



EFFECT OF Nd_2O_3 AND Fe_2O_3 ADDITION ON GADOLINIA DOPED CERIA (GDC) SOLID ELECTROLYTE SYSTEM FOR INTERMEDIATE TEMPERATURE-SOLID OXIDE FUEL CELL (IT-SOFC) APPLICATIONS

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Abstract

GDC (gadolinia doped ceria) is a solid electrolyte contender for intermediate-temperature SOFCs (solid oxide fuel cell). However, more development of this solid electrolyte is required to improve its ionic conductivity. We will investigate the effect of Nd_2O_3 and Fe_2O_3 addition on GDC solid electrolytes to boost ionic conductivity. Solid electrolytes of the composition $\text{Ce}_{0.9}\text{Gd}_{0.2}\text{M}_x\text{O}_{1.9}$ ($M = \text{Nd}, \text{Fe}$) ($x = 0\% ; 2.5\% ; 5\%, \text{ and } 7.5\%$) were synthesized using mixed oxide method and formed into pellets with a diameter of 1 cm. The pellets were sintered at 1200 °C and 1400 °C for 4 hours in an Argon environment then the EIS (electrochemical impedance spectroscopy) test was performed at 450-650°C. The results showed that the Nd_2O_3 and Fe_2O_3 added were totally dissolved in the ceria structure and produced single-phase cubic fluorite CeO_2 . GDC solid electrolyte with Fe_2O_3 addition produces higher densification than Nd_2O_3 addition, where the value reaches 75% in the GDC sintered at 1400 °C. However, the addition of Nd_2O_3 further increased the value of ionic conductivity and decreased the activation energy of the GDC solid electrolyte compared to the addition of Fe_2O_3 . The highest ionic conductivity and the lowest activation energy were obtained in the GDC with 2.5% Nd_2O_3 in 650 °C operating temperature, with the values achieved were 1.2 mS/cm and 0.41 eV, respectively. Therefore, it can be concluded that Nd_2O_3 addition is more effective to improve the performance of solid electrolyte GDC.

Keywords: SOFC (solid-oxide fuel cell), solid electrolyte, GDC (gadolinia doped ceria), ionic conductivity

1. INTRODUCTION

SOFC (solid oxide fuel cell) is a type of fuel cell or electrochemical device that can transform chemical energy from a fuel into electrical energy and heat without using a combustion stage [1]. SOFCs have several advantages over other fuel cells, including higher electrode corrosion resistance, higher fuel flexibility, and high power efficiency (40-60%) [2]. However, SOFC still has several operational issues, particularly the high operating temperature (1000 °C). The high operating temperature causes many problems in its operation, such as rapid material degradation, making it less economical [3]. Therefore, the current research focuses on lowering the operating temperature of SOFCs while maintaining good performance quality.

One effort that may be made is choosing the appropriate solid electrolyte material. The solid electrolyte must have a high ionic conductivity to be used at an intermediate temperature (400-700 °C) SOFC or IT-SOFC [4]. YSZ (Yttria stabilized zirconia) is a solid electrolyte commonly utilized in commercial SOFCs that operate at 1000 °C [5]. Nevertheless, the ionic conductivity value of YSZ diminishes in the medium operating temperature range, preventing it from being employed on IT-SOFC [6]. GDC (gadolinia doped ceria) has the potential to be employed as a solid electrolyte in IT-SOFC because it has sufficient ionic conductivity, low activation energy, and higher catalytic activity at medium temperatures [7]. However, further

development of this solid electrolyte is required to improve its ionic conductivity.

