Indian Journal of Pure & Applied Physics
Vol. 59, July 2021, pp. 477-482

Oxygen Production Through an Efficient Electrochemical Process

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Received 1 June 2021; accepted 15 June 2021

Whenever we think about life sustainability, the most crucial thing that comes first in our mind is oxygen. In the current scenario, oxygen production has grown tremendously due to its increasing demand from health sectors: the complexity, availability, and high cost of oxygen taken as a drawback. So, the development of an efficient, durable, and cost-effective oxygen production technology is necessary. Oxygen evolution reaction (OER) is the process of generating molecular oxygen via a chemical reaction. Scientists nowadays focus more on OER-based methods for portable device fabrication to generate breathable oxygen due to its economic and eco-friendly properties. In this article, we demonstrated the simple design and fabrication of an electrochemical-based oxygen evolution setup. The setup involves a plastic jar of (5 Litre) containing 1 M sodium hydroxide (NaOH) aqueous solution, and at the top portion, two holes were created for the immersion of the stainless-steel rod cathode and an anode electrode, which were connected to the power supply. The oxygen generation started in the bubbles form on the supply of voltage of 13V and 9.5A current. The produced oxygen is collected through the plastic tube. It also gives hydrogen, which can be separately stored. At the initial stage, the rate of oxygen production was 2.0 liter/min.

Keywords: Water splitting; Electrochemical oxygen generator; Electrolyte; Oxygen evolution**.**

1 Introduction

Oxygen is the second most abundant (~21%) gas present in the earth's atmosphere. It is of the chief constituents of life on the planet. Dioxygen provides the necessary energy through aerobic cellular respiration and combustion. Moreover, it has applications in many industrial and technological processes like cutting, welding, and buzzing uses oxygen to produce high-temperature gas flames[1-2]. Many critical raw materials are recovered using the oxidation process in chemical and petrochemical industries. Glass, oil, and gas, the metal industry also needs a heavy amount of oxygen for various purposes. Medical uses and life support systems are hardly unknown to anyone these days. All such applications urge a need to produce oxygen at a large scale. The photosynthesis reactions of the plants naturally produce oxygen. However, it does not help in meeting various industrial needs. Thus, various methods are developed to produce oxygen employing other sources. There is also a separation technique, which uses chemical processes. These processes are based on the capability of materials to absorb oxygen at a specific temperature and pressure. The cryogenic distillation method has been wieldy used for oxygen extraction from the air at an industrial scale. However, this method suffers from the disadvantages like its high cost and energy consumption because it works at low temperature and high pressure[3].

The other method to produce oxygen is non-cryogenic vacuum pressure swing adsorption (VPSA), where selectivity methodology adopted as N_2 adsorbed on molecular sieve made up of carbon and zeolite, and oxygen-rich gas mixture is received. Although this method can be applicable for medium amounts of oxygen production, it also requires a high production cost[4].

In ceramic air separation technology, a ceramic membrane is used as oxygen-ion conducting materials, which has advantages over contamination removal efficiency and gives high purity oxygen[5]. Recently, an electrochemical-based method to produce oxygen is getting attention due to its economical, eco-friendly, simple, and low cost. Electrolysis of water, electro-catalytic evolution of oxygen, and chemical catalytic evolution of oxygen

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*****Corresponding author (E-mail director@ampri.res.in, surenderjanagal@gmail.com) are the easiest ways to produce quality oxygen. A self-healing catalyst composed of nickel or cobalt that can split the water under ambient conditions to produce oxygen has been reported recently. They produced oxygen by splitting seawater and 0.5 M NaCl water solution. The absence of halogens in the produced oxygen is shown using differential electrochemical mass spectroscopy[6]. In one of the report various chemical strategies capable of producing high-quality oxygen have been discussed. These chemical methods are based on the fact that first, we store the oxygen in a form that differs from the gaseous diatomic form, and then chemical reactions are used to generate diatomic gaseous oxygen[7]. The early demonstration of photo-electrochemical water splitting was done in 1970s in which electrocmical decomposition of water at a potential difference of more than 1.23 V between one electrode was examined[8]. Oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) is a four electron-proton coupled reaction and two electron-transfer reaction, respectively. Hence, OER needed a higher energy (higher overpotential) to overcome the kinetic barrier of OER to occur. Metal chalcogenides have shown excellent OER activities with low overpotentials and good long-term stability in alkaline electrolyte[9].

