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Perovskite Type Lanthanum Oxide and Graphene Oxide ($LaFeO_3$-GO) Composite as (Low Overpotential) ORR Catalyst for Low and High Temperature Fuel Cells

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ABSTRACT

Due to depletion of fossil fuel and their environmental effect, renewable resources are the prime interest in which fuel cell technology competes with fossil fuels but their ORR at cathode restrict its efficiency. Perovskite type orthorhombic structure of $LaFeO_3$ incorporated GO has ability to use as catalyst in low and high temperature fuel cells having non-agglomerated particles in nanometers range (20- 100), high temperature stability, low overpotential of about 0.6V, ECSA of about 13.22 m^2g^{-1} and current density of about 0.012 $mAcm^{-1}$. Results open further research directions in supercapacitors, batteries, biological fuel cells, and applications which requires high temperature stability.

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1. Introduction

Environmental Pollution and global warming both are the main causes of increasing earth's atmosphere and millions of deaths every year(Fuller et al., 2022). According to (wen Ju et al.) globalization is undeniably connected with CO_2 discharge intensified by non-renewable energy resources like fossil fuels by producing 21.3 billion tons of CO_2 every year(Saleem, 2022; Wen et al., 2021). Fossil fuel reserves are diminishes day by day prompting the utilization of sustainable sources (water, sun, wind, biomass, geothermal, hydrogen) and innovations and a possibility in contrast to standard wellspring of energy like fuel cells (Boudghene Stambouli & Traversa, 2002).

Fuel cell is an electrochemical device converts chemical energy into electrical energy. Fuel cell is an environmentally friendly, clean, green technology with no flame, combustion, emission, noise and vibration along with high power density and energy conversion efficiency(Dincer, 2012; Holladay, Hu, King, & Wang, 2009). Oxidation Reduction Reaction (ORR) in the fuel cell decides the efficiency and cost of fuel cell. Fuel cells are divided in low temperature and high temperature fuel cells. Low temperature fuel cells require precious metal catalyst for ORR while high temperature fuel cells require metals that can stable at relatively high temperature. Platinum (Pt) used as common catalyst in the fuel but due to difficulty of oxygen adsorption on the surface of Pt requires their high loading whichincreases its cost making them uneconomical. Pt also possesses undesirable stability in the corrosive environment and at high temperatures.

Perovskite oxides (general formula ABO_3) have large catalytically active sites, tunable compositions and structures, redox properties, extremely high thermal stability, oxygen transport, ionic and electronic conductivity and oxygen vacancies due to which perovskites are extensively used in electrochemical applications(Bu et al., 2020; Huang, Zhao, Wang, & Irvine, 2018; Lo Faro, Campagna Zignani, & Aricò, 2020).

Lanthanum ferrite ($LaFeO_3$)have perovskites type structure shows good efficiency for ORR because of their oxygen adsorbing capability, higher surface area, better stability in corrosive and higher temperature environment, and alteration possibilities in the material making them a suitable candidate for using in lower as well as higher temperature fuel cells which not only increases its efficiency but also reduces the cost makes them economical (Afifah & Saleh, 2016; Dumitru et al., 2020; Hessien, Mersal, Mohsen, & Alosaimi, 2017; Li, Zhang, Yuan, & Su, 2017; Lo Faro et al., 2020; Nandikes, Pathak, Karthikeyan, Abahussain, & Singh, 2023; Paiva et al., 2020; Peña & Fierro, 2001; Thirumalairajan, Girija, Mastelaro, & Ponpandian, 2015).

Graphene oxide (GO) nanoparticlesare known for their large surface area, high electrical and electronic conductivity, adsorption capacity and exceptional strength making them suitable for applications like electronic devices, composites and environmental remediations(Chowdury, Cho, Park, Lee, & Park, 2023; Janaki & Punithamurthy, 2022; Sudha et al., 2023; Zhongguan et al., 2023).