According to Herle et. al., [8], the addition of double doping (co-doping) with two cations can enhance the ionic conductivity of a ceria-based solid electrolyte compared to single doping. Co-doping to the GDC solid electrolyte can enhance ionic conductivity and stability by preventing partial reduction of the GDC solid electrolyte [9]. According to Andersson et. al., [10], the optimal dopant for a ceria-based solid electrolyte is Promethium (Pm) (atomic number 61). Based on modeling with vienna ab initio, the addition of Pm to the ceria matrix will result in the lowest vacancy formation energy and binding energy between defects compared to other elements. Consequently, the activation energy value of the ceria-based solid electrolyte system will be lower. On the other hand, Pm is a synthetic element that does not exist in nature. As a result, the most likely approach is to use elements with atomic numbers near to 61, or more commonly, rare earth metal oxides. This is because the binding energy is a function of the atomic number. Hence the atomic number of the dopant element should be as close to Pm as possible. Neodymium (atomic number 60) is an element that can be used for co-doping of ceria-based solid electrolytes because it has an atomic number close to Pm. This co-doping aims to enhance the vacancy concentration in cubic fluorite ceria, hence increasing the ionic conductivity[11]. Apart from doping, various research has investigated the addition of a sintering aid to the GDC solid electrolyte to improve the densification [12]. With increasing densification, it is expected that the ionic conductivity will also increase. One type of sintering aid that is widely used is transition metal oxides such as Fe_2O_3 [13].

In this study, the effect of the addition of rare earth metal oxides (Nd_2O_3) and transition metal oxides (Fe_2O_3) on the physical and electrical characteristics of GDC solid electrolyte will be compared to determine the more effective material to improve the performance of solid electrolytes.

2. MATERIALS AND METHODS

Solid electrolytes with compositions $\text{Ce}_{0.9}\text{Gd}_{0.2}\text{Nd}_x\text{O}_{1.9}$ and $\text{Ce}_{0.9}\text{Gd}_{0.2}\text{Fe}_x\text{O}_{1.9}$ ($x = 0, 2.5\%, 5\%$ and 7.5%) were synthesized using CeO_2 , Gd_2O_3 , Nd_2O_3 , and Fe_2O_3 precursor with high purity (99.9%). All powders were mixed in 96% ethanol media. The ethanol-powder mixture was heated to 70°C on a hotplate and stirred using a magnetic stirrer until all of the ethanol had evaporated and there was a powder deposit at

the bottom of the beaker. Then the powder was dried in an oven at 150°C for 1 hour. The powder was then ground using a mortar with a PVA binder of 3 wt.% and 70% alcohol. After grinding, the powder was pressed into a 1 cm thick green pellet using a hydraulic compacting machine with a pressure of 40 kN. The pellets were then sintered in a tube furnace (Thermolyne) at two temperature variations of 1200°C and 1400°C for 4 hours in an Argon environment. Then several tests were carried out on the sintered pellets, including EIS (electrochemical impedance spectroscopy), XRD (x-ray diffraction), and SEM (scanning electron microscope) tests.

EIS test was carried out using a potentiostat (Corrtest) with silver paste as the standard electrode and current collector at a temperature of $450\text{--}650^\circ\text{C}$ with a frequency range of 100 kHz-0.1 Hz using an AC signal of 20 mV. The Nyquist and Bode curves obtained from the EIS test are then fitted with an equivalent circuit model using ZView software to bring the grain (R_g) and grain boundaries (R_{gb}) resistance of solid electrolytes. The total resistance (R_{total}) value can be calculated by Equation (1), and then can be used to determine the ionic conductivity and activation energy value.

$$R_{total} = R_g + R_{gb} \quad (1)$$

3. RESULT AND DISCUSSION

3.1 Phase Analysis

Figure 1 shows the diffraction pattern of the solid electrolyte of GDC (gadolinia doped ceria) with the addition of Nd_2O_3 (Nd-GDC) and Fe_2O_3 (Fe-GDC). The diffraction pattern of the three GDCs has the same pattern where the peaks are identified as planes (111), (200), (220), etc.

