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Chemical looping glycerol reforming for hydrogen production by Ni@ZrO₂ nanocomposite oxygen carriers

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ABSTRACT

The research describes the synthesis of nanocomposite Ni@ZrO₂ oxygen carriers (OCs) and lanthanide doping effect on maintaining the platelet-structure of the nanocomposite OCs. The prepared OCs were tested in chemical looping reforming of glycerol (CLR) process and sorption enhanced chemical looping reforming of glycerol (SE-CLR) process. A series of characterization techniques including N₂ adsorption-desorption, X-ray diffraction (XRD), inductively coupled plasma optical emission spectrometry (ICP-OES), high resolution transmission electron microscopy (HRTEM), H₂ temperature-programmed reduction (H₂-TPR), H₂ pulse chemisorption and O₂ temperature-programmed desorption (O₂-TPD) were used to investigate the physical properties of the fresh and used OCs. The results show that the platelet-stack structure of nanocomposite OCs could significantly improve the metal support interaction (MSI), thus enhancing the sintering resistance. The effect of lanthanide promotion on maintaining this platelet-stack structure increased with the lanthanide radius, namely, La³⁺ > Ce³⁺ > Pr³⁺ > Yb³⁺. Additionally, the oxygen mobility was also enhanced because of the coordination of oxygen transfer channel size by doping small radius lanthanide ions. The CeNi@ZrO₂ showed a moderate 'dead time' of 220 s, a high H₂ selectivity of 94% and a nearly complete glycerol conversion throughout a 50-cycle CLR test. In a 50-cycle SE-CLR stability test, the CeNi@ZrO₂-CaO showed high H₂ purity of 96.3%, and an average CaCO₃ decomposition percentage of 53% without external heating was achieved.

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Introduction

In recent years, hydrogen has been widely applied as a clean energy source for electrical power generation and transportation fuel [1,2]. According to many projections, hydrogen plants are the major source of CO₂ emissions to the atmosphere, and this trend will continue in the foreseeable future [3]. However, the conventional steam reforming process for H₂ production has some disadvantages such as catalysts deactivation resulted from coke deposition and high energy consumption [4,5]. Researchers have proposed a chemical looping reforming (CLR) process, where the catalyst is repeatedly regenerated by air, as one of the possible routes to solve these problems [6,7]. The CLR is performed by cycling the oxygen carrier (OC) between a fuel feed step, where the OC contacts fuel, and an air feed step, where the OC and deposited coke are oxidized by air [8,9]. Additionally, the oxidation of oxygen carrier in air feed step could provide heat for the calcination of the sorbent in a sorption enhanced chemical looping reforming (SE-CLR) process [10–13]. A distinguished OC should accomplish some characteristics such as excellent redox property, high catalytic reactivity and improved resistance to coke and sintering [8].

Extensive efforts have been dedicated to design OCs adaptable for the CLR process. Zafar et al. [14] have reported that Ni-based oxygen carrier shows highest redox reactivity compared with other oxygen carriers. Additionally, Ni is renowned for its excellent ability for C–C and C–H bond rupture, resulting in high catalytic reactivity in fuel feed step [15,16]. Nevertheless, the major obstacle to Ni-based OCs application is the fast deactivation derived from active phase sintering and coke formation [17,18]. Stabilization of Ni nanoparticles by solid-phase crystallization and enhancing the removal of coke deposition by high oxygen mobility oxides are two typical strategies to improve stability of OCs [19,20]. Zirconia is widely employed as an OC support because of its amphoteric characteristic and oxygen storage capacity (OSC) [21,22]. Xu et al. [23] have demonstrated that the metal-support interaction (MSI) could significantly improve when the particle size of ZrO₂ decreased to an extent that it was comparable to that of Ni particles. Because the amount of ZrO₂ nanoparticles are dominant in the catalyst, each Ni nanoparticle would be confined by several ZrO₂ nanoparticles, generally showing a platelet-stack structure in transmission electron microscopy (TEM) images. This kind of catalysts with size-comparable Ni and ZrO₂ particles (denoted as Ni@ZrO₂) would be named as a metal/oxide nanocomposite catalyst better than a conventional metal-supported catalyst (denoted as Ni/ZrO₂) [23]. Additionally, it has been demonstrated that smaller particle size of OCs could shorten the distance of oxygen releasing from bulk to surface, and thereby, more oxygen vacancies are beneficial to improve coke resistance [24]. Compared with the conventional metal-support Ni/ZrO₂ OC, the Ni@ZrO₂ OC with nanocomposite structure is assumed to possess higher coke resistance and sintering resistance.

