Hydrogen Production from Ethanol Reforming by a Microwave Discharge Using Air as a Working Gas

Wei Guo,* XianFeng Zheng,* ZhengBo Qin, QiJia Guo, and Lei Liu

1. INTRODUCTION

Hydrogen has recently attracted significant attention as a new energy source.1−3 Domestic hydrogen production depends principally on the reforming of hydrocarbons and alcohols. Ethanol is a liquid at atmospheric pressure and room temperature with good renewability and low toxicity, so it is widely used in the field of reforming. However, steam reforming has some disadvantages, such as high energy consumption, many byproducts, catalyst deactivation, and a slow reaction rate.

Hydrogen production from fuel reforming using plasmas is an advanced technology. A large number of high-energy electrons, active groups, and excited ions can be obtained through plasma technology, which greatly promotes the reforming reaction.4 In addition, the plasma device is small and the energy density is high, so it can be used without catalysts, which has great development potential.

In recent years, plasma has been used for ethanol reforming5−12 to produce hydrogen. Xin et al.13 studied hydrogen production from ethanol solution by pulsed discharge coupled with TiO2 in this work. The reforming effect of coupled TiO2 discharge is better than that of general pulse discharge. Czylikowski et al.14,15 introduced ethanol liquid at the tip of the plasma flame in the form of droplets. Their method effectively prevents the generation of soot in the plasma generating area to shorten the life of the reactor. In their research, they also compared the effects of different air intake methods on the reforming results. Among them, the swirl air intake method had a good effect. The hydrogen production rate was 1016 NL(H2)/h, which was equivalent to an energy yield of 203 NL(H2)/kWh. Ulejczyk et al.16 studied the process of producing hydrogen from a mixture of water and ethanol in plasma and plasma-catalytic reactors. The results showed that the highest energy efficiency was 7.7 mol(H2)/kWh, and the maximum concentration of hydrogen in the product was 56%. Muoz et al.17 compared the performance of
two atmospheric pressure microwave plasma sources, namely, TIAGO torch plasma and surfatron discharge plasma, in ethanol reforming using plasmas. Their research found that the method of using a plasma torch has a better reforming effect when the ethanol flow rate is large. Qiu et al. used different discharge gases to reform ethanol to produce hydrogen. It is found that air has a better reforming effect. However, the ethanol conversion rate obtained in their experiment was low.

Fulcheri conducted research on hydrogen production from ethanol using plasma. Their research found that air has a better reforming effect and its change rule are investigated under different conditions with air as the working gas in this work. The reasons for these changes are analyzed, and the energy consumption and cost of hydrogen production are discussed. This paper can provide certain theoretical guidance and technical support for the development of catalysts and optimization of the hydrogen production process.

2. RESULTS AND DISCUSSION

We studied the reforming effect of applying air to ethanol reforming and investigated the influence of experimental conditions on the reforming results from the three aspects of oxygen-to-ethanol molar ratio, ethanol flow rate, and absorbed microwave power.

In this research, without an additional external heating source, the heat in the reaction chamber mainly comes from the oxidation reaction, which is an exothermic process. Therefore, the extent of the oxidation reaction determines the amount of heat released by the reaction. In this study, the degree of oxidation reaction mainly depends on the type and quantity of free radicals and the electron temperature and electron density of high-energy electrons. Therefore, the reaction exotherm and temperature of the reaction system can be controlled mainly by controlling the oxygen-to-ethanol molar ratio and absorbed microwave power.

