

# Purification of the gas after pyrolysis in coupled plasma-catalytic system

Michał Młotek\*, Bogdan Ulejczyk, Joanna Woroszył, Irmina Walerczak, Krzysztof Krawczyk

Warsaw University of Technology, 3 Noakowskiego, 00-664 Warsaw, Poland

\*Corresponding author: e-mail: mmlotek@ch.pw.edu.pl

Gliding discharge and coupled plasma-catalytic system were used for toluene conversion in a gas composition such as the one obtained during pyrolysis of biomass. The chosen catalyst was G-0117, which is an industrial catalyst for methane conversion manufactured by INS Pulawy (Poland). The effects of discharge power, initial concentration of toluene, gas flow rate and the presence of the bed of the G-0117 catalyst on the conversion of  $C_7H_8$ , a model tars compounds were investigated. Conversion of toluene increases with discharge power and the highest one was noted in the coupled plasma-catalytic system. It was higher than that in the homogeneous system of gliding discharge. When applying a reactor with reduced G-0117 and CO (0.15 mol%),  $CO_2$  (0.15 mol%),  $H_2$  (0.30 mol%),  $N_2$  (0.40 mol%), 4000 ppm of toluene and gas flow rate of 1.5  $Nm^3/h$ , the conversion of toluene was higher than 99%. In the coupled plasma-catalytic system with G-0117 methanation of carbon oxides was observed.

**Keywords:** gliding discharge, non-equilibrium plasma, plasma-catalytic system, tar decomposition.

## INTRODUCTION

Due to climate change and environmental hazards, it has become necessary to use renewable energy sources. One of them is biomass, which can be converted into a gaseous fuel<sup>1</sup>. Pyrolysis and gasification are two most common processes which convert chemical energy contained in biomass into useful energy<sup>2, 3</sup>. As a result of these processes carbon oxides and hydrogen are formed, which can be used as fuel to drive engines and turbines and for chemical synthesis. In these processes small amounts of tars are formed, which may deposit on the engine and turbine walls, what leads to lower efficiency of the process and may cause damage to these parts<sup>4</sup>. The amount of tars in gas used as a feedstock for engines, according to different sources, must be no higher than 50–100  $mg/Nm^3$  and 5  $mg/Nm^3$  for turbines<sup>5</sup>. Therefore, the gas obtained in pyrolysis and gasification requires further purification<sup>4</sup>. The tars are mainly the complex mixture of condensable hydrocarbons, which includes single or multiple ring aromatic compounds also with oxygen, containing hydrocarbons and complex polycyclic aromatic hydrocarbons<sup>6</sup>. Such a mixture is very difficult to analysis, so it has been accepted in the scientific community that toluene or naphthalene was chosen as a model compound<sup>7–11</sup>.

Many gas cleaning processes have been proposed for the removal of tars from biomass gasification gas, including thermal cracking<sup>12, 13</sup>, mechanical separation<sup>14, 15</sup> and catalytic reforming<sup>8–11</sup>. Thermal cracking process requires high temperatures ( $>800^\circ C$ ) to decompose tars, and causes a high energy cost. Removal of tars from syngas with a physical separation process will decrease the overall processing efficiency. Catalytic reforming can convert tars into valuable products at relatively lower temperatures ( $\sim 600^\circ C$ ) compared to thermal cracking process<sup>11</sup>. The best results were obtained in catalytic methods of gas purification, however because of unfavorable conditions of reactions, the catalysts were quickly deactivated<sup>16–19</sup>. Decomposition of tars was conducted on nickel based catalysts supported on CaO, MgO and calcined dolomite, ceria-zirconia and a nickel-mayenite catalyst<sup>20–22</sup>. However, deactivation of the catalysts by poisoning or carbon deposits has been a major disadvantage for industrial applications. Adsorption on activated carbon, filtration,

catalytic method and plasma techniques are the most studied methods for decomposition of tars.

Plasma of gliding discharge and a plasma-catalytic system has also been used for the conversion of toluene<sup>23, 24</sup>. In the coupled plasma-catalytic system high conversion of toluene was obtained<sup>25</sup>. It may suggest that the use of active catalysts coupled with plasma of gliding discharge can lead to a higher conversion of tars and the activity of the catalysts could be stable.