Although the platelet-structure of nanocomposite OCs shows a potential to provide better performance in CLR, the thermal stability of this structure is low due to the phase transformation. Li et al. [25] have compared the conventional

Ni/ZrO₂ catalyst with the nanocomposite Ni@ZrO₂ catalyst, and they discovered that the Ni/ZrO₂ is mainly comprised of monoclinic zirconia (*m*-ZrO₂) while the Ni@ZrO₂ is composed of the metastable tetragonal zirconia (*t*-ZrO₂). It has been reported the phase transformation from *t*-ZrO₂ to *m*-ZrO₂ is complete at about 650–700 °C, leading to the loss of the unique platelet-stack structure in nanocomposite OCs [26]. Lanthanide oxide as a stabilizer, which could dissolve into zirconia and slow down or prevent the process, has been widely investigated. Rydén et al. [27] have studied the stabilization effect of Ca, Mg and Ce on nanocomposite ZrO₂ in CLR process and concluded that ZrO₂ stabilized with Ce performs well. Apart from being stable, the stabilized zirconia with lanthanide would contribute to the formation of defect sites, thus improving the oxygen mobility [28].

In this paper, we synthesize a series lanthanide promoted Ni@ZrO₂ nanocomposite OCs and investigate the stabilization effect of lanthanide promotion on nanocomposite Ni@ZrO₂ OCs in CLR. The physical-chemical properties of fresh and used OCs are characterized by N₂ adsorption-desorption, X-ray diffraction (XRD), inductively coupled plasma optical emission spectrometry (ICP-OES), high resolution transmission electron microscopy (HRTEM), H₂ temperature-programmed reduction (H₂-TPR), H₂ pulse chemisorption and O₂ temperature-programmed desorption (O₂-TPD). Moreover, the activity and stability tests of CLR and SE-CLR are performed to evaluate these OCs.

Experimental

Preparation of OCs

NiO and lanthanide oxide (CeO₂, LaO₂, YbO₂ and PrO₂) nanoparticles were prepared by a homogeneous precipitation method. In a typical synthetic process, NiCl₂·6H₂O (Aladdin, 99.9%), sodium dodecyl sulfate (SDS, Aladdin, 99.9%) and urea (Aladdin, 99.5%) were first dissolved in de-ionized water with molar ration of 1:2:30:60. This solution was continuously stirred to transparent at 40 °C and then kept for 20 h at 80 °C. The insoluble substance was washed with the de-ionized water and ethanol to remove the chloride species. The resultant was dried at 90 °C for 9 h. Then it was further calcined for 3 h at 300 °C.

As for the preparation of nanocomposite oxygen carriers, the NiO and lanthanide oxide nanoparticles were dispersed in the ZrOCl₂·8H₂O (Aladdin, 98%) solution with the help of PEG (Aladdin, 3 wt% solution) under sonication treatment. The Ni and lanthanide loading were fixed to 20 wt% and 5 wt%, respectively. The precipitant agent (Aladdin, NH₄OH, 1 M) was dropped into the mixture till a pH of 9.5. The obtained solid was then transferred to an autoclave and kept at 140 °C for 10 h. Subsequently, it was washed with ethanol and de-ionized water, dried for 9 h at 90 °C and calcined for 3 h at 800 °C. The synthesized nanocomposite OCs, which were promoted by Yb, Pr, Ce and La, were denoted as YbNi@ZrO₂, PrNi@ZrO₂, CeNi@ZrO₂ and LaNi@ZrO₂, respectively.

As for the conventional Ni/ZrO₂ oxygen carrier, ZrO₂ particles were prepared as the same way as the nanocomposite oxygen carrier except without addition of Ni and lanthanide

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