Scientist have developed advanced methods and measurement techniques to reduce the intrinsic properties of materials or to probe the OER at the atomic level in order to elucidate a detailed mechanism. Through the application of nanotechnology, the size and the morphology of the compounds are tunable and even a multisheet material is able to be exfoliated into a single-sheet conformation or re-arrange single-sheets with another material (graphene) to form an alternately stacked conformation. This method not only increases the number of active sites on the materials, but also improves their intrinsic properties such as electro conductivity, binding energy to the OER intermediate[9].

This paper gives a brief overview of chemical-based oxygen production techniques and a practical demonstration of a fabricated electrochemical device for oxygen generation by using an anode and cathode electrode setup along with a power supply. A simple electrochemical device for oxygen production has been in shown in Fig. 1 along with the oxygen evolution reaction.

2 Classification of oxygen production methods

Our focus is on chemical-based methodology for oxygen production. So, here we are discussing some chemical-based methods for oxygen production. There are mainly three types of methods widely used for oxygen productions as shown in Fig. 2.

2.1 Chemical-based oxygen generator

In this method, oxygen is produced via a chemical process, in which the oxygen source is usually inorganic superoxide, chlorate, and perchlorate. In this oxygen generator, a firing pin is used to ignite the process; the chemical reaction in this process is usually exothermic. In commercial airliner, the chemical oxygen generator is usually used to provide emergency oxygen to the passengers. The canisters and oxygen masks are mounted in the top portion of the plane. If some decompression occurs, the panels are opened automatically, and the masks are released. The passengers pull down the mask and remove the trigger to get oxygen.

A chlorate candle is a cylindrical chemical generator in which sodium chlorate and iron powder are mixed . It is ignited at around 600℃, producing

