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Structural and Electrochemical Characterisation of NBZFO Cobalt-Free Cathode Material for Intermediate-Temperature Solid Oxide Fuel Cells: An Experimental Investigation

Environmentally friendly energy conversion devices

Lukman Ahmed Omeiza*

Faculty of Integrated Technologies, Universiti Brunei Darussalam, Jalan Tungku Link, Gadong BE1410, Brunei Darussalam

Ukashat Mamudu

Centre for Advance Material and Energy Science, Universiti Brunei Darussalam, Jalan Tungku Link, Gadong BE1410, Brunei Darussalam

Yathavan Subramanian, Anitha Dhanasekaran, Md. Mosfiqur Rahman, Saifullah Abu Bakar, Abul Kalam Azad[§]

Faculty of Integrated Technologies, Universiti Brunei Darussalam, Jalan Tungku Link, Gadong BE1410, Brunei Darussalam

Email: *21h8401@ubd.edu.bn, §abul.azad@ubd.edu.bn

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Compared to other energy-generating technologies and energy conversion devices, intermediatetemperature solid oxide fuel cells (IT-SOFCs) have gained significant attention from energy experts due to its high energy density, moderate operating temperature (600–800°C), low emissions and reliability. Enhancing the performance of IT-SOFCs requires suitable and excellent cathode materials. Thus, a perovskite-type $Nd_{0.5}Ba_{0.5}Zr_{0.8}Fe_{0.2}O_{3+\delta}$ (NBZFO) material was synthesised via traditional solid-state reaction technique and analysed as a potential cathode material for IT-SOFCs. Analysis of X-ray diffraction data (XRD) revealed a singlephase perovskite material that crystallises in cubic space group (pm-3m). The thermal and electrochemical properties were analysed with the aid of thermogravimetric analysis (TGA) and electrochemical impedance spectroscopy (EIS). NBZFO has an electrical conductivity in air of 80 S cm⁻¹ at 400°C and a polarisation resistance (Rp) of 0.106 Ω cm² at 800°C. TGA reveals a slight loss in weight of about 0.58%, thereby suggesting a highly stable cathode material for IT-SOFC. Electrochemical investigation shows that NBZFO has good electronic and ionic conductivity and excellent oxygen stichometry. Further studies are required to understand the effects of varying B-site composition of the cathode material.

Keywords

perovskite, cathode materials, electrical conductivity, oxygen reduction reaction, solid oxide fuel cell

1. Introduction

The SOFC is an environmentally friendly technology that produces electricity through an electrochemical interaction between oxidant and fuel. The three main components that make up SOFCs are the cathode (for oxygen reduction to produce ions), electrolyte (pathway for ions transport) and the anode (for electrochemical reaction of ions with fuel to produce electricity) (1-4). The excellent electrochemical performance of conventional SOFCs is hampered by high operating temperature (800-1000°C), leading to fast degradation of electrode and electrolyte materials. Other issues such as material compatibility and stability are also frequent at high operating temperature of SOFCs (5). Recently, effort has been geared towards reducing the working temperature of SOFCs and the development of IT-SOFCs remains the only viable option towards achieving material stability, reducing the operational cost and ensuring a quick electrochemical process (6, 7). Moderate operating temperature of IT-SOFCs can increase material selection for cathodes and ensure good thermal stability. However, at moderate operating temperature, IT-SOFCs suffer from sluggish cathodic reactions, which can be attributed to low electrochemical performance. Improving the electrochemical performance of electrodes in the cathode region is one of the most essential criteria for enhancing the overall efficiency of IT-SOFCs (8, 9). Thus, cathode materials must possess good ionic-electronic conductivities in order to exhibit sufficient catalytic activity for oxygen reduction reaction (ORR). Figure 1 gives an insight into the working mechanism of SOFCs.