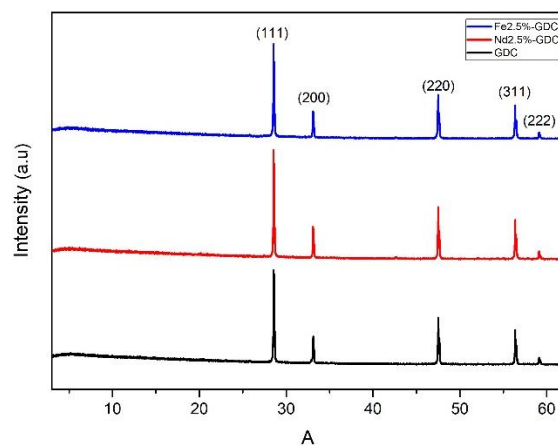


Figure 1. X-ray diffraction pattern of GDC, Nd2.5%-GDC, and Fe2.5%-GDC sintered at 1200°C

These peaks indicate that the structure generated in the three GDCs is single-phase cubic

fluorite CeO_2 [14] (JCPDS no. 34-394), and Gd_2O_3 , Nd_2O_3 , and Fe_2O_3 doping have been entirely dissolved into the ceria structure (CeO_2). Adding Gd^{3+} , Nd^{3+} , and Fe^{3+} to the ceria matrix results in substituting of Ce^{4+} ions, forming a solid solution [15]. This ion substitution changes the lattice parameter of cubic fluorite ceria due to the difference in ionic radii between Ce^{4+} (0.097 nm) as the host atom with Gd^{3+} (0.105 nm), Nd^{3+} (0.110 nm), and Fe^{3+} (0.069 nm) as doping atom [16].

3.2 Densification and Microstructure Analysis

Densification is a powder shrinkage process that occurs during the sintering process. Densification is observed with % relative density or by comparing the density of the sintered product with its theoretical density.

Figure 2 shows the relative density of solid electrolyte GDCs sintered at 1200 °C and 1400 °C. Based on the graph, the densification increased up to concentration of 2.5%, and then decreased with further increases in the co-doping concentration. Densification increases owing to mass transfer during the sintering process, which can remove pores in the solid electrolyte system. Furthermore, the rise in densification was driven by changes in the ceria lattice properties due to dopant addition. However, densification decreases as the concentration exceed 2.5%. The decrease in densification was caused by an excessive co-doping concentration, which causes more Nd^{3+} or Fe^{3+} atoms to substitute Ce^{4+} atoms, resulting in excessive lattice distortion [17].

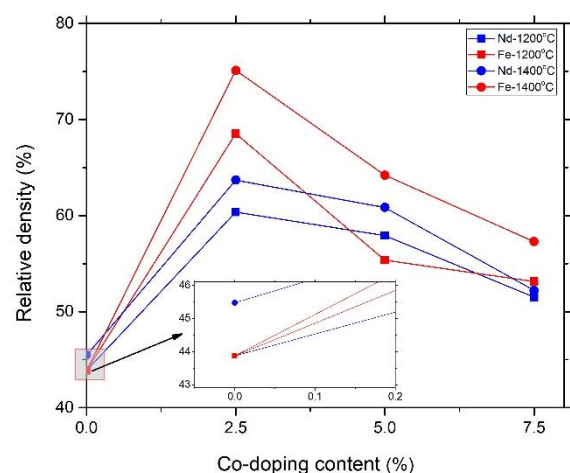


Figure 2. Relationship of co-doping concentration with relative density at sintering temperatures of 1200 °C and 1400 °C

Lattice distortion occurs along with changes in lattice parameters due to differences in radii and static forces between atoms. In GDC solid electrolyte, adding Fe_2O_3 at both 1200 °C and 1400 °C sintering temperatures results in higher

densification than adding Nd_2O_3 . This is because Fe_2O_3 acts as a sintering aid that can accelerate densification kinetics during the sintering process [18]. The highest densification (75%) was found in the GDC with the addition of Fe_2O_3 2.5%, which was sintered at 1400 °C.