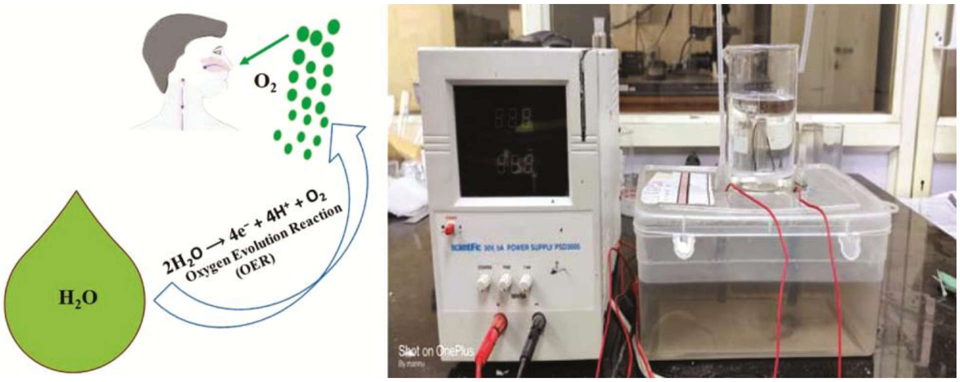

Fig. 1 — Oxygen evolution reaction setup

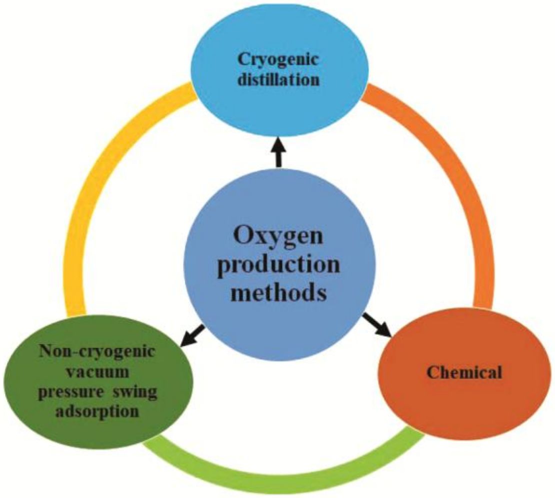

Fig. 2 — Types of oxygen production methods

NaCl and Fe_2O_3 and a fixed rate of oxygen. Now the produced oxygen can store separately. The candles can be stored for 20 years without decreasing oxygen output. The key reaction is;

$$2NaClO_3 \rightarrow 2NaCl + 2O_2 \quad \ldots (1)$$

Potassium chlorate, lithium chlorate, sodium perchlorate, lithium perchlorate can also be used as candles to generate oxygen.

Another example is hydrogen peroxide and potassium permanganate; it also releases oxygen when treated with heat, but it gives a lower amount of oxygen.

$$2\ KMnO_4 \rightarrow MnO_2 + K_2MnO_4 + O_2 \quad \ldots (2)$$

Hydrogen peroxide (H_2O_2): H_2O_2 is a well-known oxidizing agent applied as a sanitizer in food and medical productions. Exothermic decomposition of H_2O_2 gives the water and oxygen gas[11]:

$$2H_2O_2 \rightarrow 2H_2O+O_2 \quad \ldots (3)$$

The chemical reaction happens on its own but time-consuming. Manganese dioxide as a catalyst can significantly speed up the reaction. When H_2O_2 is added to a cut, catalase catalyzes the reaction. The fizzing and bubbling are caused by the catalytically increases rate of oxygen production by H_2O_2 decomposition. The presence of the highest amount of catalase in humans is possibly due to the requirement to quickly catalytically kill H_2O_2 released by metabolic activity to avoid significant cellular damage.

Potassium superoxide (KO_2): In the mining industry, potassium superoxide (KO_2) is used in rescue breathing systems. In the exhaled air of a user, KO_2 reacts exothermically with water to generate KOH and oxygen. The use of KOH is to absorb CO_2 in the exhaled air while also substitute air with oxygen[12].

$$4KO_2 + 2H_2O \rightarrow 4KOH + 3O_2 \quad \ldots (4)$$

$$2KOH + CO_2 \rightarrow K_2CO_3 + H_2O \quad \ldots (5)$$

$$K_2CO_3 + CO_2 + H_2O \rightarrow 2KHCO_3 \quad \ldots (6)$$

Since the exothermic reaction continuously heats the circulating air, the rebreathing cycle recommended having a heat exchanger for the cooling process. Unfortunately, if enough water is added to KO_2, the reaction becomes explosive. Potassium hydroxide, a byproduct, has several severe challenges like skin burning, mainly in the eyes. As a result, KO_2 canister disposal or recycling is difficult and requires expertise. Therefore, KOH leakage from these systems is a toxic threat to the environment and unacceptable. As a result, these devices are avoided for domestic or medical use due to safety and environmental disposal concerns.

3 Oxygen concentrator

It is a device that concentrates the oxygen from the ambient atmosphere by extracting nitrogen and other gas. An oxygen concentrator is based on the pressure swing adsorption (PSA) technique widely used in the medical field. A molecular sieve is used in the PSA technique to adsorb gas and operate on rapid PSA of atmospheric nitrogen onto zeolite minerals at high pressure. PSA technique is reliable and economical to produce oxygen on a mid-scale. The highly porous zeolite adsorbs a large quantity of nitrogen at high pressure due to high surface area. The oxygen concentrator compressed the air and passed it toward zeolite. Now, the zeolite adsorbs nitrogen, and the remaining gas (normal oxygen) is collected.

