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Produced Gas Conversion of Microwave Carbon Receptor Reforming

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***Abstract*—**Carbon dioxide and methane, the major components of biomass pyrolysis/gasification gas and biogas, top the list of substances that cause climate change, but they are also among the most important renewable energy sources in modern society. The purpose of this study is to convert carbon dioxide and methane into high-quality energy using char and commercial activated carbon obtained from biomass pyrolysis as a microwave receptor. The methane reforming process produces hydrogen and carbon. This carbon is deposited in the pores of the microwave receptor and lowers catalytic activity, thereby reducing the methane conversion rate. The deposited carbon was removed by carbon gasification due to the supply of carbon dioxide, which solved the problem of microwave receptor inactivity. In particular, the conversion rate remained stable at over 90% when the ratio of carbon dioxide to methane was 1:1. When the reforming results of carbon dioxide and methane were compared after fabricating nickel and iron catalysts using commercial activated carbon as a carrier, the conversion rate was higher in the iron catalyst than in the nickel catalyst and when no catalyst was used.

***Keywords*—**Microwave, gas reforming, greenhouse gas, microwave receptor, catalyst.

I. INTRODUCTION

CARBON dioxide and methane are major components of biomass pyrolysis/gasification gas and biogas generated from sewage treatment plants or landfill sites, which are basic environmental facilities, and are representative substances that cause climate change. As they are carbon-neutral fuels that circulate without increasing carbon dioxide; however, they are recognized as among the most important renewable energy sources at present, and thus attract much attention [1], [2]. To convert carbon dioxide and methane into high-quality syngas, various studies have been conducted on reforming technology.

Steam reforming is the most commonly used method because it is advantageous for the gas throughput and hydrogen production yield. This method produces hydrogen from natural gas at a crude refinery, but the reaction rate is relatively slow, and thus, a large reactor, and a large and strong endothermic reaction are required, resulting in a high temperature and pressure. The dry reforming reaction produces syngas through the reaction between carbon dioxide and methane. This method has attracted much attention because it is easier to control than steam reforming, reduces methane consumption, and consumes carbon dioxide, a greenhouse gas [3]. Partial oxidation reforming can use internal heat as an exothermic reaction, but it requires a limited amount of oxygen and exhibits low reaction efficiency [4]. Autothermal reforming has a higher hydrogen production rate than partial oxidation reforming, and requires less power than steam [5].

To convert methane and carbon dioxide to hydrogen or carbon monoxide, high-temperature reaction conditions or appropriate catalysts must be selected and applied. As the use of noble metal catalysts entails a cost problem, however, as well as a problem of the inactivation of catalysts by catalyst poison, a different type of catalyst is required [6], [7]. The use of carbon-based catalysts has been proposed as new catalysts with the advantages of higher availability, higher durability, and lower cost compared to metal catalysts in hydrocarbon pyrolysis reactions [8]. Moreover, the carbonation reaction between carbon-based catalysts and carbon dioxide, and the methane thermal decomposition reaction, can be promoted by the application of the microwave heating method [9]. Activated carbon and coal char as well as biochar, a carbide generated by biomass pyrolysis, can be used as carbon-based catalysts.

The microwave heating method has higher energy efficiency and greater excellence in rapid heating, selective heating, and uniform heating compared to the conventional hot-air or electric heating method [10]. Microwave heating has been widely used for the reduction of environmental pollutants, the pyrolysis/gasification of biomass, and the drying of materials. The microwave heating method has been applied of late to methane-carbon dioxide reforming, using the aforementioned carbon-based catalysts [9]. From such studies, it was confirmed that carbon materials are excellent as microwave receptors, and that the conversion rate of gas reforming is higher than that of the conventional heating method. It has also been reported that the selectivity of the generated gas is improved, and that the carbon deposition is also reduced. Few studies have been conducted, however, on the characteristics of reforming using sludge char, a sewage sludge carbide, as a microwave receptor. Therefore, in this study, to investigate the reforming characteristics of the microwave heating method using sludge char, which is produced through the pyrolysis of the sludge generated in sewage treatment plants, and commercial activated carbon as a microwave receptor (MR), the properties and composition ratios of the supplied carbon dioxide and methane gases, the carbon dioxide:steam ratio, and the reforming characteristics according to the catalyst type were studied.

