadings for each type of examination.

### 2.2. Steam Gasification in a Gasifier and Subsequent Analytical Methods

The apparatus employed for the continuous gasification experiments is illustrated schematically in Figure 1. More specifically, a laboratory-scale fixed-bed downdraft gasifier was employed. The reactor was composed of stainless steel (SUS) 316 with an outer diameter of 25.4 mm, an inner diameter of 21.8 mm, and a length of 750 mm. A thickness perforated grate of 2 mm was placed at a height of 18 mm from the bottom of the SUS316 tube reactor, and alumina balls were placed on the perforated plate to give a thickness of ~10 mm. The SUS tube was heated using electric furnaces by controlling the temperature at the center of each furnace. The reaction temperature was monitored using a thermocouple installed below the grate.

The sample was supplied to the gasifier using a screw feeder at a rate of 0.7 g/min. Nitrogen gas was fed into the gasifier at a rate of 200 mL/min using a flowrate regulator with a mass flow meter, and distilled water was preheated to 200 °C using a flexible heater to produce steam; and the resulting steam was supplied to the gasifier using a plunger pump at a rate of 0.01–1.0 mL/min. The gasifying agent was composed of nitrogen gas and steam, where the steam-to-carbon ratio (S/C) was set to 1 and defined as the molar ratio of the water vapor to the feedstock carbon, as expressed by the following formula

$$S/C = \frac{\text{water vapour supplied (mol)}}{\text{carbon content in feedstock supplied(mol)}}$$  \hspace{1cm} (6)

Following gasification, the generated producer gases, chars, and tars were collected. In the gas cleaning section after the first water-soluble byproduct collection impinger, the producer gas was passed through two more liquid tar collecting impingers, which were filled with acetone and isopropanol. The produced gas was recovered using a gas collecting bag. The air gas flow discharged from the sealed box by the expanding volume of the gas collecting bag inside the box was measured using a dry gas meter (DC-1C-M, Shinagawa Corporation, Tokyo). After recovering the produced gas, the reactor and pipe arrangements were disassembled, and any char remaining in the reactor was collected. The major product gases in the gas collection bags, including H₂, CO, CH₄, and CO₂, were quantitatively determined using a gas chromatograph equipped with a thermal conductivity detector (GC–TCD, GC-8, Shimadzu Corporation, Kyoto). The char products were analyzed by X-ray fluorescence (XRF) (RIX100, Rigaku Corporation, Tokyo) to determine the metal contents of the char. XRF analysis was conducted under the following conditions: X-ray source (Rh Kβ), wavelength (0.6133 Å), X-ray tube current (50 mA), X-ray tube voltage (50 kV), and dispersive crystal (LiF, Ge, pentaerythritol, thallium acid phthalate).

Cs L₃-edge X-ray absorption near-edge structure (XANES) and Cs L₃-edge extended X-ray absorption fine structure (EXAFS) data were collected at the Kyushu University Beamline (BL06) at the Kyushu Synchrotron Light Research Centre (SAGA-LS, Tosu, Japan) with a Si(111) dispersive crystal and an ionization chamber in the transmission mode. The quick-scanning XAFS technique was used for these measurements. XAFS spectra were analyzed using the ATENA.0.8.054 software.

The steam gasification char surface structure and geometry were observed by field emission scanning electron microscopy (SU8000, Hitachi, Ltd., Tokyo). The elemental distribution of the char surface was analyzed by energy-dispersive X-ray spectroscopy (EDX, X-Max, Oxford Instruments, Abingdon-On-Thames).

The experiments were analyzed based on the producer gas yield, Y, the carbon conversion to gas, X_{gas} the char yield, and the yields of other products, defined by the following formulae

$$Y_{(NL/g)} = \frac{\text{total producer gas (L)}}{\text{feedstock weight supplied (g)}} \times 100$$  \hspace{1cm} (7)

$$X_{gas}(C\text{-mol}%) = \frac{\text{carbon content in producer gas (C-mol)}}{\text{carbon content in feedstock (C-mol)}} \times 100$$  \hspace{1cm} (8)

$$X_{char}(C\text{-mol}%) = \frac{\text{carbon content in char products (C-mol)}}{\text{carbon content in feedstock (C-mol)}} \times 100$$  \hspace{1cm} (9)

$$X_{others}(C\text{-mol}%) = 100 - X_{gas} - X_{char}$$  \hspace{1cm} (10)

The carbon content of the metallic carbonate (e.g., Cs₂CO₃ and K₂CO₃) was counted in the denominator of the carbon conversion to gases.