 $La_xSr_{1-x}MnO_3$ (LSM) is one of the most commonly used cathode materials in SOFCs operating at high-temperature (800-1000°C), due to its excellent electrical conductivity and compatibility with yttriastabilised zirconia (YSZ) electrolyte (10). However, due to the low ionic conductivity of this material, the ORR is confined to the region near the triple-phase boundary between LSM and electrolyte YSZ. As a result, the material is unable to provide excellent electrochemical performance within the intermediate temperature range. Perovskite material such as LaCoO₃ doped with strontium (LSC) (11), $La_{1-x}Sr_{x}Fe_{v}Co_{1-v}O_{3}$ (LSCF) (11), $Sm_{1-x}Sr_{x}CoO_{3}$ (SSC) (12) and LaFeO₃ (LSF) (13) have been hugely studied as potential cathode materials for IT-SOFCs owing to their mixed ionic and electronic conductivities. Nevertheless, challenges such as instable, degradable performance and incompatibility with frequent use electrolyte material YSZ need to be resolved. Hence, further studies on high-performance and compatible cathode materials for IT-SOFCs are still very much needed in the sustainable and clean energy sector. Several cobalt-containing cathode materials have been investigated, showing improved electrocatalytic activities for oxygen reduction reaction at intermediate-temperature range. This is mainly due to the fact that cobalt is an excellent catalyst for oxygen activation. A large oxygen vacancy concentration was reported in the study of



Fig 1. Schematic diagram of a traditional SOFCs showing its different components

Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O₃ (BSCF) perovskite material for IT-SOFCs and the electrochemical active site can be extended to the entire porous surface, which makes BSCF cathode material to show higher ORR compared with classical cathode materials like La_{0.8}Sr_{0.2}MnO₃ (LSMO) and SmBaCo₂O_{5+x} (SBCO) (14). Regrettably, cobalt-containing perovskite cathode materials have a high thermal expansion coefficient (TEC) due to cobalt redox flexible behaviour. Moreover, high temperature-dependent coupled valency and spin transition states of cobalt can bring about a symmetry switch from cubic to hexagonal structures during prolonged working periods at high temperatures. This transition is usually complemented by a large deterioration in the transport mechanism. Furthermore, cobalt-synthesised perovskite cathode materials encounter some other challenges such as quick diffusion and evaporation under prolonged use as cathodes in IT-SOFCs. Thus, it is necessary to develop cobalt-free perovskite cathode materials that will exhibit good electrocatalytic potentials for ORR when utilised in IT-SOFCs.

Sakaki et al. (15) revealed that substituting neodymium in the A-site of perovskite cathode materials demonstrates high oxygen exchange credentials, especially in Ln_{1-x}Sr_xMnO₃ and LnSrCoMnO_{6- δ} series. Liu *et al*. (16) also demonstrated that the stability and cell performance drastically improved after substituting of A-site with neodymium. Tsvetkov et al. (17) and Zhu et al. (18) reveal that the introduction of $Ce_{0.8}Sm_{0.2}O_{1.9}$ (doped with cerium) in perovskite cathode materials will lead to an improved oxygen permeability due to the presence of cerate interfacial phase, which was formed during sintering temperature. The interfacial phase brings about structural integrity of the composition by restricting interfacial diffusion of ions. The selection of NBZFO as a cathode material for IT-SOFCs is justified by its distinct composition and properties compared to Mo-doped $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ and $Nd_{0.5}Sr_{0.5}Co_{0.5}Fe_{0.5}O_{3-\delta}$ (18, 19). Substituting cobalt with zirconium and the incorporation of neodymium will enhance specific properties such as ionic conductivity, improved stability and better compatibility within SOFC environments at intermediate temperatures. The presence of zirconium will also contribute to superior ORR kinetics and structural stability, making NBZFO a promising choice for SOFC cathode material. Hence, in this study, we have carried out a brief experimental investigation on newly synthesised cobalt-free perovskite as a potential cathode material for IT-SOFCs. The studied material showed good ionic and electronic conductivities,

low specific area resistance and excellent thermal stability compared to other reported cobalt-free cathode materials for IT-SOFCs. Results obtained indicate that NBZFO is an excellent potential cathode material for IT-SOFCs.