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II. EXPERIMENT APPARATUS AND METHOD

A. *Experiment Apparatus*

The experiment apparatus for microwave reforming that was used in this study consists of a microwave reformer, a gas/ steam feed line, a monitoring/control equipment, and a sampling/analysis line, as shown in Fig. 1 [11].

The microwave reformer has a structure in which a quartz tube reactor (40 mm in diameter and 320 mm in length) is vertically installed in a cavity oven with two 1 kW-capacity magnetrons. The temperature of the reformer was finely controlled up to 1,000 °C using a thermocouple (k-type, 2 mm in diameter) installed in the MR and a controller, and the temperature changes in the MR were continuously monitored using a data logger (Hydra Data Logger 2625A, Fluke, USA). A MR sample basket was placed inside the quartz tube reactor, and the basket was vertically variable so that the receptor sample could flow into and out of the reactor.

As for the gas/steam feed line, carbon dioxide, methane, and nitrogen were supplied from the cylinder of each gas via MFC (Line Tech, M3030V, South Korea), which controls the flow, to a mixer, from which the gases were supplied as the simulated reforming gas. The steam generated by the evaporation of a certain amount of water supplied by a syringe pump (KD SCIENTIFIC, KDS-100, USA) was supplied to the reactor without condensation through the heating line, together with the simulated reforming gas.

The monitoring/control equipment continuously monitored the gas flow rate and temperature using LabVIEW (LabVIEW 8.6, National Instruments, USA). The sampling/analysis line consisted of an impinger and a cooler (ECS-30SS, Eyela Co., Japan) containing glass fiber and anhydrous calcium chloride, respectively, for the removal of soot and moisture. GC-TCD (CP- 4900, Varian, Netherlands) was installed to analyze the gas generated by reforming.

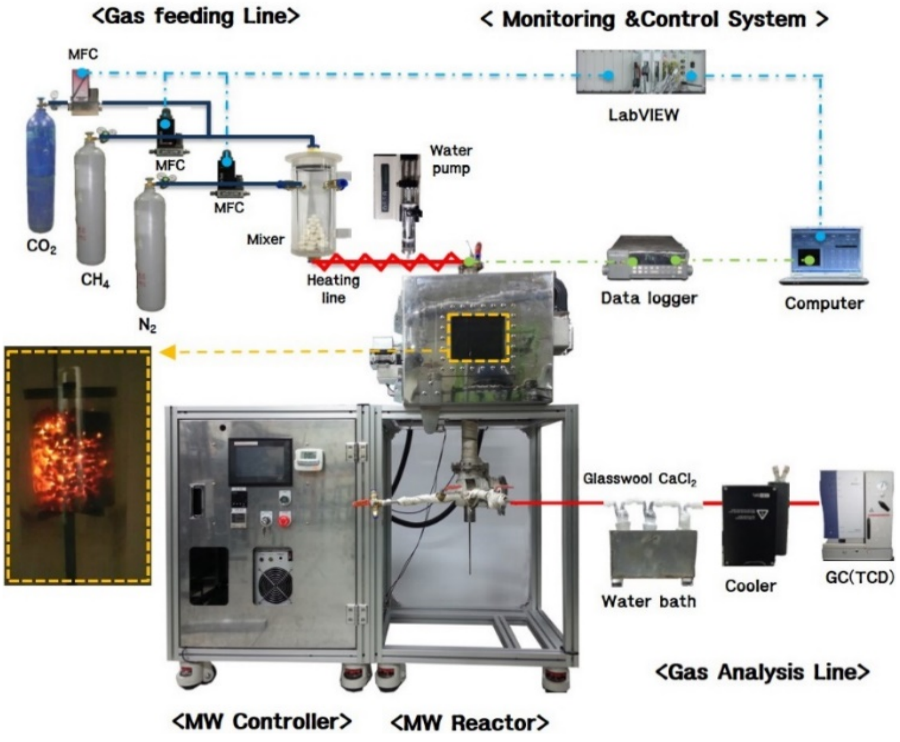


Fig. 1 Experiment apparatus for microwave reforming

B. *Experiment Method*

In the microwave reforming experiment, the MR in the sample basket located in the quartz tube reactor installed in the microwave reformer was heated through microwave irradiation, and the reformed gas generated when the simulated gas supplied into the quartz tube reactor passed through the receptor layer was collected and analyzed.

