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Conversion of CO₂ in microwave discharge in liquid methanol

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The dependence of the degree of CO₂ decomposition and the concentration of the main products (H₂ and CO) on the CO₂ flow rate at the reactor inlet is shown. The maximum degree of CO₂ decomposition reached 75 %.

Keywords: microwave discharge in liquid methanol, CO₂ decomposition, chromatography of discharge products, production of synthesis gas.

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Introduction

Currently, technologies for the decomposition and conversion of carbon dioxide (CO₂) into valuable chemical products are being studied. This task is important for ensuring the ecological safety of industrial processes, where carbon dioxide is one of the by-products. An effective way to solve this problem is to use a low-temperature plasma generated by various types of electrical discharges [1].

The study of the CO₂ conversion process is carried out mainly in a gas-discharge plasma. It is known that the addition of components such as methane and hydrogen allows one to significantly increase the degree of CO₂ decomposition. Thus, the positive effect of hydrogen and methane on CO₂ conversion is confirmed in dielectric barrier discharges, sliding arcs, and microwave discharges in the gas phase (see, for example, [2–8]). The introduction of methanol vapor in a mixture with CO₂ contributes not only to an increase in the decomposition degree but also to the formation of synthesis gas [8, 9]. The obtained CO₂ conversion degrees do not exceed 20 %.

Study [9] demonstrated a new approach to the plasma conversion of a mixture of CO₂ and methanol vapor in a device with a sliding arc at atmospheric pressure, which makes it possible to obtain synthesis gas with simultaneous CO₂ conversion with an efficiency of up to 20 %. By analogy with traditional dry methane reforming, this process is called “dry methanol reforming” by the authors.

A high degree of CO₂ conversion is demonstrated by microwave discharges initiated in the liquid phase, where the discharge is ignited in a gas bubble at the end of a microwave antenna. One of the most important advantages of such systems is the natural implementation of the products’ quenching process at the stage of the discharge afterglow (after the separation of the gas bubble from the antenna, caused by liquid evaporation into a hot gas bubble). This significantly increases the efficiency of CO₂ decomposition. In particular, in study [10] at atmospheric pressure, using a microwave discharge in liquid hydrocarbons, a 60 % decomposition degree of CO₂ was achieved. The degree of CO₂ conversion reached 45 % [11] in a microwave discharge in an aqueous solution. It is

worth noting that under these conditions, the product is synthesis gas, which serves as a raw material for obtaining different chemical compounds. The disadvantage of using such liquids was the formation of soot. In [11], to suppress soot formation, instead of pure ethanol, its aqueous solution was used, which negatively affected the CO₂ conversion.

Therefore, it is promising to study CO₂ conversion in a microwave discharge in liquid methanol, which is the simplest representative of the homologous series of alcohols, and in plasma of which H₂ and CH₄ are formed without soot generation. [12].

This study presents for the first time the results of an experimental study of CO₂ conversion when passing it through a microwave discharge in methanol. Special emphasis is placed on determining the decomposition degree of CO₂ and the composition of the main gaseous products.

Experimental Setup

Experimental setup used for the generation and study of a microwave discharge in liquids is described in detail in [10, 11]. The microwave system includes a magnetron microwave oscillator (2.45 GHz) and the elements necessary for the controlling and measuring the incident microwave power. The experiments were carried out at an incident microwave power of 490 W. The discharge section is a coaxial-to-waveguide adapter, the central conductor of which serves as an antenna for microwave energy input into the reactor. The discharge was formed near the antenna tip in a quartz reactor (diameter 55 mm) filled with methanol and placed in a protective metal mesh (wire spacing 0.5 mm). To facilitate the discharge, a tungsten rod with a diameter of 3 mm was located over the central conductor at a distance of 3 mm. The central conductor of the coaxial line was a tungsten tube (outer diameter 3.0 mm, channel diameter 1 mm), through which carbon dioxide was supplied to the liquid. The methanol volume in the reactor was about 40 mL, which ensured that the end of the central electrode of the coaxial line was below the liquid surface.

CO₂ with a flow rate from 110 to 670 mL/min passed through the RRG-20 mass

flow controller and was introduced into the reactor through the channel in the antenna. The pressure on the liquid surface was close to atmospheric pressure. The duration of the experiment was about one minute. During this time, the gas flow at the reactor outlet was measured, and after stationary conditions were established, a gas sample was taken for the analyzing the products' composition. During a single experiment, the composition of the liquid in the reactor was replenished.