2. Experimental

NBZFO powders were synthesised via the traditional solid-state reaction technique. Stoichiometric amount of commercial precursors of neodymium(III) oxide, barium carbonate, zirconia and ferric oxide was weigh and grounded in an agate mortar with a pestle for 2 h in the presence of acetone as mixing reagent. The obtained dried homogenous mixture of NBZFO was then calcined at 1000°C for 8 h in air with no intermediate grinding. Subsequently, the calcined powder was pressed into pellets (diameter: 20 mm; thickness: 2 mm) at 220 MPa and sintered at 1200°C for 4 h.

The phase and structural makeup of NBZFO fired at 1000°C was analysed using SIEMENS Diffractometer (D5000 Kristalloflex, Cu Ka1, λ = 1.5406 Å) in the range of 20 from 10° to 80° . Data obtained was analysed for phase formation using chekcell software (2). Scanning electron microscopy (SEM) was used to investigate the surface morphology of the cathode material. TGA of NBZFO was performed using ASTM E1131-20 (20) in order to understand the percentage weight loss as a function of temperature. EIS measurements were carried out using PamSens3 Analyser. The frequency scanned was from 1 MHz to 0.05 Hz and set to an amplitude of 0.02 V. The tests were carried out at 600°C and 800°C in air and the cell was investigated without normalising the data with the electrode surface area. Conventional four-probe technique was used to measure the electrical conductivity of the NBZFO on the dense pellet sintered at 1200°C and the linear thermal elongation/thermal expansion coefficient (TEC) was investigated with the aid of a dilatometer.

3. Results and Discussion

3.1 Phase and Structural Analysis

Figure 2 shows the XRD profile of NBZFO obtained at room temperature. The peak positions of NBZFO correspond to Joint Committee on Powder Diffraction Standards (JCPDS) file 01-074-1299 and can be indexed as Pm-3m. **Table I** summarises the results of the cell parameters obtained *via* chekcell software. A cubic (pm-3m) perovskite structure was formed when NBZFO was calcined at 1000°C for 8 h. The structure obtained is consistent with the cubic-phase perovskite oxides reported in the literature (21). Diligent analysis of the XRD profile reveals a sharp and well-defined peak position at 20 angles, which can be attributed to the crystallinity nature of NBZFO (22, 23).

3.2 Thermogravimetric Analysis and Linear Thermal Expansion Investigation

Figure 3 illustrates the temperature dependency of percentage weight changes of NBZFO powder in air. TGA of NBZFO shows that synthesised cathode material is highly stable, with negligible total weight loss of about 0.58% between 0°C to 800°C. This weight loss can be attributed to the evaporation of moisture from NBZFO and the formation of oxygen vacancies as temperature rises from 20°C to 800°C. At around 280°C, the TGA curve begins to steeper significantly, with negligible weight loss of about 0.44%, which is primarily due to the evaporation of moisture from the sample (24). The slight loss in weight continues with an increase in temperature due to the loss of oxygen lattice and the formation of oxygen vacancy, in conjunction with the reduction of iron (Fe^{4+} to Fe^{3+}) that occurs at around 800°C (25). As the temperature rises from 300°C to 800°C, approximately 0.14% weight



Fig 2. XRD profile of NBZFO at room temperature

of NBZFO is lost. However, as the temperature rises above 800°C, the weight loss increases drastically and the oxygen distribution remains constant at a relatively low weight loss. The weight loss due to an increase in temperature from 20°C to 280°C is denoted as ΔW_1 and between 280°C to 800°C is denoted as ΔW_2 , respectively. The weight loss above 800°C was not taken into consideration as IT-SOFCs work within a temperature range of 600°C to 800°C.