4 Device fabrication for oxygen generation

4.1 Materials & Methods

Sodium hydroxide (NaOH), Stainless steel rods, DI water, electric wire, plastic tubes, plastic jar with lid (5 ltr.), plastic pipes, sealing agent, and power supply.

4.2 Device fabrication

A plastic jar (5 ltr.) was used in the experimental part, in which a 1M NaOH aqueous solution was poured. Two holes were created on the jar's lid, in which two pipes were fitted to separate both electrodes (Fig. 3). In the mid of these pipes, two stainless steel (SS) rods were placed as described in

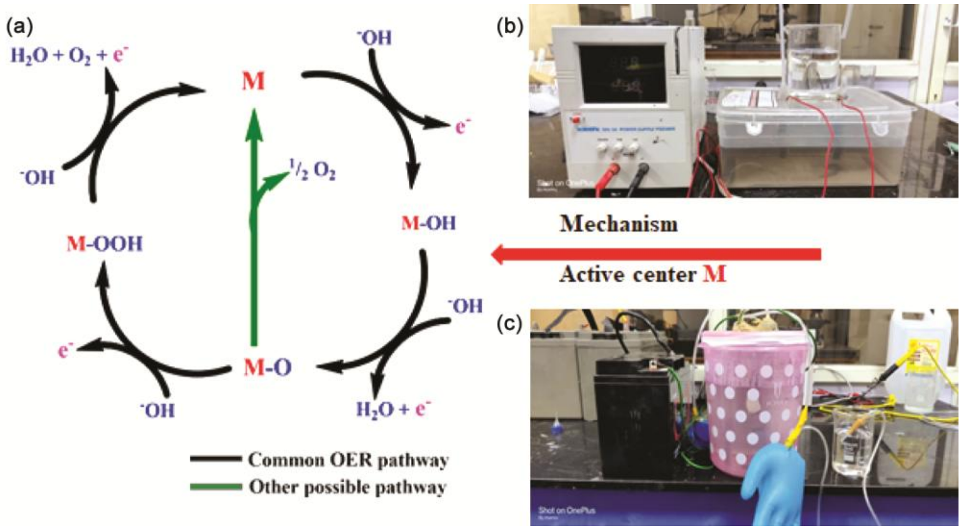

Fig. 3 — a) Electrochemical oxygen evolution reaction mechanism and b, c) experimental setup for oxygen generation

the schematic. A power supply was used to perform an electrolytic reaction. The positive and negative terminal of the power supply was connected to the SS rods, separately. When a voltage (13V) was applied, oxygen and hydrogen bubbles can be seen nearly to the electrodes on the positive electrode. The produced gases were flowed through the fitted pipes and collected at outlets. The outlet's mouth was connected with a plastic tube. This tube can be directly used for oxygen utilization. On the other hand, produced hydrogen gas can be store separately. It gives the 2 liters/min of oxygen at the initial stages.

4.3 Working principle of a fabricated device

A DC power supply is connected with two SS electrodes which are placed in NaOH containing aqueous electrolyte. When the potential is applied, hydrogen has appeared at the cathode and oxygen at the anode electrode. It was noticed that the generated amount of hydrogen is twice the generated oxygen, which was proportional to the electrical charge conducted by the solution.

5 Results and Discussions

5.1 Electrochemical water splitting

It is a simple and effective process to produce oxygen/hydrogen by the decomposition of water. The produced gases can be used for many applications, *i.e.*, hydrogen for fuel cells, oxygen for breathing. The mixture of both gases makes oxyhydrogen gas which can be used for welding and many applications. An electro chemical water splitting cell setup shown in Fig. 4. Dmitry lachinov firstly developed a device by electrolysis of water to produce oxygen and hydrogen gases at the industrial level. Here, we have fabricated a device for the production of oxygen. In water splitting, electrochemical reaction observed at the anode or oxygen evolution reaction (OER) domain and hydrogen evolution reaction (HER) occurs at the cathode in an acidic and basic medium as given below