The purpose of this work was to investigate the effects of power of discharge, initial concentration of toluene and the presence of the reduced form of the G-0117 catalyst on the conversion of  $C_7H_8$ .

## EXPERIMENTAL

In the course of this work the conversion of toluene, a model tars compound, was studied. The study was conducted in homogeneous and heterogeneous plasma-catalytic systems with G-0117, an industrial catalyst for methane conversion synthesis, in the reduced form. Measurements were conducted for two gas flow rates 1.0  $Nm^3/h$  (2000 and 6000 ppm of toluene) and 1.5  $Nm^3/h$  (1000 and 2000 ppm of toluene) in a homogeneous system and in a heterogeneous coupled plasma-catalytic system with a flow rate 1.5  $Nm^3/h$  (2000 and 4000 ppm of toluene). The composition of the inlet stream was: CO (0.15 mol%),  $CO_2$  (0.15 mol%),  $H_2$  (0.30 mol%),  $N_2$  (0.40 mol%). The calorific value of this gas was c.a. 5.8  $MJ/m^3$ .

The conversion of toluene was investigated in a three-phase gliding discharge reactor under normal pressure. The reactor was made of a quartz–glass tube of 60 mm in diameter and was equipped with three converging duralumin electrodes. The gas inlet was located at the bottom, between the electrodes. The power supply consisted of ferroresonant transformers and a transistor inverter, which resulted in a poly-phase multi-electrode reactor system. The electric power supplied to the gliding reactor was in the range of 1–2 kW and was measured by an energy meter. The reactor and the power system were described previously<sup>26–28</sup>.

The gas temperature measured at the vessel axis over the upper ends of the electrodes reached 160–540°C.

It exhibits a dependence on the discharge power and presence of catalyst.

Two kinds of gas chromatographs were used for gas analysis: a Thermo-Scientific Trace 1300 instrument with a HP5 column and a Flame Ionized Detector (FID) to determine the toluene concentration and a mass spectrometer for other higher than C5 hydrocarbons analysis, and an Agilent 6890N instrument with a ShinCarbon column and the following detectors: TCD and FID for the following gases: CO<sub>2</sub>, CO, H<sub>2</sub>, N<sub>2</sub>, O<sub>2</sub>, CH<sub>4</sub> and C2 hydrocarbons analysis. The water vapor content was measured by an APAR moisture meter.

Toluene conversion was calculated using the equation:

$$x[\text{C}_7\text{H}_8] = \frac{W_0[\text{C}_7\text{H}_8] - W[\text{C}_7\text{H}_8]}{W_0[\text{C}_7\text{H}_8]}$$

where:  $x[\text{C}_7\text{H}_8]$  – overall conversion of toluene;  
 $W_0[\text{C}_7\text{H}_8]/\text{mol} \cdot \text{h}^{-1}$ ,  $W[\text{C}_7\text{H}_8]/\text{mol} \cdot \text{h}^{-1}$  – toluene flow rate at the inlet and outlet respectively.

The calorific value of the fresh and outlet gas was calculated from the formula:

$$W = \frac{\sum Q_{pi} \times a_i}{1000}$$

where:

$W$  – calorific value [MJ/m<sup>3</sup>]

$i$  – H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>,

$Q_{pi}$  – heat of combustion of the specific gas [kJ/m<sup>3</sup>]

$a_i$  – molar fraction of the specific gas

The X – ray diffraction (XRD) analysis was carried out with a Seifert 3003 diffractometer (GE Inspection Technologies) using CuK $\alpha$  radiation.

The specific surface area of the catalyst prior to and after the catalytic tests was determined using an ASAP2020 instrument (Micromeritics).

#### Preparation of the catalyst

For the experiments a bed of reduced G-0117 was used. The results of our previous studies were taken into account during the selection of the catalyst. To obtain catalyst particles suitable for our studies, the industrial catalyst was crushed and the fraction 1.6–3.15 mm was used. The catalyst was reduced for 4 hours at 400°C in hydrogen (20 NI/h) and cooled in this atmosphere. The catalyst particles were packed into the reactor in a single batch of 36 ml. The height of the bed was 1.5 mm. The specific surface area of G-0117 was 3.06 before and 3.46 m<sup>2</sup>/g after activity measurements.

