

Enhancement of Water Resistance by Methanation Catalysts with LDHs Structure

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ABSTRACT

In order to enhance the efficiency of the synthetic methane production process, the authors developed an innovative catalyst that converts synthesis gas (syngas) obtained from the co-electrolysis reaction of water vapor and CO₂ into methane, and studied the allowable proportion of water vapor in the source gas, i.e., water resistance. Evaluation of the activity using the continuous-flow method showed that NiAl-LDHs utilizing layered double hydroxides exhibited higher catalytic reactivity for the generation of methane from syngas consisting of CO and H₂ at 0.1 MPa and 300°C. In addition, the authors experimented with the methane conversion reaction of syngas containing water vapor, which is an issue in the synthetic methane production process, when combined with a co-electrolysis reactor. The results showed that when up to 20% water vapor was present, CO conversion and CH₄ selectivity were equivalent to cases without water vapor, and the catalyst exhibited enhanced performance compared to the conventional metal-supported catalysts.

1. Introduction

The conversion of carbon dioxide (CO₂) into valuable chemicals continues to attract much attention in terms of supporting the separation, capture, and effective utilization of carbon dioxide⁽¹⁾. Among the approaches tried, the production of synthetic methane as a gas fuel is attracting attention in Japan and abroad. In Japan, the Ministry of Economy, Trade and Industry is leading a methanation project, while gas companies and automobile-related companies have also undertaken efforts⁽²⁾.

There are three main methods of synthetic methane generation: one method produces methane from biogas-derived CO₂, and the other two are Power-to-Gas (P2G) methane production (methanation) technologies. The first P2G process is the Sabatier reaction, shown in Fig. 1(a), which uses as feedstock CO₂ recovered from the atmosphere or factory exhaust gases and hydrogen (H₂) obtained by electrolyzing room-temperature water to produce methane in a catalytic thermochemical reaction. The second process, called innovative methanation, is shown in Fig. 1(b). Here, a mixture of synthesis gas (syngas), i.e., carbon monoxide (CO) and H₂, is obtained by co-electrolysis, which is a simultaneous electrolytic reaction of CO₂ and higher-temperature water vapor (H₂O). Then, using this syngas as feedstock, methane is produced by a catalytic

thermochemical reaction⁽³⁾. In this study, the authors investigate innovative methanation, which has the potential to produce methane very efficiently while consuming little energy outside of the reaction pathway⁽⁴⁾.

As a concrete example of innovative methanation, the system shown within the dashed lines of Fig. 2 has been investigated⁽⁴⁾. To generate the end product methane, the system consists of a co-electrolysis reactor with stacked solid oxide electrolysis cells (SOEC), which are expected to provide higher electrolysis efficiency, upstream and a

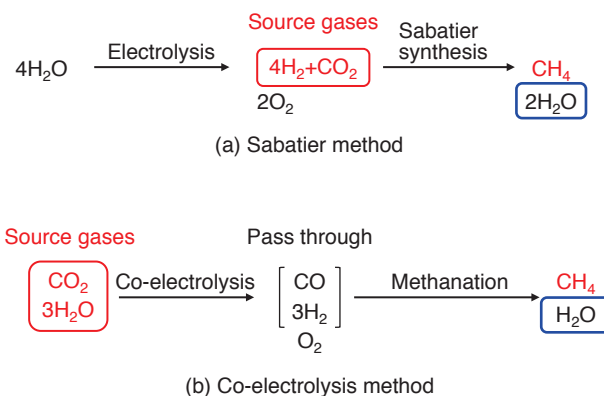


Fig. 1 Comparison of methane synthesis processes

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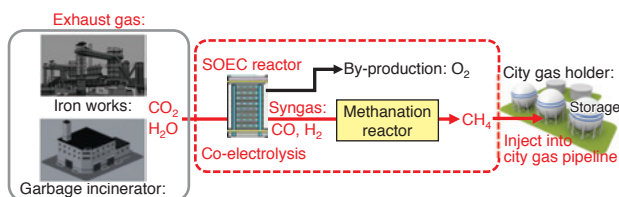


Fig. 2 Co-electrolysis methanation system via syngas

thermochemical reactor (methanation reactor) downstream to effectively utilize the heat generated by the methanation process, thereby converting the syngas obtained from the co-electrolysis reactor to methane.