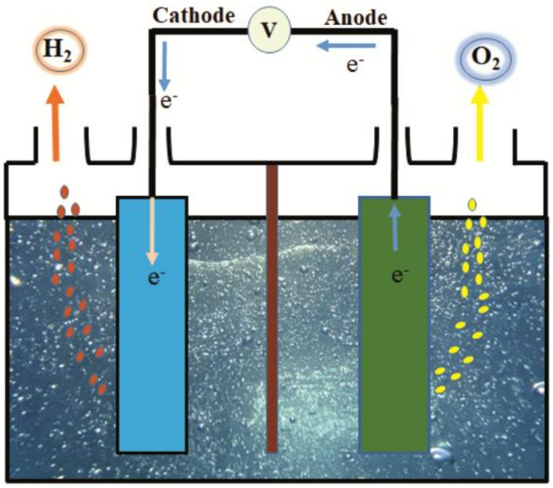

Fig. 4 — Electrochemical water splitting cell setup

In Acidic medium:

$$4e^{-}+4H^{+}(aq) \rightarrow 2H_2(g) \quad \ldots (7)$$

$$2H_2O\ (l) \rightarrow 4H^{+}(aq) + 4e^{-} + O_2(g) \quad \ldots (8)$$

In Basic medium:

$$4e^{-}+4H_2O(aq) \rightarrow 4^{-}OH(aq) + 2H_2(g) \quad \ldots (9)$$

$$4^{-}OH(aq) \rightarrow 2H_2O\ (l) + 4e^{-} + O_2\ (g) \quad \ldots (10)$$

To convert 1 mole of water molecules into hydrogen and oxygen gases requires the standard state free energy change (ΔG°) of +237.2 kJ mol^{-1} and +286 kJ mol^{-1} of enthalpy change (ΔH°). The current density and potential measure the activity of the electrocatalyst. A cell voltage of 1.23 V, 0V for HER, and 1.23 V for OER is needed from the thermodynamic perspective. The potential higher than the equilibrium potential is known as overpotential (η) is a decisive parameter for evaluating electrocatalysts' performance. Various parameters are essential for catalytic processes like the Tafel plot, Faradaic efficiency, turnover frequency (TOF), and electrochemical stability. A cell setup discussing the mechanism of HER and OER under the influence of catalyst has been given in Fig. 5.

Overpotential (η): Linear sweep voltammetry (LSV) or cyclic voltammetry (CV) was used to evaluate water splitting activity. The actual overpotential is compared at 10 mA cm^{-2} or 20 mA $cm^{-2;}$ the current density and a smaller value are favorable, resulting in a higher electrochemical activity.

Tafel Plot: It is also taken from the LSV output and recorded with linear forms of overpotential, and fitted with the Tafel equation

$$\eta = a + b \log (j) \qquad \ldots (11)$$

In the equation, η is overpotential, j is the current density, and b is a Tafel slope. It gives the significance of the reaction kinetics mechanism. A high exchange current (when $\eta = 0$) and low Tafel slope value are necessary for a high-performance electrocatalyst.

Turnover Frequency (TOF): It is the quantity of reactant a catalyst that makes molecules reactive per catalytic site per unit time and represents in the equation as follows:

$$TOF = (jA)/(\alpha Fn) \qquad \ldots (12)$$

J, A, α, F, n stands for current density, electrode surface area, number of electrons of the product, Faraday's constant, and moles of coated material atoms. TOF is used for a similar type of catalytic performance comparison.