The catalyst did not change during the activity measurements. Carbon deposits were not formed. The XRD analysis (Fig. 1) showed nickel as the main component of catalyst, it was not oxidized during experiments. NiO signals after measurements haven't been detected. Other signals originated from the support of the catalyst – mainly alumina (corundum) and hibonit (CaO(Al<sub>2</sub>O<sub>3</sub>)<sub>3</sub>).

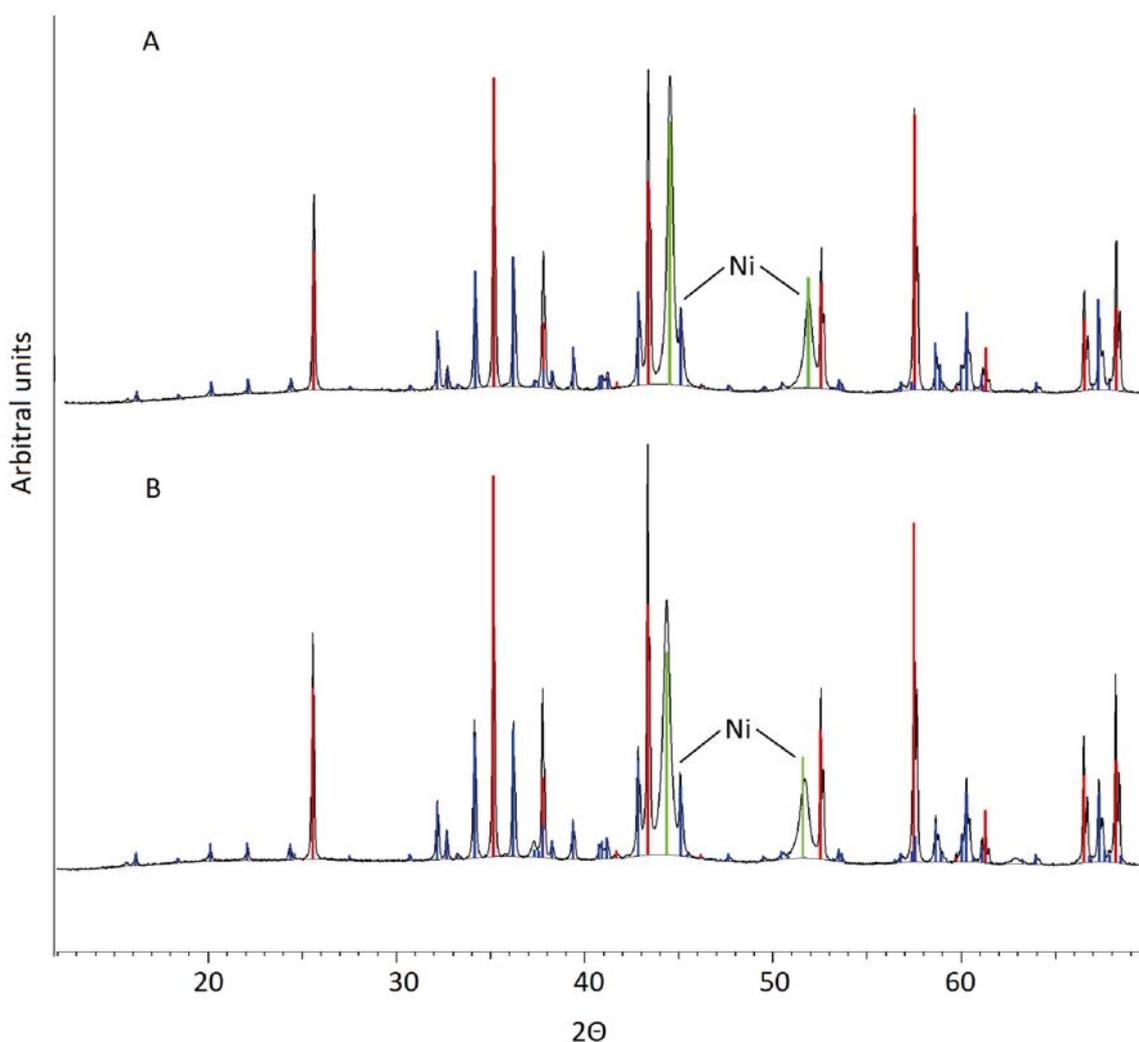
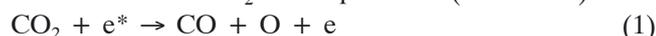