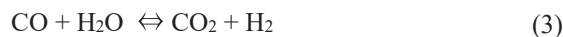
2. Achieving Higher Methane Production Efficiency

2.1. Synthetic Methane Production Process

In a co-electrolysis reactor, the reactions in Eqs. (1) and (2) occur simultaneously at the fuel electrode to generate CO, H₂, and oxygen ions (O²⁻) from the source gas and electrons (e⁻).



In addition, the water gas shift reaction shown in Eq. (3) occurs in parallel as a side reaction.



At the oxygen electrode, oxygen (O₂) is generated from O²⁻ as shown in Eq. (4).



During methanation using syngas as feedstock, 3 moles of H₂ are needed for 1 mole of CO, as shown in Eq. (5). The CO:H₂ ratio adjustment should ideally be achieved by controlling the electrolytic reaction in the co-electrolysis reactor and the flow rates of CO₂ and H₂O at the inlet.



2.2. Methanation Operating Challenges

The co-electrolysis reactor consists of multiple stacked solid oxide electrolysis cells, and when the source gas passes through the stacked cells, gas that comes in contact with the cells is converted to CO and H₂ through electrolytic reaction. In other words, source gas that does not come in contact with cells does not react and is discharged out of the reactor as unreacted gas. To help prevent cell damage due to over-voltage occurring, for example, when the source gas is depleted,

5% to 20% of the source gases (CO₂ and H₂O) in use are often passed through during the syngas generation process. Therefore, gas that has not been passed through and converted also remains in the syngas as an unreacted residual component⁽⁵⁾⁻⁽⁷⁾. Water vapor, the H₂O in the above residual component, is usually separated and removed before it gets to the methanation reactor because it is a factor that reduces catalytic activity. In the innovative methanation process as studied in the past, the proportion of water vapor in the generated gas after the co-electrolysis reactor was assumed to be 16% to 19%, and a drain was installed before the methanation reactor to cool, liquefy, and separate the water vapor, thereby reducing the proportion to 5% to 6%, after which the gas was fed into the methanation reactor⁽⁸⁾. The energy consumption associated with separation was estimated to be up to 58 kW, accounting for several percentage points of system energy efficiency. This study sought to develop a reliable water-resistant catalyst suitable for innovative methanation systems, thereby reducing the energy needed to separate the water vapor in the residual component, as well as reducing the overall process size and cost.

3. Catalyst Development Goals

There are three performance requirements for methanation catalysts, and two of the three concern the reaction shown in Eq. (5). The first is conversion, which indicates the proportion of the source gas CO that is converted to methane and other substances. The second is selectivity, which indicates the proportion of the converted material that is converted to the target product, methane. The CO conversion and CH₄ selectivity should be higher because the generated synthetic methane is to be used again as gas fuel. In addition, in methanation, H₂O is present around the catalyst as a byproduct of methanation, as shown in Eq. (5), as well as in the unreacted portion from the co-electrolysis reactor at the earlier step, and the CO obtained from the co-electrolysis reactor is converted back to CO₂ by the water gas shift reaction in Eq. (3). As a result, CO₂ entering the process is emitted from the process without being converted to valuable chemicals. Therefore, the third performance requirement for the catalyst in this study was that the catalyst should have a higher tolerance to water vapor contamination (water resistance), so that the water gas shift reaction does not occur even if water vapor is contained.

In summary, the following three performance requirements should be met.

- (1) Higher CO conversion
- (2) Higher CH₄ selectivity
- (3) Higher tolerance to water vapor contamination (water resistance)

In conventional methanation catalysts, Ni-based catalysts prepared by the coprecipitation method or impregnation method are widely used because of