The used MR was sieved to 1-3 mm, and 15 g of the receptor was placed into the sample basket, which was positioned at the center of the quartz tube reactor for each experiment. The simulated gas was constantly supplied at a total flow rate of 75 mL/min to keep the volumetric hourly space velocity (VHSV) constant at 0.3 L/g·h. The microwave power supply was 2 kW, and the sample temperature was linearly increased by initial start-up from room temperature to the set temperature of 900 °C, and then the temperature was kept constant.

Experiments for each variable were conducted according to the properties and ratios of the simulated gas, with the reforming temperature and VHSV fixed. The remaining balance, except CO_2 and CH_4 which are the major components of the simulated gas, was N_2 which is an inert gas. The reformed gas collected using Tedlar bags for gas collection at regular time intervals from the beginning of the experiment was analyzed. The injected simulated gas and reformed gas were analyzed using GC-TCD. Molecular sieve 5A (80/100 mesh) was applied to the H_2, CH_4, CO, O_2, and N_2 gases, and the PoraPlot-Q column was applied to the CO_2 gas.

The conversion rates of CO_2 and CH_4, the major gases to be reformed, were calculated using (1) and (2).

$$CH_4\ conversion\ (\%) = \frac{100[(H_2)_{out}/2]}{[(CH_4)_{out}+(H_2)_{out}/2]} \qquad (1)$$

$$CO_2\ conversion\ (\%) = \frac{100[(CO)_{out}/2]}{[(CO_2)_{out}+(CO)_{out}/2]} \qquad (2)$$

where $(CH_4)_{out}$, $(H_2)_{out}$, $(CO_2)_{out}$, and $(CO)_{out}$ represent the concentrations of methane, hydrogen, carbon dioxide, and carbon monoxide measured at the outlet of the microwave reformer, respectively.

Equations (3)-(7) show the reaction equations for the reforming of each gas.

- Carbon gasification reaction

$$C(char) + CO_2 \Leftrightarrow 2CO, \qquad \triangle H_{298} = +173\ kJ/mol \qquad (3)$$

- Methane thermal decomposition reaction

$$CH_4 \Leftrightarrow C + 2H_2, \qquad \triangle H_{298} = +75\ kJ/mol \qquad (4)$$

- Methane partial oxidation reaction

$$CH_4 + ½O_2 \Leftrightarrow 2H_2 + CO, \qquad \triangle H_{298} = -8.5\ kJ/mol \qquad (5)$$

- Methane and carbon dioxide dry reforming reaction

$$CH_4 + CO_2 \Leftrightarrow 2CO + 2H_2, \qquad \triangle H_{298} = 260.5\ kJ/mol \qquad (6)$$

- Water-gas shift reaction

$$CO + H_2O \Leftrightarrow CO_2 + H_2 \qquad \triangle H_{298} = -41\ kJ/mol \qquad (7)$$

III. RESULTS AND DISCUSSION

To produce syngas through methane and carbon dioxide reforming, the characteristics of carbon dioxide reforming, methane reforming, and methane-carbon dioxide mixed-gas reforming were investigated by conducting microwave reforming experiments using the MW receptor.

A. Carbon Dioxide Reforming Characteristics

To investigate the carbon dioxide reforming characteristics, carbon dioxide 25% and nitrogen as balance gas were supplied as the simulated gas. Fig. 2 shows the reforming results.

The CO_2 conversion rate increased as the experiment started, and it reached the maximum conversion rate of 67% at 60 minutes, and then gradually decreased. This is because the carbon of the MR reacted with carbon dioxide and was converted to carbon monoxide according to the carbon gasification reaction of (3), as shown by the fact that the concentration of carbon dioxide decreased while that of carbon monoxide increased until 60 minutes.

In particular, in the microwave heating method, as microwave energy is transmitted to the inside of the receptor and kinetic energy is converted to heat energy by the vibration of the material, microplasma is generated inside the receptor, and hot spots with temperatures higher than the thermal receptor layer temperature are maintained at specific locations, facilitating the gasification reaction, which is a heterogeneous reaction.

When the CO_2 conversion rate reached the maximum value, the conversion rate decreased, resulting in a decrease in carbon monoxide and an increase in carbon dioxide. This is because the carbon component of the MR was consumed according to (3), and the carbon gasification reaction was lowered [12].