Figure 1. G-0117 catalyst structure before – A, and after measurements – B

## RESULTS AND DISCUSSION

The measurements of toluene decomposition were performed in a homogeneous (with gliding discharge plasma) and a coupled plasma-catalytic system (gliding discharge plasma and reduced G-0117 catalyst). The study was carried out in a stream containing carbon oxides, nitrogen and hydrogen for two gas flow rates 1.0 Nm<sup>3</sup>/h (2000 and 6000 ppm of toluene) and 1.5 Nm<sup>3</sup>/h (1000 and 2000 ppm of toluene) in a homogeneous system and in a heterogeneous coupled plasma-catalytic system with a flow rate 1.5 Nm<sup>3</sup>/h (2000 and 4000 ppm of toluene). The composition of the inlet gas was (in molar fractions): CO (0.15), CO<sub>2</sub> (0.15), H<sub>2</sub> (0.30), N<sub>2</sub> (0.40). In the gas before the measurement, oxygen was not found, and the humidity was approx. 2%. The obtained results are presented in Tables 1–3 and Figures 2 and 3.

In the homogeneous system the toluene conversion increases with the increase of initial concentration of C<sub>7</sub>H<sub>8</sub> and with increasing power (Table 1 and Fig. 2) when the gas flow rate is fixed at 1000 NI/h. At high concentration of toluene, locally the temperature may increase due to toluene oxidation. This increase the reaction rate of CO<sub>2</sub> decomposition<sup>7</sup> (reaction 1).



The more oxygen radicals and high temperature leads to increase the toluene decomposition reactions rates.

In the outlet gas the presence of the following components was observed: CO<sub>2</sub>, CO, H<sub>2</sub>, CH<sub>4</sub>, oxygen, C2–C3 hydrocarbons, H<sub>2</sub>O and trace amounts of C4

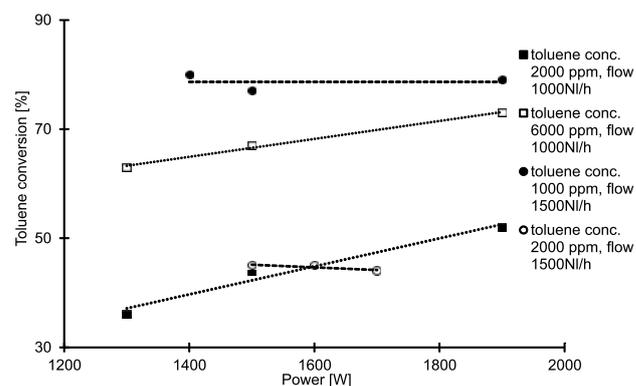


Figure 2. Toluene conversion in the homogeneous system

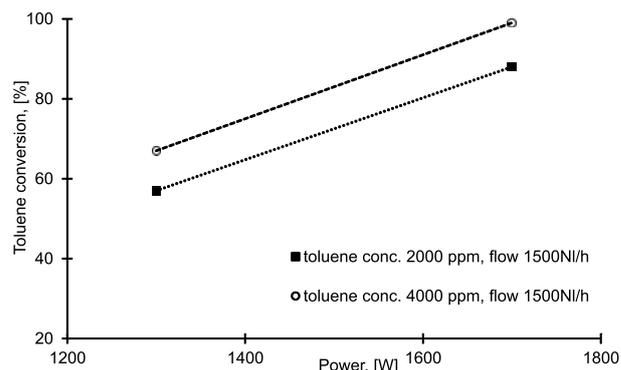


Figure 3. Toluene conversion in the plasma-catalytic system

Table 1. Results of toluene decomposition in the homogeneous system. Gas flow rate 1 Nm<sup>3</sup>/h

