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Microwave-assisted ethanol decomposition over pyrolysis residue of sewage sludge for hydrogen-rich gas production

Wenyi Deng^{*}, Shugang Liu, Jingchen Ma, Yaxin Su

School of Environmental Science and Engineering, Donghua University, Shanghai, 201620, PR China

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ABSTRACT

Microwave-assisted ethanol decomposition over pyrolysis residue of sewage sludge (PRSS) was investigated in a multi-mode microwave (MW) oven, and was compared with ethanol decomposition over a commercial activated carbon (AC). The results indicated that PRSS showed higher MW heating performance than AC. Under the same microwave power (MP), the temperature of catalyst bed, hydrogen production and ethanol conversion over PRSS were higher than that over AC. Both hydrogen production (per unit volume of ethanol) and ethanol conversion increased with the increase of MP, whereas decreased with the increase of the volumetric hourly space velocity of ethanol ($VHSV_{eth}$). Additionally, comparison between ethanol decomposition by MW and conventional heating was also conducted. Results indicated that MW heating was much more beneficial for hydrogen production than conventional heating, and the energy efficiency of hydrogen production under MW heating was much higher than conventional heating. Finally, carbon nanofilaments formation over PRSS and AC were observed under MW heating. The size and structures of carbon nanofilaments over PRSS and AC were significantly different with each other, which may be strongly correlated with the formation of micro-plasmas during MW heating.

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Introduction

Fast depletion of natural reserves of fossil fuels and concerns about the emission of CO₂ and other pollutants into atmosphere have promoted growing interest in search of new and safe energy sources. Hydrogen is regarded as one of the most promising clean energy, which has drawn much attention in recent years [1,2]. Owing to its non-toxicity, high calorific value, light weight, and zero pollutant emission, hydrogen is

widely utilized in hydrogen fuel cells, ammonia synthesis, metallurgical engineering, fertilizer production, etc. [3].

There are a variety of technologies for hydrogen production. The more developed are conventional technologies, such as coal gasification, hydrocarbon reforming, water electrolysis, etc. [4]. Nowadays, steam reforming of natural gas is the most widely adopted technology for large scale hydrogen production. The main drawback of the steam reforming technology is that a large amount of CO₂ emissions will take place.

^{*} Corresponding author.

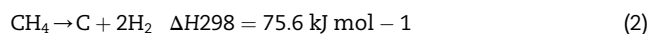
E-mail address: dengwy@dhu.edu.cn (W. Deng).

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Ethanol has been considered as an excellent bio-resource for hydrogen production, because of its low toxicity, CO₂ neutrality, easy generation from a variety of renewable feedstocks, low production costs and relatively high hydrogen content [5,6]. There are wide reports about hydrogen production from ethanol by catalytic reaction pathways, such as decomposition [3,7–9], steam reforming [2,5,6], partial oxidation [10,11], and auto-thermal reforming [12,13]. In particular, ethanol decomposition catalyzed by noble or transition metals has been widely reported, and it is considered as an important method for the production of hydrogen and carbon nanotubes (CNTs) [8,9,14]. A main drawback of metal-catalyzed ethanol decomposition is the formation of carbon that deactivates the metal catalysts [15]. Presently, hydrogen production from ethanol decomposition by discharge plasma has gained much attention. Sarmiento et al. [16] studied hydrogen production from ethanol vapor by dielectric barrier discharge, and the energy efficiency was about 75 NL H₂ kWh⁻¹. MW plasma torch is another technique used for hydrogen production from ethanol decomposition, and the energy efficiencies in the range of 4.1–72 NL H₂ kWh⁻¹ have been reported [4,17,18]. More recently, hydrogen production by discharge plasma in liquid ethanol is found to be more energy efficient. Wang et al. [19] applied MW discharge in liquid ethanol for hydrogen production, and the energy efficiency was about 137 NL H₂ kWh⁻¹. Sun et al. [1] also conducted MW discharge plasma in liquid ethanol for large capacity hydrogen production, with the energy efficiency of about 541 NL H₂ kWh⁻¹. Xin et al. [20,21] utilized pulsed spark discharge in ethanol solution for hydrogen production, which can produce about 2083 NL H₂ kWh⁻¹ of energy used.