their lower cost, higher activity, and higher methane selectivity⁽⁹⁾. The activity of catalysts prepared by the impregnation method usually depends on the basic properties of the carrier and on the preparation method. Various carriers such as SiO_2 , ZrO_2 , $\gamma\text{Al}_2\text{O}_3$, and CeO_2 have been studied⁽¹⁰⁾. However, the porous structure of these carriers makes them susceptible to H_2O adsorption, which covers the active sites of the catalyst and reduces its activity. Therefore, H_2O needs to be separated and removed upstream of the methanation reactor by a drain or other means. In this study, the authors focused on the structure of layered double hydroxides (LDHs), usually called hydrotalcites, which already contain H_2O . LDHs have a two-dimensional layered structure, as shown in Fig. 3⁽¹¹⁾, and it is well known that there is a great diversity of structures, including the metal cation ratios that can be prepared, various interactions of the layers composed between host and guest, and higher specific surface area⁽¹²⁾. Furthermore, since each layer of LDHs contains H_2O molecules and the H_2O adsorption sites are already buried, the catalyst has water repellency; that is, there is no adsorption when new water vapor is introduced, which is considered to be an advantage over conventional supported catalysts. In addition, because the structure of the LDHs results in much more highly dispersed Ni nanoparticles containing more reducible Ni species (Ni^{2+} to Ni^0) than conventional $\text{Ni}-\gamma\text{Al}_2\text{O}_3$ catalysts (where a small amount of nickel ions support the formation of mixed oxides with alumina in the form of Ni^{3+})⁽¹³⁾, the authors believed this would increase the number of reaction sites and increase CO_2 conversion and CH_4 selectivity. Therefore, LDHs are expected to be applied to hydrogen generation reactions and photoelectrochemical water oxidation processes and as functional catalysts^{(14), (15)}.

In this study, the LDHs catalyst developed by the authors, which uses Ni as the active metal, is compared to catalysts prepared by conventional coprecipitation and impregnation methods, and the performance and characteristics of the LDHs catalyst and its effect on the system are described.

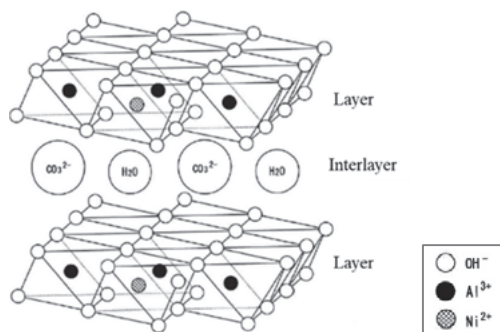


Fig. 3 LDHs structure model

4. Experimental Method

4.1. Catalyst Preparation

4.1.1. Preparation of Ni coprecipitation catalyst

A Ni-based coprecipitation catalyst was prepared using the following procedure. $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was dissolved in distilled water, and the resulting solution was dropped into a beaker together with Na_2CO_3 solution (1 mol/L, room temperature) at a slow rate of 2 mL/min. At this time, the pH value of the mixture was maintained at 8.5 at 80°C. The precipitate was then allowed to stand at room temperature for 24 hours, followed by filtration and washing to completely remove residual Na_2CO_3 from the precipitate. The precipitate was then dried at 60°C in air for 12 hours. Finally, it was heated at 2°C/min and maintained at 550°C for 4 hours for calcination.

4.1.2. Preparation of Ni supported catalyst

The catalyst in which Ni was supported by carriers with the impregnation method was prepared using the following procedure. Commercially available carriers (SiO_2 , $\gamma\text{Al}_2\text{O}_3$, ZrO_2 , CeO_2) were calcinated at 550°C for 4 hours to remove surface impurities, and Ni was introduced into the prepared carriers by the wet impregnation method. $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was dissolved in deionized water and impregnated uniformly onto the carrier. Each catalyst was prepared so that the weight of Ni was 20 wt% of the total. The resulting mixture was then exposed to a vacuum at room temperature for 1 hour and then dried at 60°C in air for 12 hours. Finally, it was heated at 2°C/min and maintained at 550°C for 4 hours for calcination.

4.1.3. Preparation of NiAl-LDHs

To achieve higher dispersion of Ni nanoparticles and increase the reaction sites, NiAl-LDHs were synthesized by a hydrothermal method using urea. First, 2.18 g of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 0.94 g of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were dissolved together in 60 ml of deionized water. Separately, 6 g of urea was dissolved in 60 ml of deionized water. Each solution was stirred at room temperature for 30 minutes at a stirring speed of 300 rpm, and then the two solutions were mixed and stirred for another 5 minutes. The mixture was then sealed in two 100 ml Teflon-coated stainless-steel autoclaves, heated at 3°C/min, and maintained at 120°C for 12 hours. The mixture was allowed to stand without stirring during this period. Then, the prepared precipitates were washed with 200 ml of deionized water each time by centrifugation until the pH of the solution was neutral. The resulting product was dried at 60°C in air for 12 hours. Finally, to help prevent the dried catalyst from losing its structure due to bonding with oxygen, it was heated at 2°C/min in a nitrogen atmosphere and calcined at 500°C for 4 hours.