2.1. Oxygen-to-Ethanol Molar Ratio (O2/Et). According to previous experiments, the products of ethanol reforming are H2, CO, CO2, CH4, C2H2, C2H4, C2H6, C3H6, C3H8, C4H10n, C4H10, and solid carbon. Figure 1 shows the change of the content of several main components in the gas phase product with the increase of the O2/Et when the absorbed microwave power was 700 W and the ethanol flow rate was 42.1 mL/min. It can be seen from this figure that with the increase of the O2/Et, the contents of H2, CO, and C2H4 all gradually decreased. The H2 content decreased from 31.21 to 27.19% and the content of CO increased from 3.75 to 8.33%. However, the content of CH4 increased slightly at first and then decreased gradually. During the experiment, as the O2/Et increased, the number of water droplets on the wall of the condensing tank increased. Also, as the O2/Et increased, the temperature of the reaction chamber also increased. It is worth noting that a small amount of soot was formed during the reaction. However, soot was adsorbed on the wall of the disk-shaped short tube and the cavity wall of the lower reaction chamber with the airflow, which had no effect on the microwave torch. When the O2/Et was relatively large (O2/Et > 0.8), we found only a very small amount of carbon deposit on the wall of the lower reaction chamber after 40 h of experiment. This shows that the design of the reactor has a significant effect on the inhibition of the formation of carbon deposits.

In addition, the influence of the flow rate of discharge gas on the reforming results is studied in this article. The typical value “12 + 5” and the contrast value “14 + 6” were selected for the study. The “12+5” mode indicates 12 NL/min peripheral gas and 5 NL/min middle gas. The “14 + 6” mode indicates 14 NL/min peripheral gas and 6 NL/min middle gas. Figure 2 shows the relationship between the O2/Et and ethanol conversion rate. It can be seen that these two different inlet modes had almost the same change trend of ethanol conversion rate. The ethanol conversion rate increased with the increase in the O2/Et and increased rapidly when the O2/Et was in the range from 0.5 to 0.7. When the O2/Et reached 0.9, the ethanol conversion rate reached 100%. Also, as the O2/Et increased, the temperature of the reaction chamber...
increased. The equilibrium temperature of the reaction chamber increased from 692.4 to 989.1 K with the increase in the O₂/Et. This shows that the increase in the O₂/Et can increase the reaction exotherm and the temperature of the reaction system.

Figure 3 shows the effect of the O₂/Et on hydrogen selectivity and hydrogen yield. The hydrogen selectivity and hydrogen yield increased with the increase in the O₂/Et. The hydrogen selectivity first decreased slightly, then increased rapidly. In the "12 + 5" and "14 + 6" inlet modes, the maximum hydrogen selectivities were 66.78 and 66.33%, respectively. The hydrogen yield first increased significantly and then decreased slightly. This is primarily because when the O₂/Et was less than 0.7, the ethanol conversion rate increased with an increase in the O₂/Et, and more CH₄ was generated. However, due to the limited content of oxygen, CH₄ could not be further oxidized, thereby leading to a decrease in hydrogen selectivity. With a further increase in O₂/Et, more oxygen radicals, excited oxygen atoms, and high-energy electrons participate in the reaction. CH₄, C₂H₄, etc. were further oxidized to H₂, CO, and CO₂, thereby increasing the hydrogen selectivity and yield. Between the two inlet modes, "14 + 6" inlet mode had a better reforming effect at low O₂/Et. When the O₂/Et was greater than 0.9, the "12 + 5" inlet mode is better. The optimal O₂/Et of the experiment is 0.9–1.0.

We also calculated hydrogen production rate under different O₂/Et to show the reforming effect more intuitively. As shown in Figure 4, with the increase in he O₂/Et, the hydrogen production rate first increased and then decreased. When O₂/Et was 1.0, the maximum hydrogen production was 2.19 mol(H₂)/mol(C₂H₅OH). The inlet modes had little effect on the hydrogen production rate (Figure 4).

In this work, the effect of carrier gas residence time on ethanol conversion rate was studied. As shown in Figure 5, when the residence time was between 0.64 and 0.81, ethanol was completely converted. When the residence time was longer, the conversion rate decreased. This is mainly because the long residence time means that the oxygen flow rate is low. Also, a relatively small amount of O radicals and high-energy electrons react with C₂H₅OH, resulting in a low degree of oxidation of C₂H₅OH and a low conversion rate of C₂H₅OH. Therefore, appropriately increasing the airflow rate means reducing the carrier gas residence time, which is beneficial to the ethanol conversion rate.