The increases in the densification of the GDC with the addition of Fe_2O_3 were caused by viscous flow sintering occurred that can accelerate the densification kinetics [19]. Also, Figure 2 indicates that the densification will increase as the sintering temperature rises. At a higher sintering temperature, the atoms in the GDC get more energy to diffuse and form denser granules. However, if the sintering temperature is too high, the grain size will increase, which causes poor mechanical properties of the solid electrolyte.

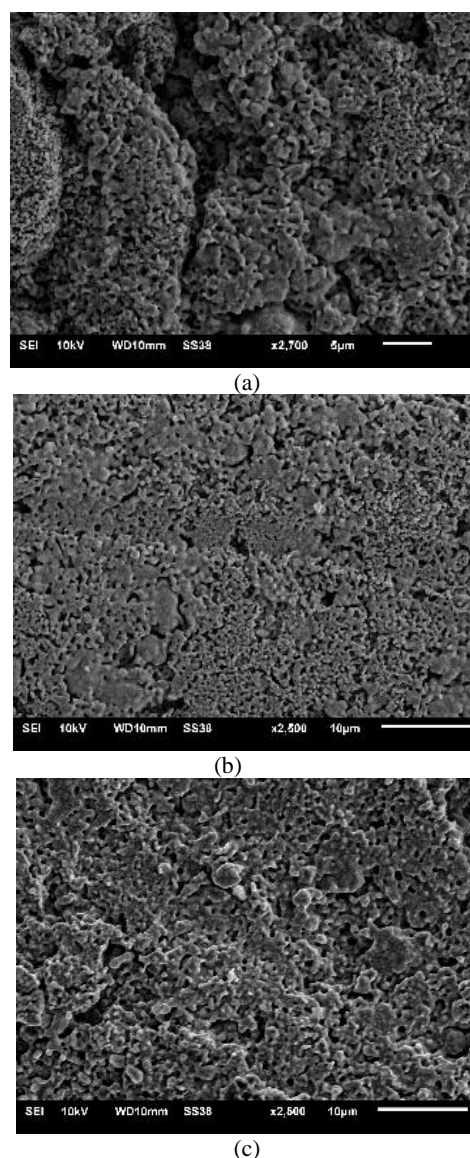


Figure 3. Microstructure of (a) GDC, (b) Nd-GDC, and (c) Fe-GDC sintered at 1400 °C and at 2.5% co-doping concentration

Figure 3 shows the microstructure of the GDC solid electrolyte GDC with Nd_2O_3 and Fe_2O_3 addition. In the microstructure of the GDC, large agglomerates were formed, which still have many pores and large grain sizes ($1.227\mu\text{m}$). The agglomerates disappeared when Nd_2O_3 was introduced to the GDC, producing finer granules with smaller grain sizes ($0.642\mu\text{m}$). This is consistent with Fig. 2, demonstrating that the GDC densification with Nd_2O_3 has a more excellent value than the GDC. According to studies by Priyadharsini et. al., [20], the presence of Nd in the ceria lattice could inhibit the agglomeration process, resulting in less aggregation of elements in specific places and smaller grain size. The microstructure generated in the GDC with the addition of Fe_2O_3 has fewer pores and a comparatively smaller grain size ($0.906\mu\text{m}$). In the Fe-GDC, it was also observed that the wet grain boundary cluster or the wetted grain boundary indicated the occurrence of viscous flow sintering [21]. This process occurs because of the liquid phase formed by the melting of the sintering aid. The liquid phase will fill the pores with capillary forces, which will then be reprecipitated at the final stage of sintering so that densification will increase.

