Catalytic study of efficient nanocomposites $\{\text{Ni}_{0.5} \text{Zn}_{0.5-x} \text{Ce}_x \}$-oxides electrodes for natural gas-fed fuel cells

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Keywords: catalyst, fuel cell, natural gas, energy materials

Abstract

Highly catalytic active anode materials are one of the scorching zones in recent solid oxide fuel cells (SOFCs). In order to develop high performance SOFCs compatible anode materials are extremely required. In this work, $\text{Ni}_{0.5} \text{Zn}_{0.5-x} \text{Ce}_x$-oxides (NZC-oxide) nanocomposite anode material is synthesized by employing facile, low cost wet chemical technique. The phase structure before and after $\text{CH}_4$ environment is studied by x-ray diffraction (XRD) and Raman spectroscopy. However, no significant structural phase change is observed in $\text{Ni}_{0.5} \text{Zn}_{0.3} \text{Ce}_{0.2}$-oxides after $\text{CH}_4$ treatment. Additional characterizations are investigated by UV-visible spectroscopy, Fourier transform infrared spectroscopy and Scanning electron microscope (SEM). The dc electrical conductivities are measure by four probe method. The ideal and suitable $\text{Ni}_{0.5} \text{Zn}_{0.3} \text{Ce}_{0.2}$-oxides among all nanocomposites using as anode in fuel cell devices, show maximum power density of 500 mWcm$^{-2}$ with open circuit voltage (OCV) of 1.0 V at 600 °C. By empowering enhanced catalytic assets of $\text{Ni}_{0.5} \text{Zn}_{0.3} \text{Ce}_{0.2}$-oxides nanocomposite anode may be useful for fuel cell applications.

1. Introduction

Solid oxide fuel cell (SOFCs) is one of the most promising type of fuel cell due to its high-conversion efficiency (over 70 % with regeneration), low emission contaminant (e.g. $\text{NO}_x$ and $\text{SO}_x$) and fuel flexibility (e.g., methane, hydrogen, gasoline, carbon monoxide, natural gas and hydrocarbons) than that of conventional electric power generators [1, 2]. Ecological effectiveness of SOFCs devices can be enhanced using biofuels i.e. ethanol because of its remarkable carbon neutrality behavior in nature as compared to traditional fossil fuels devices, at operational temperature between 800 K to 1300 K [3]. In SOFCs the electrochemical performance is dominantly dependent on the nature of both electrode and electrolyte. It is highly desired that SOFC electrodes should possess high electronic conductivity and porous microstructure, which facilitates rapid transport of reactant gases at the triple phase boundary (TPB), which is the region where reactant gases (atmosphere), ions (electrolyte), and electrons (electrode) meet. Furthermore, the anode and cathode should, exhibit high catalytic activity for $\text{H}_2$ oxidation reaction (HOR) and $\text{O}_2$ reduction reaction (ORR), respectively [4, 5]. However, Ni-oxide mixed with oxide ion conductor (yttria-stabilized zirconia (YSZ) or samarium–doped ceria (SDC)) composite is one of the most frequently used anode materials for SOFCs [6]. The ion-conducting phase is generally used to adjust the thermal expansion coefficient (TEC) with electrolyte and to extend TPBs. As TPBs plays an important role to extend active sites for the reaction, thereby results in improvement of fuel cell performance [7, 8]. Conversely direct utilization of fuel in anode can cause the decrease in the life of fuel cell due to the deposition of carbon layer. Moreover, Nickel-oxide provides good catalytic activity for HOR with good conduction path way for electrons in desired temperature range. By contrast Ni-based (Ni/ YSZ or Ni/GDC (Gadolinium-doped ceria))
anode materials with hydrogen as a fuel demonstrate the best fuel cell performance [9, 10] but major concern with Ni metal nanocomposites anode is that it fails to complete oxidize hydrocarbons which leads to the deposition of carbon layer (i.e. in the form of graphitic carbon or carbon filaments) while fed with (biofuels/natural gas) [11, 12]. It has been contended that rapid gas pyrolysis of fuel takes place at significant temperature of SOFCs which significantly enhance carbon deposition. Mainly two pyrolysis products such as acetylene and ethylene are strong agents to form carbon deposits on the surface of the anode. Development of anode materials with good catalytic activity is so far perceived one possible way to improve the fuel cell performance at lower temperature, as reported by many groups [3, 13]. Therefore, study the catalytic effect of such materials is highly desirable to obtain the overall high and stable performance of device. In previous study it is reported that Nickel-Copper (Ni–Cu) alloy could not only help to reduce carbon deposition at anodes, but due to low melting temperature its use is limited [6, 14]. In addition, Palladium-Nickel (Pd–Ni) composites anode showed favorable tolerance to carbon deposition however severe carbon deposition was still observed due to larger amount of Ni-oxide [15].

