Fabrication of a SnO$_2$-Based Hydroelectric Cell for Green Energy Production

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ABSTRACT: The generation of electricity by dissociating water into H$_3$O$^+$ and OH$^-$ ions through a hydroelectric cell (HEC) without liberating any toxic waste has achieved a groundbreaking feat. Nanoporous magnesium-doped SnO$_2$ and cobalt-doped SnO$_2$ materials have been prepared via a novel sol–gel method. The X-ray diffraction patterns of Mg-doped SnO$_2$ and Co-doped SnO$_2$ completely match with those of pure SnO$_2$, which confirms the interstitial substitution of Mg and Co in the pristine SnO$_2$. The results shown by Brunauer–Emmett–Teller theory curves illustrate the surface area of Mg-doped SnO$_2$ and Co-doped SnO$_2$ to be 46.22 and 46.81 m$^2$/g, respectively, with their pore radii being ∼3 nm. The synthesized nanoparticles were pressed into square pellets of area 4.08 cm$^2$. A zinc electrode was pasted on one side of each pellet and silver was painted on the other side to develop the HECs. The fabricated HECs of Mg-doped SnO$_2$ and Co-doped SnO$_2$ with 4.08 cm$^2$ area deliver short-circuit current, open-circuit voltage, and off-load output power of 41.69 mA, 0.787 V, and 32.81 mW and 77.52 mA, 0.454 V, and 35.19 mW, respectively. Cyclic voltammetry of both materials exhibited cathodic and anodic peaks in relation to the redox reactions taking place at Zn and silver electrodes. Nyquist curves of both HECs in the wet state confirm the ionic diffusion of split H$_3$O$^+$ and OH$^-$ ions as compared to the dry state. An off-load output power of 35.19 mW delivered by the HEC of Co-doped SnO$_2$ with 4.08 cm$^2$ area is quite promising and has great potential to replace other green energy sources.

1. INTRODUCTION

The pursuit of new sources of green energy has become a subject of thorough study in recent times, taking into account the rapidly increasing energy demand across the globe. In the present scenario, a major portion of the power generated in our country is derived from nonrenewable sources such as coal, reserves of fossil fuels, wood, and so forth, which are depleting at a great pace. The net addition of renewable energy to the global power capacity in 2017 was estimated to be around 70%, but the global energy-related carbon dioxide emissions went up to 1.4% in 2017, after being constant for three consecutive years. The rapid increase in carbon emission was attributed to robust global economic growth (of 3.7%), lower fossil fuel prices, and weaker energy efficiency efforts. Therefore, the need of the hour is to look for alternative green energy sources, which are renewable and eco-friendly. The newly designed hydroelectric cell (HEC), using the ferrite material, has attracted a lot of attention globally as it has proved to be a promising alternative for the harvesting of energy which is green and clean, without disturbing the earth’s environmental equilibrium. This ferrite HEC generates electricity by splitting molecules of water at oxygen vacancies and unsaturated cations, which are octahedrally coordinated on Li$_{0.2}$Mg$_{0.8}$Fe$_2$O$_4$. Initially, the chemidissociation of water molecules takes place on the porous surface of Li$_{0.2}$Mg$_{0.8}$Fe$_2$O$_4$ which lacks oxygen, followed by water molecule physidissociation at the nanopores. In the present work, the fabrication of Mg- and Co-doped SnO$_2$-based HECs has been presented by replacing ferrite for its low-cost availability and adaptability and to gain substantial electrical output. SnO$_2$ is an n-type semiconductor which has a direct energy band gap of 3.6 eV at room temperature. It has rutile tetragonal structure and belongs to the P4$_2$/mm space group. The SnO$_2$ unit cell comprises four O$^{2-}$ ions and two Sn$^{4+}$ ions. Each Sn$^{4+}$ ion is enclosed by six O$^{2-}$ ions residing in the corners of the octahedron, and O$^{2-}$ ions are enclosed by three Sn$^{4+}$ ions occupying the three equilateral triangle corners. SnO$_2$ has turned out to be one of the finest materials in the class of transparent conducting oxides. Some of the fascinating properties of SnO$_2$ include its reflectivity in the infrared region, high transparency in the visible region, high chemical, mechanical, and thermal stability, and low electrical resistivity. Such properties make the compound quite crucial for its usage in magneto-optic devices, solar cells, gas sensors, spintronics, photocatalysts, and lithium-ion batteries and supercapacitors.