In this study, we prepare $LaFeO_3$powder as ORR catalyst. GO nanoparticles are incorporated during their preparations to further increase its surface area, desired stability and electrochemical properties

2. Experimental Details

All chemicals are taken analytical grade graphene oxide are taken from chem reagents (no. 980345), Electronic Oven (ESCO, OFA-54-8), Weighing balance (Shimadzu ATX 224), Ultrasonicator (Elmasonic 30 H), Centrifuge (Hettich EBA 21), Potentiostat (AUTOLAB), and Furnace (Ney* VULCAN D550) is used for experiment

2.1. Preparation of $LaFeO_3$-GO Composite

$LaFeO_3$ powder can be prepared by N,N dimethylformamide sol gel method (Li et al., 2017), thermal decomposition of lanthanum ferroxalate (Dumitru et al., 2020), microwave sintered method by using urea as fuel (Sasikala et al., 2020), polymerized complex method by using citric acid and ethylene glycol solution as medium (Phokha, Pinitsoontorn, Maensiri, & Rujirawat, 2014), polymer pyrolysis method (Phokha, Hunpratup, et al., 2015; Phokha, Pinitsoontorn, Rujirawat, & Maensiri, 2015), and glucose-assisted hydrothermal strategy (Ji et al., 2013) etc. but we use citric acid sol gel method because of their easy synthesis and absence of toxic chemicals (Lebid & Omari, 2014).

As shown in Fig 1,0.01M Aqueous solution of lanthanum oxide were dissolved in 10 ml ethanol under stirring. Few drops of nitric acid were added to the above mixture. Proceed the reaction upto the extent that reaction mixture turns nearly colorless. Then 0.01M aqueous solution of Iron nitrate nano hydrated was added into the previously prepared mixture with continous stirring. Then 0.02M (equals to total molar amount taken for metals) citric acid was added into the solution under vigorous stirring. After about 10 – 15 minutes of vigorous stirring, stirring was kept at about 350 rpm and 0.01g of graphene oxide nanoparticles were added to the mixture. Then the solution was heated for 12 hours at 110°C under continous stirring.

After that, the obtained sample was washed with ethanol and water mixture by sonication followed by centrifugation.Washing process repeats at least three times. Then the sample was calcinated at 800°C for 4 hours (Lebid & Omari, 2014; Levasseur & Kaliaguine, 2008).

After calcination, the obtained sample was powdered by using mortar and pestle. While powdering the sample, acetone was added to protect the powdered form to

agglomerate again. Acetone is volatile and easily separated out at room temperature but sample was kept at 60°C for about 10 – 15 minutes to obtain dry $LaFeO_3$ powder.