### 2.3. Pyrolysis and CO₂ Gasification

Pyrolysis tests were performed by using TG and differential thermal analysis (DTA, Thermo plus EVO2-FKH TG-DTA8122, Rigaku). Temperature-programmed pyrolysis of the TW sawdust was performed under a dry N₂ atmosphere at a flow rate of 300 mL/min. Approximately 6 mg of the sample placed in a platinum pan was positioned on a sample holder in the TG analyzer. After
rapidly changing the atmospheric gas from Ar to CO₂, the CO₂
min, the temperature was increased from room temperature to
extent of the pyrolysis reactions, the conversion factor,
β
, was
defined as follows

\[ X = \frac{m_0 - m_t}{m_0} \]  

(11)

where \( m_0 \) is the sample weight at the start of pyrolysis, and \( m_t \)
is the sample weight at reaction time \( t \).

CO₂ gasification of the char feedstock was performed using
the TG–DTA. Approximately 6 mg of the TW char sample placed
in a platinum pan was positioned on a sample holder in the TG
analyzer. After maintaining the temperature at 30 °C for 30
min, the temperature was increased from room temperature to
800 °C at a rate of 30 °C/min. To determine the
temperature as a

Di = timber waste; conv. = conversion; all measurements were conducted at least twice and an average value reported.

| alkali salt      | \( \text{H}_2 \) | \( \text{CO} \) | \( \text{CO}_2 \) | \( \text{CH}_4 \) | char yield (C-mol%) | carbon conv. to gas (C-mol%) | other product yields (C-mol%) |
|------------------|-----------------|-----------------|-----------------|-----------------|----------------------|-----------------------------|-----------------------------|
| no addition      | 0.73            | 0.48            | 0.17            | 0.09            | 2.40                 | 78.2                        | 19.4                        |
| Cs₂CO₃          | 0.88            | 0.58            | 0.16            | 0.07            | 2.82                 | 86.3                        | 10.9                        |
| CsCl            | 0.77            | 0.53            | 0.15            | 0.07            | 4.76                 | 79.5                        | 15.8                        |
| CsNO₃          | 0.84            | 0.49            | 0.21            | 0.07            | 3.95                 | 81.6                        | 14.4                        |
| Cs₂SO₄         | 0.77            | 0.45            | 0.20            | 0.07            | 5.48                 | 76.3                        | 18.2                        |

2RT/E ≪ 1 and a reaction order \( n = 1 \), the following
expression can be obtained

\[ \ln \left[ \frac{-\ln(1 - X)}{T^2} \right] = \ln \left[ \frac{AR}{\beta E_A} \right] - \frac{E_A}{RT} \]

(14)

2.5. Modified Random Pore Model for CO₂ Gasification. The RPM is a gas–solid reaction kinetic model\(^3\)\(^4\)\(^5\)\(^6\) that is applicable to biomass gasification and can be expressed as

\[ \frac{dX}{dt} = k_{\text{RPM}}(1 - X)\sqrt{1 - \Psi \ln(1 - X)} \]

(15)

where \( k_{\text{RPM}} \) and \( \Psi \) are the kinetic rate constant and the pore
structure parameter, respectively. The analytical solution is

\[ -\ln(1 - X) = k_p + \frac{\Psi k_p^2}{4} t \]

(16)