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In this work, we have synthesized Mg-doped SnO$_2$ and Co-doped SnO$_2$ nanomaterials through a novel sol–gel technique and further developed their HECs. The ability of both these HECs was tested for the purpose of generating power by utilizing the concept of water splitting. It was found that Co-doped SnO$_2$ and Mg-doped SnO$_2$-based HECs delivered off-load output powers of 35.19 and 32.81 mW, respectively, which is much greater than the off-load output power of pure SnO$_2$ (16.6 mW). The output powers obtained through Mg-doped SnO$_2$ and Co-doped SnO$_2$ are promising and prove that the generation of power through the HEC can turn out to be a viable substitute to other green energy sources. Energy generation using the HEC is completely eco-friendly with the liberation of no toxic waste in the environment.

2. EXPERIMENTAL SECTION

2.1. Materials. Stannous chloride dihydrate (SnCl$_2$ 2H$_2$O, 99%), ethylene glycol (C$_2$H$_4$O$_2$, 99%), cobalt acetate tetrahydrate [Co(CH$_3$COO)$_2$ 4H$_2$O, 99%], magnesium acetate tetrahydrate [Mg(CH$_3$COO)$_2$ 4H$_2$O, 99%], and Millipore deionized water were used as starting materials. All the chemicals used for the synthesis of Mg- and Co-doped SnO$_2$ were of analytical grade and used as received.

2.2. Synthesis of Magnesium- and Co-Doped SnO$_2$. Mg- and Co-doped SnO$_2$ were synthesized via a facile sol–gel route. In the typical synthesis of Mg-doped SnO$_2$ and Co-doped SnO$_2$, 1 M SnCl$_2$ 2H$_2$O was dissolved at room temperature in 4 M ethylene glycol. Next, citric acid of 1 M concentration was mixed in the abovementioned solution, which dissolved at 50 °C. Furthermore, the aqueous solutions of 0.1 M Co(CH$_3$COO)$_2$ 4H$_2$O and 0.1 M Mg(CH$_3$COO)$_2$ 4H$_2$O were separately dissolved in the above-prepared solution to prepare the doped solutions of Co-doped SnO$_2$ and Mg-doped SnO$_2$, respectively. Now, the solutions containing Mg-doped SnO$_2$ and Co-doped SnO$_2$ samples were kept for stirring at 80 °C for 5 h (for obtaining a clear solution), which were further kept for few hours at 100 °C until the formation of thick gel occurred. The gels were dried in an oven at 150 °C for 2 days. The dried gels were calcined at 350 °C for 3 h in a muffle furnace to obtain the final product.

2.3. Fabrication of the HEC. The as-obtained powders of Mg-doped SnO$_2$ and Co-doped SnO$_2$ were made in the form of square pellets (area $= 4.08$ cm$^2$), as shown in Figure 1, by pressing them in a hydraulic press machine. The pellets were hardened by sintering them at 250 °C for 1 h. Silver paint was applied on one face of all the pellets in a comb-patterned fashion. Zinc sheets were pasted on the other side of each of the pellets. Finally, electrical contacts were provided to all the pellets in order to develop them in the form of fully functional HECs.

2.4. Characterization Techniques. The crystal structure and phase of the as-prepared Mg- and Co-doped SnO$_2$ powders were examined through the X-ray diffraction method using a Rigaku X-ray diffractometer with $ku$ radiation of Cu having a wavelength of 1.542 Å at 30 kV/15 mA with a scan rate of 0.2°/min. The morphology of the prepared samples was inspected through a JEOL (JSM-6390LV) scanning electron microscope at an accelerating voltage of 10 kV. The analysis pertaining to the details of pores, such as pore size, pore volume, the specific surface area of pores, and so forth of Mg-doped and Co-doped SnO$_2$ samples, was carried out via a nitrogen adsorption–desorption isotherm using the Brunauer–Emmett–Teller (BET) technique by the Quantachrome AutosorbIQStation2 instrument. The voltage and current of the developed HECs of Mg-doped SnO$_2$ and Co-doped SnO$_2$ samples were measured using a Keithley-2400 source meter. The electrochemical performance of both doped samples was evaluated by the cyclic voltammetry (CV) technique using a Biologic electrochemical workstation, which has a conventional three-electrode setup. The same electrochemical workstation was used to obtain the Nyquist plots of the HECs of Mg- and Co-doped SnO$_2$ in dry and wet states for carrying out their impedance analysis.