Fig 3. NBZFO weight change as a function of temperature



Fig 4. The linear thermal elongation curve of NBZFO measured in air at 30–800 $^{\circ}\mathrm{C}$

Table I Space Group, Cell Parameters and Symmetry of Synthesised NBZFO						
Chemical composition	Space group	Symmetry	a, Å	b, Å	c, Å	V, Å
$Nd_{0.5}Ba_{0.5}Zr_{0.8}Fe_{0.2}O_{3+\delta}$	pm-3m	Cubic	4.177(5)	4.177(5)	4.177(5)	72.89(14)

The TEC of NBZFO was investigated with the aid of a dilatometer, which showed a favourable average TEC of 7.2 \times 10⁻⁶ K⁻¹ in comparison to those reported in other studies (26-28). Figure 4 illustrates the linear thermal elongation analysis of NBZFO in air, at 30–800°C. Based on the obtained profile, a sharp inflation point was noticed at around 550°C, which can be attributed to the reduction of Fe^{4+} to Fe^{3+} (29). Similar plots were obtained in the investigation of $LaFe_{0.8}Cu_{0.2}O_{3-\delta}$ and $BaFe_{0.8}Cu_{0.2}O_{3-\delta}$, which the authors also attributed to the reduction of Fe⁴⁺ to Fe³ driven by the thermal release of lattice oxygen, as found in several perovskite oxides (30). The average TEC of 7.2 \times 10⁻⁶ K⁻¹ obtained for NBZFO is less than the values of $11-13 \times 10^{-6} \text{ K}^{-1}$ obtained for commonly used electrolytes (yttria-stabilised zirconia and gadolinium-doped ceria) in IT-SOFCs applications. Change in length is denoted as ⊽L and ∇T represents the change in temperature. The linear TEC is depicted by a in the plot.



Fig 5. (a) Electrochemical impedance profile of NBZFO|YSZ|NBZFO; (b) electrical conductivity in air of NBZFO as a function of temperature

3.3 Electrochemical Investigation

Figure 5(a) illustrates the electrochemical impedance profile of the NBZFO cathode obtained at 600°C and 800°C in air. The electrochemical performance assessment was carried out using symmetrical cell configuration NBZFO|YSZ|NBZFO. Impedance response for ORR on the NBZFO cathode was characterised by high and low frequencies. This observation signifies that the ORR occurring at the electrode interface involves a minimum of two distinct processes. Figure 5(b) illustrates the electrical conductivity in air of NBZFO as a function of temperature. Notably, a distinct change in the conductivity slope occurs at around 400°C, transitioning from a semiconducting-like conduction pattern to a metallic-like conduction pattern (31). At this temperature, the electrical conductivity reaches its peak, attaining a maximum value of 80 S cm⁻¹. Subsequently, as the temperature continues to rise beyond 400°C, the sample's conductivity experiences a decline. This reduction in conductivity is predominantly attributed to the loss of lattice oxygen at high temperatures (32). At intermediate-temperature range (600-800°C), the NBZFO sample exhibits conductivity values ranging from 38 S cm⁻¹ to 54 S cm⁻¹. It is noteworthy that these values surpass the maximum conductivity observed for $SrCo_{0.8}Fe_{0.1}Ga_{0.1}O_{3-\delta}$ -SDC at 600°C (33). Table II summarises the electrical conductivity of other reported studies on cathode materials for SOFCs.

In order to obtain the Rp, the impedance profile was subsequently analysed using Zview software, which revealed that the cathode material has low resistance at the electrode-electrolyte interphase. **Table III** provides a comparison overview of the Rp obtained for NBZFO and those reported in other cobalt-free cathode materials investigations. Lower polarisation resistance leads to reduced overpotentials, which means the cell can operate more efficiently and also translate to higher voltage

Table II Electrical Conductivity of Related Cathode Materials for Solid Oxide Fuel Cells				
Composition	Electrical conductivity, S cm ⁻¹	Temperature, °C	Ref.	
$PrBa_{0.5}Sr_{0.5}Co_{1.5}Fe_{0.5}O_{5+\delta}$	265	600	(34)	
$BaFe_{0.7}Co_{0.2}Nb_{0.1}O_{3-\delta}$	2.59	800	(35)	
$Pr_2Ni_{0.85}Cu_{0.1}Al_{0.05}O_{4+\delta}$	55	800	(36)	
PrBa _{0.92} CoCuO _{6-δ}	155	800	(37)	
La _{0.6} Ca _{0.4} Fe _{0.8} Ni _{0.2} O ₃	22	800	(38)	
$NdBaMn_2O_{5+\delta}$	0.75	850	(39)	
$La_{0.595}V_{0.005}Sr_{0.4}CoO_{3-\delta}$	843	400	(40)	

