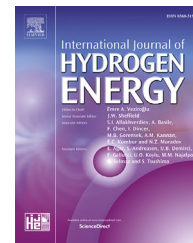




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Hydrogen production by aqueous phase reforming of phenol derived from lignin pyrolysis over NiCe/ZSM-5 catalysts

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ABSTRACT

Incipient wetness impregnation was used to synthesize the NiCe/ZSM-5 catalysts with different ratio of Ni:Ce, and CeO₂ was added as an assistant in the synthesis process. The physicochemical properties of the prepared catalysts were characterized by Transmission electron microscopy (TEM), X-ray diffraction (XRD), N₂-Sorption, H₂ temperature programmed reduction (H₂-TPR), X-ray photoelectron spectroscopy (XPS), Fourier Transform infrared Spectroscopy (FTIR) and ultraviolet–visible diffuse reflectance spectra (UV–Vis DR). The catalytic activities of the obtained catalysts were tested by using the reaction of aqueous phase reforming of phenol to produce hydrogen. Adding appropriate dose of Ce to the catalysts can increase the dispersion of nickel on the ZSM-5 support. The results showed that hydrogen selectivity was higher over 8Ni8Ce/ZSM-5 than using 8Ni/ZSM-5 as aqueous phase reforming catalysts. The content of carbon monoxide in the products after reaction over different catalysts was very low. However, the dispersion of carbon dioxide and hydrocarbons was significantly different over the two catalysts.

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Introduction

Hydrogen is one of the most important raw materials for chemical engineering, it can be used as raw materials for hydrotreating in petroleum refining industry, ammonia synthesis and many fine chemicals and as reductant in metallurgical industry [1–3]. Hydrogen as a kind of clean energy with

high heating value will play an important role in the future energy system. In recent years, on account of the requirements of environmental protection, the quantities of hydrogen needed for raw oil refining increased rapidly [4], and the development of fuel cell car raised a larger claim for on-board hydrogen supply. Technology with high efficiency and low cost to produce hydrogen had aroused with attention. There were

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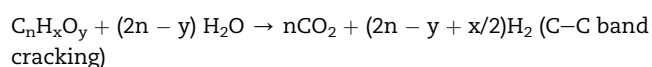
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many kinds of methods for hydrogen producing, such as organics reforming, hydrolysis, photolysis, electrolysis of water and biomass reforming and so on. Based on the shortage of natural gas, high cost of electric power supplement and also the high emission of pollutant during the process of power generation in China, the technologies of methane reforming and electrolysis of water to produce hydrogen were limited. In consideration of the very resourceful biomass and the excellent merit of biomass as raw materials to produce hydrogen, such as renewability and low pollution and so on, hydrogen production from biomass is a promising environmental and economic method of energy supply [5].

Among the different hydrogen produce routes, the mainly methods for hydrogen product from biomass are thermochemical method and biochemical method [6,7]. Thermochemical method can be used with kinds of biomass as raw materials and suitable for large scale of hydrogen production. The major technologies including biomass gasification to produce hydrogen and bio-oil steam reforming to produce hydrogen and so on [8]. Among which the method of biomass gasification to produce hydrogen is relatively mature and the process is relatively simple, however, there is always some deficiency exist, such as the high react temperature [9]. The two steps method processes are usually employed for bio-oil steam reforming. Firstly, biomass is pyrolyzed, and the obtained bio-oil will be transformed to hydrogen and other by-product by catalytic steam reforming. In comparison with biomass directly gasification to produce hydrogen, using bio-oil as reactant and the reforming method to produce hydrogen has the advantage of easier gaining higher hydrogen yield. Meanwhile, bio-oil has the advantage of easy collection, storage and transportation. The products from bio-oil can be easily separated to obtain the purified hydrogen.

Aqueous-phase reforming (APR) is the great promising process for biomass reforming to produce hydrogen because: 1) in comparison with the traditional process of alkane steam reforming, it can be carried out at relatively low temperature, such as 500 K, and 2) relatively higher pressure which avoided the process of water and the reactant gasification and thereby decreased the energy cost. This method is suitable for raw materials with water, such as biomass. 3) On account of the reaction is occurred in water, the water-gas shift is easier carried out, low content of CO can be obtained in the product [10] which is fit for supplying hydrogen to proton exchange membrane fuel cell, this usage supply new application ways for efficient utilization of biomass and hydrogen producing technique [11,12]. The undesirable decomposition reactions are minimized [13], there is high content of H₂ in products. APR method was first discovered by professor Dumsic and coworkers in Wisconsin university of America at 2002 [14]. Pt, Pd or Sn modified Raney Ni can be used as catalysts and the reaction was conducted under mild condition. With reaction temperature between 423 and 530 K and the pressure slightly higher than the saturated vapor pressure of water, oxygenated hydrocarbons can be transformed to H₂ and CO₂ with high selectivity [15–17].

The overall reactions of APR are as follow [14]:



Many kinds of noble metal supported catalysts are applied in the APR reaction, such as Pt, Pd, Ru and Ir and so on [18–21]. Noble metal supported catalysts exhibited high reforming activity [22], however the high price limited the industrial application. Non-noble metals, especially the transition metals of VIII group, such as Ni, with the advantages of low price and wide range of application, show preferable catalytic activity of hydrogen reforming [23–26]. Ni is widely employed in the industry for its low cost and good activity and H₂ selectivity in reforming processes owing to the excellent ability of C–C and C–H cleavage and sufficient oxygen transport capability [27–29]. Zhang and coworkers found that Ni/ZSM-5 showed high catalytic activity during the process of aqueous phase reforming of sorbitol to produce hydrogen [30].

For further optimizing the performance of catalyst, small quantity of assistant which can improve the dispersion rate of active species and catalytic activity was added to the catalysts. Among all the assistant, Ceria is of great significance and serious interest for researchers owing to its two important functions on which the applicability of CeO₂ depends: (1) the redox couple, Ce⁴⁺/Ce³⁺ with its ability to shift between CeO₂ and Ce₂O₃ under oxidizing and reducing conditions respectively, and (2) the ease of formation of labile oxygen vacancies and oxide ion storage. (3) CeO₂ can strongly connect with support to improve the dispersion of active species and stability. Pu and coworkers reported that during the acetic acid reforming reacting, both the conversion of acetic acid and hydrogen yield were improved and the coke formation on the catalysts is significantly inhibited by the addition of CeO₂ [31]. During the toluene steam reforming process, the addition of Ce to Ni₂Mg₄Al_{1.8} catalysts also enhance the H₂ selectivity [32]. CeO₂ is widely applied as an assistant in the ZSM-5 based catalysts [33,34] and the anchoring effect of CeO₂ for metal particles via SMSI has been demonstrated for various metal-ceria systems [35,36]. Inaba and coworkers reported that, during the reaction of gasification of cellulose to produce hydrogen, Ni/Ce/H-ZSM-5 catalyst is a promising candidate for this reaction.

According to the above analysis, catalyst with Ni as metal active species, CeO₂ as assistant and ZSM-5 as carrier will be synthesized and the catalytic activity for APR of the phenolic model compound of bio-oil will be studied in the present work.

Materials and methods

Experimental materials

Phenol was chosen as the phenolic model compound of bio-oil. ZSM-5 was purchased from the catalyst Plant of Nankai University with Si/Al ratio of 25. Nickel nitrate hexahydrate (Ni(NO₃)₂·6H₂O) and Cerium (III) nitrate hexahydrate (Ce(NO₃)₃·6H₂O) were purchased from J&K Scientific LTD. All the reagents were used without further purification.

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