3. RESULTS AND DISCUSSION

3.1. X-ray Diffraction Analysis. The X-ray diffraction pattern of pristine, Mg-doped, and Co-doped SnO$_2$ has been plotted in the 2θ range of 20–80°, as shown in Figure 2. The obtained X-ray diffraction peaks of Mg-doped SnO$_2$ and Co-doped SnO$_2$ are in complete agreement with the standard diffraction pattern of pure SnO$_2$ having the JCPDS card number #880287, which confirms the successful incorporation of Mg and Co at the Sn site in pure SnO$_2$. All the peaks of X-ray diffraction of the Mg-doped SnO$_2$ and Co-doped SnO$_2$ confirm their structure to be rutile tetragonal. The average crystallite size for the Mg-doped SnO$_2$ and Co-doped SnO$_2$ nanoparticles is calculated using the Debye Scherrer formula

$$D = \frac{k\lambda}{\beta \cos \theta}$$

where $D$ is the crystallite size, $k$ is the shape factor, $\beta$ is the full width at half-maximum, and $\lambda$ is the wavelength of the X-ray.
radiation used. The diffraction peaks for Mg- and Co-doped SnO₂ occur nearly at the same position as that of pristine SnO₂, which shows that the two dopants have been perfectly implanted into the lattice of pure SnO₂ without distorting the phase and structure of pure SnO₂. The average crystallite size calculated for the Co-doped SnO₂ sample turned out to be ~15 nm, while it is ~10 nm for the Mg-doped SnO₂ sample.

3.2. XPS Analysis. To further confirm the presence of Co and Mg elements with their valences in the Co- and Mg-doped SnO₂ samples, we recorded the XPS spectra, as presented in Figures 3 and 4, respectively. Figure 3 shows the peaks of electron binding energies at 486.5 and 493.8 eV, which corresponds to the Sn⁴⁺ state of SnO₂. It also can be observed through the inset of Figure 3 that the peaks at 780.7 and 796.8 eV are identified as Co 2P½ and 2P¾, respectively, which possibly can be attributed to Co²⁺ ions. Moreover, the positions of the Co 2P½ and Co 2P¾ peaks ruled out the presence of metallic Co and Co₂O₃ in the Co-doped SnO₂ sample. Similarly, the peaks observed at 486.5 and 493.8 eV for the Mg-doped SnO₂ sample correspond to the Sn⁴⁺ state of SnO₂, as shown in Figure 4. Furthermore, the inset of Figure 4 shows the peaks at 50.7 and 91 eV, which correspond to Mg 2P½ and Mg 2S, respectively, which possibly can be attributed to Mg²⁺ states in the Mg-doped sample.

3.3. Surface Morphology Analysis. The surface morphology of the as-prepared Co-doped SnO₂ and Mg-doped SnO₂ samples is shown in Figure 5a,b through scanning electron microscopy (SEM) images. The morphology of both Co-doped SnO₂ and Mg-doped SnO₂ exhibits the presence of a porous network in them, and the grain size of Mg- and Co-doped samples is in the range of 300 nm to 1 μm. The Co-doped SnO₂ sample shows the aggregation of differently sized grains, whereas the Mg-doped SnO₂ sample displays nonuniform particles of rod-like structures. Further analysis of the porous network present in both the samples has been carried out through the BET technique. Furthermore, transmission electron microscopy (TEM) images are recorded for Co- and Mg-doped SnO₂ samples to measure the particle size accurately and are presented in Figure 6a,b, respectively. The TEM images show that the particle size for Co- and Mg-doped samples is 16 and 12 nm, respectively, which are very close to the crystalline size calculated through XRD data.