P [W]	Ed kJ/l	T <sub>R</sub> [°C]	H. [%]	X <sub>C<sub>7</sub>H<sub>8</sub></sub> [%]	Molar fraction									Calorific value [MJ/m <sup>3</sup> ]
					H <sub>2</sub>	O <sub>2</sub>	N <sub>2</sub>	CO	CO <sub>2</sub>	CH <sub>4</sub>	C <sub>2</sub> H <sub>2</sub>	C <sub>2</sub> H <sub>4</sub>	C <sub>2</sub> H <sub>6</sub>	
2000 ppm of toluene														
1300	4.7	200	68.6	36	0.313	0.017	0.322	0.201	0.132	0.010	0.004	0.001	0.001	7.35
1500	5.4	290	80.0	44	0.254	0.038	0.367	0.197	0.128	0.010	0.004	0.001	0.001	6.54
1900	6.8	345	84.5	52	0.299	0.030	0.319	0.219	0.115	0.011	0.004	0.001	0.002	7.51
6000 ppm of toluene														
1300	4.7	169.5	56.5	63	0.310	0.015	0.331	0.193	0.137	0.006	0.006	0.001	0.001	7.17
1500	5.4	257.4	61.6	67	0.262	0.031	0.369	0.193	0.130	0.006	0.006	0.002	0.001	6.61
1900	6.8	324.5	81.5	73	0.287	0.019	0.344	0.213	0.122	0.007	0.007	0.002	0.001	7.29

Table 2. Results of toluene decomposition in the homogeneous system. Gas flow rate 1.5 Nm<sup>3</sup>/h

P [W]	Ed kJ/l	T <sub>R</sub> [°C]	H. [%]	X <sub>C<sub>7</sub>H<sub>8</sub></sub> [%]	Molar fraction									Calorific value [MJ/m <sup>3</sup> ]
					H <sub>2</sub>	O <sub>2</sub>	N <sub>2</sub>	CO	CO <sub>2</sub>	CH <sub>4</sub>	C <sub>2</sub> H <sub>2</sub>	C <sub>2</sub> H <sub>4</sub>	C <sub>2</sub> H <sub>6</sub>	
1000 ppm of toluene														
1400	3.4	200	68.6	80	0.305	0.019	0.346	0.173	0.150	0.010	0.006	0.001	0.000	6.94
1500	3.6	290	80.0	77	0.278	0.028	0.367	0.175	0.143	0.010	0.007	0.001	0.000	6.68
1900	4.6	345	84.5	79	0.268	0.030	0.374	0.179	0.139	0.011	0.007	0.001	0.000	6.64
2000 ppm of toluene														
1500	3.4	139	51.0	45	0.215	0.067	0.465	0.115	0.127	0.007	0.002	0.001	0.001	4.76
1600	3.6	243	62.1	45	0.288	0.030	0.424	0.095	0.149	0.009	0.002	0.001	0.002	5.60
1700	4.6	296.6	69.8	44	0.280	0.038	0.457	0.057	0.155	0.009	0.002	0.001	0.002	5.02

Table 3. Results of toluene decomposition in the coupled plasma-catalytic system. Gas flow rate 1.5 Nm<sup>3</sup>/h

P [W]	Ed kJ/l	T <sub>R</sub> [°C]	H. [%]	X <sub>C<sub>7</sub>H<sub>8</sub></sub> [%]	Molar fraction									Calorific value [MJ/m <sup>3</sup> ]
					H <sub>2</sub>	O <sub>2</sub>	N <sub>2</sub>	CO	CO <sub>2</sub>	CH <sub>4</sub>	C <sub>2</sub> H <sub>2</sub>	C <sub>2</sub> H <sub>4</sub>	C <sub>2</sub> H <sub>6</sub>	
2000 ppm of toluene														
1300	3.1	240	79	57	0.289	0.011	0.331	0.212	0.137	0.011	0.001	0.000	0.000	6.91
1700	4.1	540	30	88	0.185	0.026	0.415	0.318	0.172	0.055	0.004	0.000	0.000	8.83
4000 ppm of toluene														
1300	3.1	240	64	67	0.236	0.0	0.353	0.150	0.188	0.061	0.000	0.000	0.000	7.37
1700	4.1	540	100	99	0.165	0.019	0.414	0.120	0.183	0.072	0.004	0.000	0.000	6.74

hydrocarbons. Generally, the molar fraction of the main compounds was similar for both initial concentrations of toluene (Table 1). The methane content for the initial concentration of toluene equal to 2000 ppm was higher than that for 6000 ppm. It was 0.010–0.011 and 0.006–0.007 for 2000 and 6000 ppm, respectively. In the outlet gas the acetylene content was about 50% higher for the higher toluene content. For both initial concentrations of toluene the molar fractions of ethylene and ethane were the same. The concentration of hydrocarbons above C3 and C4 does not exceed 20 ppm.