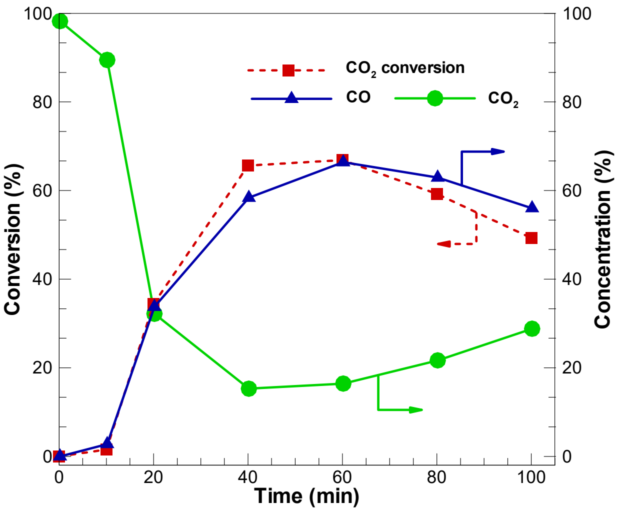


Fig. 2 Results of carbon dioxide reforming at 900 °C

B. Methane Reforming Characteristics

To investigate the methane reforming characteristics, methane (25%) and nitrogen as balance gas were supplied as the simulated gas. Fig. 3 shows the reforming results.

The methane conversion rate increased sharply over time. It reached the maximum conversion rate of 76% at 60 minutes, and then decreased. The hydrogen concentration showed a similar tendency as the methane conversion rate, and decreased after reaching the maximum concentration at 60 minutes. This is because methane was decomposed and converted to hydrogen according to (4). At this instance, carbon was also generated, and as part of the generated carbon was deposited on the surface of the MR, the porosity was lowered and the conversion rate decreased after the maximum value was reached at 60 minutes. At the beginning of the reaction, the carbon monoxide increased and then decreased. This is because hydrogen and carbon monoxide were formed by the partial oxidation reaction of sludge char by the residual oxygen (5). In addition, as already mentioned, the irradiation of microwaves on the MR layer leads to a high temperature, which increases the reactivity of the ambient gas. This high temperature particularly has a higher impact on the methane thermal decomposition reaction of (4). As a result, the problem of microwave methane reforming using a microwave carbon catalyst receptor is that the carbon generated by the methane thermal decomposition reaction is deposited on the surface of the receptor and lowers the activity of the receptor. Similar results have been obtained by other researchers on various carbonaceous-based catalysts, such as char [13], activated carbon [14], and carbon black [15].

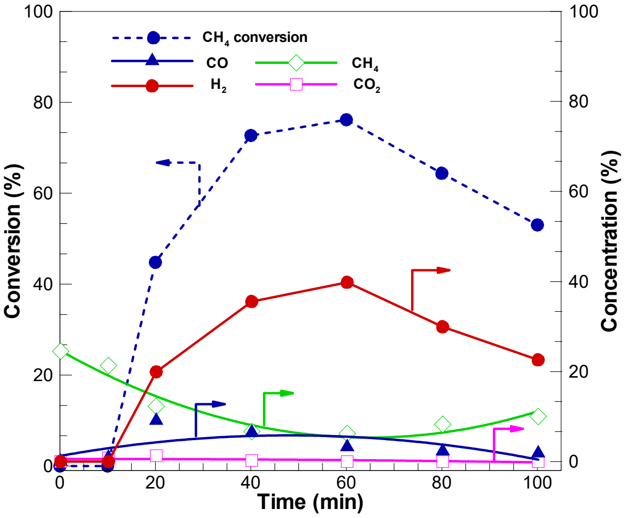


Fig. 3 Results of methane reforming at 900 °C

C. Carbon Dioxide and Methane Mixed-Gas Reforming Characteristics

To investigate the carbon dioxide and methane mixed-gas reforming characteristics, carbon dioxide (12.5%), methane (12.5%), and nitrogen as balance gas were supplied as the simulated gas. Fig. 4 shows the reforming results.