3.4. Mechanism of Mg- and Co-Doped SnO₂-Based HECs. The dissociation of water molecule takes place at the oxygen vacancies and on the unsaturated cations present on the surface of the SnO₂ lattice. Electrons are trapped in these oxygen vacancies, so now, the oxygen vacancy acts as the unsaturated/dangling bond. The trapped electrons pull the water molecule closer to it, and then, the unsaturated surface tin cations immediately take the OH⁻ ion out of it. Once the OH⁻ ion is taken away, the H₃O⁺ ion remains, and these H₃O⁺ ions are trapped in the nanopores of the SnO₂ HEC. The trapping of H₃O⁺ ions in the nanopores leads to the generation of the very high electric field inside the pores. This electric field is high enough to directly dissociate other water molecules coming in contact with it, into H₃O⁺ and OH⁻ ions, spontaneously. The dissociation of water into H₃O⁺ and OH⁻ ions proceeds in the form of an electrolytic chain reaction, which is often termed Grotthus chain reaction. The zinc sheet acts as an anode and the comb-patterned silver paint acts as an inert cathode. The hydronium ion (H₃O⁺) hops toward the silver electrode and the hydroxide ion (OH⁻) diffuses toward the Zn electrode. The movement of these ions toward their respective electrodes takes place through surface
and capillary diffusion. Zinc gets oxidized upon reacting with the OH\(^{-}\) ion and produces Zn(OH)\(_2\) and two electrons. The reduction of H\(_3\)O\(^{+}\) ions occurs at the inert Ag cathode by accepting the two electrons released from the Zn anode and further liberating H\(_2\) gas as a byproduct. The occurrence of redox reactions between both the electrodes leads to the generation of voltage and current in the cell.

At the surface of the HEC

\[
4\text{H}_2\text{O} \rightarrow 2\text{H}_2\text{O} + 2\text{OH}^{-}
\]  

(2)

At the anode

\[
\text{Zn} + 2\text{OH}^{-} \rightarrow \text{Zn(OH)}_2 + 2e^-E_{\text{oxid}} = -0.79 \text{ V}
\]

(3)

At the cathode

\[
2\text{H}_2\text{O}^+ + 2e^- \rightarrow \text{H}_2(g) + 2\text{H}_2\text{O}_{\text{red}} = +0.22 \text{ V}
\]

(4)

In the case of Co-doped SnO\(_2\) and Mg-doped SnO\(_2\), the oxygen vacancies created in their lattices are much greater than the oxygen vacancies created in the lattice of pure SnO\(_2\). Creation of a greater number of oxygen vacancies in both the doped compounds is due to the substitution of divalent Mg and Co at the site of tetravalent Sn.\(^3\) This phenomenon results in the generation of a large amount of current by both Co-doped SnO\(_2\) and Mg-doped SnO\(_2\)-based HECs as compared to the pure SnO\(_2\)-based HEC.

3.5. BET Analysis. The inspection pertaining to the details of the pores and specific surface area of Mg-doped SnO\(_2\) and Co-doped SnO\(_2\) is examined by the BET technique, as shown in Figures 7 and 8, respectively. Nitrogen gas has been employed as the probing gas to determine the adsorption–desorption isotherms. The nitrogen adsorption–desorption isotherm curve represented a type I isotherm with hysteresis for Mg-doped SnO\(_2\) and a type II hysterical isotherm for Co-doped SnO\(_2\) according to the IUPAC convention.\(^27\) Type I isotherms are an indication of materials, which are porous and are known to consist of agglomerates of uniform spheres in a fairly regular array, resulting into the narrow distribution of pore size, while type II isotherms represent pores that have wide bodies but narrow necks.\(^28\) The cumulative pore volume for Mg-doped SnO\(_2\) is found to be 0.0377 cm\(^3\)/g for pores smaller than 188.61 nm radius at a relative pressure of 0.99490 \((P/P_0)\), and similarly, the cumulative pore volume for Co-doped SnO\(_2\) is observed to be 0.03118 cm\(^3\)/g for pores smaller than 223.13 nm radius at a relative pressure of 0.99569 \((P/P_0)\).

Both Mg-doped SnO\(_2\) and Co-doped SnO\(_2\) exhibited mesoporous size distribution.\(^27\) The specific surface area of Mg-doped SnO\(_2\) and Co-doped SnO\(_2\) samples is calculated using the multipoint BET method and is estimated to be 46.22 and 46.81 m\(^2\)/g, respectively.