2.2. Ethanol Flow Rate. The effect of the ethanol volume flow rate on the reforming effect was investigated under the O₂/Et of 0.9 and absorbed microwave power of 700 W.

As shown in Figure 6, the ethanol conversion rate first increased and then decreased with an increase in the ethanol flow rate and achieved a maximum value of 100% at an ethanol flow rate of 42.1 mL/min. The reason for this is that when the ethanol flow rate is low, the intensity of the vortex flow formed in the upper reaction chamber is small, and C₂H₅OH enters the lower reaction chamber earlier owing to the updraft of the plasma jet, resulting in insufficient contact. However, when the flow rate is considerably high, the residence time of C₂H₅OH is short, and the excessive flow rate causes heat loss in the reaction chamber. This leads to a decrease in the temperature of the reaction chamber and the ethanol conversion rate. Also, this temperature change leads to a low degree of reaction and an evident increase in the content of CH₄, C₂H₄, and other hydrocarbons, resulting in a reduction in the hydrogen selectivity and hydrogen yield, as shown in Figures 6 and 7. The maximum value of energy efficiency was
46.96% at an ethanol flow rate of 42.1 mL/min. However, when the flow rate was considerably high, the ethanol conversion rate, hydrogen selectivity, and energy efficiency all decreased.

The cost of hydrogen production was also estimated in this article. As shown in Figure 8, the cost of hydrogen production was minimum at an ethanol flow rate of 42.1 mL/min, amounting to $3.66 /kg(H₂), which was lower than that of many processes in the field of hydrogen production from ethanol reforming using plasma. Among them, the cost of the raw material ethanol forms a major part. Moreover, the cost of hydrogen production can be further reduced by reducing the price of raw ethanol and optimizing the design of the subsequent reaction chamber to achieve the target [$2/ \text{kg}(\text{H}_2)] of Hydrogen Program Plan released by the U.S. Department of energy in 2020.

2.3. Absorbed Microwave Power. Figures 9 and 10 show the relationship between the reforming effect and the absorbed microwave power. The ethanol conversion rate, hydrogen selectivity, hydrogen yield, and energy efficiency increased with an increase in the absorbed microwave power. This is because the increase in the absorbed microwave power tends to increase the energy density, number of high-energy electrons, and average energy of high-energy electrons in the discharge area, and the thickness of the plasma sheath also increases. This results in an increase in reaction channel, thereby
increasing the collision probability of high-energy electrons, producing additional high-energy electrons, and free radicals. Finally, there were more active particles that participated in the hydrogen production reaction, which improves the reforming effect. When the absorbed microwave power was 700 W, the ethanol conversion rate, hydrogen selectivity, and energy efficiency respectively were 100, 63.51, and 46.96%.

The effect of microwave power on the energy yield was also calculated and analyzed, as shown in Figure 11. The energy yield first increased and then decreased, reaching a maximum of 861.91 NL(H_2)/kWh at an absorbed microwave power of 700 W. This value is higher than those given in most of the literature in the Introduction.

3. EXPERIMENTAL PRINCIPLE AND INSTRUMENT

3.1. Experimental Instrument and Process. This plasma reforming device is mainly oriented to distributed hydrogen supply and on-board hydrogen production. Although the cost of large-scale centralized hydrogen production is relatively low, the equipment start-up is slow and the cost of hydrogen transportation and storage is high. Distributed hydrogen supply and vehicle-mounted hydrogen production models are based on the concept of “produce it now and use it now” to minimize the high costs and safety risks caused by the hydrogen storage and transportation process. This means that hydrogen is produced in densely populated areas, so the hydrogen production process requires clean raw materials, compact equipment, and a gentle and safe reaction process. Compared with methanol, ethanol is less toxic, and it is a liquid that is easy to store and transport at room temperature. The application of plasma in reforming avoids frequent replacement of catalysts. The temperature of the plasma reaction system is low, which avoids the thermal pollution caused by the high-temperature conditions of traditional hydrogen production methods.