4.2. Catalyst Characterization

4.2.1. Specific surface area

The specific surface area of the prepared catalysts

was measured by N_2 physisorption using a Micromeritics 3Flex specific surface area and microporous gas adsorption analyzer. Prior to measuring, all samples were pretreated at 250°C for 10 hours under vacuum to remove surface contaminants.

4.2.2. Structural analysis by X-ray diffraction

To confirm the crystal structure of the prepared catalysts, X-ray diffraction (XRD) measurements were taken using a Rigaku RINT 2400 system X-ray diffractometer.

4.3. Evaluation of Catalyst Activity

Catalytic activity was evaluated by the continuous-flow method in a fixed-bed reactor with an inner diameter of 8 mm, as shown in Fig. 4. The catalyst was loaded into the reaction tube according to the following procedure. First, an appropriate amount of quartz wool was put in, then a mixture of 2 g of quartz sand and 0.25 g of catalyst prepared to a particle size of 400 to 850 μm was poured in. Then, 2 g of glass beads were added to preheat the gas during the reaction, and finally quartz wool was put on the top surface.

The catalyst was reduced with H_2 at 40 ml/min for 8 hours at 400°C prior to the reaction. After maintaining at 300°C for 8 hours at 0.1 MPa, 20 mL/min, $H_2/CO = 3.0$, the activity of each catalyst was measured.

The gases generated were detected by three on-line gas chromatographs equipped with a Shimadzu Corporation GC-2014AT thermal conductivity detector (TCD) and GC-2014AF flame ionization detector (FID). The TCD measures CO_2 upstream and H_2 downstream, respectively, while the FID measures CO and hydrocarbons.

CO conversion was calculated based on Eq. (6), CH_4 and CO_2 selectivity on Eqs. (7) and (8), and CH_4 yield on Eq. (9). Ar in Eq. (6) is used as an internal standard substance. Here, Eq. (6) is the proportion of CO converted to valuable products, Eq. (7) is the proportion of CH_4 to total converted hydrocarbons and CO_2 , and Eq. (8) is the proportion of CO_2 . Equation (9) shows the proportion of the reactants from which the target product CH_4 was obtained.

$$CO \text{ conversion } [\%] = \frac{Area(\frac{CO}{Ar})^{in} - Area(\frac{CO}{Ar})^{out}}{Area(\frac{CO}{Ar})^{in}} \times 100 \quad (6)$$

$$CH_4 \text{ selectivity } [\%] = \frac{n(CH_4)}{n(\text{Total hydrocarbon} + CO_2)} \times 100 \quad (7)$$

$$CO_2 \text{ selectivity } [\%] = \frac{n(CO_2)}{n(\text{Total hydrocarbon} + CO_2)} \times 100 \quad (8)$$

$$CH_4 \text{ yield } [\%] = CO \text{ conversion} \times CH_4 \text{ selectivity} \times 100 \quad (9)$$

$n: [C\text{-mol/h}]$

5. Experiment Results and Discussion

5.1. Specific Surface Area, XRD Measurement Results

Results for the specific surface area of the prepared catalysts are shown in Fig. 5. The specific surface area of NiAl-LDHs was lower than that of 20 wt% Ni/SiO₂, but was comparable or higher than that of the other carriers.

XRD results for the prepared NiAl-LDHs are shown in Fig. 6. The positions of the peaks at 37°, 43°, and 62° are attributed to the Ni oxide NiO on the LDHs, and the clear presence of the peaks indicates that Ni-based LDHs have been prepared.