3.6. V–I Polarization Curve. The functioning of the HECs of Mg-doped and Co-doped SnO\(_2\) has been demonstrated by plotting a standard V–I curve, where the behavior of cell voltage is explained with respect to operating current. The HECs of Mg-doped SnO\(_2\) and Co-doped SnO\(_2\) comprises square pellets of area 4.08 cm\(^2\), which has its one face painted with a comb-patterned silver electrode and the other face has a zinc sheet pasted on it. Electrodes and wires are connected to the square pellets to eventually give them the form of a fully developed HEC. The Mg-doped SnO\(_2\) HEC gives a short-circuit current of 41.69 mA, an open-circuit voltage of 0.787 V, and an off-load power of 32.81 mW when distilled water was sprinkled over it (see Figure 9, Table 1). Similarly, the Co-doped SnO\(_2\) HEC results in a short-circuit current of 77.52 mA, an open-circuit voltage of 0.454 V, and an off-load power of 35.19 mW (see Figure 10, Table 1). The whole polarization curve has been categorized into four types of control segments.\(^29\) The segment UV represents internal loss where 0.98 V is the theoretical maximum voltage,\(^1\) and because of internal losses in the cells, the maximum voltage obtained presently in both the cells is less than the theoretical maximum.
maximum voltage. The segment VW at low current density illustrates activation loss, which is the potential needed to overcome the energy of the electrochemical reaction on the nanopores and surface of both Mg-doped SnO₂ and Co-doped SnO₂. The intervening segment WX exhibits voltage degradation because of Ohmic losses in the cell, and this segment is mainly accountable for providing resistance to the flow of ions through the porous structure of Mg-doped SnO₂/Co-doped SnO₂. A swift decrement in voltage is seen in the high current density segment XY. This rapid decrease in voltage is observed because of the abundant aggregation of ions on the surface of electrodes. The occurrence of this sharp degradation in voltage is termed as mass transport loss or concentration loss.

The occurrence of mass transport loss can be attributed to the concentration polarization which is found in the region of high current density. The on-load peak power depicted by the polarization plot of Mg-doped SnO₂ is 4.34 mW, whereas the on-load peak power shown by the polarization plot of Co-doped SnO₂ is 7.11 mW. Furthermore, the current response of Mg-doped SnO₂ and Co-doped SnO₂ HECs has been recorded for 2 h, as shown in Figure 11. The Mg-doped SnO₂ and Co-doped SnO₂ HECs exhibit 37 and 75 mA current, respectively, up to 2 h.

### Table 1. Open-Circuit Voltage, Short-Circuit Current, and Off-Load Power of Co-Doped SnO₂ and Mg-Doped SnO₂ HECs

| no. | HEC                | open-circuit voltage (V) | short-circuit current (mA) | off-load power (mW) |
|-----|--------------------|--------------------------|---------------------------|---------------------|
| 1   | Mg-doped SnO₂-based| 0.787                    | 41.69                     | 32.81               |
| 2   | Co-doped SnO₂-based| 0.454                    | 77.52                     | 35.19               |

3.7. Cyclic Voltammetry. The electrochemical behavior of Co-doped SnO₂ and Mg-doped SnO₂ is studied by carrying out CV analysis of both the samples at room temperature, as shown in Figures 12 and 13, respectively. CV helps us to comprehend the type of electrochemical reactions occurring at an electrode’s surface. In this case, Co-doped SnO₂/Mg-doped SnO₂ has been employed as the working electrode, platinum, here, works like the counter electrode, and Ag/AgCl acts as the reference electrode. The entire analysis has been accomplished in an electrolytic solution of 6 M KOH. A potential window between −0.3 and 0.6 V was set for Co-doped SnO₂ to carry out scanning, and the scan rate used was 20 mV/s. Similarly, a potential window of −0.4 to 0.8 V was set for Mg-doped SnO₂ at a scan rate of 20 mV/s. The CV curve of Co-doped SnO₂ illustrated a peak anodic current at 5.25 mA and peak cathodic current at 5.93 mA. The Co-doped SnO₂ electrode exhibited a peak anodic potential at 0.254 V and peak cathodic potential at 0.138 V. Likewise, the Mg-doped SnO₂ electrode exhibited a peak anodic current at 3.06 mA and peak cathodic current at 2.758 mA. The peak anodic potential and peak cathodic potential shown by Mg-doped SnO₂ electrode are 0.286 and 0.172 V, respectively. The behavior of the as-obtained CV
curves of Co-doped SnO$_2$ and Mg-doped SnO$_2$ is of pseudocapacitive type, where desorption/adsorption of electrons occur in a reversible and fast way, without any change in the phase of the electrode.\textsuperscript{31} The faradaic reactions that occur on the surface of Co-doped SnO$_2$ and Mg-doped SnO$_2$ electrodes can be correlated in terms of the reactions that take place for SnO$_2$ nanoparticles, which are mentioned below:\textsuperscript{32}