This work aimed solely to synthesized Ni-oxide nano-composites based on Zinc and Ceria such as Ni_{0.5}Zn_{0.5-x}Ce_{x}-oxides (0.1 ≥ x ≥ 0.5) and evaluated them as an anode material in SOFCs. The key purpose for using Ceria and Zinc is to enhance the catalytic activity as well as to reduce the operating temperature of fuel cells. At this stage, Ce has affinity to reduce the operating temperature and completely oxidize the dry hydrocarbons fuels directly which may reduce the carbon deposition layer. Moreover, Zn oxide is likely to tune the electronic states of as prepared material which can enhance the catalytic activity of the anode material to improve the overall fuel cell performance. [7, 9, 10, 16, 17]. The catalytic activity of Ni_{0.5}Zn_{0.5-x}Ce_{x}-oxides (NZC-oxides) nano-composites were studied in CH_4 environment. The dc electrical conductivities and fuel cells performance are also investigated.

2. Experimental

2.1. Synthesis of NZC-oxides by wet chemical technique

Various Ni_{0.5}Zn_{0.5-x}Ce_{x}-oxides (0.1 ≥ x ≥ 0.5) nano-composites were synthesized by wet chemical technique. The stoichiometric amount of nickel nitrate hexa-hydrate Ni(NO_3)_2.6H_2O (Sigma Aldrich 99.99%), zinc nitrate hexa-hydrate Zn(NO_3)_2.6H_2O (Sigma Aldrich 99.99%) and cerium nitrate hexa-hydrate Ce(NO_3)_3.6H_2O (Sigma Aldrich 99.99%) were castoff. A suitable molar fraction of nickel, zinc and cerium were liquifled in 100 ml distilled water and let them stirrer at 80 °C for 30 min In next step, oxalic acid was added into given precursor and continued heating and stirring till to get black gel. The obtained gel was dried in drying oven for 4 h at 150 °C. The dried gel was thoroughly grinded and followed by sintering at 900 °C for 5 h. Finally, the sintered material was grinded yet again for characterization and cell fabrication. The schematic diagram of synthesis method is shown in figure 1.

The typical ionic electrolyte samarium doped ceria, SDC (Sm_{0.2}Ce_{0.8}O_2) was prepared as reported in literature [18]. In the same way, Lithium transition metal oxide (LiNiCuZn-oxide) was used as a cathode material and prepared as reported somewhere [19].

2.2. Fabrication of single cell and fuel cell device

The fuel cell was fabricated by using CARVER Hydraulic press (USA) under a pressure of 220 MPa. The appropriate amount of powder of anode (NZC-oxides), electrolyte (SDC) and cathode (LiNiCuZn oxide) were transfer one by one into a die and compressed via a Hydraulic press. The thickness and active area of the fabricated cell was roughly 1.5 mm and 0.64 cm^2, respectively. The fabricated cells were sintered at 700 °C for 1 h. The silver paste was applied on both sides of cells as current collector prior to measurement. For DC conductivity measurement, single cells of NZC-oxides were fabricated with 2 mm in thickness and 13 mm in diameter via a CARVER Hydraulic press (USA) under a pressure of 220 MPa.

2.3. Characterization

The crystalline structure of synthesized Ni_{0.5}Zn_{0.5-x}Ce_{x}-oxides (0.1 ≥ x ≥ 0.5) nano-composites was verified by using PAN analytical X'pert PRO dicultrometer using Cu Kα radiation (λ = 1.54 Å) at 40 KV and 30 mA with a scanning rate and scan speed of 0.01 °/s and 1°/min in a 2θ range of 10°–80°. The detailed microscopic view of the prepared nanocomposites was examined by scanning electron microscopy (SEM-Philips XL-30). The UV-Visible absorption and transmittance spectra of synthesized NZC-oxides were measured using a double beam Perkin Elmer UV-Visible Diffuse Reflectance Spectroscopic Lambda-35 (UV-35, Perkin Singapore) with the bandwidth setting of 1 nm at a scan speed of 960 nm min^-1 in the range of 300 to 1100 nm. Fourier transform infrared spectroscopy spectra (FTIR) was recorded to study the composition of NZC-oxides using Thermo Fisher Scientific (Nicolet 6700) spectrometer over the wave range of 600–4000 cm^{-1}. Raman
Spectroscopy and XRD was also used to investigate the stable phase structure and amount of carbon deposition before and after CH₄ treatment.