The conversion rates of carbon dioxide and methane increased sharply after the start of the experiment, and remained constant over time. The carbon dioxide conversion rate reached the maximum value of 94% at 60 minutes and was maintained at 93% at 100 minutes, which was the end of the experiment. As for the methane conversion rate, it reached the maximum value of 94% at 40 minutes and was maintained at 91% at 100 minutes. Unlike the reforming by supplying carbon dioxide or methane alone, the conversion rate was maintained without reduction over time when the mixed gas was supplied. This is because hydrogen was generated by the methane thermal decomposition (4), and the carbon ($C_{(CH4)}$) deposited on the surface of the MR was reduced and carbon monoxide was generated by the carbon gasification (3) due to carbon dioxide. The conversion rate was continuously maintained because the problem of receptor inactivation due to carbon deposition was solved by cleaning with carbon dioxide. It is believed that the reason that methane reached the maximum conversion rate earlier than carbon dioxide did was that the methane thermal decomposition reaction required less activation energy than the carbon gasification reaction. Furthermore, as carbon monoxide and hydrogen were generated by the dry reforming reaction, such as the methane and carbon dioxide gas uniform reaction of (6), in addition to the heterogeneous solid-gas reaction, the methane and carbon dioxide mixed-gas reforming showed a higher conversion rate than the reforming by supplying carbon dioxide or methane alone. Due to this reforming reaction, the concentrations of methane and carbon dioxide decreased, and those of hydrogen and carbon monoxide increased, as a result of microwave reforming.

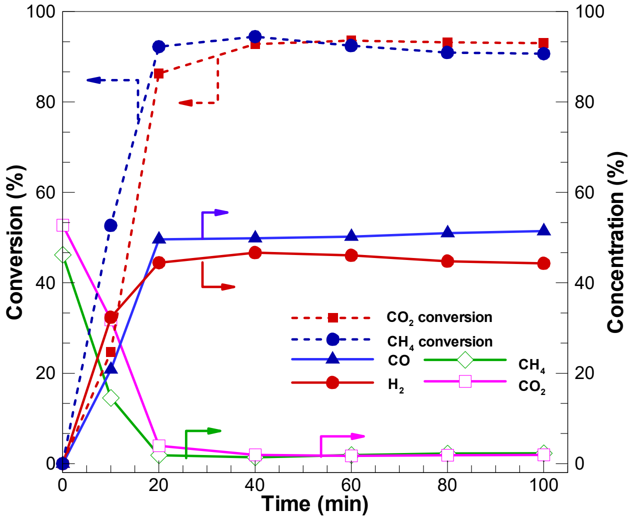


Fig. 4 Results of carbon dioxide and methane mixed-gas reforming at 900 °C

D. Effect of the Carbon Dioxide: Methane Ratio

To investigate the reforming characteristics according to the mixed-gas ratio, the results according to the composition ratios of carbon dioxide and methane are shown in Fig. 5.

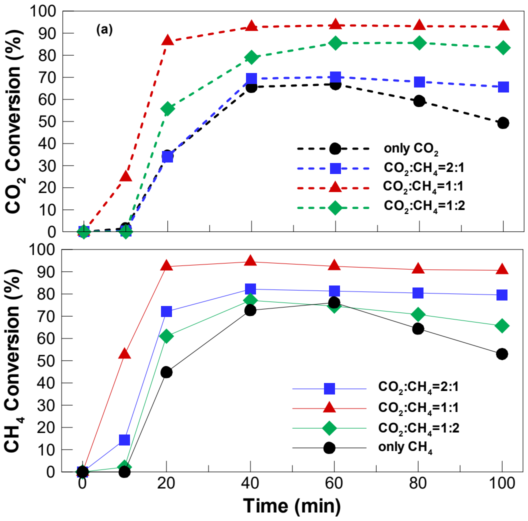
Figs. 5 (a) and (b) show the conversion rates and the average conversion rates of carbon dioxide and methane according to the gas composition ratios, respectively. When the CO_2:CH_4 ratio was 1:1, the conversion rates of carbon dioxide and methane remained constant at over 90% after 20 minutes. As can be seen in Fig. 5 (a), the methane conversion rate was higher than the carbon dioxide conversion rate when the CO_2:CH_4 ratio was 2:1. Fig. 5 (b) shows that the average conversion rate of methane was 79% and that of carbon dioxide was 61%. This was caused by the dry reforming reaction, and this was because the molecular bond energy of methane (439 kJ/mol) was lower than that of carbon dioxide (532 kJ/mol), and thus, methane decomposition occurred faster [16]. When the CO2:CH4 ratio was 1:2, the carbon conversion rate decreased because carbon was deposited through the methane thermal decomposition expressed in (4), and the carbon dioxide conversion rate increased because carbon dioxide gasification was caused by the deposited carbon ($C_{(CH4)}$), and carbon monoxide was generated.