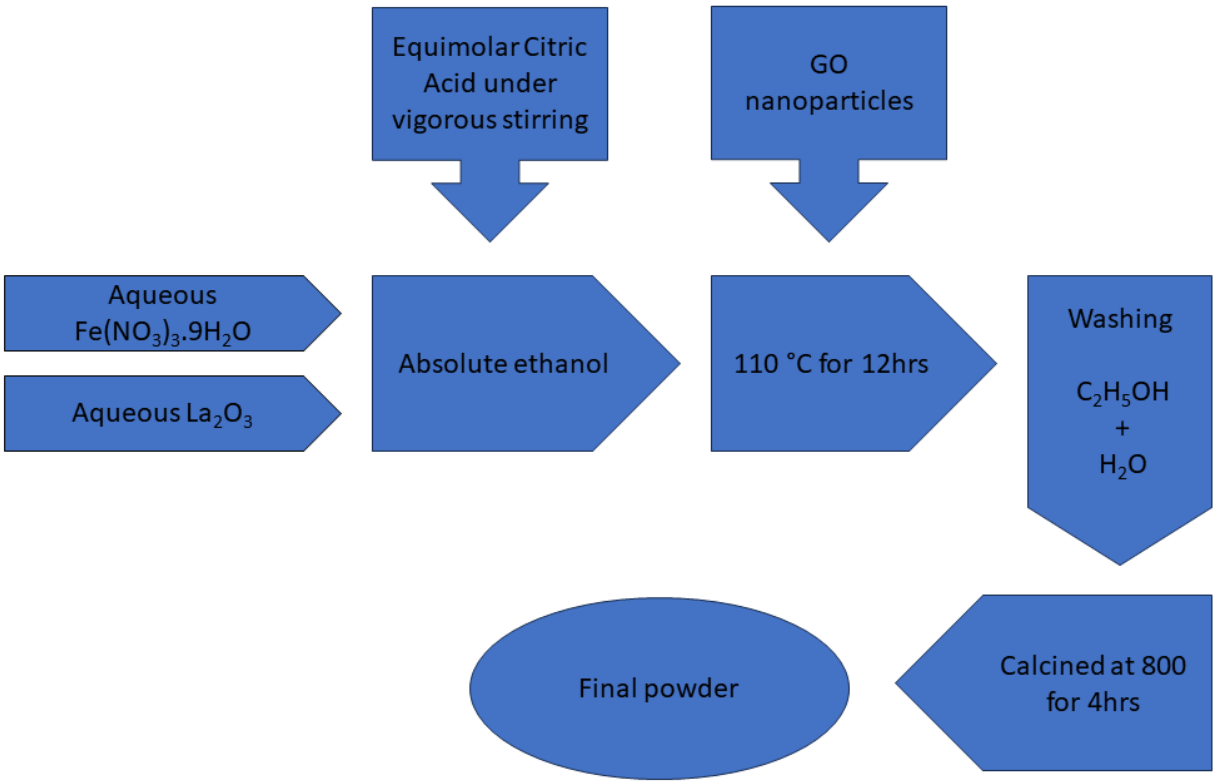

Figure 1: Schematic diagram of preparation of perovskite type $LaFeO_3$-GO ceramic powder

3. Results and Discussion

X-Ray Diffraction (XRD), Energy Dispersive X-ray spectroscopy (EDX), Scanning Electron Microscopy (SEM), and Thermogravimetric Analysis (TGA) are held in the National Centre of Physics (NCP) Pakistan.

XRD is used to calculate crystallite size, lattice parameters, degree of crystallinity etc.XRD data shows that our material is successfully prepared having Degree of crystallinity (DOC) is 44.88% and Amorphous content (weight %) is 55.12% evenprepared at 800 °C which shows that our material is not agglomeratedand is stayed in the nanometers range having particle sizes are ranging from 20 to 60 nanometers and average particle size noticed is 23 nanometers.Match 3 softwareindicates that our material has orthorohmbic structure withrefrence to the marterial in the literature (Vinila & Isac, 2022) with JCPDS card no. 96-152-6451 as shown in fig 2 (a, b). Lattice parameters arealso calculated and matched with the refrence (Lebid & Omari, 2014). All lattice parameters are found in our sample with respect to the refrence while peak at almost 65° (2 theta position) having miller indices are (224) confirms the calcination temperature. As calcination temperature increases peak formed which is like a fingerprint of the calcination temperature of lanthanum ferrite material shown in fig 2 (c, d). Peak at almost 30° (2 theta position) is probably due to GO indicates the successful incorporation of nanoparticles into the ceramic powder.

SEM images are used to gather morphological information. Figure 3 shows that the material has different type and size of crystals in the nanometers range but the majority crystals are orthorhombic. Size of crystals measured manually by Nano measure software ranges between 20 to 120 nm having maximum particles size ranges from 60 to 100 nm. This will indicate that even calcination at 800°C particles are not agitatedand found in the nanometers range predicts its high temperature catalytic applications.