3. RESULTS AND DISCUSSION

3.1. Fixed-Bed Downdraft Gasification. 3.1.1. Influence of Cesium Compounds. To elucidate the effect of cesium compounds on woody-biomass conversion by steam gasification, four different feedstocks containing different cesium salts were loaded onto the wood (10 wt% Cs₂CO₃, CsCl, CsNO₃, or Cs₂SO₄) and gasified at \( T = 1000 °C \), where \( S/C = 1 \). Table 2 presents the carbon conversion to gas of the Cs-loaded and raw TW materials. More specifically, the carbon conversion to gas was \( X_{\text{gas}} = 78.2 \) C-mol % for the raw sample but was 86.3, 79.5, 81.6, and 76.3 C-mol % for Cs₂CO₃, CsCl, CsNO₃, and Cs₂SO₄, respectively. These results indicate that the Cs₂CO₃, CsCl, and CsNO₃ cesium salts enhanced the conversion, while Cs₂SO₄ had the opposite effect. In particular, the addition of Cs₂CO₃ resulted in the greatest improvement in the conversion. Moreover, the addition of Cs₂CO₃ resulted in decreased yields for char and the other products, suggesting...
that Cs₂CO₃ promotes char conversion (reactions 2 and 3) and tar reforming (reaction 5).

As shown in Table 2, the product gas yields (i.e., H₂/CO/CO₂/CH₄) for raw TW gasification were 0.73, 0.48, 0.17, and 0.09 NL/g, respectively, while the gas yields obtained upon the addition of the various Cs salts were 0.88, 0.58, 0.16, and 0.07 NL/g for Cs₂CO₃, 0.77, 0.53, 0.15, and 0.07 NL/g for CsCl, 0.84, 0.49, 0.21, and 0.07 NL/g for CsNO₃, and 0.77, 0.45, 0.20, and 0.07 NL/g for Cs₂SO₄, respectively. All Cs-loaded samples produced greater amounts of hydrogen (0.77–0.88 NL/g) than the raw sample (0.73 NL/g), thereby implying the catalytic effect of the Cs salts on H₂ production, as described in an earlier study.¹⁵

3.1.2. Influence of Cs₂CO₃ Addition. As Cs₂CO₃ was found to be the most effective salt for the four examined Cs salts, the effects of the Cs₂CO₃ addition on the gasification performance were further investigated. Figure 2a shows the carbon conversion to gas versus Cs₂CO₃ addition for the TW steam gasification at T = 800 °C with or without Cs₂CO₃ addition (10 wt %). TW = timber waste.

Table 3. Producer Gas Yields, Carbon Conversion to Gas, Char yields, and Yields of Other Products with Cs₂CO₃ Loaded to the Feedstock during N₂/Steam Gasiification at T = 900 and 1000 °C and at S/C = 1⁴

| Cs₂CO₃ addition (wt %) | H₂ (NL/g) | CO (NL/g) | CO₂ (NL/g) | CH₄ (NL/g) | char yield (C-mol%) | carbon conv. to gas (C-mol%) | other product yields (C-mol%) |
|------------------------|----------|----------|----------|-----------|-------------------|------------------------|-----------------------------|
|                        |          |          |          |           | T = 800 °C        | T = 900 °C              | T = 1000 °C                 |
| 0                      | 0.30     | 0.22     | 0.13     | 0.08      | 9.83          | 46.5                   | 43.7                        |
| 1                      | 0.36     | 0.22     | 0.14     | 0.07      | 11.4          | 45.0                   | 43.5                        |
| 5                      | 0.60     | 0.25     | 0.23     | 0.06      | 14.1          | 56.9                   | 28.9                        |
| 10                     | 0.77     | 0.34     | 0.25     | 0.06      | 15.8          | 68.7                   | 15.6                        |
|                        |          |          |          |           | T = 900 °C        | T = 900 °C              | T = 1000 °C                 |
| 0                      | 0.65     | 0.36     | 0.22     | 0.08      | 5.34          | 69.6                   | 25.0                        |
| 1                      | 0.70     | 0.39     | 0.18     | 0.08      | 3.70          | 68.7                   | 27.6                        |
| 5                      | 0.86     | 0.49     | 0.22     | 0.07      | 4.84          | 82.3                   | 12.9                        |
| 10                     | 0.87     | 0.49     | 0.21     | 0.06      | 5.79          | 81.5                   | 12.7                        |
|                        |          |          |          |           | T = 1000 °C        | T = 1000 °C              | T = 1000 °C                 |
| 0                      | 0.73     | 0.48     | 0.17     | 0.09      | 2.40          | 78.2                   | 19.4                        |
| 1                      | 0.78     | 0.52     | 0.16     | 0.08      | 3.32          | 80.4                   | 16.3                        |
| 5                      | 0.83     | 0.56     | 0.15     | 0.08      | 3.84          | 83.5                   | 12.7                        |
| 10                     | 0.88     | 0.58     | 0.16     | 0.07      | 2.82          | 86.3                   | 10.9                        |