DC conductivity measurements were carried with the help of four probe Keithly 2450 source meter at 400 °C–700 °C. The total electrical conductivity was calculated by following equation.

\[ \sigma = \frac{L}{RA} \]

Where \( \sigma \) is the conductivity, \( L \) is the thickness of the pellet, \( R \) is the internal resistance and \( A \) is the active area of the single cell or pellet [20]. The active area of the pellet was 0.64 cm². Finally, fuel cell performance was measured by using H₂ and air as fuel and oxidant, respectively, with a flow rate of 100 ml min⁻¹. In order to depict I-V (current-voltage) and I-P (current density-power density) curves the values of current and voltage were recorded.

3. Results and discussion

3.1. Crystal structure and microstructure analysis

The x-ray diffraction pattern of prepared Ni₀.₅Zn₀.₅₋ₓCeₓ-oxides (0.1 ≤ x ≤ 0.5) nanocomposites are shown in figure 2(a). The measured peaks depicted that composite oxide has multi-phase structure. The characteristic peaks were analyzed by using the MJAD 6.5 software. The peaks were indexed with different JCPDS cards (81–0792) such as CeO₂, Ni-oxide (75–0271) and Zn-oxide (79–0205) with cubic fluorite, cubic and hexagonal structures, respectively. The average crystallite size was calculated by using Scherer’s formula:

\[ D = \frac{k\lambda}{\beta \cos \theta} \]

Where \( D \) is the crystalline size, \( k \) is a constant whose value is 0.94, \( \lambda \) is the wavelength and \( \beta \) is the full width at half maximum (FWHM). The obtained results rendered that the average crystallite size was decreased earlier from 30 to 22 nm as shown in table 1 and later it starts increases to some extent, which may be attributed due to the quantum confinement effect. Moreover, the slight variation in the peak position with increase of ceria.
contents representing the change in the lattice parameters due to different in size of ionic radii of Ni$^{2+}$, Zn$^{1+2}$ and Ce$^{3+}$ ions [21]. The ionic radii of Ce$^{3+}$ (1.143 Å) is greater than the both of Zn$^{1+2}$ (0.82 Å) and Ni$^{1+2}$ (0.78 Å).

Similarly, XDR patterns for Ni$_{0.5}$Zn$_{0.5-x}$Ce$_x$-oxides (0.1 $\leq x \leq$ 0.5) nanocomposites after testing in CH$_4$ environment for 40 min at 700 °C are shown in figure 2(b). On Comparison both the XRD pattern (before and after treatment) shows no visible change except that the peak intensity of (111) of Ni$_{0.5}$Zn$_{0.5}$Ce$_{0.0}$-oxides was slightly decreased, might be demonstrating its chemical stability. Remarkably, after CH$_4$ treatment the (200), (101) and (200) diffraction peaks vanished in Ni$_{0.5}$Zn$_{0.3}$Ce$_{0.2}$-oxides, Ni$_{0.5}$Zn$_{0.2}$Ce$_{0.3}$-oxides and Ni$_{0.5}$Zn$_{0.1}$Ce$_{0.4}$-oxides respectively, it may be due to reducing phase transition during treatment. The diffraction peaks are still the combination of cubic, cubic fluorite and hexagonal structure, depict no noticeable change before and after treatment. In order to give in deep information, the plot against particle size (D), lattice constant (a) and different composition of NZC-oxide is shown in the figure 3.

Further, the XRD results are validated by SEM micrographs of Ni$_{0.5}$Zn$_{0.5-x}$Ce$_x$-oxides (0.1 $\leq x \leq$ 0.5) nanocomposites. The agglomeration and smaller particles are indicating the presence of ZnO and NiO nanoparticles and small nano-sphere presented as CeO$_2$ nanoparticles as shown in figures 4(a)–(f). The figure 4, correspond to zinc and cerium fillings with x = 0.3, 0.2, and 0.1 in powders, respectively. The average particle size is to be 30–300 nm. These micrographs characterize the small particle size and porous structure which provides the good path of electrons to flow easily through it and is responsible to improve the overall cell performance [17].
Table 1. XRD analysis of Ni$_{0.5}$ Zn$_x$ Ce$_{0.5-x}$-oxide nanocomposites.