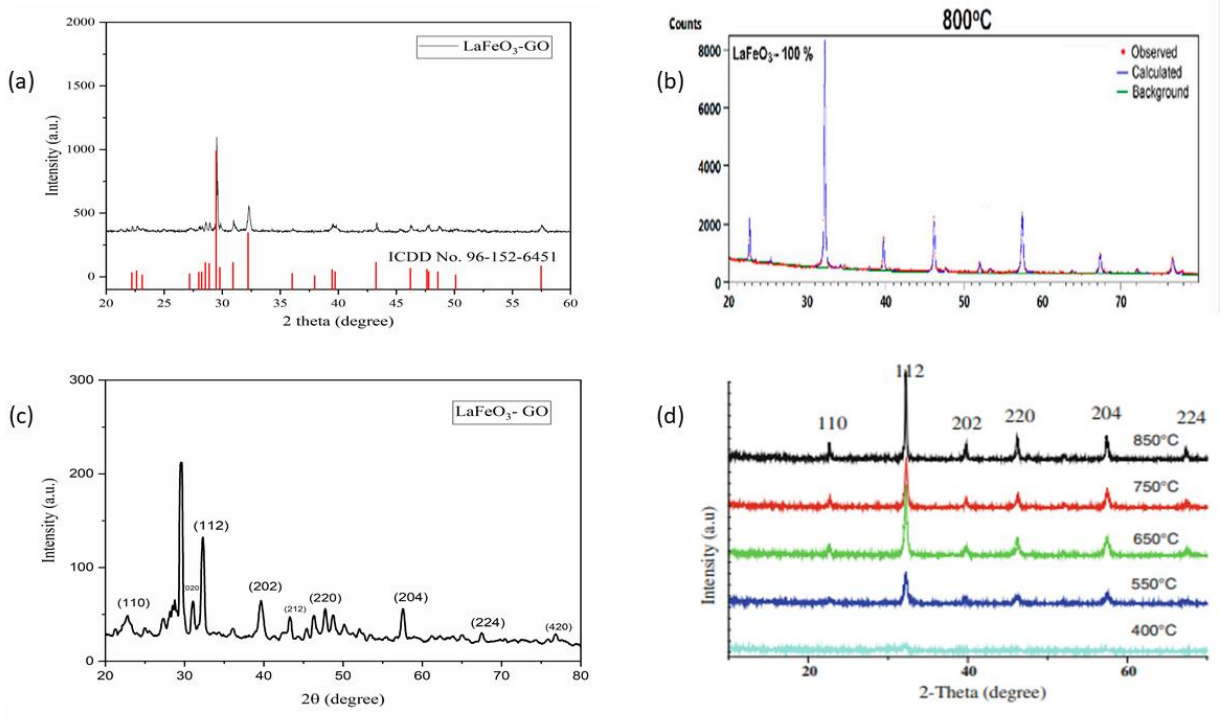

Figure 2: XRD pattern (a, b) xrd data of $LaFeO_3$-GO and refrence calcined at 800°C, (b, c) miller indices of $LaFeO_3$-GO and refrence calcined at 850°C