*Conv. = conversion; all measurements were conducted at least twice and an average value reported.*
gasification process at $T = 800, 900$, and $1000 \, ^\circ C$ and S/C = 1. The conversion at $T = 800 \, ^\circ C$ was 57.4 and 68.5 C-mol % for the addition of 5 and 10 wt % Cs$_2$CO$_3$, respectively, representing 10.1 and 21.2 percentage point increases from that obtained for the raw TW (i.e., 47.3 C-mol %). In addition, the conversion at $T = 900 \, ^\circ C$ was 82.9 C-mol % for 5 wt % Cs$_2$CO$_3$ addition and 81.4 C-mol % for 10 wt % Cs$_2$CO$_3$ addition, corresponding to 12.0 and 10.5 percentage point enhancements compared with the raw TW (i.e., 70.9 C-mol). Furthermore, at $T = 1000 \, ^\circ C$, the conversions obtained for the addition of 5 and 10 wt % Cs$_2$CO$_3$ were 84.1 and 86.1 C-mol %, respectively, which reflect 4.5 and 6.5 percentage point improvements compared to the raw TW sample (i.e., 79.6 C-mol %). Overall, the greatest increase in conversion was observed at a relatively low reaction temperature (i.e., $T = 800 \, ^\circ C$) for both 5 and 10 wt % Cs$_2$CO$_3$ addition.

Figure 2b shows the product gas yield with Cs$_2$CO$_3$ following the TW steam gasification at $T = 800 \, ^\circ C$ and S/C = 1. More specifically, the yields of H$_2$, CO, and CO$_2$ increased with the addition of Cs$_2$CO$_3$, and such a trend was observed for all of the studied reaction temperatures ($T = 800, 900$, and $1000 \, ^\circ C$), as shown in Table 3. In particular, the H$_2$ yield improved significantly from 0.30 to 0.77 NL/g upon increasing the Cs$_2$CO$_3$ loading from 0 to 10 wt %, representing an increase of 157%. Similarly, the CO yield increased from 0.22 to 0.34 NL/g (+55%), and the CO$_2$ yield increased from 0.13 to 0.25 NL/g (+92%). These improvements can be attributed mainly to the hydrogen-producing WG (reaction 3) and WGS reactions (reaction 4), which were enhanced by the addition of Cs$_2$CO$_3$. Furthermore, the WG reaction promotes char gasification and increases the CO yield, while the WGS reaction enhances the CO$_2$ yield, resulting in an overall increase in the gas yield.

As shown in Table 3, the char yields are 9.83–15.8 C-mol % at $800 \, ^\circ C$, 3.70–5.79 C-mol % at $900 \, ^\circ C$, and 2.40–3.84 C-mol % at $1000 \, ^\circ C$, indicating that the char yield decreased with increasing reaction temperature due to acceleration of the char gasification reactions (reactions 2 and 3).

For the relatively low temperature of $800 \, ^\circ C$ (Table 3), upon Cs$_2$CO$_3$ loading, the yields of other products (i.e., tar and soot) decreased, while the char yield and carbon conversion to gas increased with increasing Cs$_2$CO$_3$ loading. In the experiments with Cs$_2$CO$_3$ loading, decreased quantities of tar and increased quantities of soot as products were also observed. These results therefore indicate that Cs$_2$CO$_3$ exhibited a catalytic effect on the tar reforming process (reaction 5).