3.3 Ionic Conductivity Analysis

Ionic conductivity is a quantity that expresses the ease of a material to transport ions. Ionic conductivity is determined by Equation (2), where σ is total ionic conductivity (S/cm), R_{total} is total resistance of solid electrolyte (Ω), L is thickness of solid electrolyte (cm) and A is area of solid electrolyte (cm^2).

$$\sigma = \frac{1}{R_{total}} \times \frac{L}{A} \quad (2)$$

The total resistance of a solid electrolyte is the sum of the grain and grain boundary resistance. The grain and grain boundary conductivity can be determined to identify the contribution of grain and grain boundaries to the total conductivity. Grain and grain boundary conductivity can be calculated using the same equation shown in Equation (2), but the resistance value used is the resistance of each grain and grain boundary. Figure 4 shows the contribution of grain and grain boundaries to the total ionic conductivity of solid electrolytes GDC at an operating temperature of 450°C . From Figure 4, it can be seen that the conductivity of the grain was larger than those of the grain boundary. Variances caused this variation in conductivity the concentration of vacancy within the grains and grain boundaries. The vacancy concentration

inside the grains was more significant than the grain boundaries, resulting in a higher conductivity [22]. However, the grain boundary conductivity has a value and trend nearly identical to the total conductivity, implying that the grain boundary contribution was more prominent than the grain contribution to the total conductivity. It can also be explained by the microstructure shown in Fig. 3, where the GDC solid electrolyte with Nd_2O_3 and Fe_2O_3 addition has a tiny grain size, resulting in more grain boundaries than grains. Therefore, the contribution of grain boundaries is more dominant than grains.

At 5% co-doping concentration, there was a data anomaly. The grain boundary conductivity and total ionic conductivity of the GDC with Fe_2O_3 addition were higher than the GDC with Nd_2O_3 addition. This occurs because the grain boundary resistance in the GDC with Fe_2O_3 addition is lower than in the GDC with Nd_2O_3 addition, resulting in increased grain boundary conductivity.

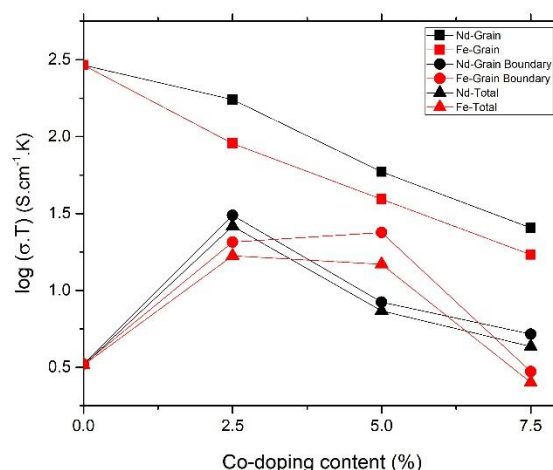


Figure 4. Grain and grain boundary contribution to total conductivity of GDCs sintered at 1200°C and at 450°C operating temperature

Figure 5 shows the relationship of co-doping a concentration with the total ionic conductivity. Ionic conductivity increases with the addition of Nd_2O_3 and Fe_2O_3 up to concentration of 2.5% and then decreases with further concentration increases. The decrease in ionic conductivity occurs because the density of the solid electrolyte drops at concentrations above 2.5%, as discussed in sub-section 3.2. It causes the mobility of oxygen ions to be slower, so the ionic conductivity decreases. Furthermore, high co-doping concentrations, it causes defects to interact with one another. Oxygen vacancy (V_{O}^{2+}) would interact with other charged species and

produce a cluster defect, which causes the oxygen vacancy to be immobile [23]. Figure 6 compares the effect of adding Nd₂O₃ and Fe₂O₃ at 2.5% concentration and various operating temperatures on the total ionic conductivity. The highest ionic conductivity value obtained in this study was 1.2 mS/cm with the addition of 2.5% Nd₂O₃ at an operating temperature of 650 °C. This value is lower than the research conducted by Dikmen et.al., [24] and Kim et.al., [25], obtained ionic conductivity of 4.9 mS/cm and 4.3 mS/cm. This value is lower than the two previous studies because the two previous studies used a higher sintering temperature and a longer sintering time so that fewer pores were produced. The fewer pores cause the ionic conductivity to be higher than in this study.