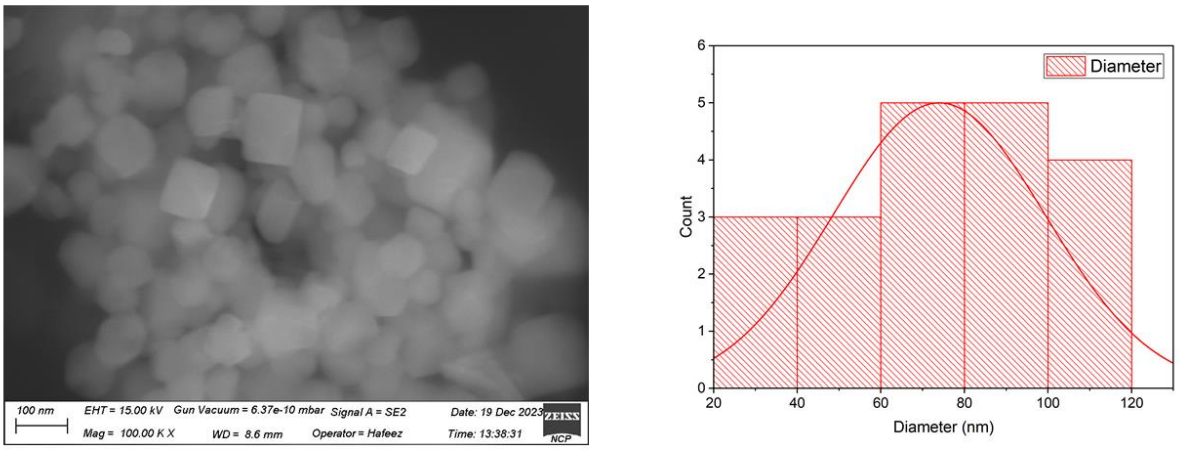

Figure 3: SEM image and their crystals size

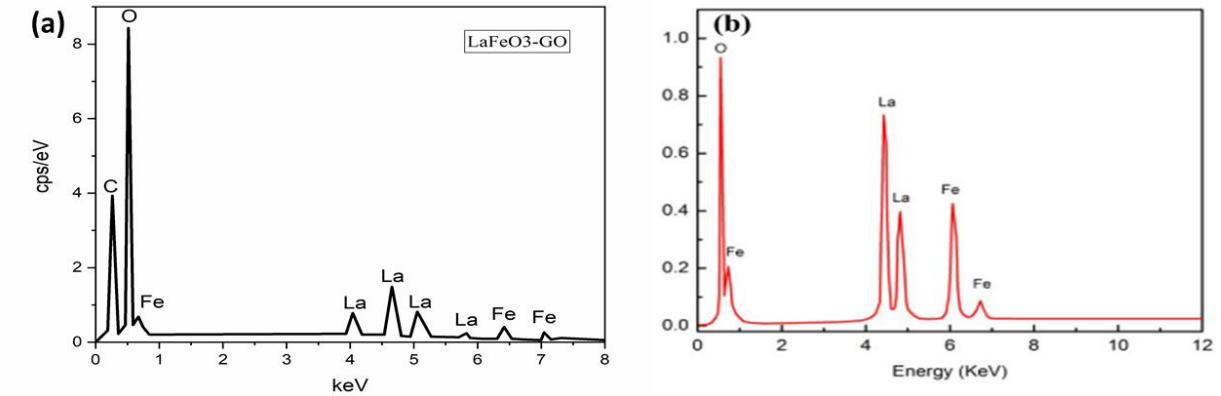

Figure 4: EDX data, (a) EDX pattern of $LaFeO_3$-GO, (b) EDX data of $LaFeO_3$ reference

EDX is used for elemental analysis. Fig 4 shows that EDX spectra of our material consists of La, Fe, O, and C which confirms the sample composition (Afifah & Saleh, 2016). Prepared material has an extra C peak indicating GO presence in the sample.

TGA is used to see the effect of temperature on the prepared sample. Fig 5 shows that while temperature increases from 40 to 1000 °C no significant weight change in the material. At first, the material is hypothesized to increase the adsorption species like oxygen and then gradually desorb from the lanthanum ferrite powder. But this adsorption is on the dopant surface which is graphene oxide because the material has no significant phase shift, degradation or decomposition evident by CobanOzkan and Dumitru in 2020 (Çoban Özkan, Türk, & Celik, 2020; Dumitru et al., 2020). Their high temperature stability predicts that the material can easily and efficiently used in high temperature fuel cells for ORR.