**3.1.3. Influence of the Amount of Steam.** Figure 2c shows the carbon conversion to gas versus the S/C ratio in the TW steam gasification process at $T = 800 \, ^\circ C$ both with and without the Cs$_2$CO$_3$ addition. Upon increasing the S/C ratio, the conversion increased regardless of whether Cs$_2$CO$_3$ was added. At S/C = 1 (Figure 2c), the carbon conversion to gas for Cs$_2$CO$_3$ addition was 63.9 C-mol %, which is 14.2 points higher than that of the raw TW (i.e., 49.7 C-mol %). In contrast, when the steam gasification process was carried out at S/C = 0.1, the conversion decreased from 45.7 to 42.1 C-mol %, which is a 3.29 point decrease. It was therefore clear that the enhancement in the amount of the generated product gas depends on the amount of steam employed during the steam gasification process.

Figure 2d shows the producer gas yield versus the S/C ratio in the TW steam gasification process at $T = 800 \, ^\circ C$. It is observed that the yields of H$_2$, CO, and CO$_2$ increased with increasing amount of steam during gasification, while only a small variation was observed for CH$_4$. The quantities of the generated producer gases (i.e., H$_2$, CO, and CO$_2$) reached a maximum at S/C = 1 for Cs$_2$CO$_3$ addition (10 wt %), as shown in Figure 2d (i.e., 0.62, 0.24, and 0.26 NL/g, respectively). These values are larger than those recorded for the raw TW (i.e., 0.46, 0.20, and 0.21 NL/g, respectively). This enhancement in producer gas generation was also attributed to the WG (eq 3) and WGS reactions (eq 4).

**3.1.4. Analysis of the Chemicals Present in the Char Products.** To elucidate the form of Cs involved in the steam gasification process, the chemical content of the byproduct char was analyzed. Thus, Table 4 shows the metal compositions of the char produced from the continuous gasification of TW with 5 wt % Cs$_2$CO$_3$ addition at $T = 800, 900$, and $1000 \, ^\circ C$, whereby the Cs mass remaining in the char product was <50%. It was therefore considered likely that Cs may have been volatilized and condensed on the inner wall of the gasifier, and/or that it passed through the pipe during steam gasification.

In an actual operating plant in Fukushima, particle-collecting filters have been installed that can almost completely recover the volatilized Cs via an established technique.

In a previous study, Cs$_2$CO$_3$-loaded TW was gasified at $T = 650–1050 \, ^\circ C$, and it was found that >80% of Cs$_2$CO$_3$ remained in the char when the moisture content (MC) was 11.4% in N$_2$ with no steam supply. In the current study, steam was supplied with an S/C ratio of 1, which affected the dissolution of the fraction of the Cs that was volatilized during gasification, leading to a lower Cs amount remaining in the char (<<50%). The dissolved Cs can be easily recovered by adsorbents such as zeolites and iron ferrocyanide.

To identify the form of Cs present in the char, XRD was carried out; however, the pattern was not sufficiently clear to identify the Cs products, and therefore, identification by XAFS analysis was attempted. Generally, gasification reactions occur...
at elevated temperatures in a reducing atmosphere; therefore, the Cs may be present in the residual char in the form of coordination compounds containing ligands with O and/or C as the coordinating atoms, due to the stability of such products. The Cs L3-edge radial structure functions (RSF) obtained from the EXAFS oscillations are shown in Figure 3b.

Figure 3. Cs–L3 edge (a) XANES spectra and (b) RSF from the EXAFS oscillations of the reference Cs salt powders (i.e., CsBPh4, CsNO3, Cs2SO4, and Cs2CO3), feedstock, and the chars from the gasification of TW using 5 wt % Cs2CO3 at T = 700, 800, and 900 °C.