The entire reforming system has numerous subsystems dedicated to different functions including gas supply, microwave plasma generation, reforming reaction chamber, temperature and pressure detection, and gas gathering and analysis.

The reforming chamber, as shown in Figure 12, is divided into an upper reaction chamber and a lower reaction chamber.
through the microwave window, waveguide coaxial converter, and waveguide tube. Subsequently, the torch transmits and compresses the energy of the electromagnetic field to the nozzle to generate the excitation, resulting in the working gas being ionized to form plasma.

Compared with the previous experiments, this experiment uses air as the working gas. The O free radicals produced by the ionization of O, in the air can destroy the C=C bond of C2H4, reducing the possibility of C2H4 forming polymers on the wall of the reaction chamber. The generated O radicals and OH radicals can react with C, thereby eliminating part of the carbon deposits. Therefore, as working gas, air has great potential in inhibiting the formation of carbon deposits. In previous studies, as working gas, nitrogen, argon, and pure oxygen are more expensive, and the evaporation of water to generate water vapor requires equipment and high power consumption. The air is simple, easy to obtain, and pollution-free and has great potential in reducing hydrogen production costs and green hydrogen production process. Also, we use the waste heat of the reaction chamber to heat and vaporize ethanol, eliminating the electricity consumption of the original heating vaporization equipment. Therefore, such a design can also effectively reduce the cost of hydrogen production.

The experimental process is shown in Figure 15. Ethanol flows into the copper tube wrapped in the lower reaction chamber through the advection pump for preheating and vaporization. This step ensures a faster and thorough reaction. The air is split into three paths through a flowmeter. One path is mixed with ethanol vapor, subsequently entering the reaction chamber tangentially. In the other two paths, air is fed into the reaction chamber in a vortex as discharge gas from the end of the microwave plasma torch, one of which is middle gas and the other is peripheral gas. Plasma is formed under the action of microwaves. The product of reforming flows out from the outlet of the lower reaction chamber and is divided into two paths after being condensed in the condensing tank. One path is connected to the arc igniter and the reforming situation in the reaction chamber is preliminarily assessed by observing the combustion phenomenon. The other path is connected to the gas-collecting port to collect gas samples and analyze the specific composition of the reforming products via gas chromatography. We had set thermocouples at the torch end, upper reaction chamber, lower reaction chamber, and gas outlet. We can get the temperature of the reaction chamber through a thermocouple.

### 3.2. Experimental Principle

The air used in this study is mainly composed of O2 and N2. The main overall reactions involved in the experiment are as follows.

**Partial oxidation of ethanol**

\[
\text{C}_2\text{H}_5\text{OH} + 0.5\text{O}_2 \rightarrow 2\text{CO} + 3\text{H}_2
\]

**Thermal decomposition of ethanol**

\[
\text{C}_2\text{H}_4\text{OH} \rightarrow \text{CH}_4 + \text{H}_2 + \text{CO}
\]

**O2 is dissociated by the bombardment of high-energy electrons**

\[
\text{O}_2 + e \rightarrow \text{O} + \text{O} + e
\]

The O radicals produced by the dissociation have a strong oxidation ability, and they react with C2H5OH or free radicals to form oxygen-containing compounds. O radicals can also capture H atoms in some particles to form OH radicals and destroy the C=C bonds of some C2.

The high-energy electrons in the plasma flame are captured by the ethanol molecules that enter the plasma flame in the form of vortex airflow. The C=C bonds, C=O bonds, and O=H bonds in the ethanol molecules are destroyed to form various free radicals, mainly CH3CHOH, C2H2, CH3CH, CH3, CH2OH, OH, H, CH2O, etc., and small molecule products such as C2H4, CH4, H2O, H2, etc. Some of these small molecules undergo oxidation reactions with O radicals, and the other part further collides with high-energy electrons to form smaller molecules and free radicals.