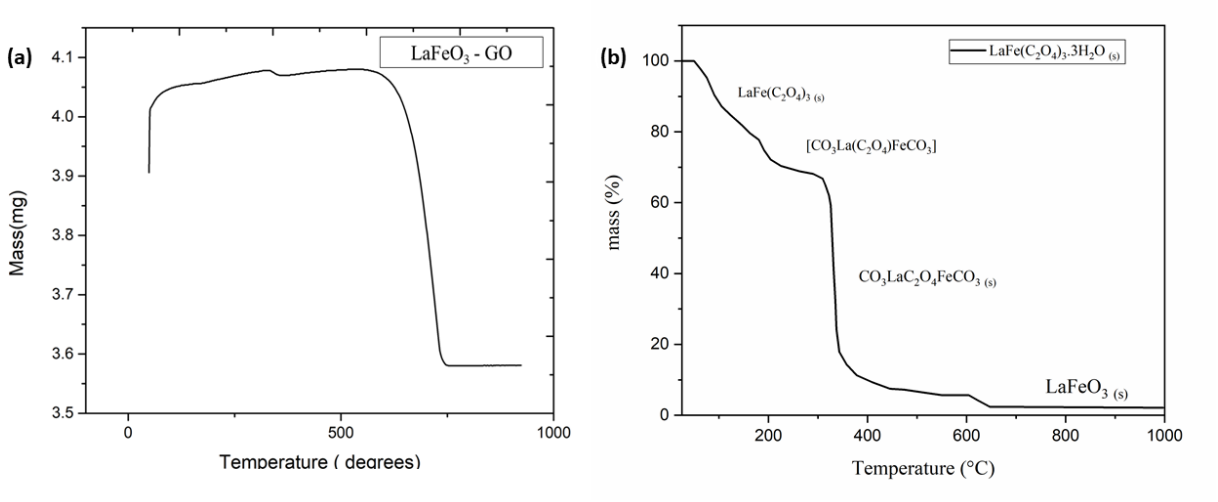

Figure 5: TGA graph (a) $LaFeO_3$-GO graph (b) Lanthanum ferrioxalate graph indicating stability of $LaFeO_3$ at high temperature.

4. Electrochemical Analysis

To check the electrochemical properties Autolab PGSTAT 302 Potentiostat is used along with GPES software 4.9. Prepared material was coated on the glassy carbon electrode acting as working electrode, Ag/AgCl electrode was used as refrence electrode while Pt wire acts as counter electrode and perform cyclic voltammetry, linear sweep voltammetry, and chronoamperometry to test the electrochemical characteristics like onset potential, current density, overpotential, mass activity, Electrochemically Active Surface Area (ECSA) and electrochemical stability of $LaFeO_3$-GO.

In Fig 6a, Cyclic Voltammetry (CV)values shows that potential window is from 0.02 to 2.01 which is constant throughout the scan rates and peak to peak separation is constant which is 1.99 V that shows that the material produces current by surface adsorbed species. As scan rate increases, the peak current value increases (from 3.55 – 11.7 μA) because it decreases the diffusion layer and uncompensated resistance produced by the electrolyte which indicates that when scan rate increases less amount of analyte are diffused and more amount is adsorbed on electrode surface. Peak ratio ranges from 0.64 – 0.73 indicating comparable efficiency of material in both anodic and cathodic scans while ECSA is about 13.22 m^2/g.