Figure 4. SEM images of the char from steam gasification at T = 800 °C and S/C = 1 (a) without Cs2CO3 addition and (b) with Cs2CO3 addition (5 wt %). (c) Cs distribution in the char and (d) carbon distribution in the char.
The RSF of the residual char (produced from 5 wt % CsCO3-loaded TW steam gasification at 700−1000 °C) exhibited a peak corresponding to the neighboring atoms (or atoms within the primary coordination sphere) at \( R = 2.3−2.4 \) Å; this value is similar to the peak distances corresponding to the O-containing reference salts (Cs2CO3, Cs2SO4, and CsNO3), suggesting that the Cs compounds present in the char products (obtained after gasification at \( T = 700−1000 \) °C) contain O acting as the coordinating atoms. In contrast, the reference cesium tetraphenylborate compounds [i.e., CsB(C6H5)4 or CsBPh4] are completely ionic (ionic crystals) with no ligand; the peak of their neighboring atoms is located at \( R = 2.9 \) Å, which is a relatively large distance from Cs. It is therefore unlikely that the Cs present in the char products exists in the form of ionic crystals due to the lower distance of the peak compared to that of CsBPh4.

Figure 3a shows the Cs L3-edge XANES data for the reference Cs salt powders (i.e., the Cs2CO3, Cs2SO4, and CsNO3 O-containing salts and the CsBPh4 ionic crystal salt), the Cs-loaded feedstock, and the residual TW chars produced from the steam gasification of the 5 wt % Cs2CO3-loaded TW at \( T = 700, 800, \) and 900 °C. The XANES spectra of the chars were similar to that of the reference Cs2CO3. The RSF (Figure 3b) shows that with the exception of CsBPh4, the RSF values of the reference Cs salts are similar to one another. However, this result confirms that the chars were more similar to the reference Cs2CO3 than to the other reference Cs salts containing O as the coordinating atom. Thus, these results indicate that the Cs2CO3 loaded onto the TW remains as Cs2CO3 in the char after steam gasification.

The char surfaces were then analyzed using SEM, as shown in Figure 4a (\( T = 800 \) °C, S/C = 1). More specifically, Figure 4a shows the SEM image of the char surface without the Cs2CO3 addition, while Figure 4b shows the corresponding image for the 5 wt % Cs2CO3 addition. Thus, as shown in Figure 4b, many spherical particles with a diameter of \( \sim 0.1 \) μm distributed throughout the char surface are observed; no such particles are observed without Cs2CO3 addition. The small spherical particles were therefore attributed to Cs2CO3. The overlapping of these particles on the char surface was caused by the repeated spraying of the Cs2CO3 aqueous solution onto the TW sawdust surface during feedstock preparation.

The EDX mapping analyses for Cs and carbon are shown in Figure 4c,d, whereby the similar Cs and carbon distributions indicate that Cs is present on the same locations as carbon. According to the EDX elemental analysis, the main species present on the char surface were C, K, Ca, and Cs with the loadings of 66, 0.63, 0.0, and 32 wt %, respectively. The EDX-based K and Ca contents (i.e., 0.63 and 0.0 wt %) were 1−2 orders of magnitude lower than those obtained by XRF (i.e., 4.3 and 3.9 wt %), while that of Cs (32 wt %) was larger (i.e., 21 wt % obtained by XRF). These results confirm the presence of a large number of Cs atoms on the char surface that act as active sites during steam gasification.

### 3.2. Pyrolytic Kinetics of TW

The TG curves (obtained in a nitrogen atmosphere) for the raw TW and the Cs2CO3-loaded TW samples are compared in Figure 5. For the Cs2CO3-loaded TW, the temperature at which decomposition began was lower than that for the raw TW, enabling the occurrence of low-temperature pyrolysis. The pyrolysis kinetic parameters for the four Cs salts, including Cs2CO3, are presented in Table 5, and it is observed that only Cs2CO3 lowered the pyrolytic activation energy and frequency factor (\( E_a = 72.0 \) kJ/mol, \( A = 10^{3.85} \) s−1) from the corresponding values of the raw TW (\( E_a = 88.8 \) kJ/mol, \( A = 10^{5.12} \) s−1). Therefore, only Cs2CO3 accelerated the decomposition through the promotion of the pyrolytic reaction, which is similar to the results described above for the continuous gasification process.