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Figure 14. Schematic of a microwave plasma torch.

Figure 15. Schematic of experimental equipment and process.
H radicals have a great effect on the generation of H₂. Two H radicals can generate H₂ through complex reactions. H radicals can also form H₂ by trapping H in ethanol and ethanol molecular fragments such as HCO, CH₃HCO, C₂H₆, CH₄, etc. However, OH and CH₃ radicals also react with H radicals to generate H₂O and CH₄, respectively, thereby reducing hydrogen selectivity.²⁵

OH radicals and CH₃ radicals have dehydrogenation ability, which can make the ethanol molecules lose H and increase the ethanol conversion rate. O radicals, OH radicals, CH₃ radicals, and other particles (the ground state or excited state of molecules, atoms, or other free radicals) give H₂, CO, CO₂, CH₄, and other reforming products through a series of free radical collisions. In addition, during the reforming process, carbon deposits mainly come from the following reactions

\[
C_2H_4(a) \rightarrow \text{polymer} \rightarrow 2C + 2H_2
\]

\[
2CO \leftrightarrow CO_2 + C
\]

\[
CO + H_2 \leftrightarrow H_2O + C
\]

\[
CH_4 \leftrightarrow 2H_2 + C
\]

\[
C_2H_4 \leftrightarrow 2C + 2H_2
\]

Deposited carbon has an adverse effect on the performance of the plasma reactor. A large amount of soot may extinguish the plasma flame. Therefore, in the process of hydrogen production by plasma reforming, it is very important to suppress the formation of deposited carbon. In the reforming reaction, O radicals can destroy the C≡C bond and reduce the possibility of C₂H₄ forming polymers on the wall of the reaction chamber. H radicals also help to convert C₂H₄ into some C₁ products. OH radicals and O radicals can react with solid carbon to form CO.²⁶ In addition, some H₂O molecules generated during the reaction will also react with C atoms to generate H₂ and CO.

### 3.3. Parameter Setting

The experiment examines the influence of experimental conditions on the reforming effect from three aspects: oxygen-to-ethanol molar ratio (O₂/Et), ethanol flow rate, and absorbed microwave power. The definition of O₂/Et is as follows:

\[
O_2/\text{Et} = \frac{n_i(O_2)}{n_i(\text{Et})}
\]

Among them, \(n_i(O_2)\) is the number of moles of O₂ in the initial reactant and \(n_i(\text{Et})\) is the number of moles of ethanol in the initial reactant.

Through the preliminary experiment, the setting of the experimental parameters is presented in Table 1.

The experimental results are evaluated from these following four aspects:

- Energy efficiency
- Ethanol conversion rate
- Hydrogen selectivity
- Hydrogen yield

#### Table 1. Values and Ranges of Experimental Parameters

|                         | absorbed microwave power (W) | O₂/Et | flow rate of ethanol (mL/min) |
|-------------------------|-----------------------------|-------|-------------------------------|
| typical value           | 700                         | 0.9   | 42.1                          |
| range                   | 300–800                     | 0.5–1.2 | 16.2–58.9                   |

\[
T = \frac{Q(H_2)}{Q_{\text{total}}}
\]

Ethanol conversion rate

\[
x = \frac{n_i(C)}{n_i(\text{C}_2\text{H}_4)}
\]

Hydrogen selectivity

\[
S = \frac{2n_i(H_2)}{n_i(H)}
\]

Hydrogen yield

\[
y = \frac{2n_i(H_2)}{n_i(\text{C}_2\text{H}_4)}
\]

Among them, \(Q(H_2)\) means the calorific value of product H₂, \(Q_{\text{total}}\) means the calorific value of consumed ethanol and electricity consumption, \(n_i(C)\) means the number of moles of carbon in gaseous products, \(n_i(\text{C}_2\text{H}_4)\) means the number of moles of H in product H₂, \(n_i(\text{H})\) means the number of moles of H in gaseous products, \(n_i(\text{C}_2\text{H}_4)\) means the number of moles of H in initial ethanol.