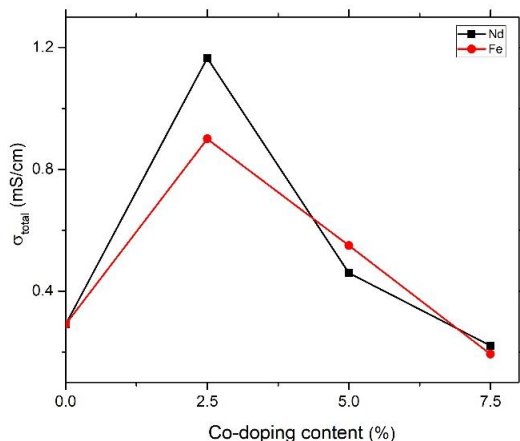
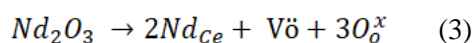


Figure 5. Relationship of co-doping concentration with total ionic conductivity

The ionic conductivity of the GDC with the addition of Nd₂O₃ has a higher value than the GDC with the addition of Fe₂O₃. The increase in ionic conductivity in the Nd-GDC was due to the rise in the vacancy concentration. When Nd₂O₃ is introduced, Nd³⁺ will replace Ce⁴⁺, forming an oxygen vacancy defect [22]. The formation of this oxygen vacancy is written with the Kröger-Vink notation shown in Equation (3). The more oxygen vacancies formed, the easier the mobility of oxygen ions in the solid electrolyte so that the ionic conductivity increases.



The maximum ionic conductivity of the GDC with the addition of Fe₂O₃ was achieved at a concentration of 2.5%, which reached 0.89 mS/cm at an operating temperature of 650 °C. This value is greater than the previous study by Dong et.al., [26], which found 6×10⁻² mS/cm of ionic conductivity. Furthermore, the Fe-GDC

exhibits a higher ionic conductivity value than the Nd-GDC because Fe-GDC solid electrolytes have higher densification than GDC. Nevertheless, the addition of Fe₂O₃ is not directly related to the formation of oxygen vacancies, so the ionic conductivity value is lower than the Nd₂O₃ addition. Therefore, it can be concluded that Nd₂O₃ is the more effective material for increasing the ionic conductivity of solid electrolyte GDC compared to Fe₂O₃.

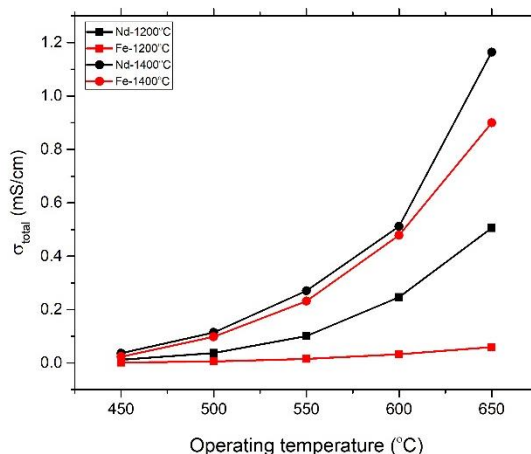


Figure 6. Comparison of the addition of 2.5% Nd₂O₃ and 2.5% Fe₂O₃ to the total ionic conductivity at various operating temperature

This also validates the research by Andersson et.al., [10] described in the introduction, where the atomic number of Nd is closer to the atomic number of Pm.

3.4 Activation Energy Analysis

The ionic conductivity of the solid electrolyte is a function of the operating temperature. The dependence of ionic conductivity with operating temperature is usually shown by the Arrhenius plot, which can be seen in Fig. 7. As shown in Figure 7, generally, the ionic conductivity increases with increasing operating temperature because of the decrease in the total resistance of the solid electrolyte [27]. This is also consistent with the data shown in Fig. 6. As the operating temperature rises, the mobility of oxygen ions will increase, causing the ionic conductivity value to increase [18].