| alkali salt (5 wt %) | activation energy \( E_a \) (kJ/mol) | frequency factor \( \log_{10} A \) |
|----------------------|-----------------------------------|-------------------------------|
| Cs2CO3               | 88.8                              | 5.12                          |
| CsCl                 | 72.0                              | 3.85                          |
| CsNO3                | 89.8                              | 5.39                          |
| Cs2SO4               | 89.6                              | 5.35                          |
| K2CO3                | 88.7                              | 5.23                          |
| Na2CO3               | 65.2                              | 3.29                          |
| Na2CO3               | 58.5                              | 2.67                          |

Table 5. Pyrolysis Kinetic Parameters
for K$_2$CO$_3$ ($E_a = 65.2$ kJ/mol, $A = 10^{2.29}$ s$^{-1}$) and Na$_2$CO$_3$ ($E_a = 58.5$ kJ/mol, $A = 10^{2.07}$ s$^{-1}$) shows that their pyrolytic reactivity values were similar to that for Cs$_2$CO$_3$ ($E_a = 72.0$ kJ/mol, $A = 10^{2.65}$ s$^{-1}$), indicating that Cs$_2$CO$_3$ plays a catalytic role in TW pyrolysis by lowering the activation energy and the frequency factor.

### 3.3. CO$_2$ Gasification Kinetics of TW Char

To elucidate the catalytic effect of Cs$_2$CO$_3$ on the CO$_2$ gasification of the TW char, the gasification process was performed by TG in the presence of Cs$_2$CO$_3$ at $T = 800–950$ °C. The plot of the reactivity ($dX/dt$) versus $X$ for the raw TW char and TW char with a 10 wt% Cs$_2$CO$_3$ loading is shown in Figure 6, where the graph shows both the observed reactivity and the calculated reactivities (RPM and MRPM). It is observed that at 800, 850, and 900 °C, the maximum reactivity for the Cs$_2$CO$_3$-loaded char is approximately 2–6 times higher than that of the raw TW char.

**Figure 6.** Observed and calculated reactivities ($dX/dt$) of the TW char (with 10 wt% Cs$_2$CO$_3$) and raw TW char (raw char) during isothermal CO$_2$ gasification at $T = 800–950$ °C. RPM = random pore model.
Table 6. Comparison of the Random-Pore Model Structure Parameter, $\Psi_i$, in CO$_2$ Gasification of the Char Samples (Raw TW Char and 10 wt % Cs$_2$CO$_3$ Loaded Char) at $T = 800$–$950$ °C

| sample                     | temperature (°C) | $\Psi$ | $c$ | $p$ | $k_{\text{RPM}}$ | $A$ (s$^{-1}$) | $E_a$ (kJ/mol) |
|----------------------------|-----------------|--------|-----|-----|-----------------|----------------|---------------|
| raw TW char                | 800             | 3.46   | 1.29| 6.79| 0.037           | 20.9           | 218           |
|                            | 850             | 2.27   | 1.19| 8.80| 0.068           |                |               |
|                            | 900             | 4.45   | 1.24| 8.83| 0.198           |                |               |
|                            | 950             | 3.14   | 1.24| 7.93| 0.741           |                |               |
| Cs$_2$CO$_3$ loaded TW char| 800             | 4.16   | 1.26| 2.54| 0.190           | 7.15           | 77.5          |
|                            | 850             | 3.01   | 1.10| 3.36| 0.437           |                |               |
|                            | 900             | 7.20   | 1.20| 5.33| 0.354           |                |               |
|                            | 950             | 3.21   | 1.18| 4.04| 0.664           |                |               |

char. However, at $T = 950$ °C, the reactivity of the Cs$_2$CO$_3$-loaded char was equivalent to that of the raw char. For the raw char, the peak of dX/dt was in the range of 0.8 < X < 0.95, and no dX/dt peak was observed for the Cs$_2$CO$_3$-loaded char. Therefore, it appeared that alkali-metal salts in the raw char accelerated CO$_2$ gasification at higher X values (0.8 < X < 0.95) due to the greater catalytic activity. On the other hand, Cs$_2$CO$_3$ increased the char reactivity from an earlier stage in the process. The RPM was used to estimate the raw char reactivity for X < 0.4, while the MRPM predicted the reactivity for 0.4 < X < 1 more accurately than the original RPM, particularly for 0.8 < X < 0.95.