Fig. 5 (c) shows the gas concentrations, heating values, and H_2:CO ratios. While the syngas produced from the dry reforming reaction between CH_4 and CO_2 had a H_2:CO molar ratio of almost 1, a similar H_2:CO ratio of 0.99 was generated when the CO_2:CH_4 ratio was 1:1 [17]. The heating values were 11.70, 12.90, and 15.14 MJ/m^3, respectively, when the CO_2:CH_4 ratios were 2:1, 1:1, and 1:2.

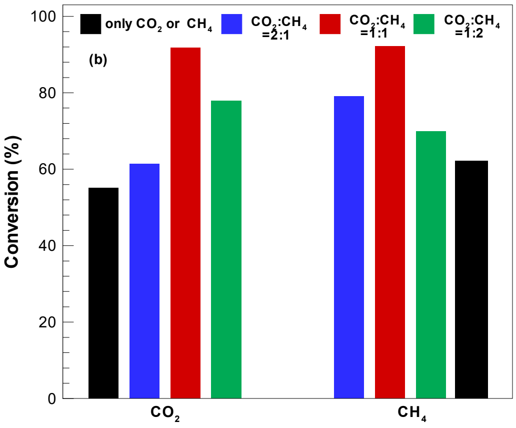
E. Effect of the Carbon Dioxide: Steam Ratio

To investigate the effect of steam on the carbon dioxide reforming characteristics, an experiment was performed using the CO_2:steam ratios of 1:1 and 1:2, and the results are shown in Fig. 6.

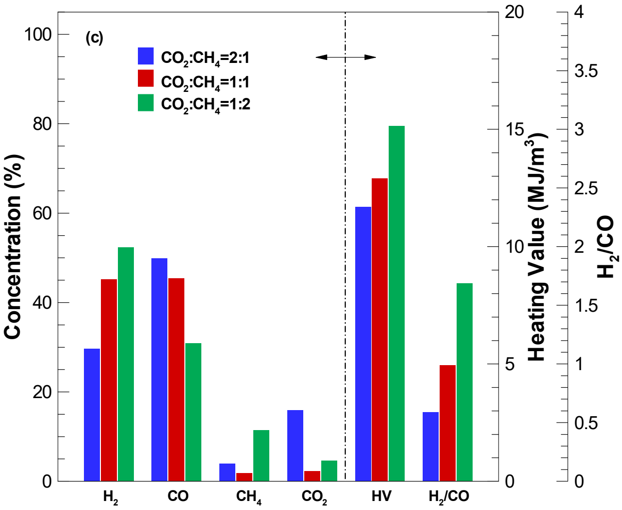
In Fig. 6 (a), when steam was supplied, the carbon dioxide conversion rate reached the maximum value at 40 minutes and then decreased, and the average conversion rate of carbon dioxide was less than 60%. As the carbon component of sludge char, a MR, was reduced by the steam gasification reaction ($C + H_2O \leftrightarrow CO + H_2$) when moisture was supplied, the carbon dioxide conversion rate slightly decreased over time. In Fig. 6 (b), as the supply of steam increased, the hydrogen gas increased and the carbon monoxide gas decreased. This is because the carbon monoxide and steam generated by the carbon dioxide reforming process produced carbon dioxide and hydrogen through the reaction shown in (7) [18]. The reason for the decrease of carbon dioxide gas was that the influence of the supplied amount was greater than that of the produced amount. The remaining amount of carbon dioxide gas was believed to be the amount of the supplied carbon dioxide gas left after the reaction.