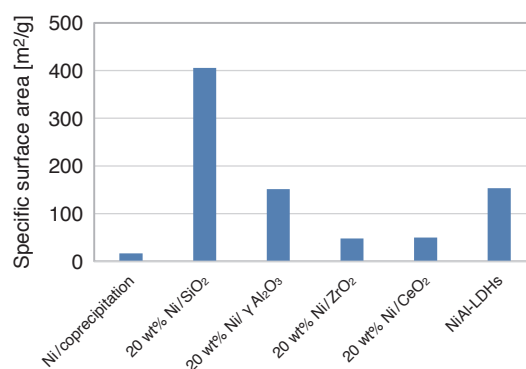


Fig. 5 Comparison of specific surface area

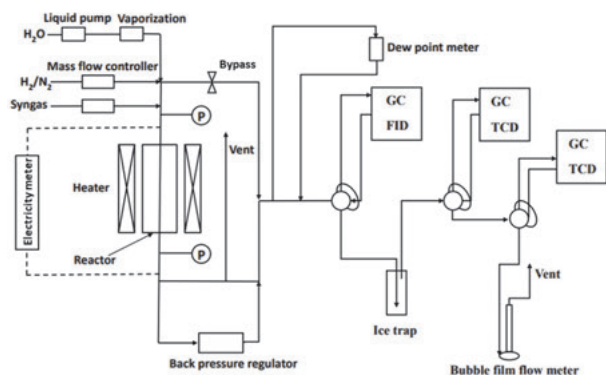


Fig. 4 Reaction devices diagram of fix-bed reactor

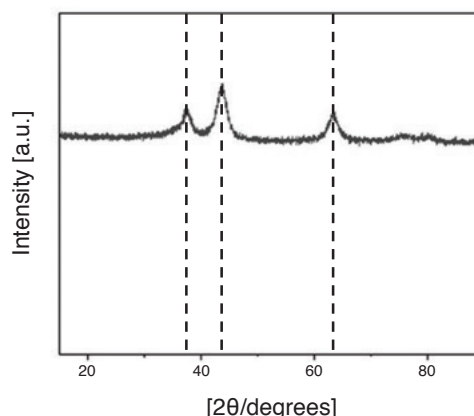


Fig. 6 XRD measurement result of NiAl-LDHs

Table 1 Results of methane synthesis processes

Catalysts	CO conversion [%]	Selectivity [%]						CH ₄ yield [%]
		CO ₂	CH ₄	C ₂	C ₃	C ₄	C ₅₊	
Ni-coprecipitation	55.9	0	97	2.8	0.3	0	0	54.2
20 wt% Ni/SiO ₂	99.5	2.9	93.7	2.9	0.4	0	0	93.2
20 wt% Ni/ γ -Al ₂ O ₃	92	0.4	87.9	7.4	3.3	0.8	0.2	80.9
20 wt% Ni/ZrO ₂	82.2	0.1	89.7	7.1	2.5	0.4	0.1	73.7
20 wt% Ni/CeO ₂	47	7	50.6	15.1	14.1	5.7	7.5	23.8
NiAl-LDHs	100	0.2	99.8	0	0	0	0	99.8

5.2. Catalytic Activity

A comparison of CO conversion, selectivity, and yield of NiAl-LDHs with catalysts with different carriers using Ni is shown in Table 1. First, other than NiAl-LDHs, 20 wt% Ni/SiO₂ performed more effectively than other catalysts, with 99.5% CO conversion, 93.7% CH₄ selectivity, and 93.2% CH₄ yield. However, CO₂ was also generated in addition to CH₄. On the other hand, NiAl-LDHs achieved 100% CO conversion, 99.8% CH₄ selectivity, and the highest yield of 99.8%. NiAl-LDHs showed greater activity than 20 wt% Ni/SiO₂ despite having lower specific surface area, indicating that it is a reliable catalyst for methanation.

5.3. Reaction Characteristics

Syngas methanation characteristics were evaluated for NiAl-LDHs at different temperatures. The reaction conditions other than temperature were the same as in the catalytic activity experiment. The results for different temperatures are shown in Fig. 7. Both CO conversion and CH₄ selectivity were lower at temperatures below 250°C, while CO conversion reached 100% at 275°C, and CH₄ selectivity reached 100% at 300°C. In general, methanation reactions often show higher reactivity at temperatures from 250°C to 500°C^{(10), (12)} and pressures from 0.1 to several MPa for Ni-based catalysts, but lower temperatures and higher pressures are advantageous for exothermic reactions. With the NiAl-LDHs developed in this study, the reaction

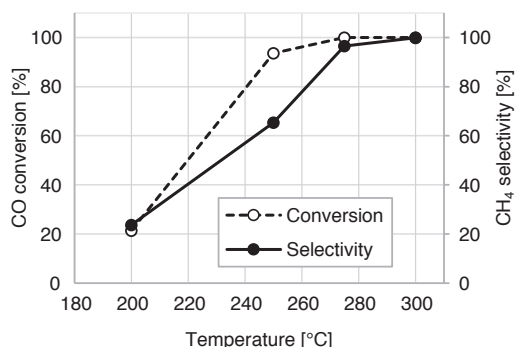


Fig. 7 Reaction characteristics of NiAl-LDHs

temperature is comparable to those values, while the above results were obtained at a reaction pressure of 0.1 MPa. This confirms the advantageous properties of higher activity at lower temperatures and lower pressures and the ability to reduce pressurization energy.