When calculating energy efficiency, we use a low calorific value. We also calculate the energy yield and hydrogen production cost. They are defined as follows:

- Cost of hydrogen production

\[
C(H_2) = \frac{C_{\text{total}}}{q(H_2)}
\]

Energy yield

\[
Q(H_2) = \frac{V(H_2)}{C_e}
\]

Among them, \(C_{\text{total}}\) is the cost of electricity and raw materials per hour, \(q(H_2)\) is the mass production of hydrogen per hour, \(V(H_2)\) is the volume production of hydrogen per hour, and \(C_e\) is the electricity consumption per hour.

Hydrogen production costs include electricity consumption costs, raw material costs, and hydrogen separation and purification costs. The electricity consumption includes the electricity consumption of reforming equipment, condensing system, and peripheral equipment. The electricity consumption is calculated based on the average industrial electricity price of 0.4 yuan per kilowatt-hour. The price of a SA molecular sieve is 15 yuan per kilogram, and the price of 99% industrial ethanol is 2000 yuan per ton in China.

### 4. CONCLUSIONS

Based on the concept of onsite hydrogen production, a microwave plasma device for hydrogen production from ethanol reforming had been designed in this research. Also, the experimental device was optimized for the full contact of reactants and reduction of carbon deposition. The effects of the O₂/Et, ethanol flow rate, and absorbed microwave power on the reforming results were investigated. The results showed that an appropriate increase in the O₂/Et can increase the ethanol conversion rate, hydrogen selectivity, and hydrogen yield, but it should not exceed 1.0. The hydrogen production rate reached 2.19 mol(H₂)/mol(C₂H₅OH) when the O₂/Et was 1.0. The best residence time is 0.64 to 0.81 s. Increasing
the ethanol flow rate was beneficial in improving energy efficiency. However, an ethanol flow rate exceeding 42.1 mL/min had a negative impact on ethanol conversion rate. An increase in absorbed microwave power had a positive effect on the ethanol conversion rate and hydrogen selectivity. The lowest cost of hydrogen production was approximately $3.66/kg(H_2)_2$, and the highest energy yield was 861.91 NL/(H_2)_2/kWh. The optimum experimental conditions were an O_2/ Et value of 0.9, an ethanol flow rate of 42.1 mL/min, and an absorbed microwave power of 700 W, which can deliver better reforming results. The design of the reaction chamber requires further optimization, and the use of catalysts and separation of H_2 from N_2 will be considered in the future.

**AUTHOR INFORMATION**

**Corresponding Authors**
Wei Guo — School of Physics and Electronic Information, Anhui Normal University, Wuhu 241003 Anhui, China; Anhui Huadong Polytechnic Institute, Wuhu 241003 Anhui, China; orcid.org/0000-0003-4401-0027; Phone: +86 17718247466; Email: 1477369047@qq.com

XianFeng Zheng — School of Physics and Electronic Information, Anhui Normal University, Wuhu 241003 Anhui, China; Email: xzgheng@mail.ahnu.edu.cn

**Authors**
ZhengBo Qin — School of Physics and Electronic Information, Anhui Normal University, Wuhu 241003 Anhui, China; orcid.org/0000-0002-1127-8070
Qijia Guo — Anhui Huadong Polytechnic Institute, Wuhu 241003 Anhui, China
Lei Liu — School of Physics and Electronic Information, Anhui Normal University, Wuhu 241003 Anhui, China

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.1c04312

**Author Contributions**
The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

**Notes**
The authors declare no competing financial interest. The data that supports the findings of this study are available within the article and its Supporting Information.

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