(a) Conversion rate of carbon dioxide and methane



(b) Average conversion rate of carbon dioxide and methane



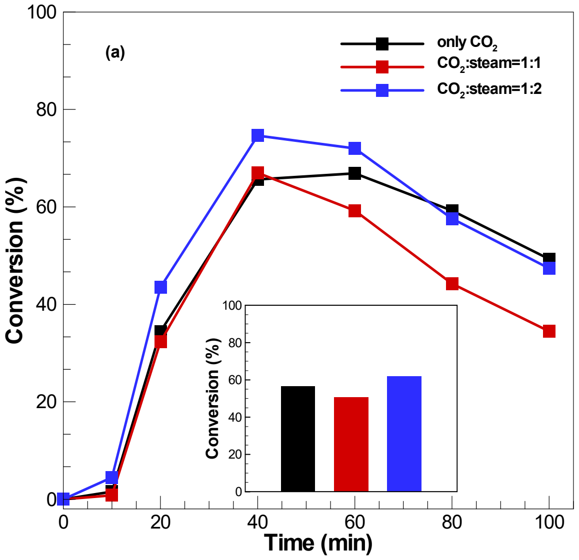
(c) Reformed-gas concentration, heating values, and H_2:CO ratios

Fig. 5 Results according to the ratios of carbon dioxide and methane

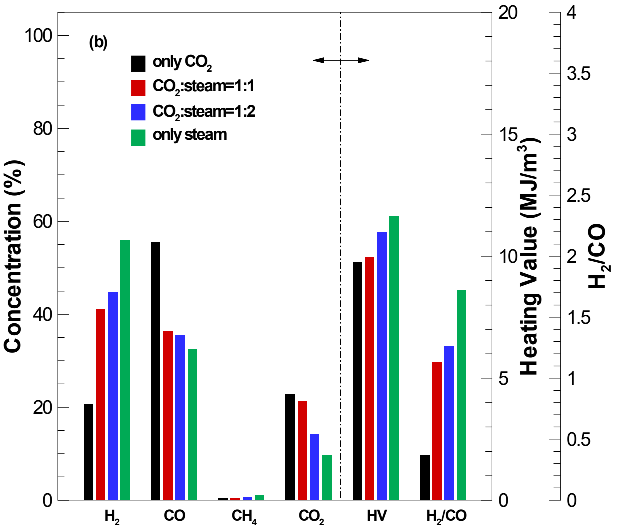
Due to the increase of hydrogen gas, the heating value and H_2:CO ratio increased along with the CO_2: steam ratio. The H_2:CO ratios were 0.37, 1.13, 1.26, and 1.72 for carbon dioxide only, CO_2: steam=1:1, CO2: steam=1:2, and steam only.

F. Effect of Catalysts

The activity of the carbon-based catalyst used in the carbon dioxide/methane microwave reforming is determined by the internal structure and surface characteristics of the MR. As the volume of the micropores of the receptor must be ensured to obtain high conversion rates of carbon dioxide and methane [10], [19], [20], the commercial activated carbon with excellent micropores was used as a carrier instead of sludge char, and Ni and Fe catalysts were prepared using the impregnation method, and the characteristics of the catalysts were investigated.



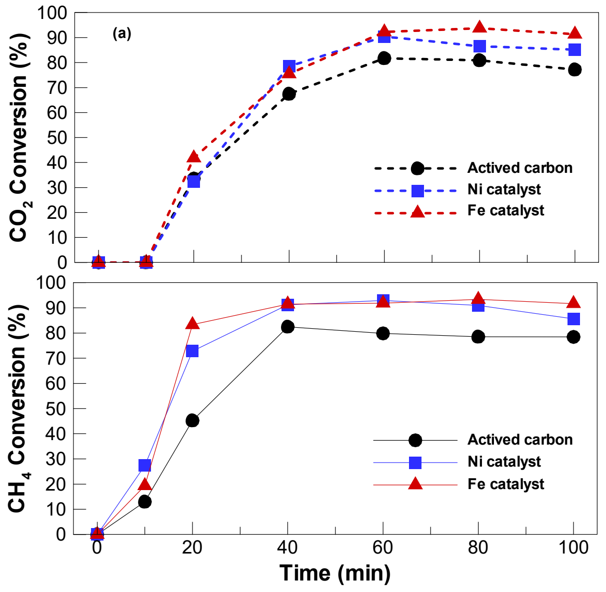
(a) Conversion rate of carbon dioxide and methane