5.4. Impact When There Is Water Contamination of the Source Gas

Under the same conditions as in the catalytic activity experiment, the proportion of water vapor supplied in the syngas was calculated based on Eq. (10).

$$\text{Water vapor proportion [\%]} = \frac{F_{H_2O}(\text{gas})}{(F_{H_2O}(\text{gas}) + F_{\text{syngas}})} \quad (10)$$

As shown in Fig. 8, the CO conversion of the benchmark 20 wt% Ni/SiO₂ decreased as the proportion of water vapor increased, and the CO conversion decreased to 50% when the water vapor content was 20%. On the other hand, the CO conversion of NiAl-LDHs remained close to 100% even when the water vapor proportion increased from 20% to 65%. As shown in Fig. 9, at water vapor proportions up to 20%, the CH₄ selectivity of LDHs was equivalent to that when no water vapor contamination was present, and started to gradually decrease after a water vapor proportion

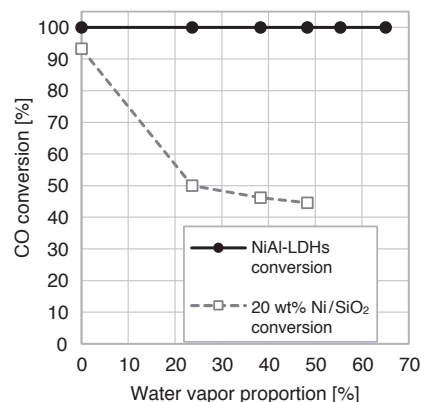


Fig. 8 Conversion of each catalyst with water vapor

of 20%. When the water vapor contamination reached 65%, CH₄ selectivity was 55%. CO₂ selectivity increased as CH₄ selectivity decreased, suggesting that a water gas shift occurred when the NiAl-LDHs exceeded the allowable moisture proportion. These results indicate that the catalyst is more water resistant than conventional supported catalysts and that the allowable proportion of water vapor in the source gas is 20%.

The use of the NiAl-LDHs developed in this study could help to eliminate the need to remove residual water vapor from the source gas, potentially reducing energy consumption by up to 58 kW.

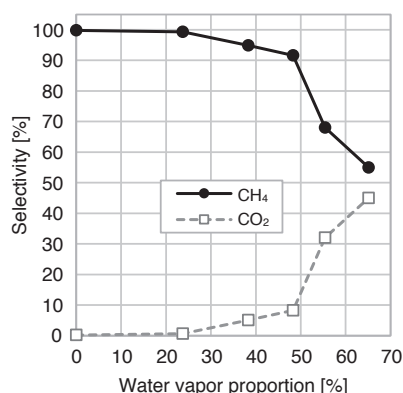


Fig. 9 Selectivity of NiAl-LDHs when mixed with water vapor

6. Conclusion

NiAl-LDHs were developed as a thermo-chemical catalyst to generate methane from syngas to increase the efficiency of the synthetic methane production process, and the following results were obtained.

- (1) NiAl-LDHs exhibited 100% CO conversion and almost 100% CH₄ selectivity at 0.1 MPa and 300°C. Among the catalysts used for comparison, 20 wt% Ni/SiO₂ had about the same CO conversion as NiAl-LDHs, but the NiAl-LDHs had higher CH₄ selectivity and CH₄ yield than even those of 20 wt% Ni/SiO₂.
- (2) Comparing NiAl-LDHs and 20 wt% Ni/SiO₂, a Ni based catalyst used for comparison, in terms of the allowable proportion of water vapor in the source gas showed that the CO conversion and CH₄ selectivity of 20 wt% Ni/SiO₂ decreased immediately when there was water vapor contamination, whereas NiAl-LDHs maintained its activity without decrease at up to 20% water vapor contamination, showing reliable water resistance.

Based on the above, the use of the NiAl-LDHs developed in this study is expected to contribute to the building of more efficient methane production processes in the future by reducing energy consumption in the dehydration process and downsizing it upstream of the

methanation reactor, thereby increasing process efficiency and reducing process costs.

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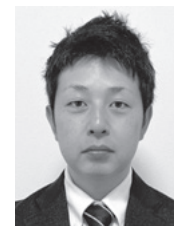
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