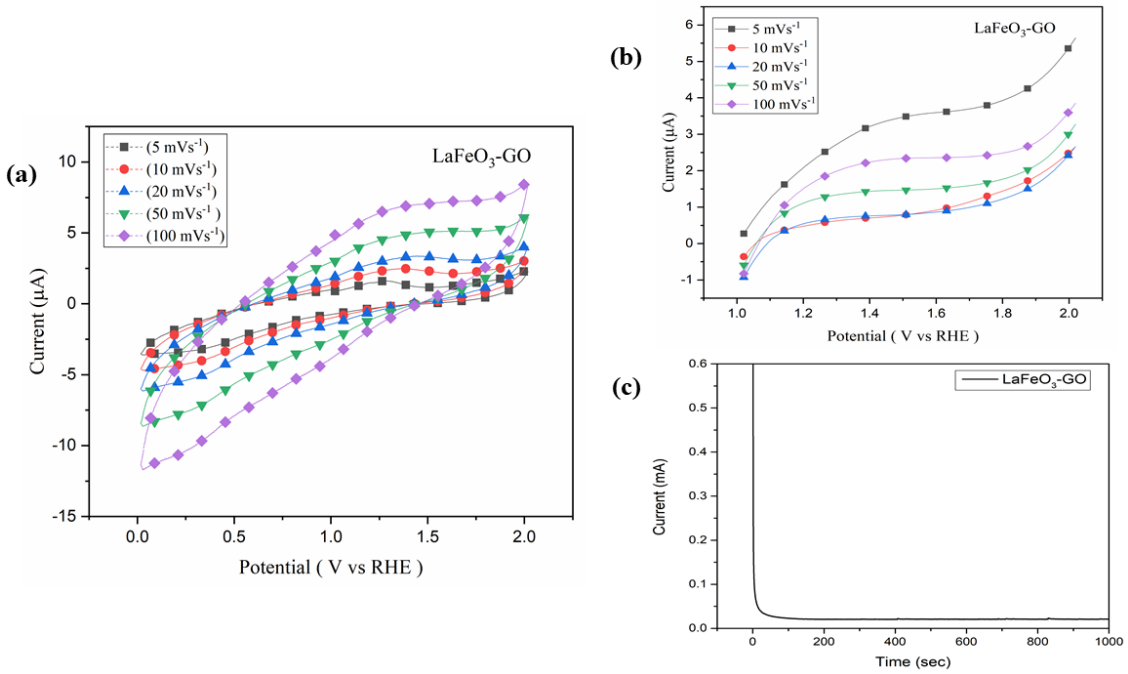

Figure 6: Electrochemical analysis of $LaFeO_3$-GO (a) cyclic voltammetry of $LaFeO_3$-GO, (b) linear sweep voltammetry of $LaFeO_3$-GO, (c) chronoamperometry of $LaFeO_3$-GO

In Fig 6b, Linear Sweep Voltammetry (LSV) curves shows that onset potential offered by $LaFeO_3$-GO is 1V having overpotential is about 0.6V, current density is 0.012 $mAcm^{-1}$, and mass activity is 0.8 Ag^{-1}.In Fig 6c, Chronoamperometry (CA) curve shows that when potential is applied to the material its gains stability in about 100 sec seconds and then becomes constant to about 1000 seconds indicating the electrochemical stability of the material.

5. Conclusion

Perovskite type $LaFeO_3$-GO with orthorhombic structure is successfully prepared by citric acid sol-gel method while calcination at 800°C creating oxygen vacancies but amorphous content is remains at 54% indicating non-agglomeration. XRD confirms the successful preparation and average particles size comes out as 23 nm. SEM images shows different types of crystals are present in the sample and after manual selection of particles average particle size is about 60-100 nm. EDX confirms elements present in the sample. CV confirms its reversible nature and adsorb species current output 0.012 $mAcm^{-1}$, potential window 0.02-2.01V, peak ratio 0.73µA, and ECSA value of about 13.22 m^2g^{-1}. LSV reveals it overpotential of about 0.6V, current density 0.012 $mAcm^{-1}$, and mass activity 0.8 Ag^{-1}. CA confirms electrochemical stability of $LaFeO_3$-GO.

6. Future Perspective

We can increase the electrochemical activity of material by increasing its electrochemically active surface area achieved by calcinated at low temperatures (400°C) so particles size reduces which gives high value of output current, ORR activity and ECSA values (Lebid & Omari, 2014). Electrochemical properties, band gap and high surface area of reduced graphene oxide provides a better agreement to use along with $LaFeO_3$ for ORR catalyst (Abdel-Aal et al., 2020). By the addition of graphene oxide and titanium dioxide or reduced graphene oxide and titanium dioxide, probably we can increase the ORR activity because of high surface area of graphene or reduced graphene oxide along with electrochemical properties of titanium dioxide can show the better electrochemical properties. We can also alter the compositions of lanthanum and iron or the dopant to alter the electrochemical properties. Sr addition in the lanthanum ferrite structure increases its electrochemical properties, so it is best suitable to add Sr in the lanthanum ferrite powder during composition which will possibly increases the ORR catalytic activity (Paiva et al., 2020; Søgaard, Vang Hendriksen, & Mogensen, 2007). Co can also be used to make layered

perovskites to increase the efficiency of ORR material (Bu et al., 2020). Ni can be used to enhance the electrochemical properties of $LaFeO_3$ perovskite due to its property of decreasing oxygen vacancy formation energy and hydroxide defects formation (Yao et al., 2024). Ir, Ag and Cu can be the candidate of the composition due to its oxygen reduction activity (Lindiwe, 2018). Ru and Ir has ability to replace Pt in basic medium (Adhikary, Sarkar, Mukherjee, & Datta, 2021). This perovskite type material can be used as hybrid electrolyte material for batteries due to its high temperature stability, conductivity(conductivity increases with high temperature calcination as probably creating oxygen vacancies) and large surface area (Foran et al., 2022; Lebid & Omari, 2014). This material can be effectively used as anode material with superior current densities and surface area values which can be further increased and explored by making composition with other elements.

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