| Sample name     | Average crystallite size nm | Average atomic distance(d) nm | Average lattice constant(a) Nm | Average volume nm$^3$ | Average FWHM   |
|-----------------|-----------------------------|-------------------------------|-------------------------------|-----------------------|----------------|
| 1: Ni$_{0.5}$ Zn$_{0.5}$ Ce$_{0.0}$ | 30.32124                    | 0.172276                      | 0.391991                      | 0.067846               | 0.35928        |
| 2: Ni$_{0.5}$ Zn$_{0.4}$ Ce$_{0.1}$ | 29.45833                    | 0.173203                      | 0.430574                      | 0.430574               | 0.363292       |
| 3: Ni$_{0.5}$ Zn$_{0.3}$ Ce$_{0.2}$ | 24.84615                    | 0.177477                      | 0.502952                      | 0.131817               | 0.380462       |
| 4: Ni$_{0.5}$ Zn$_{0.2}$ Ce$_{0.3}$ | 22.21053                    | 0.184323                      | 0.382194                      | 0.062717               | 0.426263       |
| 5: Ni$_{0.5}$ Zn$_{0.1}$ Ce$_{0.4}$ | 27.84615                    | 0.176348                      | 0.416303                      | 0.01665                | 0.353538       |
| 6: Ni$_{0.5}$ Zn$_{0.0}$ Ce$_{0.5}$ | 28.95238                    | 0.178568                      | 0.400296                      | 0.049916               | 0.35119        |
3.1.1. UV-visible analysis

The obtained absorption spectra of prepared anode material are expressed in figure 5. The figure 5(a) exhibited different absorbance peaks in ultraviolet region such as 225 nm (Ni-oxide), 280–335 nm (Ni–Zn–Ce oxide) and 380 nm (Zn oxide). The measured peaks beyond 200 nm give informative spectra as electrons excite from the p & d orbitals, pie-orbitals and particularly pie-conjugated system befalls [22]. The absorption coefficient and the energy gap were calculated by following equation:

\[(\alpha h\nu) = A(h\nu - E_g)^{1/2}\]

Where \(\alpha\) is the absorption coefficient, \(h\) is the photon energy, \(E_g\) is the optical band gap energy and \(A\) is a constant [23]. The tauc plot of \((\alpha h\nu)^2\) against photon energy \((h\nu)\) provides the band gap energy.

The absorption band edge of nickel doped ZnO perceived at 380 nm shifted toward the high wavelength, it means that no absorption was logged into visible region and this shift might be due to the sp-d exchange collaboration among the band electrons and localized d-electrons of Ni\(^{2+}\) ions [22]. However, the fluctuating peaks intensities could be due to different configuration of zinc and cerium. Whereas in ultraviolet region (200–400 nm) all the maximum peaks were attained at 225 nm (Ni-oxide), and 380 nm as shown in figure 5(a). The transmission continuums were specified in figure 5(b). The typical transmission is 42 % in visible near IR region, as the amount of zinc drops and cerium gradually rises thus the transmission also falls up to 25%. The
3.1.2. FTIR analysis
Fourier-transform infrared spectroscopy (FTIR) is used to study the functional groups and composition of prepared nanocomposites with their conformation. Figure 6 shows FTIR spectrum of Ni$_{0.5}$Zn$_{0.5-x}$Ce$_x$-oxides ($0.1 \leq x \leq 0.5$) nanocomposites. The spectra are noted in the range of 750 to 2700 cm$^{-1}$. The peak round 2360 cm$^{-1}$ and 1520 cm$^{-1}$ are recognized to C-N stretch and N–O asymmetric stretches the peak at 1040 cm$^{-1}$ is attributed to C–N stretch. FTIR spectra give us the reason that all the compositions have the same functional groups helpful in the confirmation.

3.1.3. Catalysts studies using raman spectroscopy
Raman spectroscopy has been used to depict the carbon deposition on prepared catalyst material before and after CH$_4$ treatment. The synthesized materials were tested in fuel environment (CH$_4$) at 700 °C for 40 min Then tested the raman spectra of Ni$_{0.5}$Zn$_{0.5-x}$Ce$_x$-oxides before and after treatment in CH$_4$ environment is shown in figure 7 respectively. The peaks around 445–460 cm$^{-1}$ attributed due to bending of skeletal vibrations. The peak approximately at 551–557 cm$^{-1}$ resembles to 1 P (1 phonon) oscillations in NiO and bands at 920–925 cm$^{-1}$ are related with 2 P oscillations in Ni-oxide as shown in figures 7(a), (b). On the other hand, Raman D and G bands usually indicates the presence of carbon on the surface of anode materials. However, the effective domain size of the carbon deposits was calculated by measuring the ratio of these two band intensities [24]. For untreated NZC oxide nanocomposites, no D and G bands are found as shown in figure 6(a). However, the significant amount of graphitic carbon was found in all four treated Ni$_{0.5}$Zn$_{0.5-x}$Ce$_x$, Ni$_{0.5}$Zn$_{0.4}$Ce$_{0.1}$, Ni$_{0.5}$Zn$_{0.1}$Ce$_{0.4}$ and Ni$_{0.5}$Zn$_{0.0}$Ce$_{0.5}$ nanocomposite oxides due to the presence of two quite sharp modes, the G peak around 1580–1600 cm$^{-1}$ and the D peak around 1350 cm$^{-1}$ as shown in figure 7(b) [25]. Furthermore, presence of small amount of carbon in Ni$_{0.5}$Zn$_{0.5}$Ce$_{0.2}$ and Ni$_{0.5}$Zn$_{0.2}$Ce$_{0.3}$-oxides are related to other anode material. This was credited to the difference in oxide conductivity and possible effect of water intermediate interactions [26]. The sharps peaks around 457 and 461 cm$^{-1}$ may be
depicted that good stability in crystal structure of prepared anode material as shown in figure 7(b). Based on above results we can concluded that Ni0.5 Zn0.3Ce0.2-oxides has good resistance for carbon deposition as shown in figure 7(b).