Ethanol decomposition is an endothermic reaction, and the direct decomposition of ethanol can produce carbon monoxide, methane and hydrogen through reaction (1), and further decomposition of methane can promote hydrogen formation through reaction (2) [22,23].



The operation of endothermic catalytic reactors is limited by heat transfer (convective, conductive or radial resistances) through the packed bed, which will result in a pronounced temperature profile and thereby affecting reaction rate [24]. MW heating of dielectric materials provides many advantages over conventional heating, such as selective heating, volumetric heating, heating from the interior of the material, quick start-up and stopping, and higher level of automation and safety [25,26]. Carbon materials, such as AC, carbon black and pyrolysis residue from bio-solid, are usually adopted as catalysts or supports in MW-assisted catalytic reactions, owing to the excellent MW absorbing property [27–29]. Moreover, the carbon catalysts also possess the advantages of lower costs, high temperature resistance, tolerance to sulfur and other potentially harmful impurities in the feedstock [27]. However, few reports on ethanol decomposition on carbon catalysts can be found. Rechnia et al. [22,30] have studied the co-decomposition ethanol and methane over AC, but ethanol was used as an additive agent to alleviate the deactivation of AC.

As one of the main products from sludge pyrolysis, PRSS is mainly composed of fixed carbon and ash, and usually demonstrates an excellent MW absorption ability even better than AC [27]. Hence, ethanol decomposition over PRSS was investigated under MW heating in this study, and it was compared with ethanol decomposition over AC. Special attentions were paid to the change of gaseous components, ethanol conversion and hydrogen yield under the effect of MP and VHSV_{eth}. The difference between MW heating and conventional heating was also examined, as well as the formation of carbon nanofilaments on the surface of PRSS and AC.

Experimental

Preparation of catalysts

Sludge samples with moisture content of 80 wt% were sampled from a municipal wastewater treatment plant in Shanghai China, and were air dried at 105 °C for proximate and ultimate analysis. As shown in Table 1, the proximate analysis was conducted in a SDLA618 thermo-balance, while ultimate analysis of C, H, S, N was conducted in a Vario EL III apparatus, and the content of O was calculated by subtraction according to eqn. (3) [31].

$$\text{O}(\text{wt}\%) = 100 - \text{Moisture} - \text{Ash} - \text{C} - \text{H} - \text{N} - \text{S} - \text{CO}_2 \quad (3)$$

where CO₂ is mass percentage of carbon dioxide from decomposition of carbonate in sludge samples. PRSS was produced from pyrolysis of the dried sludge in N₂ atmosphere at 800 °C for 30 min in an electric furnace, and was utilized as a catalyst for ethanol decomposition. For comparison purpose, a commercial AC (Sinopharm Chemical Reagent Co.,Ltd) which was made from coconut shell and activated with steam was also used. The particle diameters of PRSS and AC were in the range of 0.45–1.0 mm. The metal elements (Al, Fe, Ca, K, Mg, Na, Ni, Co) in PRSS and AC were measured by an inductively coupled plasma emission spectrometer (ICE-AES, Leeman Prodigy), and Table 2 shows the elemental analysis of PRSS and AC. Textural characterization of the catalysts were carried out by a Micromeritics Tristar 3020 apparatus. Table 3 shows the Brunauer-Emmett-Teller (BET) surface area (S_{BET}), the micropore area (S_{mic}), the total pore volume (V_T), the micropore volume (V_{mic}), and the average pore diameter (d_{avg}) of PRSS and AC.

Experimental system and procedure

Ethanol decomposition was conducted in a multi-mode MW oven, or in an electric furnace (conventional heating with thermal power of 3 kW, not show in Fig. 1). Fig. 1 shows the schematic of the conducted reaction system. The MW frequency is 2.45 GHz, and the maximum power output of the MW oven is 800 W, divided into 10 different power levels. In other words, the output power can be manually regulated at 80, 160, 240, 320, 400, 480, 560, 640, 720, and 800 W. A quartz reaction tube (40 cm length × 2.2 cm i.d.) charged with 6 g of catalyst was inserted into the MW oven. Liquid ethanol was injected into the tube and decomposed in the catalyst. The

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