Table 6 presents the RPM pore structure parameter, $\Psi_i$, the reaction rate constant, $k_{\text{RPM}}$, and the MRPM dimensionless constants, $c$ and $p$, for the char samples (raw TW char and Cs$_2$CO$_3$-loaded TW char) at $T = 800$, 850, 900, and 950 °C. In this context, we note that a higher value of $\Psi$ represents a developed pore structure. Thus, the $\Psi$ values for Cs$_2$CO$_3$-loaded char gasification at $T = 800$, 850, 900, and 950 °C were 4.16, 3.01, 7.20, and 3.21, respectively, while the corresponding values for the raw char were 3.46, 2.27, 4.45, and 3.14, respectively. Notably, at $T = 900$ °C, the $\Psi$ value for the Cs$_2$CO$_3$-loaded char is 1.6 times greater than that of the raw char, although equivalent values were obtained at all other temperatures. These results indicate that the addition of 10 wt % Cs$_2$CO$_3$ did not accelerate the development of the TW char.

Furthermore, the RPM reaction rate constants, $k_{\text{RPM}}$, for the raw TW char at $T = 800$, 850, 900, and 950 °C were determined to be 0.037, 0.068, 0.198, and 0.741 s$^{-1}$, respectively, while the corresponding values for the 10 wt % Cs$_2$CO$_3$-loaded char were 0.190, 0.437, 0.354, and 0.664 s$^{-1}$, respectively. Therefore, at the relatively low reaction temperatures of 800 and 850 °C, $k_{\text{RPM}}$ showed a drastic single-digit increase for the 10 wt % Cs$_2$CO$_3$-loaded char, while at a higher temperature of 950 °C, equivalent values were found. These results indicate that Cs$_2$CO$_3$ accelerates the reactivity at the relatively low temperatures of 800 and 850 °C.

Moreover, the MRPM dimensionless constant $c$ was determined in the range from 1.19 to 1.29 (Cs$_2$CO$_3$-loaded char) and from 1.10 to 1.26 (raw char), while the dimensionless constant $p$ ranged from 6.79 to 8.83 (Cs$_2$CO$_3$-loaded char) and from 2.54 to 5.33 (raw char). At the same reaction temperature, $p$ decreased upon Cs$_2$CO$_3$ addition, which is consistent with the previous research that suggested that $p$ decreases with increasing quantities of alkali and alkaline-earth metals (K, Na, and Ca) in the char samples.

The kinetic parameters [i.e., activation energy ($E_a$) and frequency factor (A)] given by the Arrhenius plots based on the RPM rate constants are presented in Table 6. For the raw char, $E_a = 218$ kJ/mol and $A = 20.9$ s$^{-1}$ ($R^2 = 0.962$), whereas for the Cs$_2$CO$_3$-loaded char, $E_a = 77.5$ kJ/mol and $A = 7.15$ s$^{-1}$ ($R^2 = 0.777$). According to these results, the addition of Cs$_2$CO$_3$ to the char results in a lower activation energy and frequency factor compared to those of the raw char. As a result, Cs$_2$CO$_3$ increased the reactivity during the CO$_2$ gasification of the TW char at the relatively low temperatures of 800–900 °C.

4. CONCLUSIONS

The use of wood containing radioactive cesium as a gasification feedstock was considered for energy production. We investigated the catalytic effect of Cs on the gasification reaction, and it was found that Cs$_2$CO$_3$ promoted the process of the carbon conversion to gas. The degree of enhancement depended on the Cs$_2$CO$_3$ loading, the reaction temperature, and the amount of steam. Cs existed as Cs$_2$CO$_3$ on the char surface after gasification, acting as an active site for reaction acceleration by lowering the activation energy and frequency factor. This technology can be applied for forest decontamination combined with heat and power production. Reuse of the residual Cs$_2$CO$_3$ is under investigation.

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Notes
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