3.1.4. Electrical conductivity
The fuel cell performance strongly depends on the conductivity of the material. Nevertheless, maximum conductivity will achieve, if the material has smaller crystalline size. The conductivity also depends on the temperature and porosity of the materials, the greater the porosity of the structure of the material, the higher will be the conductivity. However, from the above all the characterizations of compositions, we have selected two more suitable composition for further study and then it can be decided to choose the more ideal composition. The dc conductivity measurement of selected samples (Ni0.5 Zn0.3Ce0.2-oxides and Ni0.5 Zn0.2Ce0.3-oxides) was measured in the temperature range of 400 °C–700 °C. The Arrhenius plot (1000/T (K) versus ln σ T) of dc conductivity is shown in figure 8 which clearly depicts that the conductivity increases with the increase in temperature. The maximum conductivity achieved was 7 S cm\(^{-1}\) for Ni0.5 Zn0.3Ce0.2-oxides but obtained conductivity for Ni0.5 Zn0.2Ce0.3-oxides was 5.5 S cm\(^{-1}\) because of the reduction in charge carriers i.e., the ratio of Zn\(^{2+}\)/Zn\(_{\text{total}}\) due to increasing cerium content [27].

3.1.5. Fuel cell performance
The fuel cell performance of Ni0.5 Zn0.3Ce0.2-oxides and Ni0.5 Zn0.2Ce0.3-oxides, as anode was characterized using cell structure of NZC/SDC/ LiNiCuZn. The figures 9(a), (b), illustrates the characteristic current-voltage (I-V) and current-power (I-P) curves. The maximum power density of 500 mW cm\(^{-2}\) and open circuit voltage
(OCV) of 1.0 V are obtained at 600 °C for Ni0.5 Zn0.3Ce0.2-oxides as shown in figure 9(b). The peak power density of 220 mW cm$^{-2}$ is also obtained at 600 °C for Ni0.5 Zn0.2Ce0.3-oxide. Thus, it can be recognized that the fuel cell based on Ni0.5 Zn0.3Ce0.2-oxides composition (with very low carbon contents on anode side after testing in CH4 environment) demonstrates remarkably the better catalytic activity and tolerance to carbon deposition as anode material with improved performance even at low temperature.

4. Conclusion

In summary, the Ni0.5 Zn0.5$_x$Ce$_{x}$-oxides (0.1 $\geq$ x $\geq$ 0.5) nano-composites were effectively synthesized by wet chemical method. The x-ay diffraction patterns depicted that all the nano-composites oxides are well crystalline and have multiphase structure. Interesting quantum confinement effect is also depicted by the XRD results, as crystallite size started to decrease from (30–22 nm) and later increases with increasing contents of Ce. It could be explained as change in lattice parameters due to difference in ionic radii of Ni$^+$, Zn$^+$ and Ce$^+$ ions. UV-Visible results revealed that the maximum absorption is found to be in ultraviolet region (200–400 nm) which is promising achievement to form crystalline structure. It can be predictable that Ni0.5Zn0.3Ce0.2-oxides nanocomposite demonstrates remarkably the better catalytic activity and tolerance to carbon deposition on anode side after testing in CH4 environment. The maximum conductivity is 7 Scm$^{-1}$ at 600 °C for Ni0.5Zn0.5Ce0.2-oxides. These results propose that Ni0.5Zn0.5Ce0.2-oxides can be consider as promising anode material for SOFCs.
Acknowledgments

Authors sincerely appreciate the King Saud University for its funding of this research through Researchers Supporting Project (RSP-2019/49)

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