For the gas flow rate of 1.5 Nm<sup>3</sup>/h and the initial concentration of toluene of 2000 ppm the conversion of C<sub>7</sub>H<sub>8</sub> did not depend on the power (Fig. 2). It can be result of short residence time and consumption of the excited electrons generated in plasma on other processes i.e carbon dioxide dissociation and formation of oxygen radicals. However high toluene conversion at concentration 1000 ppm was the effect of reaction of oxygen radicals with toluene. Oxygen radicals may also recombine with each other to generate oxygen molecule. The highest toluene conversion (80%) was noted for the initial concentration of toluene of 1000 ppm and power of 1400 W.

The amount of CO decreased as the discharge power increased and in the outlet gas a higher oxygen concentration than that for 1.0 Nm<sup>3</sup>/h was detected.

The mass balance (Table 4) shows that for the homogeneous system the molar rates in the inlet and outlet streams were similar. The balance shows that nearly all products generated during the process have been quantified.

The calorific value of the outlet stream in the homogeneous system was lower while using a higher gas flow rate. The highest values were obtained in the plasma-catalytic system and the gas flow rate of 1.5 Nm<sup>3</sup>/h. It was 8.83 MJ/m<sup>3</sup> for 1700 W and initial concentration

**Table 4.** Mass balance of carbon [mol/h] in the homogeneous system. Gas flow rate 1.0 Nm<sup>3</sup>/h. Initial conc. of toluene 2000 ppm

Compounds	P [W]			
	0 <sup>(*)</sup>	1300	1500	1900
CO	7.37	8.97	8.79	9.78
CO <sub>2</sub>	7.05	5.89	5.71	5.13
CH <sub>4</sub>	0.00	0.44	0.43	0.46
C <sub>2</sub> H <sub>2</sub>	0.00	0.36	0.36	0.40
C <sub>2</sub> H <sub>4</sub>	0.00	0.08	0.08	0.08
C <sub>2</sub> H <sub>6</sub>	0.00	0.04	0.07	0.08
C <sub>7</sub> H <sub>8</sub>	0.62	0.40	0.35	0.30
Σ	15.02	16.18	15.79	15.83

(\*)Initial composition of the inlet stream

of toluene 2000 ppm. It shows that the coupled system enhanced the calorific value of the outlet gas.

On the basis of the obtained results (Table 3) it was found that for the reduced catalyst, the CO concentration decreases by about 25% and the hydrogen concentration decreases by about 43% in 4000 ppm concentration of toluene. This is probably due to multiple reactions occurring during the process. These may be, for example, oxidation reactions, reduction, conversion, dissociation and methanation<sup>4</sup>. The use of

the catalyst has resulted in a much higher efficiency of the removal of toluene, the model substance of tars in the gas after biomass pyrolysis (Fig. 3). It should be noted, however, that the use of a catalyst causes a significant reduction in the amount of hydrogen in the outlet gas. In the case of homogeneous systems, the hydrogen content is reduced, on average, by about 12%, and under the same conditions in the plasma catalytic system the hydrogen content decreases by up to 43%. Similarly, an unfavorable phenomenon was observed with the amount of carbon monoxide. In the homogeneous system, the amount of CO increased and in the plasma-catalytic system it decreased by about 20% for the discharge power of 1700 W. Based on the results of the tests it was found that the total decomposition of toluene is possible in a G-0117 catalytic converter with the reduced form of the catalyst.

The use of the plasma – catalyst system increased the calorific value of the outlet gas. It was higher than the calorific value of the inlet gas and above the minimal level demanded by engines and turbines. One can conclude that some part of energy consumed for plasma generation was recuperated in the increase of calorific value of the outlet gas.

## CONCLUSIONS

On the basis of the obtained results it has been found that the application of the gliding discharge is an effective technique for decomposition of toluene, a model compound for tar produced in biomass gasification. The obtained results were repeatable and by mass balance it can be concluded that most of the products were determined. The conversion of toluene increased with discharge power. The highest efficiency of toluene decomposition and calorific rate of outlet gas were obtained in the plasma-catalytic system with the reduced G-0117 catalyst.

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