The activation energy of a solid electrolyte system can be calculated from the Arrhenius plot using Equation (3), where σ is total ionic conductivity (S/cm), σ_0 is a pre-exponential factor (S.K/cm), T is operating temperature (K), k is Boltzman constant, and E_a is activation energy (eV).

$$\sigma T = \sigma_0 \exp\left(-\frac{E_a}{k.T}\right) \quad (3)$$

Figure 8 shows the relationship of co-doping concentration to the activation energy of the

GDC solid electrolyte system. In general, adding of Nd_2O_3 and Fe_2O_3 can reduce the activation energy of the solid electrolyte system. The lowest activation energy was achieved at a co-doping concentration of 2.5%.

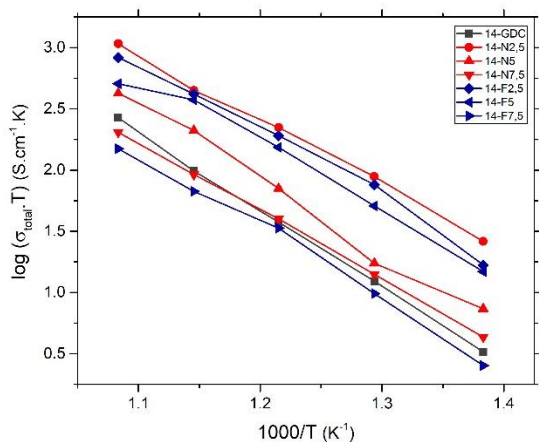


Figure 7. Arrhenius plot of the GDC sintered at 1400 °C

The GDC adding of Nd_2O_3 had a lower activation energy value than the GDC with the addition of Fe_2O_3 . The activation energy of the GDC with the addition of 2.5% Nd_2O_3 reached 0.41 eV, while the addition of Fe_2O_3 reached 0.45 eV. These values have met the requirements for SOFC applications, which are below 1 eV [7]. The decrease in activation energy indicates that the energy required to release oxygen ions from the crystal lattice and ion migration becomes smaller so that the ionic conductivity increases [27].

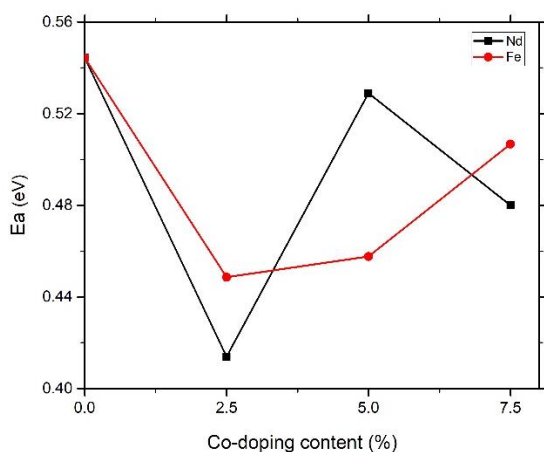


Figure 8. Activation energy of the GDC solid electrolyte

4. CONCLUSION

GDC (gadolinia doped ceria) solid electrolytes with Nd_2O_3 and Fe_2O_3 addition was successfully synthesized by mixed-oxide method. Based on the findings in this study, Nd_2O_3 and Fe_2O_3 added were dissolved in the ceria structure and produced single-phase cubic fluorite CeO_2 . The highest densification, up to 75%, was obtained in the GDC solid electrolyte with Fe_2O_3 addition.

The highest ionic conductivity and the lowest activation energy were obtained in the GDC with 2.5% Nd_2O_3 in 650°C operating temperature, with the values achieved were 1.2 mS/cm and 0.41 eV, respectively. It can be concluded that the addition of Nd_2O_3 is more effective than the addition of Fe_2O_3 in enhancing the ionic conductivity of GDC solid electrolyte and can lower the activation energy. The improvement in ionic conductivity is likely to improve SOFC cell performance at intermediate operating temperatures.

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