Table III Polarisation Resistance of Other Reported Cobalt-Free Cathode Materials				
Composition	Polarisation resistance, Ω cm ²	Temperature, °C	Ref.	
Cu _{1.4} Mn _{1.6} O ₄	0.143	800	(41)	
Bi _{0.5} Sr _{0.5} FeO _{3-δ}	0.038	700	(42)	
$Bi_{0.5}Sr_{0.5}Fe_{0.8}Cu_{0.2}O_{3-\delta}$	0.13	700	(43)	
$PrLa_{0.4}Ba_{0.6}Fe_{0.8}Zn_{0.2}O_{5+\delta}$	0.044	750	(44)	
$La_{0.4}Bi_{0.4}Sr_{0.2}FeO_{3-\delta}$	0.1	700	(45)	
$SrFe_{0.8}W_{0.2}O_{3-\delta}$ -SDC	0.036	700	(46)	



and power output. **Figure 6** shows the Rp pattern of NBZFO. The PR was observed to decrease with an increase in temperature for the symmetrical cell configuration NBZFO|YSZ|NBZFO. At 800°C, the Rp was found to be 0.106 Ω cm².

SEM was used to investigate the morphology of NBZFO and the result obtained showed a highly porous cathode material with a pore size of $2\ \mu\text{m}.$ This result is in agreement with various reported studies on cobalt-free cathode materials (41, 47, 48). Generally, pore size is not the only determining factor for a good cathode material, but PR and excellent electrical conductivity are of utmost importance in selecting cathode materials for IT-SOFC. Figure 7 gives an insight into the cross-sectional morphological analysis of NBZFO sintered at 900°C with dense electrolyte (YSZ). The good interface lamination indicates good thermal expansion compatibility between the electrode (NBZFO) and electrolyte (YSZ). The interface of NBZFO-YSZ indicates good adhesion and crack-free. It is evident from the surface morphology, that NBZFO is highly porous and contains uniformly distributed particles. The even distribution of particles and good porosity creates



Fig 7. Cross-sectional surface analysis of NBZFO with commonly used YSZ showing good interfacial adhesion

a network of interconnected pores within the cathode material. This allows for efficient oxygen diffusion through the cathode, ensuring that oxygen can readily reach the electrochemically active sites where the ORR occurs. Improved oxygen diffusion leads to better overall cell performance.

4. Conclusion

NBZFO was synthesised *via* the traditional solidstate reaction method and investigated as a possible cathode material for IT-SOFCs. The phase analysis of XRD data of NBZFO revealed a cubic space group symmetry, which was indexed as pm-3m. TGA of the synthesised cathode material revealed negligible weight loss of 0.58% between 0°C to 800°C. The electrical conductivity of NBZFO in air was maximum at 415°C, with a value of 80 S cm⁻¹ and decreased with an increase in temperature to about 38 S cm⁻¹ at around 800°C. This was attributed to the loss of oxygen content in the lattice at high temperatures. NBZFO showed promising electrochemical properties, with a Rp of 0.106 Ω cm² at 800°C. Overall, low Rp in cathode materials is a critical factor in achieving efficient and durable IT-SOFCs, making them more attractive for a wide range of applications, including stationary power generation and portable electronics. The favourable TEC of NBZFO shows that this material has good thermal compatibility with commonly used electrolytes. Cobalt-free NBZFO cathode also has the potential to reduce the high operating costs associated with its cobalt-containing counterpart, enhancing cell performance, stability and durability, thereby ensuring prolonged cell life and improved efficiency. Thus, primary finding on NBZFO shows good potential as cathode materials in IT-SOFCs. Future work will include the varying of B-site composition and doping of A-site and a comprehensive electrochemical characterisation.