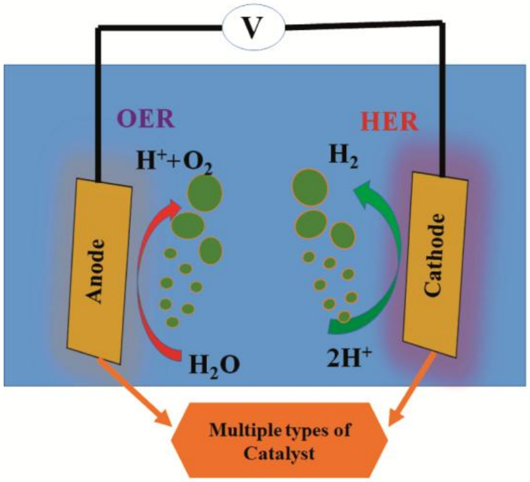

Fig. 5 — Electrochemical mechanism of HER and OER

Faradaic Efficiency: The utilization efficiency of electrons in the electrochemical reaction is measured by faradic efficiency. And water electrolysis is calculated by comparing the experimental and theoretical amounts of evolved gases.

$$\text{Faradaic efficiency} = n_t /n_e = bFn_e /(It) \qquad \ldots (13)$$

$n_{t,}$ n_e are the theoretical and experimental gas, b is the number of electrons of the product.

Stability: In water splitting, the practical applicability or durability is the crucial parameter for any devolved design. In an electrochemical setup, it is obtained by plotting current versus time curve, and the other way is repetitions of LSV, and the number of cycles should 1000 or maybe more at a scan rate of 100 mV s^{-1}.

In water-splitting, OER shows a sluggish 4 electron transfer process with high overpotential, which is not favorable, and hence for efficient OER with modifications in the anode electrode is needed for better catalytic activity and lower overpotential.

Currently, Ru and Ir-based catalysts have already set the benchmark for water splitting. However, their high cost and rarity make them difficult for their use in wide applications.

Zhang and their coworker reported CuO core and CoP based anode with superior activity and stability in essential medium with a low Tafel slope (56 mV dec^{-1}) along with the overpotentials of 290 and 330 mV respectively at the current density of 50 and 300 mA cm^{-2}. It is also evidenced with 98 % of Faraday efficiency[10]. We have summarized some of the best reported efficient OER catalysts[13-19] with their effective parameters as shown in Table 1.

The categorization of oxygen production or separation methods can be done based on its process, dynamics, purity of oxygen, and the quality of byproducts. We have listed and compare all methods for oxygen production[3] as given in Table 2.

Hence, it can be seen from the data that the chemical method for oxygen production can be used

Table 1 — Summary of some efficient OER and water splitting activity

S No	Catalyst	Electrolyte	*η (mV)*	Tafel slope (mV/dec)	Reference
1	NiS/Ni foam	1.0 M KOH	300	89	13
2	$FeNiS_2NA/Ti$	1.0 M KOH	205	43	14
3	$Ni_3S_2@NI$	0.1 M KOH	330	163	15
4	$NiCoS_2/CC$	1.0 M KOH	300	119	16
5	CuO bundles	1.0 M KOH	350	59	17
6	Annealed CuO	1.0 M KOH	430	61.4	18
7	MoO_2	1.0 M KOH	260	54	19

Table 2 — Comparative studies of oxygen production methods

Method	O_2 purity (%)	By product	Startup time	Reference
Cryogenic	99+	Nitrogen, Argon	Hours	3
Adsorption	95	Nitrogen	Minutes	3
Chemical	99+	Water, Hydrogen	Hours	3

on a large scale, and it is environmental friendly since there is no harmful side product formation, and it released hydrogen that can be used for other applications like a fuel cell.

6 Conclusion

The advantage and importance of developed electrochemical devices for oxygen production have been discussed. A brief idea about the types of oxygen production methods has been elaborated, including their restrictions and challenges. The developed electrochemical device for oxygen production is simple, low cost, and eco-friendly. The large-scale production of oxygen by this method can be possible with the design of multiple assemblies, as now people are looking for affordable and sustainable options for oxygen according to their needs.

Acknowledgement

We thankful to the Technology Mission Division of DST for providing the research grant (DST File No: DST/TMD(EWO)OWUIS-2018/TS-03 (G)).

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