\begin{align}
\text{SnO}_2 + \text{H}_2\text{O} + e^- &\leftrightarrow \text{SnOOH} + \text{OH}^- \\
\text{SnOOH} + e^- &\leftrightarrow \text{SnO} + \text{OH}^- 
\end{align}

The faradaic reactions are of the reversible nature.

3.8. Electrochemical Impedance Spectroscopy Analysis. The electrochemical impedance spectroscopy helps us understand the charge transfer mechanism that takes place in an HEC.\textsuperscript{23} Nyquist curves of the Co-doped SnO$_2$ HEC and Mg-doped SnO$_2$ HEC are plotted in dry and in the moist states, as shown in Figures 14 and 15, respectively. The

![Figure 14](https://example.com/figure14.png)

Figure 14. Nyquist curve of the Co-doped SnO$_2$ HEC in dry and moist states.

![Figure 15](https://example.com/figure15.png)

Figure 15. Nyquist curve of the Mg-doped SnO$_2$ HEC in dry and moist states.

Nyquist curves give us a clear idea about the diffusion of ions that take place in an HEC as a result of water being dissociated on the surface of the HECs. The Nyquist curves of Co-doped SnO$_2$ and Mg-doped SnO$_2$ cells in the dry state illustrate a very high resistance of the order of $\sim 10^6$ Ω. When Co-doped SnO$_2$ and Mg-doped SnO$_2$ cells are soaked in distilled water, the resistance exhibited by the Nyquist curves of the wet cells is nearly 26 Ω for the Co-doped SnO$_2$ cell and almost 54 Ω for the Mg-doped SnO$_2$ cell. The calculation of resistance of the Co-doped SnO$_2$ HEC and Mg-doped SnO$_2$ HEC has been performed using semicircular fitting of the obtained Nyquist curves for both the cells in dry and in moist conditions. The presence of sloping tails at low frequency in the Nyquist plots of both the wet Co-doped SnO$_2$ cell and wet Mg-doped SnO$_2$ cell indicates diffusion of ions at the interface of electrodes.\textsuperscript{33,34}

4. CONCLUSIONS

The Co-doped SnO$_2$ and Mg-doped SnO$_2$ nanoparticles have been prepared by the facile sol–gel method. The specific surface areas obtained by BET analysis for Mg-doped SnO$_2$ and Co-doped SnO$_2$ are 46.22 and 46.81 m$^2$/g, respectively, with their pore radii being \~3 nm. The CV curves illustrate the enhanced redox reaction on the Co-doped SnO$_2$ material. Nyquist curves of both Co-doped SnO$_2$ and Mg-doped SnO$_2$ HECs in the wet and dry states exhibit ionic diffusion in the cells and show that there is a great reduction in the impedance of the Co-doped SnO$_2$ HEC under the wet conditions. The developed HEC of 4.08 cm$^2$ area of Co-doped SnO$_2$ exhibited an off-load power output of 35.19 mW in comparison to that of 32.81 mW for the Mg-doped SnO$_2$-based HEC. A power output of 35.19 mW obtained via water splitting by the HEC of 4.08 cm$^2$ area of the Co-doped SnO$_2$ sample is quite encouraging and proves to be a cost-effective, eco-friendly, and an efficient method for generation of green energy.

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Notes
The authors declare no competing financial interest.

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