A water cooler was used to separate the gaseous products of the plasma-chemical reactions from the evaporated liquid products. The condensed liquid was returned to the reactor. The flow rate of the gas mixture flow at the discharge outlet downstream of the water cooler was measured with a mechanical flowmeter.

The composition of the gas phase (CO, CO₂, H₂, C₂H₂, C₂H₄ and CH₄) at the reactor outlet with a discharge was determined on a FIA back-flush portable gas chromatograph (SPC MEMS, Russia) with a thermal conductivity detector (TCD) and two chromatographic columns using HayeSep N adsorbents and 13X molecular sieves. The carrier gas was argon.

The main discharge products were H₂ and CO. Minor mixture components included CO₂, CH₄, C₂H₂ and C₂H₄. In this paper, we focus on studying the process of CO₂ decomposition and the production of synthesis gas.

The decomposition degree of CO₂ (α) was calculated from the product flow rates at the reactor output using the formula:

$$\alpha = \frac{\text{CO}_2^{\text{input}} - \text{CO}_2^{\text{out}}}{\text{CO}_2^{\text{input}}} \cdot 100 \% \quad (1)$$

where CO₂^{out} is the CO₂ flow rate of at the reactor output and CO₂^{input} is the CO₂ flow rate at the reactor input. These values were obtained from the measured total gas flow rate at the reactor output and the corresponding component concentrations.

Results and Discussion

Fig. 1 shows the dependences of the concentration of the main components of the gas mixture at the discharge outlet on the CO₂ flow

rate at the reactor input. The decrease in the hydrogen concentration with an increase in the CO_2 flow rate is probably related to the dilution of the methanol vapor with carbon dioxide, as well as the reduction of the residence time of the reaction mixture in the discharge zone. It should be noted that the residence time decreases due to the reduction of the lifetime of the gas bubble

containing the plasma with the increase in the CO_2 flow rate.

Fig. 2 shows the dependence of CO_2 decomposition degree on its flow rate at the reactor input. A decrease in the CO_2 decomposition degree is due to the reduction of the residence time of the reaction mixture in the discharge zone.

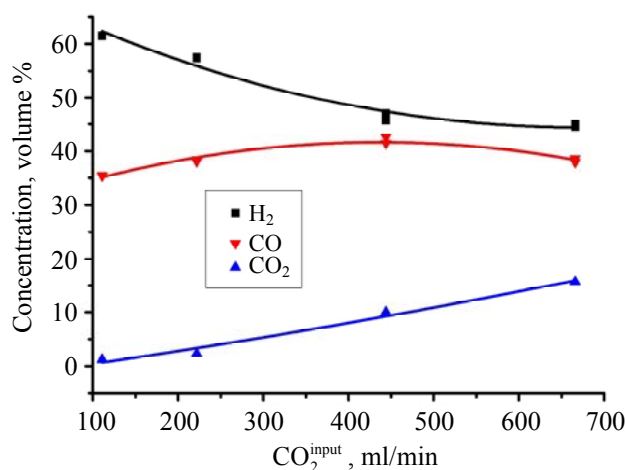


Fig. 1. Dependence of the concentrations of the main products on the CO_2 flow rate

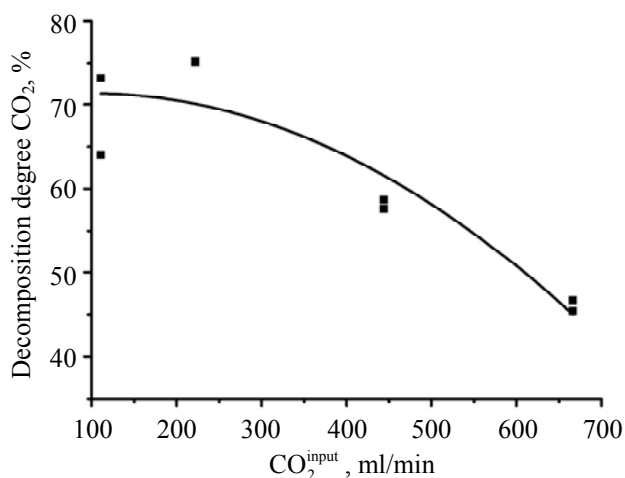


Fig. 2. Dependence of the CO_2 decomposition degree on the CO_2 flow rate at the input

Conclusion

The presented results show that a microwave discharge in liquid methanol can be an effective method for decomposing carbon dioxide while simultaneously producing synthesis gas. The obtained degree of carbon dioxide conversion exceeds the values obtained in a

microwave discharge in an ethanol aqueous solution.

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