Glossary

BSCF	$Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_3$	Rp	polarisation resistance
EIS	electrochemical impedance spectroscopy	SEM	scanning electron microscopy
IT-SOFCs	intermediate-temperature solid oxide fuel cells	SSC	Sm _{1-x} Sr _x CoO ₃
LSMO	La _{0.8} Sr _{0.2} MnO ₃	SBCO	SmBaCo ₂ O _{5+x}
LSCF	$La_{1-x}Sr_xFe_yCo_{1-y}O_3$	SOFC	solid oxide fuel cell
LSC	LaCoO ₃	TEC	thermal expansion coefficient
LSF	LaFeO ₃	TGA	thermogravimetric analysis
LSM	$La_xSr_{1-x}MnO_3$	XRD	X-ray diffraction data
NBZFO	$Nd_{0.5}Ba_{0.5}Zr_{0.8}Fe_{0.2}O_{3+\delta}$	YSZ	yttria-stabilised zirconia
ORR	oxygen reduction reaction		

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The Authors



Lukman Ahmed Omeiza is a PhD candidate in Energy Systems Engineering at the Faculty of Integrated Technologies, Universiti Brunei Darussalam. His main research focus is on developing new, novel electrode materials for energy conversion and storage devices. He has also been involved in research studies focused on the development of nanofluids for solar thermal applications, building energy systems and thermal management.



Ukashat Mamudu received his PhD in Material Science from the Centre for Advanced Material and Energy Sciences (CAMES), Universiti Brunei Darussalam with a focus on green material synthesis and characterisation for the fabrication of anticorrosion coatings for corrosion protection of metallic infrastructure. During his PhD, he worked on the extraction of cellulose nanocrystals and also performed surface functionalization of cellulose nanocrystals for anticorrosion application. He spent six months under Professor El-Sayed M. Sherif at the Center of Excellence for Research in Engineering Material (CEREM), King Saud University, Saudi Arabia synthesising novel nanocomposite inhibitors for corrosion protection.



Yathavan Subramanian is a PhD candidate specialising in Energy Systems Engineering at the Faculty of Integrated Technologies, Universiti Brunei Darussalam. He completed his BE and MS (by research) in Mechanical Engineering at Anna University, Chennai, India. His research focuses on solar fuel generation and pollutant removal *via* photocatalysis, as well as the development of novel electrode materials for batteries. Yathavan has published numerous research and review articles, contributing significantly to the fields of energy conversion and storage.



Anitha Dhanasekaran is a PhD candidate specialising in Energy Systems Engineering at the Faculty of Integrated Technologies, Universiti Brunei Darussalam. She completed her BE in Mechanical Engineering and ME in Manufacturing Engineering at Anna University, Chennai, India. Her current research focuses on the application of metal oxide-based nanofluids for battery thermal management systems in electric vehicles through modelling approaches. She also specialised in the preparation of novel electrolyte materials for protonic ceramic fuel cells. Anitha has published numerous research and review articles in reputed journals.



Md. Mosfiqur Rahman is a PhD Research Fellow in the Department of Chemical Engineering at Universiti Brunei Darussalam. His research focuses on numerical modelling and the development of materials for solid oxide fuel cells (SOFCs). The aim of the work is to advance the efficiency and application of SOFC technology through innovative material solutions and computational approaches.



Muhammad Saifullah bin Abu Bakar obtained his PhD in Chemical Engineering from Aston University in Birmingham, UK, in 2014 with a thesis titled 'Catalytic Intermediate Pyrolysis of Brunei Rice Husk for Bio-Oil Production'. He graduated with a BEng (Hons) in Chemical Engineering in 2008, and an MSc in Chemical Process Technology with Polymer Processing in 2009, also from Aston University. Completed research projects include reactor design for the oxidative dehydrogenation of butenes to 1,3-butadiene and power generation from sewage sludge *via* combustion or pyrolysis/gasification.



Abul Kalam Azad works at the Faculty of Integrated Technologies, Universiti Brunei Darussalam. Before this, he worked with Professor John T. S. Irvine at the University of St Andrews, UK, on SOFC materials development. He obtained his PhD at the University of Gothenburg, Sweden under the supervision of Professor Sten G. Eriksson on materials preparation and characterisation for magnetic memory applications. He graduated in Physics from Jahangirnagar University, Bangladesh.