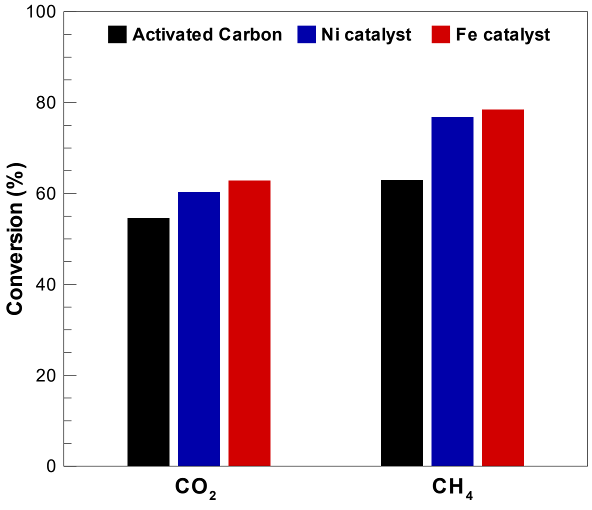
(b) Reformed-gas concentration, heating values, and H_2:CO ratios

Fig. 6 Results according to the carbon dioxide/steam ratio

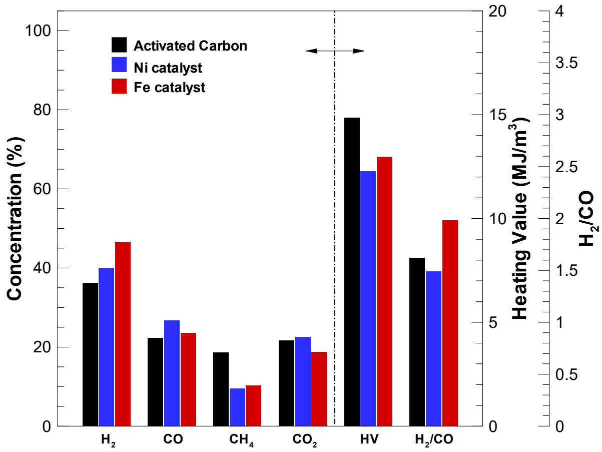
Fig. 7 shows the characteristics of the reforming using the catalysts. Fig. 7 (a) shows higher conversion rates of carbon dioxide and methane when the Ni and Fe catalysts were used than when only activated carbon was used. The Ni and Fe catalysts showed similar tendencies. In Fig. 7 (b), the average conversion rates of carbon dioxide and methane were 60.3 and 76.8% for Ni and 62.8 and 78.4% for Fe, indicating similar results. This is because, unlike sludge char, the commercial activated carbon can maintain the microporosity without the collapse of the solid internal structure. The gas concentrations in Fig. 7 (c) show that the Ni catalyst had a relatively higher amount of carbon monoxide gas, while the Fe catalyst had a relatively higher amount of hydrogen gas, and that the H_2:CO ratio of the Fe catalyst was 1.92, which was higher than that of the Ni catalyst (1.49).



(a) Conversion rate of carbon dioxide and methane



(b) Average conversion rate of carbon dioxide and methane



(c) Reformed-gas concentration, heating values, and H_2:CO ratios

Fig. 7 Results according to the catalyst type

IV. CONCLUSION

Carbon dioxide and methane are the major components of biomass pyrolysis/gasification gas and biogas, and this study was conducted on microwave reforming to convert them into high-quality energy sources.

In the case of the microwave reforming of carbon dioxide or methane using sludge char, the carbon dioxide produced carbon monoxide through carbon gasification, while the methane produced hydrogen and carbon through thermal decomposition, and the produced carbon was deposited on the MR, which reduced the conversion rate.

In the case of the microwave modification of the mixed gas of carbon dioxide and methane, the deposited carbon was cleaned through carbon gasification due to carbon dioxide, and thus, the conversion rate and the generated gas were kept stable.

When the carbon dioxide:methane ratio was 1:1, the conversion rate remained stable at over 90%. When the ratio changed, the conversion rate slightly decreased but remained stable, without a sharp decline.

The results according to the carbon dioxide:steam ratio showed that an increase in the supply of steam could not affect the carbon dioxide conversion rate but increased the heaving value and H_2:CO ratio.

The Ni and Fe catalysts using commercial activated carbon as a carrier showed higher conversion rates than commercial activated carbon only used as the MR. The Ni catalyst produced more carbon monoxide, while the Fe catalyst produced more hydrogen.

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